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INTERLABORATORY VALIDATION OF U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 1625A

Draft Final Report

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Prepared by:

Barrett P. Eynon
Christopher Maxwell
Alfonso Valdes

Prepared for:

JRB Associates
8400 Westpark Drive
McLean, Virginia 22102

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SRI International
333 Ravenswood Avenue
Menlo Park, CA 94025
(415) 326-6200
TWX: 334-486

SRI International®
333 Ravenswood Ave. • Menlo Park, CA 94025
(415) 326-6200 • TWX: 910-373-2046 • Telex: 334-486

EXECUTIVE SUMMARY

The U.S. Environmental Protection Agency is developing methods for the chemical analysis of pollutants in wastewater. This report describes the validation of Revision A of Method 1625 in laboratories which perform routine analysis of wastewater samples. The report gives the design of the validation study, the details of preparation of the samples used in the study, the results of the study, and laboratory performance specifications determined from the laboratory data using Method 1625A.

Method 1625A is designed to measure semivolatile toxic organic pollutants in water by gas chromatography-mass spectrometry (GCMS). The method employs isotope dilution, a technique in which stable isotopically labeled analogues of the pollutants are added to each water sample and serves to reduce the variability of the analysis and correct for recovery bias. The method also permits use of internal standard and external standard analytical techniques, and therefore permits comparison of method performance by three techniques.

In order to evaluate Method 1625A and to aid in the selection of contract laboratories for future wastewater analyses, EPA invited 26 laboratories, including EPA regional laboratories and commercial laboratories, to participate in the Effluent Guidelines Division June 1983 Performance Evaluation. Each laboratory was sent an identical set of standard solutions and a water sample, plus instructions specifying a series of 11 calibrations and quantitations to be performed. These standards, prepared by one central laboratory, contained known amounts of priority pollutants and their labeled analogues, and were used for preparation of calibration, verification, recovery, blank, and aqueous performance standards. The contents of the water sample, prepared by a central laboratory, were not revealed to the laboratories. Fourteen laboratories

submitted analysis reports.* Three data formats were allowed for submission: magnetic tape, a hard-copy version of the magnetic tape format, or data sheets provided in the instructions. Data submitted on magnetic tape was extracted by the EPA Sample Control Center (SCC); the remainder of the submitted data was coded by SRI and then submitted to the SCC for entry.

The data were validated and screened for outlier values to produce a final data set. This final data set was then analyzed to provide estimates of the precision and accuracy of Method 1625A. Results of the interlaboratory validation revealed superior performance of the isotope dilution technique. The median absolute value of the relative accuracy taken across all compounds in the study was improved from 22.3 percent for the internal standard method to 7.6 percent for isotope dilution, and the median precision across all compounds was improved from 29.8 percent for internal standard to 14.3 percent for isotope dilution. Thus isotope dilution methods were found to be considerably more precise and accurate than internal standard methods. However, there was also some indication that isotope dilution requires more care in its application, since the median proportion of laboratories that could not quantify or could not detect compounds in the aqueous performance sample rose from 15.4 percent for the internal standard method to 23.1 percent for the isotope dilution method. These problems may be expected to diminish as the laboratories gain experience with the isotope dilution method, and with increased use of direct computer submission of data on magnetic media, which should eliminate transcription and coding as a source of error.

The data from the study were used to develop specifications to be used in a subsequent revision of Method 1625 and other EPA methods. Specifications were developed for calibration linearity, initial precision and accuracy, calibration verification, ongoing accuracy, and absolute and relative retention time accuracy. These specifications will also be applied to data received by EPA in its analytical programs.

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* One laboratory submitted analyses on two different instruments. These were treated as separate laboratories for the analysis, for a total of 15 data sets.

CONTENTS

ILLUSTRATIONS

TABLES

ACKNOWLEDGMENTS

This work was conducted for the Analytical Branch of EPA's Effluent Guidelines Division. Special thanks are due to William A. Telliard, Analytical Branch Chief, and Russell Roegner, Statistician. Mr. Telliard was the EPA Project Officer for the interlaboratory validation; Mr. Roegner was the EPA Task Officer for statistical analysis of the results. Many thanks also to Dale Rushneck of Interface, Inc. for his contributions and suggestions; and to John Norris of Viar & Co. and Bob Elder of JRB Associates for their valued comments. Finally, the laboratories which participated in this study should be acknowledged for their contribution to the advancement of techniques for the analysis of pollutants in environmental samples.

I INTRODUCTION

In 1976 the U.S. Supreme Court issued a consent decree requiring the U.S. Environmental Protection Agency (EPA) to measure and limit 65 compounds and classes of compounds in discharged waters. The list of 65 was subsequently refined by EPA to a list of 129 specific parameters termed the "priority pollutants." In addition, EPA is responsible for developing methods for measuring toxic pollutant concentrations in water and other media. Within EPA's Office of Water, the Effluent Guidelines Division (EGD) is responsible for promulgation of nationwide standards for allowable concentrations of these pollutants in discharges from municipal and industrial facilities. To support these standards, the Division has been involved in the development of the latest, state-of-the-art methods of chemical analysis. Method 1625 employs isotope dilution gas chromatography-mass spectrometry (GCMS) to measure the concentrations of toxic semivolatile organic pollutants in water and wastewater. Revision A of Method 1625 permits measurements by isotope dilution, internal standard, and external standard techniques, and employs a capillary chromatographic column to aid in resolution of complex mixtures. Isotope dilution methods differ from previously proposed internal and external standard methods in two important ways: (1) for each compound to be measured, a specific stable, isotopically labeled analogue of the compound is used for reference, and (2) this reference compound is added to each water sample prior to extraction. Because the reference compound behaves chemically in a way identical to the pollutant, losses in pollutant concentration during the analysis process are compensated for by losses in the reference compound. As a result, the true value of the pollutant is known more accurately when isotope dilution is used.

With isotope dilution, as with other chemical analysis techniques, there is an error inherent in every measurement. This error can come from variations in the analytical conditions (temperature, pressure, flow rate), from variations in electrical signals, from imprecise operations performed by the chemist, or from other sources. In order to develop performance specifications for the method, method performance must be assessed in laboratories that will use the method. This "interlaboratory" validation is necessary because results obtainable in a single laboratory do not represent performance in all laboratories, and specifications resulting from single-laboratory data often reflect better precision and sometimes reflect better accuracy than results from laboratories as a group. This fact is especially true when the laboratory that develops the method determines the specification, because this laboratory may achieve better performance by using techniques that are not well documented for the method. Interlaboratory validation of Method 1625 was one of the objectives of the study reported here. The other objective was to evaluate laboratories to determine which laboratories were qualified to perform work for EGD. Those laboratories not meeting minimum standards, as determined by specifications resulting from the validation, would be disqualified from performing EGD work.

In order to evaluate Method 1625A and to aid in the selection of contract laboratories for future wastewater analyses, EPA invited 26 laboratories, including EPA regional laboratories and commercial laboratories, to participate in the June 1983 Performance Evaluation. Each laboratory was sent an identical set of standard solutions plus one unknown water sample, and was asked to perform five calibrations and six analyses. A detailed set of instructions was given to the laboratories, along with a copy of the method, a listing of the data elements to be reported, and a specification for reporting results on magnetic tape. These documents are attached as appendices to this report. Also included in the appendices is the task order for preparation of the performance evaluation standards and sample.

The 14 laboratories listed in Table I-1 responded to EGD's request for participation. Data were received in the forms of quantitation reports on magnetic tape, hard copy of quantitation reports, and data reporting sheets. The data elements collected were aimed at assessing data quality. Upon receipt of data from the laboratories, the data elements were entered into a data base in the IBM computer at EPA's National Computer Center. The EPA computer was chosen over alternates so that all data would be available for future use by the Agency. Data were preserved in the state in which they were received so that editing rules other than those used in this study can be applied, if required. These data were then verified and cleaned through editing and error-checking procedures. The verified data were used to produce the analysis results presented in this report. Because of its length, a listing of the data from this study is not included in this report. Separate computer listings of the data have been submitted to EPA.

The final data set was used to provide estimates of the precision and accuracy in the interlaboratory validation. The data were also used to construct quality control limits specified in Method 1625B, and to be applied to quality assurance tests for data received by EGD. These limits are given in the tables in sections III and VI. In some cases, parallel sets of numbers were generated for analysis by the internal standard method. These numbers may be used by EPA in setting quality control specifications for Method 625 for priority pollutants by internal standard. These numbers are also given in the tables in sections III and VI.

Table I-2 has been provided to assist readers of this report seeking information on a particular subject. This table provides an added link between the body of the report and the appendices, so that information on a given subject can be located easily.

Table I-1

PARTICIPATING LABORATORIES

Radian Corp.

S-CUBED

EMS Laboratories, Inc.

ACUREX Corp.

Envirodyne

Southern Research Institute

IT Analytical Services

Arthur D. Little

Gulf South Research Institute

Environmental Science and Engineering

Shell

Midwest Research Institute

EPA Region II

EPA Region VII

Table I-2

INFORMATION CONCORDANCE

Subject	Reference: Report page	Method section: 1625A	Method section: 1625B	Appendix
Accuracy of analysis (median)	ii, 47			
Calibration				
Techniques	21-23			
External standard	21	7	7	
Internal standard	22	7	7	
Isotope dilution	22	7	7	
Linearity	24-26	7	7	
Log-Log	26, 34			
Compound m/z	13	Tbl 6,7	Tbl 6,7	
Compound number (EDG, CAS, NPDES)	10	Tbl 1,8	Tbl 1,8	
Reference	15-16	Tbl 3,4	Tbl 3,4	
Data				
Elements	17			B and E
Formats	17			B and D
Screening	39-45			
Kurtosis (FSCREEN)	40			H
Laboratory ranking	39			G
Robust (QSCREEN)	40			H
Description of analyses	9, 11-12	6-14	6-14	
Injections for perf. evaluation	9			B
Not detected entries	19			
Participating laboratories	3, 4			
Submitting results	17			
Precision of analysis (median)	ii, 48			
Retention time outlier labs	70, 71			
Specifications				
Accuracy				
Start-up	56-59	7.10	Tbl 8	J,K,L
On-going	67-69	12	Tbl 8	K
Calibration				
Linearity	25-33	7	7	
Verification	60-67	12	Tbl 8	
Precision				
Start-up	56-59	7.10	Tbl 8	J,K,L
Retention time	70-80	12	Tbl 3,4	H
Standard preparation	10	6	6	C

II STUDY DESIGN

The Effluent Guidelines Division (EDG) June 1983 Laboratory Performance Evaluation and Interlaboratory Method Validation was designed around the use of Method 1625A by laboratories performing analysis of water and wastewater samples. EPA had proposed methods for the analysis of water and wastewater in the Federal Register in December of 1979. Method 625 is a GCMS method for the analysis of the priority pollutants in water and wastewater which closely parallels Method 1625 (see the December 9, 1979 Federal Register for details of this method). The methods proposed in the Federal Register in 1979 have been validated in the intervening years through interlaboratory studies. The 1979 proposed methods did not contain quality control/quality assurance (QA/QC) specifications internal to each method, but a suggested QA/QC program was given in the proposed method package. Therefore, an objective of the interlaboratory validation studies for the proposed methods was to develop performance specifications for the methods, so that the QA/QC could be stated within each method. In developing isotope dilution methods for the analysis of pollutants in water, EGD recognized the need to develop QA/QC specifications for these methods, also. Thus, a major objective of the work described in this report was to produce performance specifications for Method 1625A. The specifications resulting from the interlaboratory evaluation of Method 1625A are explained in detail below, and are developed in subsequent sections of this report and in the Appendices. The resulting method which includes these specifications (Method 1625B) is also given in an Appendix.

Calibration Linearity--used to specify under which circumstances the response of the GCMS instrument to a given compound would be linear, or that a calibration curve was to be used. Specifications were to be developed for calibration by isotope dilution, internal standard, and external standard calibration techniques. The study design included the requirement to

calibrate the instrument by injecting the pollutants at concentrations of 10, 20, 50, 100, and 200 µg/mL along with the labeled compounds at a constant concentration of 100 µg/mL.

Calibration Verification--used to periodically verify that the GCMS instrument remains in calibration. Specifications were to be developed to measure the allowable deviation from a single point on the calibration curve. The study design required that the laboratory verify calibration of the GCMS instrument by analyzing the 100 µg/mL calibration solution after the instrument has been calibrated.

Retention Time Precision--used to aid in the identification of a pollutant, and to determine that sufficient time would be allowed for separation of pollutants in complex mixtures. Specifications were to be developed for the absolute retention time of the internal standard, for the relative retention time of each labeled compound to the internal standard, and for the relative retention time of each pollutant to its labeled analogue. The study design required each laboratory to report the retention time for each compound in every analysis performed.

Initial Precision and Accuracy, and On-going Accuracy--used to determine that the laboratory could perform analyses of the pollutants and labeled compounds in a reagent water matrix. Specifications were to be developed for the precision and recovery of four replicate analyses of 100 µg/L samples of the pollutants and labeled compounds in reagent water, and for periodic single analyses of a reagent water sample containing all pollutants and labeled compounds at this concentration. (Because of the necessity to allow for the simultaneous test of a large number of compounds in Method 1625A, the specifications were subsequently modified to permit two sets of four analyses for the initial precision and accuracy test, and one set of two analyses for the on-going accuracy test. The details of these modifications are given later in this report.) The study required that each laboratory spike and analyze a single reagent water sample containing all pollutants and labeled compounds at this concentration.

NOTE: The use of a fixed concentration (100 µg/L) and of a reagent water matrix (rather than an actual wastewater matrix) was based on the rationale that laboratory performance is best quantified by repetitive analysis of the same sample under the same analysis conditoons.

Labeled Compound Recovery--used to assess that the method would perform properly on any particular sample tested. A specification was to be developed for the permissable range of recovery for each labeled compound from each sample. The study design used the recovery for labeled compound from the reagent waters under test to develop this specification.

NOTE: The application of specifications developed from reagent water data to actual wastewater samples was based on the rationale that treated wastewaters behave very similar to reagent water, that wastewaters which did not behave similarly to reagent water would be diluted with reagent water so that they would (see section 15 of Method 1625A or B), and that data obtained on wastewaters which did not behave similarly to reagent water after dilution could not be reported for regulatory compliance purposes (see section 15 of Method 1625B).

The Performance Evaluation was implemented by requiring a series of 11 injections into a GCMS instrument by each laboratory. The purposes of the 11 injections were for calibration, calibration verification, measurement of the performance response ratio,* and analysis of extracts of a standard, a blank, and a sample of unknown composition. Further details of the 11 injections are given by the method description, attached to this report as Appendix A, and the "Instructions for Preparation and Analysis of Performance Evaluation Samples," attached as Appendix B. The 11 injections were to simulate the steps a laboratory would take when applying the method to analysis of water samples; i.e., the laboratory would first obtain spectra of the pollutants and labeled compounds, then calibrate, then analyze samples. Periodic calibration verification would be used to show

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* Ratio of peak area for the compound to peak area for the labeled compound.

that instrument performance had not changed. Table II-1 lists the injections and analyses. The solutions to be injected for calibration were to be prepared by combining appropriate amounts of solutions of the pollutants and of the labeled compounds, diluting this mixture to the appropriate volume, then adding the internal standard. The solution to be used for measurement of the response ratio was to be prepared by measuring out a known volume of the solution provided and adding a known volume of the internal standard. Preparation of the solutions to be used for the blank, aqueous performance standard, and the sample are detailed in the flow chart in Figure II-1.

Table II-2 lists the pollutants included in this study, and the numbering scheme used for the quantitation reports to distinguish the compound and analysis method. The first digit of the three-digit compound number indicates the method and pollutant type: 0 indicates priority pollutants measured by the internal standard method; 1 is used only for compound 164 (2,2-difluorobiphenyl), the reference compound for the internal standard method; 2 indicates the labeled analogue of a priority pollutant, measured by the internal standard method; 3 indicates priority pollutants measured by the isotope dilution method; 5 indicates other non-priority-pollutant compounds, analyzed by the internal standard method; 6 indicates labeled analogues of the other compounds, measured by internal standard; and 7 indicates the other compounds measured by isotope dilution. This numbering scheme permits identification of the quantitation method and the compound simultaneously, so that confusion over the quantitation method or spelling of compound names is avoided.

All standards were prepared by a central laboratory to eliminate variability from this source. The labeled compounds were furnished to the central laboratory from EPA's supply of these compounds at the Sample Control Center. The labeled compounds were from the same lot as those used for analyses in EGD's analytical programs. The standards for the pollutants were obtained from commercial sources and were analyzed by GCMS to certify their purity. The standards and sample were prepared according to the "Task Order for Preparation of Performance Evaluation Samples" (attached to this report as Appendix C).

Table II-1

SAMPLES AND ANALYSES

Sample	Designation	Unlabeled (Native) Compound Concentration	Labeled (Isotope) Compound Concentration	Operations	Fractions Analyzed*
10ug/mL calibration	CAL 10	10ug/mL	100ug/mL	Injection	C
20ug/mL calibration	CAL 20	20ug/mL	100ug/mL	Injection	C
50ug/mL calibration	CAL 50	50ug/mL	100ug/mL	Injection	C
100ug/mL calibration	CAL 100	100ug/mL	100ug/mL	Injection	C
200ug/mL calibration	CAL 200	200ug/mL	100ug/mL	Injection	C
Performance response ratio	PRR	100ug/mL	100ug/mL	Injection	C
Verification	VER	100ug/mL	100ug/mL	Injection	C
Blank	BLK	0	100ug/L	Extraction and Injection	C
Aqueous performance standard	APS	100ug/L	100ug/L	Extraction and Injection	C
Test sample	EPA	Various**	100ug/L	Extraction and Injection	A, B

* A = Acid, B = Base/Neutral, C = Combined fraction

** Up to 30 pollutants prepared at 10-200ug/L by the central laboratory.

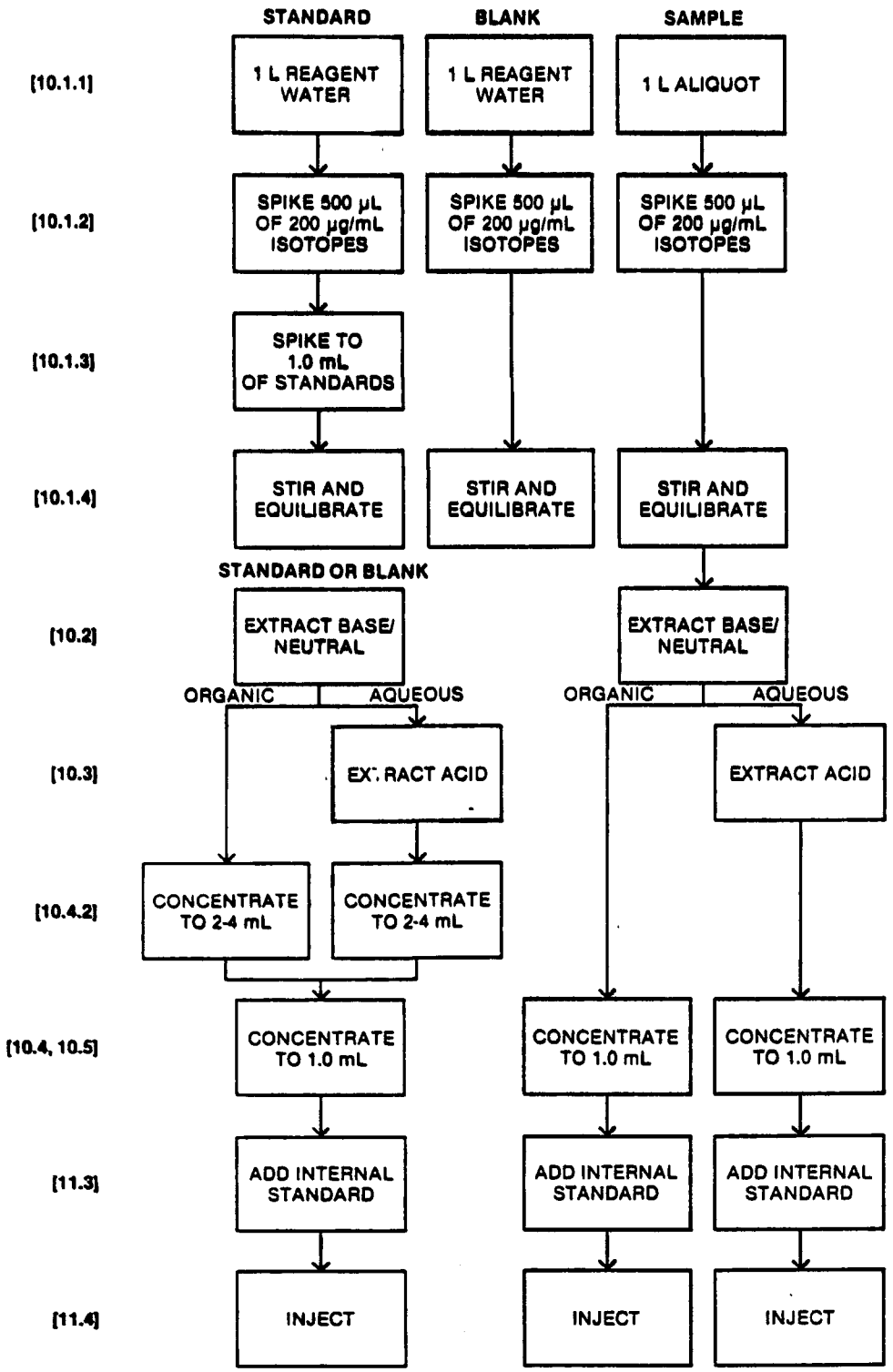

Figure II-1 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

Table II-2

COMPOUNDS, COMPOUND NUMBERS, AND MASS/CHARGE RATIOS

COMPOUND	EPA FRACTION	REF CMPD	CORRECT M/Z	ALTERNATE M/Z
001B ACENAPHTHENE	B	164	154	.
005B BENZIDINE	B	164	184	.
008B 1,2,4-TRICHLOROBENZENE	B	164	180	.
009B HEXACHLOROBENZENE	B	164	284	.
012B HEXACHLOROETHANE	B	164	201	.
018B BIS(2-CHLOROETHYL)ETHER	B	164	93	.
020B 2-CHLORONAPHTHALENE	B	164	162	.
021A 2,4,6-TRICHLOROPHENOL	A	164	196	.
022A P-CHLORO-M-CRESOL	A	164	107	.
024A 2-CHLOROPHENOL	A	164	128	.
025B 1,2-DICHLOROBENZENE	B	164	146	.
026B 1,3-DICHLOROBENZENE	B	164	146	.
027B 1,4-DICHLOROBENZENE	B	164	146	.
028B 3,3'-DICHLOROBENZIDINE	B	164	252	.
031A 2,4-DICHLOROPHENOL	A	164	162	.
034A 2,4-DIMETHYLPHENOL	B	164	122	.
035B 2,4-DINITROTOLUENE	B	164	165	.
036B 2,6-DINITROTOLUENE	B	164	165	.
037B 1,2-DIPHENYLHYDRAZINE	B	164	77	.
039B FLUORANTHENE	B	164	202	.
040B 4-CHLOROPHENYL PHENYL ETH	B	164	204	.
041B 4-BROMOPHENYL PHENYL ETHE	B	164	248	.
042B BIS (2-CHLOROISOPROPYL) E	B	164	121	.
052B HEXACHLOROBUTADIENE	B	164	225	.
053B HEXACHLOROCYCLOPENTADIENE	B	164	237	.
054B ISOPHORONE	B	164	82	.
055B NAPHTHALENE	B	164	128	.
056B NITROBENZENE	B	164	123	.
057A 2-NITROPHENOL	A	164	139	.
058A 4-NITROPHENOL	A	164	139	.
059A 2,4-DINITROPHENOL	A	164	184	.
060A 4,6-DINITRO-O-CRESOL	A	164	198	.
062B N-NITROSODIPHENYLAMINE	B	164	169	.
064A PENTACHLOROPHENOL	A	164	266	.
065A PHENOL	B	164	94	.
066B BIS (2-ETHYLHEXYL) PHTHAL	B	164	149	.
068B DI-N-BUTYL PHTHALATE	B	164	149	.
069B DI-N-OCTYL PHTHALATE	B	164	149	.
070B DIETHYL PHTHALATE	B	164	149	.
071B DIMETHYL PHTHALATE	B	164	163	.
072B BENZO(A)ANTHRACENE	B	164	228	.
073B BENZO(A)PYRENE	B	164	252	.
074B BENZO(B)FLUORANTHENE	B	164	252	.
075B BENZO(K)FLUORANTHENE	B	164	252	.
076B CHRYSENE	B	164	228	.
077B ACENAPHTHYLENE	B	164	152	.
078B ANTHRACENE	B	164	178	.
079B BENZO(GHI)PERYLENE	B	164	276	.
080B FLUORENE	B	164	166	.
081B PHENANTHRENE	B	164	178	.
084B PYRENE	B	164	202	.
164B 2,2'-DIFLUOROBIPHENYL	A	164	190	.
164B 2,2'-DIFLUOROBIPHENYL	B	164	190	.
201B ACENAPHTHENE-D10	B	164	164	.

Table II-2 (Continued)

COMPOUND	EPA FRACTION	REF CMPD	CORRECT M/Z	ALTERNATE M/Z
205B BENZIDINE-D8 (RINGS-D8)	B	164	192	.
208B 1,2,4-TRICHLOROBENZENE-D3	B	164	183	.
209B HEXACHLOROBENZENE-13C6	B	164	292	.
212B HEXACHLOROETHANE-1-13C	B	164	204	.
218B BIS(2-CHLOROETHYL)-D8 ETH	B	164	101	.
220B 2-CHLORONAPHTHALENE-D7	B	164	169	.
221A 2,4,6-TRICHLOROPHENOL-3,5	A	164	200	202
222A 4-CHLORO-3-METHYLPHENOL-2	A	164	109	.
224A 2-CHLOROPHENOL-3,4,5,6-D4	A	164	132	.
225B 1,2-DICHLOROBENZENE-D4	B	164	152	150
226B 1,3-DICHLOROBENZENE-D4	B	164	152	150
227B 1,4-DICHLOROBENZENE-D4	B	164	152	150
228B 3,3'-DICHLOROBENZIDINE-D6	B	164	258	.
231A 2,4-DICHLOROPHENOL-3,5,6-	A	164	167	165
234A 2,4-DIMETHYLPHENOL-3,5,6-	B	164	125	.
235B 2,4-DINITROTOLUENE-3,5,6-	B	164	168	.
236B 2,6-DINITROTOLUENE-D3	B	164	167	.
237B 1,2-DIPHENYL-D10-HYDRAZIN	B	164	82	.
239B FLUORANTHENE-D10	B	164	212	.
240B 4-CHLOROPHENYL PHENYL-D5	B	164	209	.
242B BIS(2-CHLOROISOPROPYL)ETH	B	164	131	.
252B HEXACHLORO-1,3-BUTADIENE-	B	164	231	.
253B HEXACHLOROCYCLOPENTADIENE	B	164	241	.
254B ISOPHORONE-D8	B	164	88	.
255B NAPHTHALENE-D8	B	164	136	.
256B NITROBENZENE-D5	B	164	128	.
257A 2-NITROPHENOL-3,4,5,6-D4	A	164	143	.
258A 4-NITROPHENOL-2,3,5,6-D4	A	164	143	.
259A 2,4-DINITROPHENOL-3,5,6-D	A	164	187	.
260A 4,6-DINITRO-O-CRESOL-D2	A	164	200	.
262B N-NITROSODIPHENYLAMINE-D6	B	164	175	.
264A PENTACHLOROPHENOL-13C6	A	164	272	274
265A PHENOL-2,3,4,5,6-D5	B	164	99	.
266B BIS(2-ETHYLHEXYL)PHTHALAT	B	164	153	.
268B DI-N-BUTYL PHTHALATE-D4	B	164	153	.
269B DI-N-OCTYL PHTHALATE-D4	B	164	153	.
270B DIETHYL PHTHALATE-3,4,5,6	B	164	153	.
271B DIMETHYL PHTHALATE-3,4,5,	B	164	167	.
272B BENZO(A)ANTHRACENE-D12	B	164	240	.
273B BENZO(A)PYRENE-D12	B	164	264	.
274B BENZO(B)FLUORANTHENE-D12	B	164	264	.
275B BENZO(K)FLUORANTHENE-D12	B	164	264	.
276B CHRYSENE-D12	B	164	240	.
277B ACENAPHTHYLENE-D8	B	164	160	.
278B ANTHRACENE-D10	B	164	188	.
279B BENZO(GHI)PERYLENE-D12	B	164	288	.
280B FLUORENE-D10	B	164	176	.
281B PHENANTHRENE-D10	B	164	188	.
284B PYRENE-D10	B	164	212	.
301B ACENAPHTHENE	B	201	154	.
305B BENZIDINE	B	205	184	.
308B 1,2,4-TRICHLOROBENZENE	B	208	180	.
309B HEXACHLOROBENZENE	B	209	284	.
312B HEXACHLOROETHANE	B	212	201	.

Table II-2 (Continued)

COMPOUND	EPA FRACTION	REF CMPD	CORRECT M/Z	ALTERNATE M/Z
318B BIS(2-CHLOROETHYL)ETHER	B	218	93	.
320B 2-CHLORONAPHTHALENE	B	220	162	.
321A 2,4,6-TRICHLOROPHENOL	A	221	196	.
322A P-CHLORO-M-CRESOL	A	222	107	.
324A 2-CHLOROPHENOL	A	224	128	.
325B 1,2-DICHLOROBENZENE	B	225	146	.
326B 1,3-DICHLOROBENZENE	B	226	146	.
327B 1,4-DICHLOROBENZENE	B	227	146	.
328B 3,3'-DICHLOROBENZIDINE	B	228	252	.
331A 2,4-DICHLOROPHENOL	A	231	162	.
334A 2,4-DIMETHYLPHENOL	B	234	122	.
335B 2,4-DINITROTOLUENE	B	235	165	.
336B 2,6-DINITROTOLUENE	B	236	165	.
337B 1,2-DIPHENYLHYDRAZINE	B	237	77	.
339B FLUORANTHENE	B	239	202	.
340B 4-CHLOROPHENYL PHENYL ETH	B	240	204	.
342B BIS (2-CHLOROISOPROPYL) E	B	242	121	.
352B HEXACHLOROBUTADIENE	B	252	225	.
353B HEXACHLOROCYCLOPENTADIENE	B	253	237	.
354B ISOPHORONE	B	254	82	.
355B NAPHTHALENE	B	255	128	.
356B NITROBENZENE	B	256	123	.
357A 2-NITROPHENOL	A	257	139	.
358A 4-NITROPHENOL	A	258	139	.
359A 2,4-DINITROPHENOL	A	259	184	.
360A 4,6-DINITRO-O-CRESOL	A	260	198	.
362B N-NITROSODIPHENYLAMINE	B	262	169	.
364A PENTACHLOROPHENOL	A	264	266	.
365A PHENOL	B	265	94	.
366B BIS (2-ETHYLHEXYL) PHTHAL	B	266	149	.
368B DI-N-BUTYL PHTHALATE	B	268	149	.
369B DI-N-OCTYL PHTHALATE	B	269	149	.
370B DIETHYL PHTHALATE	B	270	149	.
371B DIMETHYL PHTHALATE	B	271	163	.
372B BENZO(A)ANTHRACENE	B	272	228	.
373B BENZO(A)PYRENE	B	273	252	.
374B BENZO(B)FLUORANTHENE	B	274	252	.
375B BENZO(K)FLUORANTHENE	B	275	252	.
376B CHRYSENE	B	276	228	.
377B ACENAPHTHYLENE	B	277	152	.
378B ANTHRACENE	B	278	178	.
379B BENZO(GHI)PERYLENE	B	279	276	.
380B FLUORENE	B	280	166	.
381B PHENANTHRENE	B	281	178	.
384B PYRENE	B	284	202	.
502B BETA NAPHTHYLAMINE	B	164	143	.
503B ALPHA PICOLINE	B	164	93	.
504B DIBENZOTHIOPHENE	B	164	184	.
505B DIBENZOFURAN	B	164	168	.
506B N-DODECANE	B	164	57	.
507B DIPHENYLAMINE	B	164	169	.
508B DIPHENYLETHER	B	164	170	.
509B ALPHA TERPINEOL	B	164	59	.
510B STYRENE	B	164	104	.

Table II-2 (Concluded)

COMPOUND		EPA FRACTION	REF CMPD	CORRECT M/Z	ALTERNATE M/Z
511B DI-N-BUTYL AMINE		B	164	86	.
512B BIPHENYL		B	164	154	.
513B P-CYMENE		B	164	119	.
517B N-DECANE	C10	B	164	57	.
519B N-HEXADECANE	C16	B	164	57	.
521B N-EICOSANE	C20	B	164	57	.
523B N-TETRACOSANE	C24	B	164	57	.
526B N-TRIACONTANE	C30	B	164	57	.
602B 2-NAPHTHYL-D7-AMINE		B	164	150	.
603B 2-METHYLPYRIDINE-D7		B	164	100	.
604B DIBENZOTHIOPHENE-D8		B	164	192	.
605B DIBENZOFURAN-D8		B	164	176	.
606B N-DODECANE-D26		B	164	66	.
607B DIPHENYL-D10-AMINE		B	164	179	.
608B DIPHENYL-D10 ETHER		B	164	180	.
609B ALPHA-TERPINEOL-D3		B	164	62	.
610B STYRENE-2,3,4,5,6-D5		B	164	109	.
611B DI-N-BUTYL-D18-AMINE		B	164	96	.
612B DIPHENYL-D10		B	164	164	.
613B P-CYMENE-D14		B	164	130	.
617B N-DECANE-D22		B	164	66	.
619B N-HEXADECANE-D34		B	164	66	.
621B N-EICOSANE-D42		B	164	66	.
623B N-TETRACOSANE-D50		B	164	66	.
626B N-TRIACONTANE-D62		B	164	66	.
702B BETA NAPHTHYLAMINE		B	602	143	.
703B ALPHA PICOLINE		B	603	93	.
704B DIBENZOTHIOPHENE		B	604	184	.
705B DIBENZOFURAN		B	605	168	.
706B N-DODECANE	C12	B	606	57	.
707B DIPHENYLAMINE		B	607	169	.
708B DIPHENYLETHER		B	608	170	.
709B ALPHA TERPINEOL		B	609	59	.
710B STYRENE		B	610	104	.
711B DI-N-BUTYL AMINE		B	611	86	.
712B BIPHENYL		B	612	154	.
713B P-CYMENE		B	613	119	.
717B N-DECANE	C10	B	617	57	.
719B N-HEXADECANE	C16	B	619	57	.
721B N-EICOSANE	C20	B	621	57	.
723B N-TETRACOSANE	C24	B	623	57	.
726B N-TRIACONTANE	C30	B	626	57	.

Fourteen laboratories, including the central laboratory,* submitted data. One laboratory submitted two complete sets of data on different analytical equipment, which were treated as separate data sets for the purposes of this study. For study purposes, each set of data was assigned a letter code, from A to O, for use in reporting study data. In this report this code cannot be correlated with any of the data; i.e., the order of data presentation is not the same as the list of the laboratories in Table I-1.

Three data formats were allowed for submissions: quantification reports on magnetic tape, a hard copy version of the magnetic tape format, or data sheets provided in the instructions. The tape format, specified in "Quantitation Reports on Magnetic Tape," (attached to this report as Appendix D) is a specific data format which is being developed by EPA for computer-readable submission of GCMS quantitation reports. Fields in this format are specified to allow the submission of complete information on the quantitation process, including time and date extracted and analyzed, method, column type and temperature program, reference compound, peak area, retention time, mass-to-charge ratio, calculated amount, and units, plus reference library information. A specific definition of the data elements collected in the study is given in "Effluent Guidelines Division (EGD) Data Elements" (attached to this report as Appendix E). Participating laboratories were encouraged to use the tape format to test this method of data submission and to reduce coding time and transcription errors. Six laboratories submitted data on magnetic tape and three laboratories submitted data in hard copy equivalent to the tape format. The remaining laboratories submitted data in the format shown in Figure II-2. Data submitted on magnetic tape was extracted by the EPA Sample Control Center (SCC). The remainder of the data was coded and verified by SRI, then joined with the tape data to form the raw data set.

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* The central laboratory, S-CUBED, was exempted from the laboratory evaluation, but submitted analyses for use in the method evaluation.

Figure II-1

QUANTITATION DATA SHEET FORMAT

In addition to the quantitation reports, each laboratory submitted library spectrum information for each compound in the study, including at a minimum the five highest spectral peaks and any additional peaks greater than 1/10th the size of the highest peak. This data was not analyzed for this report but may be subsequently analyzed.

A number of processing steps were then necessary to complete the data set before analysis. In particular, laboratories were allowed to report only detected compounds in their quantitation reports, and to report only the isotope dilution results (or internal standard if isotope dilution was not possible). For study purposes, "not detected" entries were created for compounds which were not reported, and parallel entries were created for both the isotope dilution and internal standard methods for each compound.

A data frame was constructed by taking the list of compounds measured in this study (216, counting labeled compounds and the standard, and counting compounds measured by internal standard and by isotope dilution twice), and crossing it with the list of 15* laboratories and with the list of 11 injections. The data frame was then compared with the study data from the raw data base so that (1) compounds in the frame that were not reported by a laboratory were entered as "not detected" (peak area = 0), and (2) references to compounds outside of the frame were removed. Frame records for cases that were not reported (i.e. CAL 200 samples for one laboratory, labeled analogues for another laboratory, and EPA, APS, and BLK samples for a third laboratory, plus all records for compound 341, which had no labeled analogue) were deleted so that no artificial entries were generated.

Table II-2 includes the mass/charge value that was specified for use in quantitation of the compound. Analyses submitted using the wrong ratio were discarded; however, alternative mass/charge ratios were accepted for a few compounds, because of the absence of background interference in the prepared

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* Counting the two analysis sets from one laboratory, on different equipment, as separate laboratories.

study samples. Alternative rations are also listed in Table II-2. For the EPA samples, type A (acid fraction) compounds measured in the B (base/neutral) fraction and type B compounds measured in the A fraction were discarded. (Type A compounds are those with a nominal fraction of A on the compound list, except for phenol [65] and 2,4 dimethylphenol [34], which were treated as type B.)

Entries were created for both the internal standard and isotope dilution method measurements for each compound, carrying the peak area and retention time information from whichever type of record was actually reported. The correct reference compound numbers were also generated, being compound 164 for all internal standard records and the compound number minus 100 for isotope dilution records.

III CALIBRATION LINEARITY

The purpose of this chapter is to describe how statistical limits for testing calibration linearity were developed, and how calibration curves were constructed and applied to the data in this study. The concentration amounts calculated and reported by the laboratories on the quantitation reports were not reproducible because of variation in the calibration schemes for different analytical instruments and laboratories; and therefore, were not used in this study. As described below, a uniform calibration methodology was applied to the peak area measurements for each compound at each laboratory to obtain quantified amount values for use in this study.

Calibration Curves

In order to calculate the concentration of each compound in a sample, a calibration curve is applied to the peak area of the compound and of the reference compound obtained from the gas chromatograph. This calibration curve is constructed by the analysis of a series of calibration samples at known concentrations.

The form of the calibration curve is defined by the specific analytical method. For external standard methods, the calibration curve directly relates the peak area (A) for the compound and the known concentration (C) in the calibration sample by

$$A = f_{ES}(C) \quad .$$

The inverse of the calibration curve is applied to obtain the measured concentration of an unknown sample, i.e.,

$$C = f_{ES}^{-1} \ (A) \quad .$$

For internal standard methods, the ratios of the peak areas and concentrations to those of a reference compound are used for calibration, i.e.,

$$A/A_{ref} = f_{IS}(C/C_{ref}) \quad ,$$

and the unknown concentration in a sample is constructed from the area ratio and the known level of the standard spiked into the sample by

$$C = f_{IS}^{-1}(A/A_{ref})C_{ref} \quad .$$

Isotope dilution methods use a calibration formula parallel to that of internal standard methods, except the reference compound for each compound is its labeled analogue, rather than a single internal standard for all compounds.

In estimating the calibration curve, a range of calibration samples are used in order to evaluate the response of the instrumentation over its performance range. Method 1625 specifies five calibration standards for isotope dilution; Method 625 specifies three calibration standards for internal standard methods. In this study, five calibration points were obtained for each compound at each laboratory, at 10, 20, 50, 100, and 200 µg/mL. Because of the inherent variability in the measurement of individual samples, the determination of the calibration curve is subject to measurement error. Therefore, the more calibration samples used in the determination of the calibration curve, and the simpler the functional form of the curve (in terms of the number of parameters of the curve to be estimated), the more accurate the calibration curve and sample measurements will be. The simplest form of the response curve is a proportional ("linear") response curve

$$f(x) = ax \quad .$$

Because this function represents the theoretical response of a perfect instrument and contains only one parameter to estimate, this form of the calibration curve would be preferred in any case where the calibration data do not indicate nonlinearity in the response.

The random variation in the calibration response can be assumed to have a proportional error structure, i.e. for repeated measurements the area ratio A/A_{ref} (A for external standard) is distributed around f_{IS} (C/C_{ref}) (or f_{ES} (C) for external standard) as

$$f_{IS}(C/C_{ref})\ (1 + \varepsilon)$$

where ε has mean zero and variance σ^2 independent of C/C_{ref}. In the case of linear response curve,

$$A/A_{ref} = a(C/C_{ref})\ (1 + \varepsilon) \quad ,$$

and rewriting in terms of the response factor RF gives

$$RF = (A/A_{ref})/(C/C_{ref}) = a(1 + \varepsilon) \quad .$$

Hence the coefficient of variation of the response factor would be

$$\frac{\sqrt{a^2\sigma^2}}{a} = \sigma \quad ,$$

a constant for all concentration ratios. This assumption of proportional error may break down for low concentrations near the method detection limit, but should be a good model of the variance structure over the effective performance range of the method. If a linear proportional calibration curve is to be fitted to a calibration set containing values $A_1 \ldots A_n$, $A_{ref_1} \ldots A_{ref_n}$, $C_1 \ldots C_n$ and $C_{ref_1} \ldots C_{ref_n}$, the best estimate of the calibration coefficient a can be derived from the formula for a weighted regression (see for instance Draper and Smith, Applied Regression Analysis, Second Ed., p. 112) as

$$a = \sum_{i=1}^{N} (A_i/A_{ref_i})/(C_i/C_{ref_i})/n = \frac{1}{n} \sum_{i=1}^{N} RF_i = \overline{RF} \quad ,$$

where $\overline{RF}$ is the average response factor.

Linearity Tests

Statistical goodness-of-fit tests for a particular functional form are constructed by a comparison of the residual error of the fitted function ("lack of fit") to an estimate of error of the function obtained from replicate measurements at one value ("pure error") (see, for example, Draper and Smith, pp. 33-42). For this study, goodness-of-fit linearity limits were needed both for calibration of the study samples and for use in final method specifications. The replicate calibration-type samples available in this study were the CAL 100, VER, and PRR samples. The CAL 100 and VER samples were constructed by each laboratory by diluting the prepared isotope and priority pollutant standards supplied for the study, to produce samples containing 100µg/mL of priority pollutant and labeled compounds. The PRR sample was obtained from the solution of 100µg/mL mixed standards provided by the central laboratory. Therefore, the contents of the PRR sample should be identical to those of the CAL 100 and VER samples, though constructed by a slightly different process. Because of the similarity of the PRR sample to the CAL 100 and VER samples, and because the PRR results provide additional data values (i.e. degrees of freedom) for estimation of pure error, these three samples were considered as replicates, subject to a check for bias relative to the CAL 100 and VER samples. Details of the tests applied to the PRR sample are given in Appendix F.

The linearity test was constructed by calculation of the average intralaboratory variation in the standardized response factor of the three replicates across all laboratories in the study. Then, for each laboratory, the coefficient of variation of the five calibration samples was calculated and compared with the test specification. If the coefficient of variation was smaller than the test criterion, a linear calibration was used for that compound at that laboratory. If the linearity test was not passed, an alternate method of calibration was used, as described in the "Log-Log Calibration" section.

Linearity Specification Calculations

For each record, information was obtained from the record for the appropriate reference compound, and the following ratios were computed (if their components were all reported):

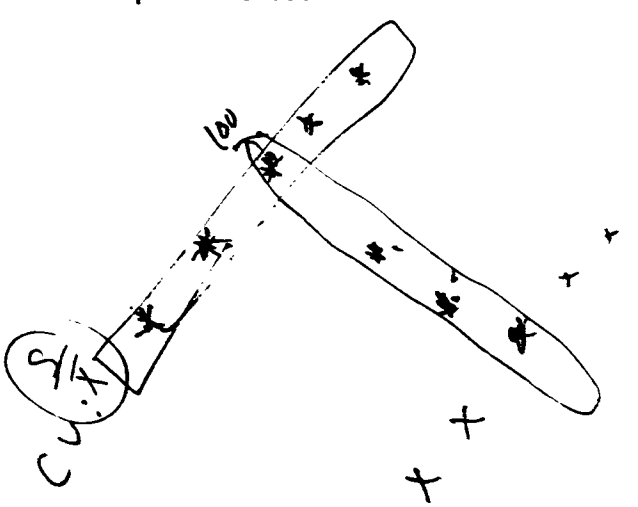
Area ratio = peak area/peak area (reference)

Input ratio = input concentration/input concentration (reference)

Response factor = area ratio/input ratio.

The area ratios (AR), response factors (RF), and input ratios (IR) for the CAL, VER, and PRR samples were then used to calculate the calibration curves. In order to prevent undue influences of outliers, a mild prescreening of the response factors was done (a QSCREEN of each set of seven values at level .0001; see Chapter IV). The 345 values (out of approximately 20,000 values screened) identified were set to "missing" for all future calculations. Then, appropriate limits for testing the goodness of fit of a linear calibration were obtained. For each compound and laboratory, the variance among RF_{100}, RF_{VER}, and RF_{PRR} was determined, and standardized by the square of the mean response factor for all seven samples (the five CAL samples, PRR, and VER). This standardized variance was then averaged across all laboratories, weighted by the degrees of freedom* for each laboratory, to come up with an overall standardized variance of response factors σ^2, with DF total degrees of freedom. Then for calibration, assuming the response is truly linear with proportional error structure, the test for linearity used was to compare the coefficient of variation of $RF_{10} \ldots RF_{200}$ with

$$\text{CV Limit} = 100 \left(\sigma^2 F_{N-1,DF}(.95)\right)^{\frac{1}{2}},$$



* Number of points (up to 3) minus 1.

where N is the number of nonmissing RF_{10}... RF_{200}, F is the inverse of the cumulative F distribution, and DF is the degrees of freedom in the estimate of σ^2 described above. These values are tabulated in Table III-1 by compound, for five, four, and three calibration points. External standard limits were constructed as described above, using only the response ratio of peak area/input concentration. Results are given in Table III-2.

Linear Calibration

These limits were applied to the actual calibration data for each compound for each lab. If fewer than four calibration points were available, no calibration was attempted. (The entry for three points may be used by EPA in setting linearity limits for internal standard methods--e.g., Method 625--but was not used in this study.) If the coefficient of variation of the response factors was less than or equal to the limit for that compound then the average response factor $\overline{RF}$ was obtained and a linear calibration was used. (The average response factor is exactly the weighted least squares solution for the linear calibration curve, under the assumption of proportional error.)

Log-Log Calibration

In the formal specification of Method 1625, if the use of the proportional linear calibration (i.e. the "averaged response to concentration ratio") is rejected, the analyst is directed to use the "complete calibration curve" for that compound over the five-point range. For purposes of this study, if the linearity test failed, then a log-log regression was performed between the area ratios and the input ratios:

$$\log (AR) = \log (b) + \gamma \cdot \log (IR),$$

which converts into a calibration curve

$$AR = b\,(IR)^{\gamma} \quad .$$

If γ ("curvature") was less than zero, the curve was rejected, and no

CALIBRATION LIMITS -- INTERNAL STANDARD AND ISOTOPE DILUTION

COMPOUND	DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)	LOG LIMIT (5 POINTS)	LOG LIMIT (4 POINTS)	LOG LIMIT (3 POINTS)
001B ACENAPHTHENE	27	28.9	30.1	32.1	0.289	0.307	0.344
005B BENZIDINE	24	97.9	102.0	108.4	1.007	1.071	1.198
008B 1,2,4-TRICHLOROBENZENE	27	31.3	32.6	34.7	0.324	0.345	0.387
009B HEXACHLOROBENZENE	27	41.4	43.1	45.9	0.416	0.445	0.498
012B HEXACHLOROETHANE	23	19.4	20.2	21.5	0.260	0.276	0.309
016B BIS(2-CHLOROETHYL)ETHER	26	31.5	32.8	34.9	0.351	0.373	0.418
020B 2-CHLORONAPHTHALENE	22	18.2	18.9	20.1	0.499	0.530	0.592
021A 2,4,6-TRICHLOROPHENOL	26	26.5	27.6	29.3	0.322	0.342	0.393
022A P-CHLORO-M-CRESOL	21	26.3	27.4	29.1	0.356	0.379	0.423
024A 2-CHLOROPHENOL	26	27.3	28.4	30.2	0.311	0.331	0.371
025B 1,2-DICHLOROBENZENE	27	32.4	33.7	35.9	0.349	0.371	0.416
026B 1,3-DICHLOROBENZENE	27	32.0	33.4	35.5	0.337	0.359	0.402
027B 1,4-DICHLOROBENZENE	25	33.6	35.0	37.2	0.342	0.363	0.407
028B 3,3'-DICHLOROBENZIDINE	28	101.2	105.5	112.3	0.944	1.005	1.127
031A 2,4-DICHLOROPHENOL	27	28.9	30.1	32.0	0.312	0.332	0.372
034A 2,4-DIMETHYLPHENOL	27	27.1	28.3	30.1	0.345	0.367	0.412
035B 2,4-DINITROTOLUENE	26	45.4	47.3	50.3	0.447	0.475	0.532
036B 2,6-DINITROTOLUENE	27	38.5	40.1	42.7	0.414	0.440	0.494
037B 1,2-DIPHENYLHYDRAZINE	25	25.9	27.0	28.7	0.297	0.316	0.353
039B FLUORANTHENE	27	58.1	60.5	64.4	0.531	0.555	0.633
040B 4-CHLOROPHENYL PHENYL ETH	26	33.8	35.2	37.5	0.352	0.375	0.420
041B 4-BROMOPHENYL PHENYL ETHE	14	31.1	32.3	34.1	0.348	0.359	0.398
042B BIS (2-CHLOROISOPROPYL) E	22	55.9	58.1	61.8	0.484	0.514	0.575
052B HEXACHLOROBUTADIENE	26	33.6	35.0	37.2	0.354	0.377	0.422
053B HEXACHLOROCYCLOPENTADIENE	24	32.0	33.3	35.4	0.341	0.363	0.406
054B ISOPHORONE	27	57.0	59.3	63.2	0.738	0.786	0.880
055B NAPHTHALENE	27	27.2	28.3	30.2	0.301	0.320	0.359
056B NITROBENZENE	11	20.1	20.8	21.9	0.216	0.228	0.251
057A 2-NITROPHENOL	24	29.2	30.4	32.3	0.323	0.343	0.384
058A 4-NITROPHENOL	23	58.8	61.2	65.0	0.587	0.624	0.698
059A 2,4-DINITROPHENOL	28	108.1	112.7	119.9	0.831	0.885	0.991
060A 4,6-DINITRO-O-CRESOL	26	44.1	45.9	48.9	0.459	0.489	0.546
062B N-NITROSODIPHENYLAMINE	13	11.8	12.2	12.9	0.134	0.141	0.156
064A PENTACHLOROPHENOL	27	46.3	48.3	51.4	0.436	0.464	0.520
065A PHENOL	26	29.0	30.2	32.1	0.331	0.352	0.394
066B BIS (2-ETHYLHEXYL) PHTHAL	27	67.3	70.1	74.6	0.683	0.642	0.719
068B DI-N-BUTYL PHTHALATE	27	47.5	49.5	52.7	0.446	0.475	0.532
069B DI-N-OCTYL PHTHALATE	27	108.0	112.5	119.8	0.923	0.983	1.101
070B DIETHYL PHTHALATE	26	35.8	37.3	39.7	0.366	0.390	0.436
071B DIMETHYL PHTHALATE	27	32.8	34.2	36.4	0.368	0.392	0.439
072B BENZO(A)ANTHRACENE	26	65.1	67.8	72.2	0.697	0.742	0.831
073B BENZO(A)PYRENE	27	122.6	127.8	136.0	1.235	1.314	1.472
074B BENZO(B)FLUORANTHENE	26	106.0	110.4	117.4	1.082	1.152	1.290
075B BENZO(K)FLUORANTHENE	25	89.6	93.3	99.2	0.948	1.008	1.129
076B CHRYSENE	27	93.3	97.2	103.5	0.901	0.959	1.074
077B ACENAPHTHYLENE	24	24.3	25.3	26.9	0.296	0.315	0.352
078B ANTHRACENE	27	34.0	36.3	38.6	0.352	0.374	0.419
079B BENZO(GHI)PERYLENE	27	109.5	114.1	121.4	1.541	1.641	1.838
080B FLUORENE	27	29.4	30.6	32.6	0.324	0.345	0.386
081B PHENANTHRENE	27	35.1	36.6	38.9	0.352	0.375	0.420
084B PYRENE	28	62.6	65.2	69.3	0.618	0.657	0.735
164B 2,2'-DIFLUOROBIPHENYL	28	0.0	0.0	0.0	0.000	0.000	0.000

Table III-1 (Continued)

COMPOUND	DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)	LOG LIMIT (5 POINTS)	LOG LIMIT (4 POINTS)	LOG LIMIT (3 POINTS)
201B ACENAPHTHENE-D10	24	15.1	15.8	16.8	0.173	0.184	0.206
205B BENZIDINE-D8 (RINGS-D8)	23	155.5	161.8	172.0	1.117	1.188	1.328
208B 1,2,4-TRICHLOROBENZENE-D3	23	27.4	28.6	30.4	0.266	0.282	0.316
209B HEXACHLOROBENZENE-13C6	23	38.5	40.1	42.6	0.379	0.403	0.451
212B HEXACHLOROETHANE-1-13C	20	25.7	26.7	28.3	0.255	0.271	0.302
218B BIS(2-CHLOROETHYL)-D8 ETH	21	29.2	30.4	32.3	0.315	0.335	0.374
220B 2-CHLORONAPHTHALENE-D7	27	31.9	33.2	35.4	0.543	0.578	0.648
221A 2,4,6-TRICHLOROPHENOL-3,5	24	23.1	24.0	25.6	0.260	0.277	0.310
222A 4-CHLORO-3-METHYLPHENOL-2	25	20.4	21.2	22.6	0.225	0.240	0.268
224A 2-CHLOROPHENOL-3,4,5,6-D4	25	27.4	28.5	30.3	0.272	0.290	0.324
225B 1,2-DICHLOROBENZENE-D4	24	34.0	35.4	37.7	0.349	0.371	0.415
226B 1,3-DICHLOROBENZENE-D4	24	26.0	27.1	28.8	0.288	0.307	0.343
227B 1,4-DICHLOROBENZENE-D4	25	24.3	25.3	27.0	0.268	0.285	0.319
228B 3,3'-DICHLOROBENZIDINE-D6	26	142.9	148.8	158.4	1.057	1.125	1.260
231A 2,4-DICHLOROPHENOL-3,5,6-	27	29.3	30.5	32.5	0.279	0.297	0.332
234A 2,4-DIMETHYLPHENOL-3,5,6-	27	27.4	28.6	30.4	0.264	0.281	0.315
235B 2,4-DINITROTOLUENE-3,5,6-	24	80.7	84.0	89.3	1.068	1.136	1.271
236B 2,6-DINITROTOLUENE-D3	16	43.7	45.3	48.0	0.523	0.554	0.616
237B 1,2-DIPHENYL-D10-HYDRAZIN	26	20.9	21.8	23.2	0.238	0.253	0.284
239B FLUORANTHENE-D10	27	79.8	83.1	88.5	0.636	0.677	0.759
240B 4-CHLOROPHENYL PHENYL-D5	27	29.7	30.9	32.9	0.305	0.325	0.364
242B BIS(2-CHLOROISOPROPYL)ETH	21	30.4	31.6	33.6	0.298	0.316	0.353
252B HEXACHLORO-1,3-BUTADIENE-	24	28.2	29.4	31.2	0.264	0.280	0.314
253B HEXACHLOROCYCLOPENTADIENE	22	94.3	98.2	104.3	0.614	0.652	0.729
254B ISOPHORONE-D8	26	36.7	38.3	40.7	0.336	0.357	0.400
255B NAPHTHALENE-D8	27	23.9	24.9	26.5	0.240	0.256	0.287
256B NITROBENZENE-D5	11	20.7	21.4	22.6	0.236	0.248	0.274
257A 2-NITROPHENOL-3,4,5,6-D4	27	19.0	19.8	21.1	0.202	0.215	0.241
258A 4-NITROPHENOL-2,3,5,6-D4	22	76.4	79.5	84.5	0.693	0.737	0.823
259A 2,4-DINITROPHENOL-3,5,6-D	27	49.3	51.3	54.7	0.455	0.484	0.543
260A 4,6-DINITRO-O-CRESOL-D2	27	42.7	44.5	47.3	0.402	0.428	0.480
262B N-NITROSODIPHENYLAMINE-D6	19	26.5	27.6	29.3	0.326	0.345	0.385
264A PENTACHLOROPHENOL-13C6	26	41.5	43.2	46.0	0.375	0.399	0.447
265A PHENOL-2,3,4,5,6-D5	27	58.5	60.9	64.8	0.454	0.483	0.541
266B BIS(2-ETHYLHEXYL)PHTHALAT	26	70.8	73.7	78.5	0.610	0.649	0.727
268B DI-N-BUTYL PHTHALATE-D4	25	50.1	52.1	55.5	0.506	0.538	0.602
269B DI-N-OCTYL PHTHALATE-D4	27	148.9	155.1	165.1	1.071	1.140	1.277
270B DIETHYL PHTHALATE-3,4,5,6	26	29.8	31.1	33.1	0.304	0.324	0.363
271B DIMETHYL PHTHALATE-3,4,5,	27	59.1	61.6	65.6	0.479	0.510	0.571
272B BENZO(A)ANTHRACENE-D12	24	94.2	98.0	104.3	0.782	0.831	0.930
273B BENZO(A)PYRENE-D12	27	152.6	159.0	169.2	1.305	1.389	1.556
274B BENZO(B)FLUORANTHENE-D12	26	132.9	138.5	147.3	1.096	1.166	1.306
275B BENZO(K)FLUORANTHENE-D12	26	108.4	112.9	120.1	0.961	1.023	1.145
276B CHRYSENE-D12	27	107.4	111.9	119.1	0.869	0.926	1.037
277B ACENAPHTHYLENE-D8	27	25.2	26.2	27.9	0.250	0.266	0.298
278B ANTHRACENE-D10	25	31.9	33.3	35.4	0.325	0.345	0.386
279B BENZO(GHI)PERYLENE-D12	27	119.6	124.6	132.7	1.559	1.659	1.859
280B FLUORENE-D10	27	38.3	39.9	42.5	0.569	0.605	0.678
281B PHENANTHRENE-D10	26	28.5	29.7	31.6	0.304	0.324	0.363
284B PYRENE-D10	25	95.7	99.7	106.0	0.892	0.949	1.062
301B ACENAPHTHENE	26	16.8	17.5	18.7	0.189	0.196	0.210
305B BENZIDINE	24	115.5	120.3	127.9	0.991	1.054	1.179

Table III-1 (Continued)

COMPOUND	DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)	LOG LIMIT (5 POINTS)	LOG LIMIT (4 POINTS)	LOG LIMIT (3 POINTS)
308B 1,2,4-TRICHLOROBENZENE	26	23.4	24.4	26.0	0.275	0.292	0.327
309B HEXACHLOROBENZENE	24	20.0	20.8	22.1	0.250	0.266	0.298
312B HEXACHLOROETHANE	20	26.3	27.3	29.0	0.594	0.631	0.704
318B BIS(2-CHLOROETHYL)ETHER	22	21.6	22.5	23.9	0.222	0.236	0.264
320B 2-CHLORONAPHTHALENE	22	10.7	11.2	11.9	0.175	0.186	0.208
321A 2,4,6-TRICHLOROPHENOL	26	26.7	27.8	29.6	0.341	0.363	0.407
322A P-CHLORO-M-CRESOL	22	19.3	20.1	21.3	0.239	0.254	0.284
324A 2-CHLOROPHENOL	26	19.5	20.3	21.6	0.248	0.264	0.295
325B 1,2-DICHLOROBENZENE	26	34.7	36.2	38.5	0.345	0.367	0.411
326B 1,3-DICHLOROBENZENE	26	22.7	23.7	25.2	0.279	0.297	0.333
327B 1,4-DICHLOROBENZENE	27	22.5	23.5	25.0	0.262	0.278	0.312
328B 3,3'-DICHLOROBENZIDINE	26	32.0	33.3	35.4	0.343	0.365	0.409
331A 2,4-DICHLOROPHENOL	27	21.1	22.0	23.4	0.262	0.279	0.312
334A 2,4-DIMETHYLPHENOL	27	19.0	19.8	21.0	0.256	0.272	0.305
335B 2,4-DINITROTOLUENE	26	79.0	82.3	87.5	0.810	0.862	0.965
336B 2,6-DINITROTOLUENE	18	64.2	66.7	70.7	0.851	0.903	1.006
337B 1,2-DIPHENYLHYDRAZINE	28	19.9	20.7	22.0	0.242	0.258	0.289
339B FLUORANTHENE	28	21.4	22.3	23.8	0.260	0.276	0.310
340B 4-CHLOROPHENYL PHENYL ETH	28	21.1	21.9	23.4	0.259	0.275	0.309
342B BIS (2-CHLOROISOPROPYL) E	22	23.0	24.0	25.5	0.292	0.311	0.347
352B HEXACHLORODUTADIENE	26	18.9	19.6	20.9	0.244	0.260	0.291
353B HEXACHLOROCYCLOPENTADIENE	22	20.0	20.8	22.1	0.269	0.286	0.320
354B ISOPHORONE	28	34.3	35.8	38.1	0.574	0.611	0.685
355B NAPHTHALENE	28	15.4	16.0	17.1	0.192	0.205	0.230
356B NITROBENZENE	12	22.5	23.3	24.6	0.186	0.196	0.217
357A 2-NITROPHENOL	28	20.5	21.4	22.8	0.256	0.273	0.306
358A 4-NITROPHENOL	23	33.8	35.2	37.4	0.498	0.538	0.592
359A 2,4-DINITROPHENOL	28	25.8	26.9	28.7	0.396	0.422	0.473
360A 4,6-DINITRO-O-CRESOL	27	44.3	46.2	49.1	0.401	0.427	0.478
362B N-NITROSODIPHENYLAMINE	15	26.7	27.7	29.3	0.259	0.274	0.304
364A PENTACHLOROPHENOL	28	22.4	23.3	24.8	0.261	0.278	0.311
365A PHENOL	28	29.3	30.5	32.5	0.572	0.609	0.682
366B BIS (2-ETHYLHEXYL) PHTHAL	27	15.5	16.2	17.2	0.251	0.268	0.300
368B DI-N-BUTYL PHTHALATE	27	14.5	15.1	16.0	0.210	0.224	0.250
369B DI-N-OCTYL PHTHALATE	28	22.7	23.7	25.2	0.281	0.299	0.335
370B DIETHYL PHTHALATE	28	18.1	18.9	20.1	0.215	0.229	0.256
371B DIMETHYL PHTHALATE	28	25.3	26.4	28.1	0.451	0.480	0.538
372B BENZO(A)ANTHRACENE	25	25.9	27.0	28.7	0.334	0.355	0.397
373B BENZO(A)PYRENE	27	35.4	36.9	39.3	0.541	0.576	0.645
374B BENZO(B)FLUORANTHENE	26	28.4	29.6	31.5	0.290	0.309	0.346
375B BENZO(K)FLUORANTHENE	24	59.2	61.7	65.6	0.627	0.666	0.746
376B CHRYSENE	26	22.4	23.3	24.8	0.278	0.296	0.331
377B ACENAPHTHYLENE	26	23.1	24.0	25.6	0.262	0.279	0.312
378B ANTHRACENE	27	19.9	20.7	22.0	0.240	0.256	0.287
379B BENZO(GHI)PERYLENE	26	44.3	46.1	49.1	0.380	0.405	0.453
380B FLUORENE	28	79.7	83.0	88.4	0.572	0.609	0.683
381B PHENANTHRENE	28	16.5	17.2	18.3	0.200	0.213	0.238
384B PYRENE	26	34.0	35.4	37.7	0.371	0.394	0.442
502B BETA NAPHTHYLAMINE	27	61.1	63.7	67.8	0.819	0.872	0.977
503B ALPHA PICOLINE	25	53.0	55.2	58.7	0.494	0.526	0.589
504B DIBENZOTHIOPHENE	24	30.6	31.8	33.8	0.305	0.325	0.363
505B DIBENZOFURAN	27	28.6	29.8	31.5	0.285	0.303	0.340

Table III-1 (Concluded)

COMPOUND		DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)	LOG LIMIT (5 POINTS)	LOG LIMIT (4 POINTS)	LOG LIMIT (3 POINTS)
506B N-DODECANE		24	29.6	30.8	32.8	0.310	0.329	0.369
507B DIPHENYLAMINE		20	26.9	28.0	29.7	0.270	0.286	0.320
508B DIPHENYLETHER		25	31.3	32.6	34.7	0.337	0.359	0.402
509B ALPHA TERPINEOL		25	32.2	33.5	35.6	0.368	0.391	0.438
510B STYRENE		24	32.7	34.1	36.2	0.352	0.374	0.418
511B DI-N-BUTYL AMINE		11	101.2	104.6	110.3	1.004	1.058	1.167
512B BIPHENYL		25	20.5	21.3	22.7	0.265	0.282	0.316
513B P-CYMENE		26	29.6	30.8	32.8	0.320	0.340	0.381
517B N-DECANE	C10	26	42.1	43.8	46.6	0.436	0.464	0.520
519B N-HEXADECANE	C16	27	29.3	30.5	32.5	0.339	0.360	0.404
521B N-EICOSANE	C20	27	38.3	39.9	42.5	0.404	0.430	0.482
523B N-TETRACOSANE	C24	28	56.5	58.9	62.7	0.527	0.561	0.629
526B N-TRIACONTANE	C30	26	114.1	118.8	126.5	0.964	1.026	1.149
602B 2-NAPHTHYL-D7-AMINE		27	116.0	120.9	128.7	0.959	1.021	1.144
603B 2-METHYLPYRIDINE-D7		23	47.7	49.6	52.8	0.441	0.468	0.524
604B DIBENZOTHIOPHENE-D8		22	26.4	27.5	29.2	0.296	0.315	0.352
605B DIBENZOFURAN-D8		26	17.6	18.3	19.5	0.203	0.216	0.241
606B N-DODECANE-D26		28	33.1	34.5	36.7	0.319	0.339	0.380
607B DIPHENYL-D10-AMINE		18	22.5	23.4	24.8	0.259	0.275	0.306
608B DIPHENYL-D10 ETHER		23	13.5	14.1	14.9	0.140	0.149	0.167
609B ALPHA-TERPINEOL-D3		24	55.9	58.2	61.9	0.572	0.609	0.681
610B STYRENE-2,3,4,5,6-D5		24	28.1	29.3	31.2	0.292	0.311	0.348
611B DI-N-BUTYL-D18-AMINE		8	91.8	94.5	99.0	1.101	1.153	1.259
612B DIPHENYL-D10		22	10.8	11.2	11.9	0.119	0.126	0.141
613B P-CYMENE-D14		24	29.0	30.2	32.1	0.272	0.290	0.324
617B N-DECANE-D22		25	32.8	34.2	36.4	0.302	0.321	0.360
619B N-HEXADECANE-D34		27	24.4	25.5	27.1	0.254	0.270	0.303
621B N-EICOSANE-D42		26	21.4	22.3	23.7	0.233	0.248	0.277
623B N-TETRACOSANE-D50		28	62.5	65.1	69.3	0.539	0.573	0.643
626B N-TRIACONTANE-D62		26	154.1	160.5	170.7	1.111	1.183	1.324
702B BETA NAPHTHYLAMINE		27	29.2	30.4	32.4	0.369	0.393	0.440
703B ALPHA PICOLINE		24	19.7	20.5	21.8	0.225	0.240	0.268
704B DIBENZOTHIOPHENE		25	17.7	18.4	19.6	0.217	0.230	0.258
705B DIBENZOFURAN		28	15.0	15.7	16.7	0.180	0.191	0.215
706B N-DODECANE	C12	26	19.5	20.3	21.7	0.227	0.241	0.270
707B DIPHENYLAMINE		20	15.9	16.5	17.6	0.176	0.187	0.209
708B DIPHENYLETHER		24	19.1	19.9	21.2	0.241	0.256	0.287
709B ALPHA TERPINEOL		23	21.6	22.5	23.9	0.267	0.284	0.318
710B STYRENE		26	19.8	20.6	21.9	0.261	0.277	0.310
711B DI-N-BUTYL AMINE		8	41.5	42.7	44.7	1.309	1.371	1.497
712B BIPHENYL		22	18.5	19.3	20.5	0.224	0.238	0.266
713B P-CYMENE		24	16.3	17.0	18.1	0.168	0.178	0.199
717B N-DECANE	C10	25	28.9	30.1	32.0	0.365	0.389	0.435
719B N-HEXADECANE	C16	28	19.4	20.2	21.5	0.241	0.256	0.287
721B N-EICOSANE	C20	28	24.8	25.9	27.6	0.271	0.289	0.324
723B N-TETRACOSANE	C24	28	26.5	27.6	29.4	0.266	0.283	0.318
726B N-TRIACONTANE	C30	28	32.9	34.3	36.5	0.411	0.437	0.490

Table III-2

CALIBRATION LIMITS -- EXTERNAL STANDARD

COMPOUND	DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)
001B ACENAPHTHENE	29	49.1	51.1	54.4
005B BENZIDINE	25	101.8	106.0	112.7
008B 1,2,4-TRICHLOROBENZENE	29	58.5	61.0	65.0
009B HEXACHLOROBENZENE	29	52.4	54.7	58.2
012B HEXACHLOROETHANE	25	41.1	42.8	45.5
018B BIS(2-CHLOROETHYL)ETHER	30	63.5	66.2	70.5
020B 2-CHLORONAPHTHALENE	24	37.1	38.6	41.0
021A 2,4,6-TRICHLOROPHENOL	29	57.5	59.9	63.8
022A P-CHLORO-M-CRESOL	23	52.4	54.6	58.0
024A 2-CHLOROPHENOL	30	61.3	63.9	68.1
025B 1,2-DICHLOROBENZENE	29	53.7	56.0	59.6
026B 1,3-DICHLOROBENZENE	30	55.1	57.5	61.2
027B 1,4-DICHLOROBENZENE	27	54.2	56.4	60.1
028B 3,3'-DICHLOROBENZIDINE	28	95.1	99.1	105.5
031A 2,4-DICHLOROPHENOL	29	55.4	57.7	61.5
034A 2,4-DIMETHYLPHENOL	29	52.6	54.8	58.4
035B 2,4-DINITROTOLUENE	29	61.1	63.6	67.8
036B 2,6-DINITROTOLUENE	29	58.5	60.9	64.9
037B 1,2-DIPHENYLHYDRAZINE	30	49.0	51.1	54.4
039B FLUORANTHENE	30	59.1	61.6	65.6
040B 4-CHLOROPHENYL PHENYL ETH	30	51.8	54.0	57.5
041B 4-BROMOPHENYL PHENYL ETHE	16	87.9	91.2	96.6
042B BIS (2-CHLOROISOPROPYL) E	24	67.8	70.6	75.1
052B HEXACHLOROBUTADIENE	27	51.6	53.7	57.2
053B HEXACHLOROCYCLOPENTADIENE	29	54.6	56.9	60.6
054B ISOPHORONE	30	68.4	71.3	75.9
055B NAPHTHALENE	29	43.3	45.2	48.1
056B NITROBENZENE	14	48.6	50.3	53.2
057A 2-NITROPHENOL	30	62.2	64.8	69.0
058A 4-NITROPHENOL	24	69.2	72.1	76.6
059A 2,4-DINITROPHENOL	28	79.7	83.0	88.4
060A 4,6-DINITRO-O-CRESOL	27	60.5	63.0	67.1
062B N-NITROSODIPHENYLAMINE	17	49.4	51.3	54.4
064A PENTACHLOROPHENOL	29	70.6	73.6	78.4
065A PHENOL	29	60.5	63.1	67.2
066B BIS (2-ETHYLHEXYL) PHTHAL	29	77.9	81.2	86.4
068B DI-N-BUTYL PHTHALATE	30	56.4	58.7	62.6
069B DI-N-OCTYL PHTHALATE	29	92.4	96.3	102.6
070B DIETHYL PHTHALATE	30	56.2	58.6	62.4
071B DIMETHYL PHTHALATE	29	52.0	54.2	57.7
072B BENZO(A)ANTHRACENE	28	65.8	68.6	73.0
073B BENZO(A)PYRENE	27	108.3	112.8	120.0
074B BENZO(B)FLUORANTHENE	25	94.2	98.1	104.3
075B BENZO(K)FLUORANTHENE	27	86.0	89.6	95.3
076B CHRYSENE	29	91.7	95.5	101.7
077B ACENAPHTHYLENE	27	46.2	48.1	51.2
078B ANTHRACENE	30	42.7	44.5	47.4
079B BENZO(GHI)PERYLENE	27	120.4	125.5	133.6
080B FLUORENE	29	50.4	52.5	55.9
081B PHENANTHRENE	30	52.1	54.3	57.9
084B PYRENE	28	66.5	69.3	73.8
164B 2,2'-DIFLUOROBIPHENYL	26	49.0	51.0	54.3

Table III-2 (Continued)

COMPOUND	DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)
201B ACENAPHTHENE-D10	26	43.2	45.0	47.9
205B BENZIDINE-D8 (RINGS-D8)	23	160.0	166.6	177.1
208B 1,2,4-TRICHLOROBENZENE-D3	23	56.5	58.8	62.5
209B HEXACHLOROBENZENE-13C6	23	49.3	51.3	54.5
212B HEXACHLOROETHANE-1-13C	20	48.2	50.1	53.2
218B BIS(2-CHLOROETHYL)-D8 ETH	22	62.2	64.7	68.7
220B 2-CHLORONAPHTHALENE-D7	28	57.2	59.6	63.5
221A 2,4,6-TRICHLOROPHENOL-3,5	25	52.9	55.0	58.6
222A 4-CHLORO-3-METHYLPHENOL-2	27	56.5	58.9	62.7
224A 2-CHLOROPHENOL-3,4,5,6-D4	25	54.6	56.9	60.5
225B 1,2-DICHLOROBENZENE-D4	26	62.3	64.9	69.0
226B 1,3-DICHLOROBENZENE-D4	26	62.3	64.9	69.0
227B 1,4-DICHLOROBENZENE-D4	27	59.6	62.1	66.0
228B 3,3'-DICHLOROBENZIDINE-D6	25	128.9	134.2	142.8
231A 2,4-DICHLOROPHENOL-3,5,6-	27	57.0	59.4	63.2
234A 2,4-DIMETHYLPHENOL-3,5,6-	27	57.9	60.3	64.2
235B 2,4-DINITROTOLUENE-3,5,6-	25	94.6	98.6	104.8
236B 2,6-DINITROTOLUENE-D3	16	44.1	45.8	48.5
237B 1,2-DIPHENYL-D10-HYDRAZIN	27	49.4	51.5	54.8
239B FLUORANTHENE-D10	28	79.6	82.9	88.3
240B 4-CHLOROPHENYL PHENYL-D5	26	51.6	53.8	57.2
242B BIS(2-CHLOROISOPPOPYL)ETH	22	63.3	65.9	70.0
252B HEXACHLORO-1,3-BUTADIENE-	25	53.9	56.1	59.7
253B HEXACHLOROCYCLOPENTADIENE	22	57.0	59.3	63.0
254B ISOPHORONE-D8	26	50.6	52.7	56.1
255B NAPHTHALENE-D8	27	47.8	49.8	53.1
256B NITROBENZENE-D5	12	43.8	45.3	47.8
257A 2-NITROPHENOL-3,4,5,6-D4	27	61.7	64.2	68.4
259A 4-NITROPHENOL-2,3,5,6-D4	23	81.0	84.3	89.7
259A 2,4-DINITROPHENOL-3,5,6-D	26	81.6	85.0	90.5
260A 4,6-DINITRO-O-CRESOL-D2	27	65.9	68.6	73.1
262B N-NITROSODIPHENYLAMINE-D6	20	55.8	58.0	61.5
264A PENTACHLOROPHENOL-13C6	28	72.5	75.6	80.5
265A PHENOL-2,3,4,5,6-D5	27	55.5	57.8	61.6
266B BIS(2-ETHYLHEXYL)PHTHALAT	26	90.1	93.9	99.9
268B DI-N-BUTYL PHTHALATE-D4	25	59.0	61.4	65.3
269B DI-N-OCTYL PHTHALATE-D4	27	133.2	138.8	147.7
270B DIETHYL PHTHALATE-3,4,5,6	26	49.6	51.7	55.0
271B DIMETHYL PHTHALATE-3,4,5,	28	87.3	91.0	96.9
272B BENZO(A)ANTHRACENE-D12	25	91.2	95.0	101.1
273B BENZO(A)PYRENE-D12	26	143.9	149.9	159.5
274B BENZO(B)FLUORANTHENE-D12	26	124.8	130.0	138.4
275B BENZO(K)FLUORANTHENE-D12	26	94.0	97.9	104.1
276B CHRYSENE-D12	27	103.2	107.5	114.5
277B ACENAPHTHYLENE-D8	26	45.3	47.2	50.2
278B ANTHRACENE-D10	25	47.6	49.5	52.7
279B BENZO(GHI)PERYLENE-D12	26	126.1	131.3	139.7
280B FLUORENE-D10	27	53.4	55.6	59.2
281B PHENANTHRENE-D10	28	50.6	52.7	56.2
284B PYRENE-D10	25	95.2	99.2	105.5

Table III-2 (Concluded)

COMPOUND		DENOM DEGR OF FREEDOM	CV LIMIT (5 POINTS)	CV LIMIT (4 POINTS)	CV LIMIT (3 POINTS)
506B N-DODECANE		27	54.2	56.5	60.1
507B DIPHENYLAMINE		24	38.5	40.1	42.7
508B DIPHENYLETHER		27	53.6	55.8	59.4
509B ALPHA TERPINEOL		28	63.5	66.2	70.5
510B STYRENE		27	65.0	67.7	72.1
511B DI-N-BUTYL AMINE		13	63.6	65.8	69.6
512B BIPHENYL		27	49.2	51.2	54.5
513B P-CYMENE		29	57.0	59.4	63.3
517B N-DECANE	C10	27	63.5	66.2	70.5
519B N-HEXADECANE	C16	30	54.2	56.5	60.2
521B N-EICOSANE	C20	30	56.4	58.7	62.6
523B N-TETRACOSANE	C24	30	70.0	73.0	77.7
526B N-TRIACONTANE	C30	29	92.1	96.0	102.3
602B 2-NAPHTHYL-D7-AMINE		27	74.2	77.3	82.3
603B 2-METHYLPYRIDINE-D7		22	48.0	49.9	53.0
604B DIBENZOTHIOPHENE-D8		24	53.4	55.5	59.1
605B DIBENZOFURAN-D8		28	47.6	49.6	52.8
606B N-DODECANE-D26		25	50.4	52.4	55.8
607B DIPHENYL-D10-AMINE		19	44.0	45.8	48.6
608B DIPHENYL-D10 ETHER		25	45.0	46.8	49.8
609B ALPHA-TERPINEOL-D3		24	71.4	74.3	79.1
610B STYRENE-2,3,4,5,6-D5		23	50.8	52.8	56.2
611B DI-N-BUTYL-D18-AMINE		7	101.1	103.8	108.4
612B DIPHENYL-D10		22	48.5	50.4	53.6
613B P-CYMENE-D14		24	41.0	42.7	45.4
617B N-DECANE-D22		26	48.4	50.4	53.6
619B N-HEXADECANE-D34		27	47.8	49.8	53.0
621B N-EICOSANE-D42		27	49.9	52.0	55.3
623B N-TETRACOSANE-D50		27	74.0	77.1	82.1
626B N-TRIACONTANE-D62		26	136.0	141.7	150.8

calibration curve was returned, as area ratio decreasing with increasing input ratio was considered to be unacceptable. The goodness of fit of the log-log calibration curve was then tested by comparing the residual root mean sum of squares from the regression with

$$\text{Log Limit} = \left(\sigma_L^2 F_{N-2,DF}(.95)\right)^{\frac{1}{2}} ,$$

where σ_L^2 is the weighted average variance of the logarithms of the response factors for CAL 100, PRR, and VER across all labs in the study; DF is the number of degrees of freedom in the estimate of σ_L^2; and N is again the number of calibration points. (Two is subtracted from N because two parameters are being estimated in the regression. The variance of the logarithms of the RFs is an approximation to the residual error of the regression in the neighborhood of $\gamma = 1$.)* These limits are also tabulated in Table III-1. If the residual error from the regression was too large, no calibration was performed; otherwise the log-log calibration curve was used.

The goodness-of-fit test was used to guard against spurious fluctuations in the calibration curve. A few curves with $\gamma < 1/4$ were disallowed because of numerical instability in applying the calibration (i.e. small variation in area ratio would produce large variation in the calculated amount).

Calculation of Amounts

The results of the calibrations are tabulated in Table III-3, by laboratory. The cell frequency indicate the number of compounds at each

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* For $\gamma = 1$ the logarithmic calibration curve becomes

$$\log (AR) = \log (b) + \log (IR) + \varepsilon_L$$

or $\log (AR) - \log (IR) = \log (RF) = \log (b) + \varepsilon_L$

hence $\text{Var} (\log (RF)) = \sigma_L^2$

Table III-3

FREQUENCIES OF CALIBRATION RESULTS

LABCODE MESSAGE

FREQUENCY	0 CALIBRATION	1 CALIBRATION	2 CALIBRATION	3 CALIBRATION	CURVATURE < 1/4	CURVATURE <= 0	LIN CAL REJ-LBL	LINEAR CALIBRATI	LOG-LOG CALIBRAT	LOG-LOG FIT REJE	TOTAL
A	3	0	0	0	0	0	1	182	13	2	201
B	1	0	0	0	0	2	2	179	12	4	200
C	8	2	3	9	0	0	2	134	37	3	198
D	5	0	0	0	2	1	2	162	18	3	193
E	6	3	7	1	3	0	14	80	41	44	199
F	13	0	0	0	1	0	4	120	55	5	198
G	10	0	0	0	0	0	1	177	8	1	197
H	17	0	0	0	0	0	1	156	10	0	184
I	18	0	0	4	0	0	0	166	6	0	194
J	12	0	0	0	0	0	0	171	12	2	197
K	1	0	0	0	0	0	0	192	4	3	200
L	2	0	0	4	0	1	1	178	16	1	203
M	6	6	0	0	0	0	21	132	18	20	203
N	134	0	0	0	0	0	0	0	0	0	134
O	8	0	0	0	1	0	0	175	5	3	192
TOTAL	244	11	10	18	7	4	49	2204	255	91	2893

laboratory experiencing the particular calibration outcome. Overall, among those situations for which sufficient data was available (i.e. four or five calibration points), 84 percent were acceptable for linear calibration, 10 percent were rejected for linearity but had an acceptable log-log fit, 4 percent had unacceptable linear and log-log fits, and 2 percent were labeled compounds rejected for linear fit (no log-log fit could be performed because the input ratio is 1 on all samples).

The calibration results were then applied to all samples in the study, with

$$\text{Amount} = \frac{\text{Area Ratio}}{\text{RF}} \times \text{Input Concentration (Reference)}$$

for linear calibrations, and

$$\text{Amount} = \left(\frac{\text{Area Ratio}}{b}\right)^{(1/\gamma)} \times \text{Input Concentration (Reference)}$$

for log-log calibrations. According to the study design, entries were calculated for compounds by internal standard, labeled analogues by internal standard, and compounds by isotope dilution. Amount values were not calculated by external standard methods.

Linearity Limits for Calibration

Across-compound summary percentile statistics on the CV limits for testing calibration linearity for each compound series are presented in Table III-4. The median number across compounds should be an acceptable number to use for deciding whether to assume the proportional calibration based on the average response factor or use a more detailed representation of the response curve. This has been done both for 3 and 5 calibration points, for each compound series.

Table III-4

SUMMARY OF COEFFICIENT OF VARIATION LIMITS
FOR CALIBRATION LINEARITY

Methods/ Series	3 Calibration Points			5 Calibration Points		
	25th %ile	Median	75th %ile	25th %ile	Median	75th %ile
External Standard	57.7	64.3	74.8	52.0	58.0	67.5
External Std. (L.A.)	54.8	63.0	89.7	49.4	57.0	81.0
Internal Standard	32.1	37.2	64.1	29.0	33.6	57.8
Internal Std. (L.A.)	28.8	36.4	84.5	26.0	32.8	76.4
Isotope Dilution	21.5	24.8	32.4	19.4	22.5	29.2

Note: L.A. = Labeled analogs.

IV DATA SCREENING

After the amounts were calculated, they were screened for outliers, both on a laboratory and individual-point basis. First, the laboratories were ranked and screened according to Youden's extreme rank method (see Appendix G) in order to identify laboratories with significantly poorer results than the majority of the study laboratories. For each compound, the absolute deviations of each laboratory's amount from the median amount across all laboratories for that sample were used for ranking. The rank sums were taken across all eleven samples for the labeled compounds, and across all samples except the BLK and EPA for unlabeled compounds. Each compound was tested separately, and laboratories that were unable to report one or more of the measurements were not evaluated for that compound.* Table IV-1 presents the results by compound and laboratory. Laboratory codes are listed for each column. A "." indicates that not all sample values were reported or quantifiable for that compound at that laboratory. A "+" indicates all data was present and the laboratory ranking results were acceptable for that laboratory. "HI" indicates the occurrence of an unacceptably high proportion of extreme values reported by that laboratory for that compound. The binomial probability at $p = .05$ of seeing the observed number of compound rejections or greater for each laboratory was computed. Laboratories with a binomial probability of less than .05 of that many rejections or more were rejected overall and removed from the study. Lab E, with 29 out of 89 rejections, was removed on this basis. Table IV-2 presents totals of the numbers of compounds, number of rejections, and overall results.

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* One laboratory did not report CAL 200 and another did not report APS, EPA, and BLK. These two laboratories, B and I, could not be screened in this protocol. However, preliminary examinations excluding the missing samples from the calculation indicated no problems with these laboratories.

Second, the amount values were then screened individually for outliers. Two methods were applied: (1) a robust quantile method based on the median and interquantile distance (QSCREEN), and (2) Ferguson's method, based on the sample kurtosis (FSCREEN). These methods are described in Appendix H. Both screening methods were applied to each sample, across laboratories on the logarithms of the amounts. QSCREEN was applied at level .001 and FSCREEN at level .01 for reasons described in Appendix H. If fewer than five laboratories reported detectable amounts of a compound, neither screening procedure could be applied. A total of 501 points were identified as outliers by the quantile method, and 251 points by Ferguson's method. Out of 26,195 points screened, 508 points were identified by at least one method (approximately 2 percent). Amounts for which either method rejected were set to missing. Details are given in Appendix H.

Table IV-1

LABORATORY RANKING RESULTS

CMPD_NO	A	B	C	D	E	F	G	H	I	J	K	L	M	O
001B ACENAPHTHENE	+	.	+	+	+	+	+	+	.	+	.	+	+	+
005B BENZIDINE	+	.	.	+	.	+	+	+	.	.	+	+	+	+
008B 1,2,4-TRICHLOROBENZENE	+	.	.	+	HI	+	+	+	.	+	.	+	+	+
009B HEXACHLOROBENZENE	+	.	+	+	+	+	+	+	.	+	.	+	+	+
012B HEXACHLOROETHANE	+	.	+	.	+	+	+	+	.	+	.	+	+	.
018B BIS(2-CHLOROETHYL)ETHER	+	.	+	+	+	+	+	+	.	+	.	+	+	.
020B 2-CHLORONAPHTHALENE	+	.	+	+	.	+	+	.	.	+	.	+	+	.
021A 2,4,6-TRICHLOROPHENOL	+	.	+	+	HI	HI	+	+	.	.	.	+	+	.
022A P-CHLORO-M-CRESOL	+	.	+	.	+	+	.	.	.	.	.	+	+	+
024A 2-CHLOROPHENOL	+	.	+	+	HI	+	+	+	.	+	.	+	+	.
025B 1,2-DICHLOROBENZENE	+	.	+	+	+	+	+	+	.	+	.	+	+	+
026B 1,3-DICHLOROBENZENE	+	.	+	+	HI	+	+	+	.	+	.	+	+	+
027B 1,4-DICHLOROBENZENE	+	.	+	+	HI	.	+	+	.	+	.	.	+	+
028B 3,3'-DICHLOROBENZIDINE	+	.	.	+	.	+	+	+	.	+	+	+	.	+
031A 2,4-DICHLOROPHENOL	+	.	+	+	+	+	+	+	.	+	.	+	+	.
034A 2,4-DIMETHYLPHENOL	+	.	+	+	HI	+	+	+	.	+	.	+	+	.
035B 2,4-DINITROTOLUENE	+	.	+	+	+	.	+	+	.	+	.	+	+	.
036B 2,6-DINITROTOLUENE	+	.	+	+	+	+	+	+	.	+	.	+	+	+
037B 1,2-DIPHENYLHYDRAZINE	.	.	+	+	.	+	+	+	.	+	.	+	.	+
039B FLUORANTHENE	+	.	+	+	HI	+	+	+	.	+	.	+	.	+
040B 4-CHLOROPHENYL PHENYL ETH	+	.	+	+	+	+	+	+	.	+	.	+	.	+
041B 4-BROMOPHENYL PHENYL ETHE	+	.	+	+	+	+	.	+	.	.	.	+	+	+
042B BIS (2-CHLOROISOPROPYL) E	+	.	+	+	+	+	.	.	.	+	+	+	+	+
052B HEXACHLOROBUTADIENE	+	.	+	+	+	+	+	+	.	+	.	.	.	+
053B HEXACHLOROCYCLOPENTADIENE	+	.	+	+	+	+	+	+	.	.	.	+	+	.
054B ISOPHORONE	+	.	+	+	+	+	+	+	.	+	.	+	+	+
055B NAPHTHALENE	+	.	+	+	HI	+	+	+	.	+	.	+	+	+
056B NITROBENZENE	+	.	.	.	.	.	.	.	.	.	.	+	+	.
057A 2-NITROPHENOL	.	.	+	+	+	+	+	+	.	.	.	.	+	+
058A 4-NITROPHENOL	+	.	.	+	.	.	+	.	.	+	.	+	HI	+
059A 2,4-DINITROPHENOL	+	.	+	+	+	+	+	+	.	+	+	+	+	+
060A 4,6-DINITRO-O-CRESOL	+	.	.	+	+	+	+	+	.	+	.	+	.	+
062B N-NITROSODIPHENYLAMINE	.	.	.	.	.	+	.	+	.	.	.	+	.	.
064A PENTACHLOROPHENOL	+	.	.	+	+	+	+	+	.	+	.	+	+	+
065A PHENOL	+	.	+	+	+	+	+	.	.	+	.	+	+	.
066B BIS (2-ETHYLHEXYL) PHTHAL	+	.	+	.	+	+	+	.	.	+	+	+	.	+
068B DI-N-BUTYL PHTHALATE	+	.	+	+	+	+	+	+	.	+	.	+	.	+
069B DI-N-OCTYL PHTHALATE	+	.	+	+	HI	+	+	+	.	+	+	+	+	.
070B DIETHYL PHTHALATE	+	.	+	+	.	+	+	+	.	.	.	+	+	.
071B DIMETHYL PHTHALATE	+	.	+	+	+	+	+	+	.	+	.	+	.	+
072B BENZO(A)ANTHRANCENE	+	.	+	+	HI	+	+	+	.	+	+	+	.	+
073B BENZO(A)PYRENE	+	.	+	+	+	+	+	+	.	.	+	+	.	+
074B BENZO(B)FLUORANTHENE	+	.	+	+	.	.	+	.	.	+	+	+	.	+
075B BENZO(K)FLUORANTHENE	+	.	+	+	+	+	+	+	.	+	+	+	.	+
076B CHRYSENE	+	.	+	+	HI	+	+	+	.	+	+	+	+	+
077B ACENAPHTHYLENE	.	.	+	+	HI	.	+	+	.	.	.	+	+	.
078B ANTHRACENE	+	.	+	+	+	.	+	+	.	+	.	+	+	+
079B BENZO(GHI)PERYLENE	+	.	.	+	.	+	+	+	.	+	+	+	.	+
080B FLUORENE	+	.	+	+	HI	+	+	+	.	+	.	+	+	+
081B PHENANTHRENE	+	.	+	+	+	+	+	+	.	+	.	+	+	+
084B PYRENE	+	.	+	+	+	+	+	+	.	+	.	+	.	.
201B ACENAPHTHENE-D10	+	.	+	.	.	+	+	+	.	+	.	+	+	.
205B BENZIDINE-D8 (RINGS-D8)	+	.	.	+	HI	.	+	+	.	.	+	+	+	+
206B 1,2,4-TRICHLOROBENZENE-D3	+	.	.	+	.	+	+	+	.	+	.	.	+	+

Table IV-1 (Continued)

CMPD_NO	A	B	C	D	E	F	G	H	I	J	K	L	M	O
209B HEXACHLOROBENZENE-13C6	+	.	+	+	+	+	.	.	.	+	.	+	+	+
212B HEXACHLOROETHANE-1-13C	+	.	+	+	.	.	.	+	.	+	.	+	+	.
218B BIS(2-CHLOROETHYL)-D8 ETH	+	.	.	+	.	+	HI	+	.	.	.	+	+	.
220B 2-CHLORONAPHTHALENE-D7	+	.	+	+	.	+	.	+	.	+	.	+	+	HI
221A 2,4,6-TRICHLOROPHENOL-3,5	+	.	+	.	.	.	+	+	.	+	.	+	.	.
222A 4-CHLORO-3-METHYLPHENOL-2	.	.	+	+	.	+	+	+	.	+	.	+	+	.
224A 2-CHLOROPHENOL-3,4,5,6-D4	+	.	+	+	.	+	+	.	.	+	.	+	+	.
225B 1,2-DICHLOROBENZENE-D4	+	.	.	+	.	+	+	+	.	+	.	+	+	+
226B 1,3-DICHLOROBENZENE-D4	+	.	+	+	.	+	+	.	.	+	.	+	+	.
227B 1,4-DICHLOROBENZENE-D4	+	.	+	+	.	.	+	+	.	+	.	+	+	.
228B 3,3'-DICHLOROBENZIDINE-D6	HI	.	HI	.	HI	+	+	+	.	+	+	+	.	+
231A 2,4-DICHLOROPHENOL-3,5,6-	+	.	+	+	.	+	+	+	.	+	.	.	+	.
234A 2,4-DIMETHYLPHENOL-3,5,6-	+	.	.	+	.	+	+	+	.	+	.	+	+	.
235B 2,4-DINITROTOLUENE-3,5,6-	+	.	+	.	.	.	+	+	.	.	.	+	HI	.
236B 2,6-DINITROTOLUENE-D3	+	.	+	.	.	.	+	.	.	.	.	+	.	.
237B 1,2-DIPHENYL-D10-HYDRAZIN	+	.	+	+	.	+	+	+	.	+	.	+	.	.
239B FLUORANTHENE-D10	+	.	+	+	HI	+	+	+	.	+	.	+	+	+
240B 4-CHLOROPHENYL PHENYL-D5	+	.	+	+	.	+	+	+	.	+	.	+	+	+
242B BIS(2-CHLOROISOPROPYL)ETH	+	.	+	+	.	+	.	.	.	+	.	+	+	+
252B HEXACHLORO-1,3-BUTADIENE-	+	.	+	+	+	+	+	.	.	+	.	.	.	+
253B HEXACHLOROCYCLOPENTADIENE	+	.	.	.	+	+	+	+	.	+	+	+	+	+
254B ISOPHORONE-D8	+	.	.	+	+	+	+	+	.	+	.	.	+	+
255B NAPHTHALENE-D8	+	.	+	+	.	+	+	+	.	+	.	+	+	+
256B NITROBENZENE-D5	+	.	.	.	.	.	.	.	.	.	.	+	.	.
257A 2-NITROPHENOL-3,4,5,6-D4	+	.	+	+	.	+	+	+	.	.	.	+	+	.
258A 4-NITROPHENOL-2,3,5,6-D4	+	.	+	+	.	.	+	.	.	+	.	+	+	.
259A 2,4-DINITROPHENOL-3,5,6-D	+	.	+	+	.	+	+	+	.	+	.	+	.	.
260A 4,6-DINITRO-O-CRESOL-D2	+	.	+	+	.	+	+	+	.	+	.	+	.	.
262B N-NITROSODIPHENYLAMINE-D6	+	.	+	+	.	.	.	+	.	.	.	+	.	.
264A PENTACHLOROPHENOL-13C6	+	.	+	+	.	+	+	+	.	+	.	+	.	.
265A PHENOL-2,3,4,5,6-D5	+	.	+	+	+	+	+	+	.	+	.	+	+	+
266B BIS(2-ETHYLHEXYL)PHTHALAT	HI	.	.	+	HI	+	+	+	.	+	+	+	.	.
268B DI-N-BUTYL PHTHALATE-D4	HI	.	+	+	+	+	.	+	.	+	.	+	.	.
269B DI-N-OCTYL PHTHALATE-D4	+	.	+	+	+	+	+	+	.	+	+	+	HI	.
270B DIETHYL PHTHALATE-3,4,5,6	+	.	.	+	.	+	+	+	.	+	.	+	+	.
271B DIMETHYL PHTHALATE-3,4,5,	+	.	+	.	.	+	.	+	.	+	.	+	+	+
272B BENZO(A)ANTHRACENE-D12	+	.	HI	+	+	+	+	+	.	+	+	+	.	.
273B BENZO(A)PYRENE-D12	HI	.	+	+	+	+	+	+	.	+	+	+	.	.
274B BENZO(B)FLUORANTHENE-D12	+	.	HI	+	+	.	+	.	.	+	+	+	.	+
275B BENZO(K)FLUORANTHENE-D12	+	.	HI	+	+	+	+	+	.	+	+	+	.	+
276B CHRYSENE-D12	+	.	HI	+	HI	.	+	+	.	+	+	+	.	+
277B ACENAPHTHYLENE-D8	+	.	+	+	.	.	+	+	.	.	.	+	+	.
278B ANTHRACENE-D10	+	.	+	.	+	.	+	+	.	+	.	+	.	.
279B BENZO(GHI)PERYLENE-D12	+	.	+	+	.	+	+	+	.	+	+	+	.	+
280B FLUORENE-D10	+	.	+	+	.	+	+	+	.	+	.	+	HI	+
281B PHENANTHRENE-D10	+	.	+	+	+	+	+	+	.	+	.	+	.	.
284B PYRENE-D10	+	.	+	+	HI	+	+	+	.	+	.	+	HI	.
301B ACENAPHTHENE	+	.	+	.	+	+	+	+	.	+	+	.	+	+
305B BENZIDINE	+	.	.	.	+	.	.	.	.	.	+	+	+	+
308B 1,2,4-TRICHLOROBENZENE	+	.	.	+	.	+	+	+	.	+	+	+	+	+
309B HEXACHLOROBENZENE	+	.	+	.	HI	+	.	.	.	+	+	+	+	+
312B HEXACHLOROETHANE	+	.	+	.	+	.	.	+	.	+	+	+	+	.
316B BIS(2-CHLOROETHYL)ETHER	+	.	+	+	.	+	+	.	.	.	+	.	+	.
320B 2-CHLORONAPHTHALENE	+	.	+	+	.	+	+	.	.	+	.	+	.	HI

Table IV-1 (Continued)

CMPD_NO	A	B	C	D	E	F	G	H	I	J	K	L	M	O
321A 2,4,6-TRICHLOROPHENOL	+	.	+	.	.	.	+	+	.	HI	+	+	+	+
322A P-CHLORO-M-CRESOL	+	.	+	.	.	.	+	.	.	.	.	+	+	.
324A 2-CHLOROPHENOL	+	.	+	+	.	+	+	.	.	+	+	+	+	+
325B 1,2-DICHLOROBENZENE	+	.	.	+	+	HI	+	+	.	+	+	.	+	+
326B 1,3-DICHLOROBENZENE	+	.	+	+	.	+	+	.	.	+	+	+	+	+
327B 1,4-DICHLOROBENZENE	.	.	+	+	.	+	+	+	.	+	+	+	+	+
328B 3,3'-DICHLOROBENZIDINE	+	.	.	.	.	+	+	+	.	+	+	.	.	+
331A 2,4-DICHLOROPHENOL	.	.	+	+	.	+	+	+	.	+	+	+	+	+
334A 2,4-DIMETHYLPHENOL	.	.	.	.	.	HI	+	+	.	+	+	+	+	.
335B 2,4-DINITROTOLUENE	+	.	+	.	+	+	+	+	.	.	+	+	HI	+
336B 2,6-DINITROTOLUENE	.	.	+	+	+	.	+	.	.	.	+	+	.	.
337B 1,2-DIPHENYLHYDRAZINE	+	.	+	+	+	+	+	+	.	+	+	.	.	+
339B FLUORANTHENE	+	.	+	+	.	+	+	+	.	+	+	+	.	+
340B 4-CHLOROPHENYL PHENYL ETH	+	.	+	+	.	HI	+	+	.	+	+	+	.	+
342B BIS (2-CHLOROISOPROPYL) E	+	.	+	+	.	+	.	.	.	+	+	+	+	+
352B HEXACHLOROBUTADIENE	+	.	+	+	.	+	+	.	.	+	+	+	.	+
353B HEXACHLOROCYCLOPENTADIENE	+	.	.	.	.	.	+	+	.	+	+	+	+	HI
354B ISOPHORONE	HI	.	.	.	.	+	+	+	.	+	+	+	.	+
355B NAPHTHALENE	+	.	+	+	.	+	+	+	.	+	+	+	+	+
356B NITROBENZENE	+	.	.	.	.	.	.	.	.	.	+	+	.	.
357A 2-NITROPHENOL	+	.	+	.	.	.	+	+	.	+	+	+	+	+
358A 4-NITROPHENOL	+	.	.	+	.	.	+	.	.	+	+	+	HI	+
359A 2,4-DINITROPHENOL	+	.	+	+	.	HI	+	+	.	+	+	+	.	+
360A 4,6-DINITRO-O-CRESOL	+	.	.	+	+	+	+	+	.	+	+	+	.	+
362B N-NITROSODIPHENYLAMINE	+	.	.	+	.	.	.	+	.	.	+	+	.	.
364A PENTACHLOROPHENOL	+	.	.	+	.	HI	+	+	.	+	+	+	+	+
365A PHENOL	+	.	+	+	.	+	+	+	.	+	+	+	+	+
366B BIS (2-ETHYLHEXYL) PHTHAL	+	.	+	+	.	+	+	.	.	+	+	.	+	+
368B DI-N-BUTYL PHTHALATE	+	.	+	+	.	+	.	+	.	HI	+	+	+	+
369B DI-N-OCTYL PHTHALATE	+	.	+	+	.	+	+	+	.	+	+	.	+	+
370B DIETHYL PHTHALATE	+	.	+	+	.	HI	+	+	.	.	+	.	+	+
371B DIMETHYL PHTHALATE	+	.	.	.	.	+	+	+	.	+	+	+	.	.
372B BENZO(A)ANTHRACENE	+	.	+	.	.	.	+	+	.	+	+	+	.	+
373B BENZO(A)PYRENE	+	.	+	HI	.	.	+	+	.	.	+	.	.	+
374B BENZO(B)FLUORANTHENE	+	.	.	+	.	+	+	.	.	+	+	+	.	.
375B BENZO(K)FLUORANTHENE	+	.	.	+	+	+	+	.	.	.	+	+	.	+
376B CHRYSENE	+	.	+	.	.	+	+	+	.	.	+	+	+	+
377B ACENAPHTHYLENE	.	.	+	+	.	+	+	+	.	+	+	+	+	.
378B ANTHRACENE	+	.	+	+	.	.	+	+	.	+	+	.	+	+
379B BENZO(GHI)PERYLENE	+	.	.	+	.	+	+	+	.	.	+	.	.	+
380B FLUORENE	+	.	+	+	+	+	+	+	.	+	+	+	+	.
381B PHENANTHRENE	+	.	+	+	.	+	+	+	.	+	+	.	+	+
384B PYRENE	+	.	+	HI	.	.	+	+	.	+	+	+	HI	.
502B BETA NAPHTHYLAMINE	+	.	.	+	+	.	HI	+	.	+	.	.	+	+
503B ALPHA PICOLINE	+	.	+	+	+	+	+	.	.	+	.	+	+	+
504B DIBENZOTHIOPHENE	+	.	+	+	+	+	+	+	.	.	.	+	+	+
505B DIBENZOFURAN	+	.	+	+	.	+	+	+	.	+	.	+	+	+
506B N-DODECANE	+	.	+	+	HI	+	+	+	.	+	.	+	+	+
507B DIPHENYLAMINE	+	.	+	+	.	.	+	.	.	+	.	+	.	+
508B DIPHENYLETHER	+	.	+	+	HI	+	+	.	.	+	.	+	+	+
509B ALPHA TERPINEOL	+	.	.	+	+	+	+	+	.	+	.	+	+	.
510B STYRENE	.	.	+	+	+	+	+	+	.	+	.	.	+	+
511B DI-N-BUTYL AMINE	+	.	+	+	.	+	+	+	.	+	+	+	+	.
512B BIPHENYL	+	.	+	+	HI	+	+	+	.	+	.	+	+	.

Table IV-1 (Concluded)

CMPD_NO		A	B	C	D	E	F	G	H	I	J	K	L	M	O
513B P-CYMENE		+	.	+	+	HI	+	+	+	.	+	.	.	+	+
517B N-DECANE	C10	+	.	+	+	+	+	+	+	.	+	+	+	+	+
519B N-HEXADECANE	C16	+	.	+	+	HI	+	+	+	.	+	.	.	+	+
521B N-EICOSANE	C20	+	.	+	+	+	+	+	+	.	+	.	+	+	+
523B N-TETRACOSANE	C24	+	.	+	+	HI	+	+	+	.	+	+	+	.	+
526B N-TRIACONTANE	C30	+	.	+	+	.	+	+	+	.	+	.	+	.	.
602B 2-NAPHTHYL-D7-AMINE		+	.	+	+	.	.	.	+	.	+	+	.	+	.
603B 2-METHYLPYRIDINE-D7		.	.	+	+	.	+	+	.	.	+	+	+	+	+
604B DIBENZOTHIOPHENE-D8		+	.	.	+	.	+	.	+	.	.	.	+	.	.
605B DIBENZOFURAN-D8		+	.	+	+	.	+	+	+	.	+	.	+	.	.
606B N-DODECANE-D26		+	.	+	+	.	+	+	+	.	HI	+	+	+	+
607B DIPHENYL-D10-AMINE		+	.	.	+	.	.	+	.	.	+	.	+	.	.
608B DIPHENYL-D10 ETHER		+	.	+	+	.	.	+	+	.	+	.	+	+	.
609B ALPHA-TERPINEOL-D3		+	.	+	+	.	+	+	.	.	+	+	.	+	HI
610B STYRENE-2,3,4,5,6-D5		+	.	+	+	.	+	+	+	.	+	.	+	+	.
611B DI-N-BUTYL-D18-AMINE		.	.	.	+	.	.	.	.	.	+	+	.	.	.
612B DIPHENYL-D10		.	.	+	.	.	.	.	.	.	+	.	.	.	.
613B P-CYMENE-D14		+	.	.	+	.	+	+	+	.	+	.	+	.	+
617B N-DECANE-D22		+	.	+	.	+	+	+	+	.	+	.	+	.	+
619B N-HEXADECANE-D34		+	.	+	+	.	+	+	+	.	+	.	+	+	+
621B N-EICOSANE-D42		+	.	+	+	.	+	+	+	.	+	.	+	.	.
623B N-TETRACOSANE-D50		+	.	+	+	HI	+	+	+	.	+	+	+	.	+
626B N-TRIACONTANE-D62		HI	.	+	+	HI	+	+	+	.	.	.	+	.	.
702B BETA NAPHTHYLAMINE		+	.	.	.	+	.	.	+	.	+	+	.	+	.
703B ALPHA PICOLINE		+	.	.	+	.	+	+	.	.	+	.	+	.	+
704B DIBENZOTHIOPHENE		+	.	.	+	.	+	+	+	.	.	+	.	+	+
705B DIBENZOFURAN		+	.	+	+	.	.	+	+	.	+	+	+	.	+
706B N-DODECANE	C12	+	.	+	+	.	.	+	+	.	+	+	+	+	+
707B DIPHENYLAMINE		+	.	.	+	.	.	+	.	.	+	+	+	.	+
708B DIPHENYLETHER		+	.	+	+	.	.	+	.	.	+	+	+	+	+
709B ALPHA TERPINEOL		.	.	.	+	HI	+	.	.	.	.	.	.	+	.
710B STYRENE		+	.	+	+	.	+	+	+	.	+	+	.	+	+
711B DI-N-BUTYL AMINE		.	.	.	.	.	.	.	.	.	+	+	.	.	.
712B BIPHENYL		+	.	+	+	.	.	+	+	.	+	.	+	.	.
713B P-CYMENE		+	.	+	+	.	+	+	+	.	+	+	.	.	+
717B N-DECANE	C10	+	.	+	+	.	+	+	+	.	+	+	+	+	.
719B N-HEXADECANE	C16	+	.	+	+	.	+	+	+	.	+	+	.	HI	+
721B N-EICOSANE	C20	+	.	+	+	.	+	+	+	.	+	+	.	+	+
723B N-TETRACOSANE	C24	+	.	+	+	.	.	+	+	.	+	+	+	+	+
726B N-TRIACONTANE	C30	+	.	+	+	.	.	+	+	.	.	+	+	.	.

Table IV-2

SUMMARY OF LABORATORY RANKING RESULTS

LAB	NUMBER OF CMPDS	NUMBER OF REJECTS	REJECT LAB
A	185	6	+
B	0	.	.
C	153	5	+
D	167	2	+
E	89	29	HI
F	151	7	+
G	174	2	+
H	157	0	+
I	0	.	.
J	164	3	+
K	94	0	+
L	166	0	+
M	127	9	+
O	124	4	+

V METHOD PRECISION AND ACCURACY

After the data set was recomputed and screened as described in the preceding chapters, a number of statistical analyses were performed to evaluate the performance of the proposed analytical method in relation to current analytical methods. The method precision and accuracy were calculated from the analyses of the aqueous performance standard (APS) sample. This water sample, containing 100 µg/L of each of the compounds in the study, was subjected to the complete method: extraction, injection, and calibration. Three measures of method performance were calculated with respect to the analysis for each compound by internal standard methods, the corresponding labeled analogue by internal standard, and the compound by isotope dilution:

(1) Percentage of cases with "not detected" or unquantifiable results.

(2) Method precision, calculated as the coefficient of variation of the reported results:

$$100\ S_n/\bar{X} \quad \text{(percent)}$$

(3) Method accuracy, calculated as the relative absolute deviation of the average measured concentration from the true value (100 ug/L):

$$100 \left| \frac{\bar{X} - 100}{100} \right| \quad \text{(percent)} \quad .$$

Here $\bar{X}$ and S_n are the sample mean and standard deviation of the reported analyses across laboratories (excluding "not detected" results). These values are tabulated in Table V-1 by compound. Figures V-1 and V-2 show the method precision and accuracy plotted versus compound number.

The median accuracy across all compounds is 22.3 percent for internal standard, 26.1 percent for labeled compounds by internal standard, and 7.6 percent for compounds by isotope dilution. This considerable

improvement in the accuracy (bias) of the isotope dilution method is due to the addition of the reference compounds (labeled analogues) prior to the extraction process. This corrects for recovery problems during the extraction, since both the compound and its labeled analogue should be extracted with similar efficiency.

The median precision across all compounds is 29.8 percent for internal standard, 33.4 percent for analogues by internal standard, and 14.3 percent by isotope dilution. This improvement presumably is due to the closer match between the response sensitivities of the compound and the analogue than between those of the compound and the general reference standard.

Thus, the isotope dilution methodology can be seen to be noticeably more precise and more accurate than the internal standard method. However, there is also some indication that isotope dilution requires more care in its application, since the median proportion of laboratories that could not quantify or detect compounds is 15.4 percent by internal standard, 8.3 percent for analogues by internal standard, but 23.1 percent by isotope dilution. These problems may be expected to diminish as the laboratories gain experience with the isotope dilution method, and with increased use of direct computer submission of data on magnetic media, which should eliminate transcription and coding as a source of error. Also, in practice, Method 1625 specifies that if a laboratory is unable to quantify a compound by isotope dilution, then the laboratory should report the quantification by internal standard methods, thus limiting the nonquantitation to a minimum.

Table V-1

PRECISION AND ACCURACY EVALUATION
AQUEOUS PERFORMANCE STANDARD

	MEASUREMENT											
	COMPOUND BY INTERNAL STANDARD				LABELLED ANALOG BY INTERNAL STANDARD				COMPOUND BY ISOTOPE DILUTION			
COMPOUND	N OF LABS	NOT DETECT /QUANT	PRECISION	ACCURACY	N OF LABS	NOT DETECT /QUANT	PRECISION	ACCURACY	N OF LABS	NOT DETECT /QUANT	PRECISION	ACCURACY
	#	%	%	%	#	%	%	%	#	%	%	%
001B ACENAPHTHENE	13	7.7	18.1	26.3	12	8.3	26.9	27.5	13	23.1	10.3	2.7
005B BENZIDINE	13	38.5	75.3	67.4	12	33.3	78.2	58.2	13	38.5	54.2	1.4
008B 1,2,4-TRICHLOROBENZENE	13	15.4	39.0	37.8	11	0.0	51.3	46.0	13	30.8	8.8	5.7
009B HEXACHLOROBENZENE	13	7.7	23.9	14.3	11	9.1	36.8	19.5	13	30.8	6.3	6.1
012B HEXACHLOROETHANE	11	9.1	68.6	55.4	9	0.0	71.7	53.9	11	27.3	86.4	62.3
018B BIS(2-CHLOROETHYL)ETHER	13	7.7	24.7	17.0	10	0.0	32.0	26.1	13	30.8	20.5	5.3
020B 2-CHLORONAPHTHALENE	11	18.2	61.3	50.3	12	0.0	34.4	31.1	11	18.2	37.2	32.4
021A 2,4,6-TRICHLOROPHENOL	13	23.1	15.7	10.7	11	18.2	42.6	2.1	13	30.8	26.8	11.3
022A P-CHLORO-M-CRESOL	11	9.1	31.3	19.8	12	8.3	16.1	14.0	11	18.2	14.1	0.0
024A 2-CHLOROPHENOL	13	7.7	28.2	17.2	11	0.0	23.9	22.6	13	23.1	9.0	3.1
025B 1,2-DICHLOROBENZENE	13	15.4	32.4	40.7	12	8.3	44.0	44.4	13	23.1	10.8	3.7
026B 1,3-DICHLOROBENZENE	13	7.7	41.4	46.4	12	8.3	50.7	53.5	13	23.1	21.7	13.9
027B 1,4-DICHLOROBENZENE	13	7.7	39.8	45.2	12	0.0	48.5	48.9	13	15.4	24.1	10.3
028B 3,3'-DICHLOROBENZIDINE	13	30.8	51.7	43.5	12	16.7	61.6	36.4	13	23.1	16.1	9.6
031A 2,4-DICHLOROPHENOL	13	7.7	21.6	13.4	12	0.0	25.4	17.8	13	7.7	7.7	6.0
034A 2,4-DIMETHYLPHENOL	13	15.4	42.9	43.3	12	0.0	36.0	36.2	13	23.1	14.3	2.2
035B 2,4-DINITROTOLUENE	13	7.7	21.2	10.1	11	9.1	36.0	19.4	13	38.5	11.8	9.0
036B 2,6-DINITROTOLUENE	13	7.7	23.1	10.9	10	20.0	21.4	12.5	13	38.5	11.2	6.1
037B 1,2-DIPHENYLHYDRAZINE	13	23.1	30.0	14.4	12	8.3	28.2	24.0	13	15.4	39.0	27.1
039B FLUORANTHENE	13	23.1	23.8	24.0	12	8.3	20.8	23.1	13	15.4	18.6	13.0

(CONTINUED)

Table V-1 (Continued)

	MEASUREMENT											
	COMPOUND BY INTERNAL STANDARD				LABELLED ANALOG BY INTERNAL STANDARD				COMPOUND BY ISOTOPE DILUTION			
	N OF LABS	NOT DETECT /QUANT	PRECI-SION	ACCUR-ACY	N OF LABS	NOT DETECT /QUANT	PRECI-SION	ACCUR-ACY	N OF LABS	NOT DETECT /QUANT	PRECI-SION	ACCUR-ACY
	#	%	%	%	#	%	%	%	#	%	%	%
COMPOUND												
040B 4-CHLOROPHENYL PHENYL ETH	13	7.7	24.5	20.2	12	0.0	30.9	26.7	13	7.7	19.4	12.5
041B 4-BROMOPHENYL PHENYL ETHE	13	7.7	20.2	18.0	.	.	.	.	.	.	.	.
042B BIS (2-CHLOROISOPROPYL) E	10	10.0	16.1	25.0	10	10.0	17.0	29.9	10	10.0	9.3	5.7
052B HEXACHLOROBUTADIENE	13	7.7	53.4	44.8	11	0.0	61.4	50.9	13	15.4	31.5	15.9
053B HEXACHLOROCYCLOPENTADIENE	13	23.1	108.7	75.7	9	11.1	134.1	74.5	13	61.5	7.8	0.1
054B ISOPHORONE	13	15.4	23.8	16.8	12	8.3	14.6	19.2	13	15.4	13.8	9.7
055B NAPHTHALENE	13	7.7	32.9	33.8	12	0.0	38.3	36.9	13	15.4	10.5	6.0
056B NITROBENZENE	6	16.7	17.6	21.3	5	20.0	13.8	30.1	6	33.3	7.5	5.3
057A 2-NITROPHENOL	13	7.7	24.6	16.5	12	0.0	22.8	20.5	13	7.7	10.5	5.0
058A 4-NITROPHENOL	11	18.2	41.4	19.6	12	16.7	36.0	9.3	11	27.3	16.3	4.3
059A 2,4-DINITROPHENOL	13	7.7	34.2	2.3	12	8.3	42.7	1.8	13	15.4	10.7	1.5
060A 4,6-DINITRO-O-CRESOL	13	15.4	74.3	22.2	12	8.3	44.5	0.8	13	30.8	9.7	1.6
062B N-NITROSODIPHENYLAMINE	13	69.2	32.2	0.9	12	41.7	15.8	21.3	13	61.5	12.2	3.9
064A PENTACHLOROPHENOL	13	15.4	34.3	18.7	12	8.3	32.1	17.2	13	15.4	11.4	3.5
065A PHENOL	13	7.7	32.2	19.7	12	0.0	24.1	19.5	13	7.7	14.3	1.2
066B BIS (2-ETHYLHEXYL) PHTHAL	13	30.8	24.4	10.7	12	25.0	19.5	20.1	13	15.4	20.7	24.5
068B DI-N-BUTYL PHTHALATE	13	15.4	36.5	29.4	12	8.3	35.0	31.6	13	30.8	12.0	12.6
069B DI-N-OCTYL PHTHALATE	13	15.4	24.4	21.3	12	8.3	33.4	28.0	13	7.7	12.5	12.0
070B DIETHYL PHTHALATE	13	15.4	48.1	42.2	12	0.0	63.0	52.7	13	7.7	20.0	22.3
071B DIMETHYL PHTHALATE	13	7.7	82.9	51.1	12	8.3	82.1	55.6	13	15.4	18.0	19.4

(CONTINUED)

Table V-1 (Continued)

COMPOUND	MEASUREMENT: COMPOUND BY INTERNAL STANDARD N OF LABS	NOT DETECT /QUANT	PRECISION	ACCURACY	LABELLED ANALOG BY INTERNAL STANDARD N OF LABS	NOT DETECT /QUANT	PRECISION	ACCURACY	COMPOUND BY ISOTOPE DILUTION N OF LABS	NOT DETECT /QUANT	PRECISION	ACCURACY
	#	%	%	%	#	%	%	%	#	%	%	%
072B BENZO(A)ANTHRANCENE	13	30.8	24.7	18.6	12	16.7	27.4	14.0	13	30.8	13.8	5.5
073B BENZO(A)PYRENE	13	23.1	29.6	16.1	12	16.7	28.1	16.6	13	15.4	20.5	11.9
074B BENZO(B)FLUORANTHENE	13	23.1	59.7	28.2	12	25.0	58.3	38.6	13	30.8	65.1	43.1
075B BENZO(K)FLUORANTHENE	13	15.4	72.4	37.1	12	16.7	53.3	31.1	13	30.8	14.3	8.0
076B CHRYSENE	13	23.1	23.9	21.9	12	16.7	36.6	26.5	13	7.7	22.6	5.9
077B ACENAPHTHYLENE	12	16.7	17.2	22.3	12	0.0	23.4	29.3	12	8.3	19.3	14.7
078B ANTHRACENE	13	7.7	25.7	28.2	12	16.7	23.8	21.9	13	15.4	20.2	2.0
079B BENZO(GHI)PERYLENE	13	30.8	43.0	13.0	12	16.7	42.0	10.8	13	30.8	12.5	7.6
080B FLUORENE	13	7.7	19.0	18.7	12	0.0	22.0	23.3	13	7.7	11.3	3.4
081B PHENANTHRENE	13	15.4	20.0	25.0	12	8.3	22.0	23.3	13	23.1	5.3	5.0
084B PYRENE	12	16.7	33.9	28.7	11	9.1	22.2	22.8	12	16.7	12.1	7.6
502B BETA NAPHTHYLAMINE	13	30.8	68.7	36.5	11	27.3	49.8	24.5	13	53.8	71.2	26.6
503B ALPHA PICOLINE	13	30.8	41.6	33.2	12	16.7	49.7	27.7	13	30.8	16.8	5.2
504B DIBENZOTHIOPHENE	13	15.4	19.8	23.8	12	16.7	22.5	24.2	13	23.1	13.3	9.2
505B DIBENZOFURAN	13	7.7	23.5	19.4	12	8.3	19.6	24.8	13	23.1	9.2	7.6
506B N-DODECANE	12	8.3	86.6	47.3	12	0.0	62.4	51.9	12	8.3	52.2	21.4
507B DIPHENYLAMINE	13	23.1	28.8	21.4	11	27.3	30.3	25.2	13	38.5	20.7	10.5
508B DIPHENYLETHER	12	8.3	17.8	30.4	11	0.0	29.5	31.3	12	25.0	8.9	5.6
509B ALPHA TERPINEOL	13	15.4	14.7	17.5	11	9.1	33.2	8.3	13	53.8	23.0	0.6
510B STYRENE	13	15.4	46.9	52.3	12	8.3	59.3	53.1	13	23.1	25.4	10.7

(CONTINUED)

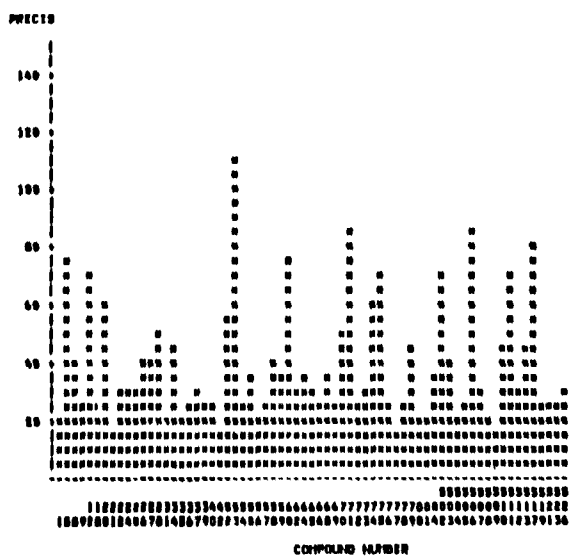
Table V-1 (Concluded)

		MEASUREMENT											
		COMPOUND BY INTERNAL STANDARD				LABELLED ANALOG BY INTERNAL STANDARD				COMPOUND BY ISOTOPE DILUTION			
		N OF LABS	NOT DETECT /QUANT	PRECI- SION	ACCUR- ACY	N OF LABS	NOT DETECT /QUANT	PRECI- SION	ACCUR- ACY	N OF LABS	NOT DETECT /QUANT	PRECI- SION	ACCUR- ACY
		#	%	%	%	#	%	%	%	#	%	%	%
COMPOUND													
511B DI-N-BUTYL AMINE		13	84.6	68.6	95.1	11	72.7	126.1	73.9	13	84.6	137.0	27.8
512B BIPHENYL		13	15.4	27.9	26.9	11	63.6	14.4	41.7	13	46.2	12.3	5.2
513B P-CYMENE		13	15.4	45.4	48.5	12	0.0	67.5	53.6	13	30.8	9.9	3.3
517B N-DECANE	C10	12	8.3	80.1	64.0	12	8.3	38.2	39.4	12	25.0	28.5	29.4
519B N-HEXADECANE	C16	13	23.1	27.5	22.0	12	0.0	38.0	28.7	13	15.4	14.6	14.5
521B N-EICOSANE	C20	13	7.7	24.0	16.1	12	8.3	25.8	23.4	13	7.7	33.6	22.1
523B N-TETRACOSANE	C24	13	23.1	25.0	11.0	12	25.0	23.3	20.5	13	30.8	8.6	5.6
526B N-TRIACONTANE	C30	13	23.1	28.1	15.5	12	16.7	22.5	21.8	13	7.7	20.5	12.3

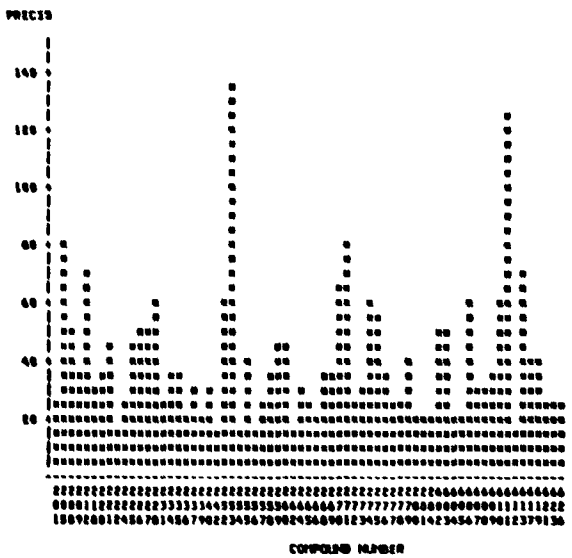
Figure V-1

METHOD PRECISION, APS SAMPLE

Compound by Internal Standard



Labelled Analog by Internal Standard



Compound by Isotope Dilution

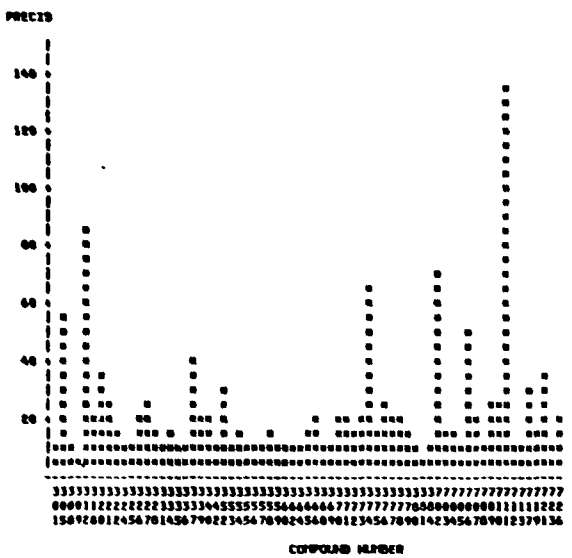
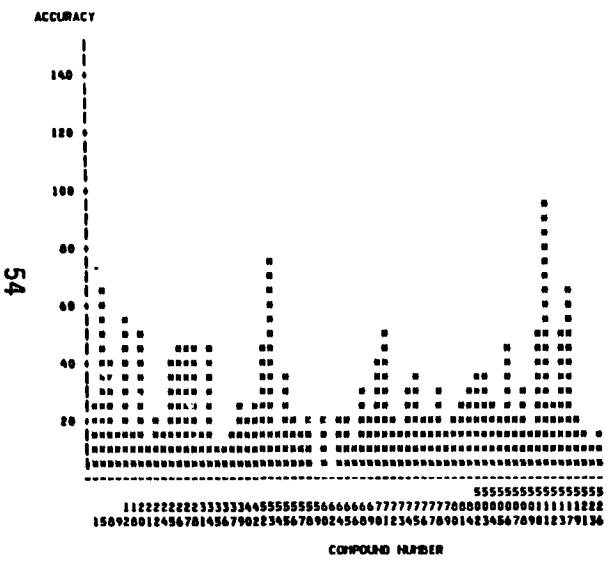


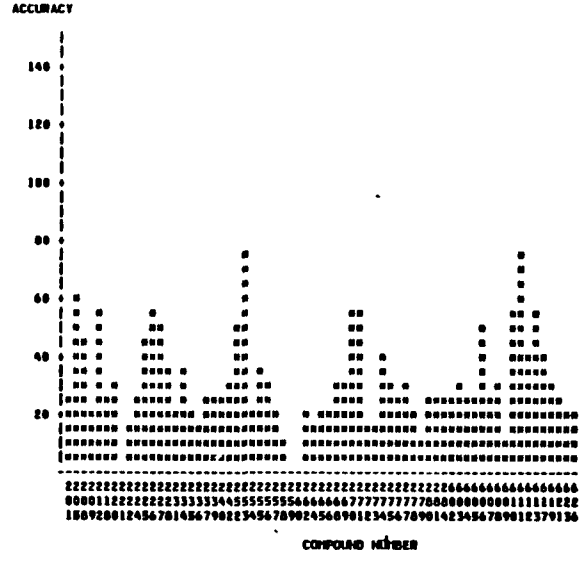
Figure V-2

METHOD ACCURACY, APS SAMPLE

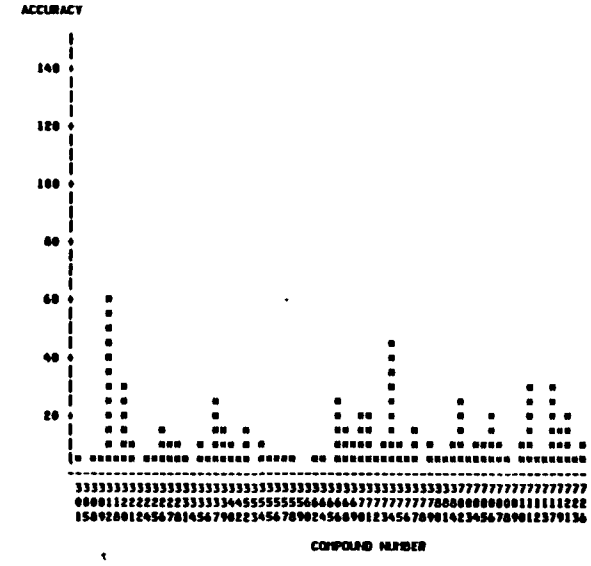
Compound by Internal Standard



Labelled Analog by Internal Standard



Compound by Isotope Dilution



VI QUALITY CONTROL LIMITS

Another important use of the data developed in the study is the calculation of quality control limits for testing and screening of priority pollutant analyses submitted to EPA for use in future analytical programs. These quality control limits were calculated by constructing statistical prediction intervals for future observations of a quantity of interest using statistical estimates determined in this study.

To help ensure the initial and ongoing quality of analytical measurement by isotope dilution and internal standard methods, a number of quality control limits are provided in this section. These limit values were developed for application in the quality control procedures being developed by EPA for final specifications of these methods. The limits are calculated using the results of a variance components analysis of the measured amounts on two subsets of the data: the calibration-type samples (CAL 100, VER, and PRR) and the extracted samples (BLK, APS, and EPA). The inter- and intralaboratory variance components of the logarithms of the amounts were estimated (S_E^2 and S_A^2) along with the logarithmic mean response (M) by maximum-likelihood techniques. Details of the variance components procedure are given in Appendix I.

The measured concentrations of these compounds have been assumed to follow a lognormal distribution throughout the analysis described in this section, as well as in other sections of this report. The lognormal distribution has been frequently and effectively applied to model pollutant concentrations, including other effluent guidelines priority pollutant data, and agrees with the physical interpretation of nonnegative concentration values. Limits derived from this assumption are always nonnegative. Descriptive and summary statistics calculated for this data support the assumption of lognormality.

In other EPA method validation studies the compound-specific performance specifications have usually been determined at individual test

levels of p = .05 (i.e. based on 95 percent confidence limits for a single future observation). Using such specifications, each compound measured would have a 5 percent chance of falling outside its QC limit.

Because of the large number of compounds (154, including labeled analogues) involved in the quality tests for Method 1625A, it would be extremely likely that one or more items on each test would be failed simply by random chance if the tests were all performed at individual test levels of p = .05. It was deemed desirable, instead, to specify test limits such that the global test level (i.e. the chance of failing on one or more of the compounds out of the whole list) was held to 5 percent. Two approaches were suggested: (1) reduce the individual test level, and (2) allow retesting of those items that failed and only indicate an out-of-control process if the same item fails twice. These remedies can be applied in conjunction. For instance, the start-up test, described below, involves both precision and accuracy testing for a total of 308 items on the test. Testing at an individual level of .01 and allowing one retest of the failed items will achieve an overall level of 5 percent. Details of the binomial calculations for these considerations are given in Appendix J.

Start-up Test Limits

When a laboratory begins operation, it is required to perform four replicate extracts and analyses of prepared samples containing 100 µg/L of all compounds. The arithmetic average and standard deviation of the four values given by

$$\bar{X} = \frac{1}{4} \sum_{i=1}^{4} X_i \quad ,$$

$$S = \frac{1}{3} \sum_{i=1}^{4} (X_i - \bar{X})^2 \quad ,$$

are then computed for each compound.

ELEMENT NAME: CONCENTRATION/DILUTION FACTOR

Definition: The concentration or dilution ratio of the sample fraction or spike before analysis.

Input	**Type/Length**
Quantitation Report	X(11)
As Stored Internally	9(5)V9(5)

Unit of Measure

Pure number.

Edit Criteria:

Format: two numbers separated by a colon, the first is the pre-extraction/dilution amount; the second is the post extraction/dilution amount. ie XXXXX:XXXXX;

Example: 1000:1 means that an initial 1000 mL volume of sample was concentrated to 1 mL extract; 1:100 means that an inital volume of sample was diluted with 99 parts reagent water.

Acceptable range: 10000:1 to 1:10000.
NA:NA - used for calibration standards

Use: When a semi-volatiles fraction (acid or base/neutral) is extracted, the normal volume of sample is 1 liter (1000 mL). The extract is normally concentrated to a volume of 1 mL. The units on the quantitation report for semi-volatiles is UG/ML. Multiplying these units by 1000 mL/L, and dividing by the concentration ratio (1000/1) yields the final volume in ug/L. Mathematically, the concentration factor (CF) can be expressed in the following equation:

Csamp (ug/L) = Cext (ug/mL) x 1000 (mL/L) / CF

for our example,

ELEMENT NAME: CONCENTRATION/DILUTION FACTOR (Continued)

Edit Criteria (continued):

Csamp (ug/L) = Cext (ug/mL) x 1000 (mL/L) / 1000

Similarly, for volatiles,

Csamp (ug/L) = Cdil samp (ug/L) / DF

For a dilution factor of 1:10,

Csamp (ug/L) = Cdil samp (ug/L) / (1/10)

and C samp (ug/L) = 10 x Cdil samp (ug/L)

As can be seen, the concentration and dilution factors are critical to computation of the correct concentrations in water.

Limits for $\bar{X}$ required to ensure method accuracy are given in Table VI-1. The limits for $\bar{X}$ were calculated by

$$\exp\left[\left(M + \frac{1}{2} S_A^2 - \frac{1}{8} \eta_A^2\right) \pm t_d\left(1 - \frac{p}{2}\right) \sqrt{S_E^2 + \eta_A^2/4 + S_E^2/L + S_A^2/N + \frac{9}{32} S_A^4/(N-L)}\right],$$

where M, S_A^2, and S_E^2 are the variance components results; $\eta_A^2 = \exp(S_A^2) - 1$; L is the number of laboratories in this study; N is the total number of observations of the compound in the set of samples for this study; t_d is the inverse cumulative t distribution with d = min (N-L, L-1) degrees of freedom; and p is the individual test level. The derivation of this formula is given in Appendix K. Values are given for p levels of .05 and .01 for labeled compounds by internal standard and for compounds by isotope dilution. In order to achieve an overall significance level of 5 percent on the precision (described below) and accuracy tests, the individual compound limits would be based on an individual significance level of .01, and one retest of those compounds that failed the first round would be allowed (for discussion of multi-round testing see Appendix J).

Table VI-1 also gives upper limits for S to ensure method precision. The limits for S were calculated by

$$\exp(M)\, Q(1-p, S_A)\, K(1-p,d) \quad ,$$

where Q(q,s) is the qth quantile of the distribution of the standard deviation of four lognormal variates with logarithmic mean 0 and logarithmic standard deviation s, and K(q,d) is

$$K(q,d) = \sqrt{\frac{F_{3,d}(q)}{C_3(q)/3}} \quad ,$$

where F is the inverse cumulative of an F distribution, C is the inverse cumulative of a chi-squared distribution, and d is the degrees of freedom in the estimate of S_A, e.g., N-L. The derivation of this formula and the details of the computer simulation used to evaluate Q are given in Appendix L. Values are given at individual test levels of .05 and .01. To obtain an overall 5 percent level over all compounds, in conjunction with the start-up accuracy test, the .01 individual levels would be used with one retest allowed for compounds that fail the first round.

Table VI-1

START-UP LIMITS FOR ACCURACY AND PRECISION

SERIES-LABELLED ANALOGS BY INTERNAL STANDARD

COMPOUND	ACCUR. P .05 LOWER	ACCUR. P .05 UPPER	ACCUR. P .01 LOWER	ACCUR. P .01 UPPER	PREC. P .05	PREC. P .01
201 B ACENAPHTHENE-D10	46	120	38	147	29	38
205 B BENZIDINE-D8 (RINGS-D8)	2	1054	0	4987	139	269
208 B 1,2,4-TRICHLOROBENZENE-D3	22	143	15	212	41	57
209 B HEXACHLOROBENZENE-13C6	47	172	36	228	59	81
212 B HEXACHLOROETHANE-1-13C	10	202	5	400	51	77
218 B BIS(2-CHLOROETHYL)-D8 ETH	38	146	29	196	26	33
220 B 2-CHLORONAPHTHALENE-D7	39	131	30	168	31	41
221 A 2,4,6-TRICHLOROPHENOL-3,5	54	146	43	183	36	47
222 A 4-CHLORO-3-METHYLPHENOL-2	38	134	30	174	76	111
224 A 2-CHLOROPHENOL-3,4,5,6-D4	45	130	36	162	18	24
225 B 1,2-DICHLOROBENZENE-D4	21	141	14	212	27	35
226 B 1,3-DICHLOROBENZENE-D4	19	135	13	203	35	48
227 B 1,4-DICHLOROBENZENE-D4	22	133	15	193	35	48
228 B 3,3'-DICHLOROBENZIDINE-D6	13	286	7	562	56	80
231 A 2,4-DICHLOROPHENOL-3,5,6-	47	133	38	164	22	28
234 A 2,4-DIMETHYLPHENOL-3,5,6-	22	153	15	228	17	22
235 B 2,4-DINITROTOLUENE-3,5,6-	32	170	22	245	28	37
236 B 2,6-DINITROTOLUENE-D3	56	146	44	184	44	59
237 B 1,2-DIPHENYL-D10-HYDRAZIN	40	134	31	173	27	35
239 B FLUORANTHENE-D10	44	129	36	161	27	35
240 B 4-CHLOROPHENYL PHENYL-D5	49	131	40	161	40	52
242 B BIS(2-CHLOROISOPROPYL)ETH	44	119	35	149	21	27
252 B HEXACHLORO-1,3-BUTADIENE-	13	182	8	316	44	63
253 B HEXACHLOROCYCLOPENTADIENE	0	601	0	3458	33	60
254 B ISOPHORONE-D8	57	114	49	133	18	23
255 B NAPHTHALENE-D8	36	122	28	157	30	39
256 B NITROBENZENE-D5	33	144	18	265	20	28
257 A 2-NITROPHENOL-3,4,5,6-D4	49	121	41	145	18	23
258 A 4-NITROPHENOL-2,3,5,6-D4	23	240	14	398	117	188
259 A 2,4-DINITROPHENOL-3,5,6-D	33	208	22	308	48	66
260 A 4,6-DINITRO-O-CRESOL-D2	48	186	36	247	48	64
262 B N-NITROSODIPHENYLAMINE-D6	62	109	54	126	28	37
264 A PENTACHLOROPHENOL-13C6	48	163	37	212	38	49
265 A PHENOL-2,3,4,5,6-D5	29	150	21	210	101	161
266 B BIS(2-ETHYLHEXYL)PHTHALAT	42	155	32	205	22	29
268 B DI-N-BUTYL PHTHALATE-D4	32	143	23	195	18	23
269 B DI-N-OCTYL PHTHALATE-D4	20	231	12	383	35	46
270 B DIETHYL PHTHALATE-3,4,5,6	15	159	9	260	53	78
271 B DIMETHYL PHTHALATE-3,4,5,	4	265	2	640	64	108
272 B BENZO(A)ANTHRACENE-D12	36	206	25	298	31	41
273 B BENZO(A)PYRENE-D12	45	142	35	181	19	24
274 B BENZO(B)FLUORANTHENE-D12	19	314	11	577	107	168
275 B BENZO(K)FLUORANTHENE-D12	25	303	15	514	79	114
276 B CHRYSENE-D12	43	165	33	219	50	69
277 B ACENAPHTHYLENE-D8	47	120	39	146	24	31
278 B ANTHRACENE-D10	41	147	31	194	37	49
279 B BENZO(GHI)PERYLENE-D12	41	193	29	268	34	45
280 B FLUORENE-D10	58	114	51	131	33	43
281 B PHENANTHRENE-D10	53	111	45	130	31	40
284 B PYRENE-D10	41	137	32	176	23	29
602 B 2-NAPHTHYL-D7-AMINE	2	969	0	3891	24	33
603 B 2-METHYLPYRIDINE-D7	18	224	11	380	88	138
604 B DIBENZOTHIOPHENE-D8	56	112	48	130	24	31
605 B DIBENZOFURAN-D8	55	116	47	136	25	31
606 B N-DODECANE-D26	12	187	7	331	37	53
607 B DIPHENYL-D10-AMINE	37	148	27	206	32	42
608 B DIPHENYL-D10 ETHER	45	125	36	155	28	37
609 B ALPHA-TERPINEOL-D3	33	198	22	292	36	48
610 B STYRENE-2,3,4,5,6-D5	14	166	8	281	35	49
611 B DI-N-BUTYL-D18-AMINE	1	1472	0	166102	197	456
612 B DIPHENYL-D10	42	110	28	165	30	43
613 B P-CYMENE-D14	11	200	6	359	47	67
617 B N-DECANE-D22	13	174	8	298	48	70
619 B N-HEXADECANE-D34	46	130	37	162	35	46
621 B N-EICOSANE-D42	43	135	34	172	26	34
623 B N-TETRACOSANE-D50	37	156	27	211	22	28
626 B N-TRIACONTANE-D62	37	174	27	242	31	41

Table VI-1 (Concluded)

SERIES=COMPOUNDS BY ISOTOPE DILUTION

	COMPOUND		ACCUR. P .05 LOWER	ACCUR. P .05 UPPER	ACCUR. P .01 LOWER	ACCUR. P .01 UPPER	PREC. P .05	PREC. P .01
301B	ACENAPHTHENE		85	123	79	134	16	21
305B	BENZIDINE		28	295	16	518	77	119
308B	1,2,4-TRICHLOROBENZENE		89	125	82	136	14	19
309B	HEXACHLOROBENZENE		95	118	90	124	11	16
312B	HEXACHLOROETHANE		38	515	21	960	144	227
318B	BIS(2-CHLOROETHYL)ETHER		67	161	55	196	25	34
320B	2-CHLORONAPHTHALENE		63	259	46	357	70	100
321A	2,4,6-TRICHLOROPHENOL		71	169	59	205	41	57
322A	P-CHLORO-M-CRESOL		83	121	76	131	27	37
324A	2-CHLOROPHENOL		85	124	79	135	9	13
325B	1,2-DICHLOROBENZENE		81	131	73	146	13	17
326B	1,3-DICHLOROBENZENE		75	168	63	201	31	43
327B	1,4-DICHLOROBENZENE		72	164	61	194	31	42
328B	3,3'-DICHLOROBENZIDINE		78	151	68	174	20	26
331A	2,4-DICHLOROPHENOL		91	123	85	131	9	12
334A	2,4-DIMETHYLPHENOL		71	133	62	153	10	13
335B	2,4-DINITROTOLUENE		84	140	75	158	13	18
336B	2,6-DINITROTOLUENE		87	129	80	141	21	30
337B	1,2-DIPHENYLHYDRAZINE		64	234	49	308	53	73
339B	FLUORANTHENE		81	154	71	177	25	33
340B	4-CHLOROPHENYL PHENYL ETH		84	148	75	166	32	42
362B	BIS (2-CHLOROISOPROPYL) E		88	127	81	138	12	17
352B	HEXACHLOROBUTADIENE		64	198	51	251	41	56
353B	HEXACHLOROCYCLOPENTADIENE		80	124	69	144	10	15
354B	ISOPHORONE		85	140	76	156	19	25
355B	NAPHTHALENE		87	128	80	139	15	20
356B	NITROBENZENE		83	132	69	161	14	25
357A	2-NITROPHENOL		85	128	78	140	12	15
358A	4-NITROPHENOL		72	127	62	146	30	42
359A	2,4-DINITROPHENOL		79	122	72	134	13	18
360A	4,6-DINITRO-O-CRESOL		84	122	77	133	14	19
362B	N-NITROSODIPHENYLAMINE		76	122	65	142	28	45
364A	PENTACHLOROPHENOL		83	128	76	140	16	21
365A	PHENOL		82	118	77	127	27	36
366B	BIS (2-ETHYLHEXYL) PHTHAL		81	185	69	220	23	31
368B	DI-N-BUTYL PHTHALATE		86	146	76	165	11	15
369B	DI-N-OCTYL PHTHALATE		86	145	77	161	12	16
370B	DIETHYL PHTHALATE		86	170	75	196	33	44
371B	DIMETHYL PHTHALATE		85	164	74	188	27	36
372B	BENZO(A)ANTHRANCENE		76	145	65	168	15	20
373B	BENZO(A)PYRENE		74	165	62	195	20	26
374B	BENZO(B)FLUORANTHENE		50	350	32	545	120	183
375B	BENZO(K)FLUORANTHENE		67	124	59	143	19	26
376B	CHRYSENE		70	157	59	186	38	51
377B	ACENAPHTHYLENE		80	161	69	186	28	38
378B	ANTHRACENE		69	148	58	174	30	41
379B	BENZO(GHI)PERYLENE		81	141	72	160	15	21
380B	FLUORENE		87	123	81	132	22	29
381B	PHENANTHRENE		96	115	93	119	10	13
384B	PYRENE		84	137	76	152	14	19
702B	BETA NAPHTHYLAMINE		23	513	10	1236	32	49
703B	ALPHA PICOLINE		69	129	59	149	27	38
704B	DIBENZOTHIOPHENE		87	136	79	150	23	31
705B	DIBENZOFURAN		91	126	85	136	15	20
706B	N-DODECANE	C12	50	260	35	369	53	74
707B	DIPHENYLAMINE		71	167	53	205	32	45
708B	DIPHENYLETHER		89	125	82	136	14	19
709B	ALPHA TERPINEOL		57	172	42	234	29	44
710B	STYRENE		66	179	53	221	31	42
711B	DI-N-BUTYL AMINE		*	*	*	*	*	*
712B	BIPHENYL		84	131	75	148	29	41
713B	P-CYMENE		84	127	76	140	13	19
717B	N-DECANE	C10	34	141	24	195	36	51
719B	N-HEXADECANE	C16	89	146	80	162	24	33
721B	N-EICOSANE	C20	67	209	53	263	44	59
723B	N-TETRACOSANE	C24	87	127	80	139	8	11
726B	N-TRIACONTANE	C30	73	168	61	200	24	32

Ongoing Calibration-Verification Limits

During each shift, a standard calibration sample containing 100 µg/mL of all compounds, is analyzed to check the method calibration. Limits for the results of this check are given in Table VI-2 for compounds by internal standard (which would be appropriate for Method 625), labeled compounds by internal standard, and compounds by isotope dilution. The limits are obtained from the variance components analysis of the calibration-type samples as

$$\exp[\ln(100) \pm t_d(1 - \tfrac{p}{2})\, S_A] \quad ,$$

where t_d is again the inverse cumulative t distribution, and d is the degrees of freedom in the estimate of S_A, e.g. N-L. The derivation of this formula is given in Appendix K. The maximum-minimum and minimum-maximum limits were set at 85 and 115, respectively for calibration verification of all compounds; i.e., for the 100 µg/mL calibration verification standard, no specification falls in the range of 85 to 115 µg/mL. These limits were chosen because isotope dilution has the potential of being too precise, and in the same way that some probability exists that wide limits can be developed, there is a remote probability that narrow limits can be developed, also. Since calibration is verified with a single analysis for all compounds, it was felt that an additional allowance for those compounds which fall in the range of 85 to 115 would not render the analysis for any given compound too imprecise.

Limits are given in Table VI-2 for individual test levels of .05, .01, .001, and .0001. To achieve an overall 5 percent level on this test for Method 625* (compounds by internal standard), either the .01 values from the first section of Table VI-2 could be used with one retest allowed for failing compounds, or a single-round test at individual level .001 could be used. To achieve an overall 5 percent level for Method 1625A, the .01 level

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* For Method 625, acid (A) and base/neutral (B) compounds are tested separately, for a total of 12 and 48 compounds tested.

Table VI-2

ONGOING CALIBRATION VERIFICATION LIMITS

SERIES=COMPOUNDS BY INTERNAL STANDARD

COMPOUND	P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER	P .0001 LOWER	P .0001 UPPER
001B ACENAPHTHENE	85	118	80	126	74	136	68	146
005B BENZIDINE	39	254	28	356	18	552	12	844
008B 1,2,4-TRICHLOROBENZENE	80	125	74	136	66	151	60	167
009B HEXACHLOROBENZENE	65	153	56	178	46	217	38	262
012B HEXACHLOROETHANE	72	140	63	158	54	185	46	217
018B BIS(2-CHLOROETHYL)ETHER	69	145	61	165	51	196	43	231
020B 2-CHLORONAPHTHALENE	74	135	67	150	57	175	49	203
021A 2,4,6-TRICHLOROPHENOL	78	128	71	141	63	159	56	180
022A P-CHLORO-M-CRESOL	85	115	85	118	80	125	75	133
024A 2-CHLOROPHENOL	77	131	70	144	62	163	55	183
025B 1,2-DICHLOROBENZENE	70	143	62	162	52	191	45	223
026B 1,3-DICHLOROBENZENE	78	129	71	141	63	158	56	177
027B 1,4-DICHLOROBENZENE	77	129	70	142	62	160	55	180
028B 3,3'-DICHLOROBENZIDINE	57	177	46	217	35	284	27	370
031A 2,4-DICHLOROPHENOL	76	131	69	144	61	163	55	183
034A 2,4-DIMETHYLPHENOL	84	119	79	127	72	138	67	150
035B 2,4-DINITROTOLUENE	67	150	58	174	48	210	40	252
036B 2,6-DINITROTOLUENE	66	151	57	175	47	212	39	253
037B 1,2-DIPHENYLHYDRAZINE	77	130	70	143	62	163	54	185
039B FLUORANTHENE	69	145	60	166	51	197	43	234
040B 4-CHLOROPHENYL PHENYL ETH	75	134	67	148	59	169	52	193
041B 4-BROMOPHENYL PHENYL ETHE	75	133	67	149	57	176	47	211
042B BIS (2-CHLOROISOPROPYL) E	70	142	62	162	52	192	44	228
052B HEXACHLOROBUTADIENE	80	126	73	136	66	152	59	169
053B HEXACHLOROCYCLOPENTADIENE	75	133	68	148	59	169	52	193
054B ISOPHORONE	73	137	65	153	56	178	49	205
055B NAPHTHALENE	74	134	67	149	59	171	51	194
056B NITROBENZENE	78	129	70	143	60	166	51	197
057A 2-NITROPHENOL	78	127	72	139	64	156	57	174
058A 4-NITROPHENOL	56	178	45	221	34	295	25	393
059A 2,4-DINITROPHENOL	64	155	55	181	45	222	37	270
060A 4,6-DINITRO-O-CRESOL	67	149	58	173	48	209	40	251
062B N-NITROSODIPHENYLAMINE	76	131	67	150	53	187	40	249
064A PENTACHLOROPHENOL	63	158	54	186	43	230	35	283
065A PHENOL	74	136	66	151	57	174	50	199
066B BIS (2-ETHYLHEXYL) PHTHAL	61	164	51	195	41	245	33	306
068B DI-N-BUTYL PHTHALATE	73	137	65	153	57	177	49	204
069B DI-N-OCTYL PHTHALATE	52	194	41	246	30	335	22	450
070B DIETHYL PHTHALATE	69	144	61	164	52	194	44	229
071B DIMETHYL PHTHALATE	69	144	61	164	51	194	44	228
072B BENZO(A)ANTHRANCENE	57	175	47	214	36	279	28	360
073B BENZO(A)PYRENE	36	277	25	400	15	646	10	1027
074B BENZO(B)FLUORANTHENE	43	235	31	320	21	483	14	723
075B BENZO(K)FLUORANTHENE	39	254	28	354	18	546	12	828
076B CHRYSENE	42	237	31	322	21	480	14	705
077B ACENAPHTHYLENE	85	117	80	125	74	135	68	147
078B ANTHRACENE	66	151	57	175	47	211	40	252
079B BENZO(GHI)PERYLENE	42	240	30	330	20	503	13	761
080B FLUORENE	71	141	63	159	54	186	46	216
081B PHENANTHRENE	66	151	57	175	47	211	40	252
084B PYRENE	67	148	58	171	48	207	40	249
502B BETA NAPHTHYLAMINE	58	171	48	209	37	271	28	352
503B ALPHA PICOLINE	65	155	55	182	45	225	36	276

Table VI-2 (Continued)

SERIES=COMPOUNDS BY INTERNAL STANDARD

COMPOUND		P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER	P .0001 LOWER	P .0001 UPPER
504B DIBENZOTHIOPHENE		71	140	63	158	54	185	46	216
505B DIBENZOFURAN		73	137	65	153	57	176	49	202
506B N-DODECANE		65	154	56	180	45	220	37	268
507B DIPHENYLAMINE		70	142	62	162	52	192	44	227
508B DIPHENYLETHER		70	143	62	162	52	192	44	226
509B ALPHA TERPINEOL		71	140	63	158	54	185	46	216
510B STYRENE		69	144	61	165	51	196	43	232
511B DI-N-BUTYL AMINE		30	328	15	646	4	2397	1	17589
512B BIPHENYL		80	125	73	136	66	151	60	168
513B P-CYMENE		72	138	64	155	55	181	48	209
517B N-DECANE	C10	59	169	49	204	39	260	31	327
519B N-HEXADECANE	C16	67	149	58	172	49	206	41	245
521B N-EICOSANE	C20	67	150	58	173	48	209	40	249
523B N-TETRACOSANE	C24	67	148	59	171	49	204	41	243
526B N-TRIACONTANE	C30	55	180	45	223	34	296	26	392

Table VI-2 (Continued)

SERIES=LABELLED ANALOGS BY INTERNAL STANDARD

COMPOUND	P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER	P .0001 LOWER	P .0001 UPPER
201B ACENAPHTHENE-D10	85	116	82	122	76	131	71	141
205B BENZIDINE-D8 (RINGS-D8)	34	295	23	438	14	738	8	1233
208B 1,2,4-TRICHLOROBENZENE-D3	81	123	75	133	68	147	61	163
209B HEXACHLOROBENZENE-13C6	66	152	56	178	46	217	38	265
212B HEXACHLOROETHANE-1-13C	73	137	65	154	55	180	47	212
218B BIS(2-CHLOROETHYL)-D8 ETH	75	133	68	147	59	169	52	194
220B 2-CHLORONAPHTHALENE-D7	85	116	82	122	77	130	72	139
221A 2,4,6-TRICHLOROPHENOL-3,5	85	116	81	123	75	133	69	144
222A 4-CHLORO-3-METHYLPHENOL-2	85	118	80	126	74	136	68	147
224A 2-CHLOROPHENOL-3,4,5,6-D4	77	130	70	142	62	160	55	180
225B 1,2-DICHLOROBENZENE-D4	81	123	75	133	68	148	61	164
226B 1,3-DICHLOROBENZENE-D4	75	133	68	147	59	168	52	192
227B 1,4-DICHLOROBENZENE-D4	83	120	78	129	71	140	65	153
228B 3,3'-DICHLOROBENZIDINE-D6	48	210	36	275	25	393	18	558
231A 2,4-DICHLOROPHENOL-3,5,6-	82	122	76	131	70	144	64	157
234A 2,4-DIMETHYLPHENOL-3,5,6-	79	127	72	139	65	155	58	172
235B 2,4-DINITROTOLUENE-3,5,6-	77	130	70	144	61	164	53	187
236B 2,6-DINITROTOLUENE-D3	66	151	57	176	45	220	36	278
237B 1,2-DIPHENYL-D10-HYDRAZIN	79	127	72	139	64	155	58	174
239B FLUORANTHENE-D10	72	139	64	157	54	184	47	215
240B 4-CHLOROPHENYL PHENYL-D5	78	128	72	140	64	157	57	175
242B BIS(2-CHLOROISOPROPYL)ETH	70	143	62	162	52	193	44	229
252B HEXACHLORO-1,3-BUTADIENE-	85	118	80	126	73	136	68	148
253B HEXACHLOROCYCLOPENTADIENE	73	137	65	154	55	180	47	211
254B ISOPHORONE-D8	75	134	67	149	59	170	52	194
255B NAPHTHALENE-D8	85	116	81	123	76	132	71	141
256B NITROBENZENE-D5	76	131	68	147	57	177	46	219
257A 2-NITROPHENOL-3,4,5,6-D4	81	124	75	134	67	148	61	163
258A 4-NITROPHENOL-2,3,5,6-D4	64	157	54	185	43	231	35	287
259A 2,4-DINITROPHENOL-3,5,6-D	66	151	57	175	47	212	39	256
260A 4,6-DINITRO-O-CRESOL-D2	78	128	71	140	63	158	56	177
262B N-NITROSODIPHENYLAMINE-D6	81	124	75	134	66	151	59	170
264A PENTACHLOROPHENOL-13C6	69	146	60	167	50	199	42	237
265A PHENOL-2,3,4,5,6-D5	72	138	65	155	56	180	48	208
266B BIS(2-ETHYLHEXYL)PHTHALAT	70	144	61	164	51	195	43	232
268B DI-N-BUTYL PHTHALATE-D4	75	133	68	147	59	168	52	192
269B DI-N-OCTYL PHTHALATE-D4	51	196	40	250	29	344	21	467
270B DIETHYL PHTHALATE-3,4,5,6	72	139	64	156	55	182	47	211
271B DIMETHYL PHTHALATE-3,4,5,	73	136	66	152	57	175	50	201
272B BENZO(A)ANTHRACENE-D12	58	173	47	211	36	275	28	357
273B BENZO(A)PYRENE-D12	40	250	29	348	19	535	12	812
274B BENZO(B)FLUORANTHENE-D12	43	233	32	317	21	474	14	703
275B BENZO(K)FLUORANTHENE-D12	41	245	30	338	19	513	13	767
276B CHRYSENE-D12	54	186	43	232	32	310	24	411
277B ACENAPHTHYLENE-D8	83	120	78	128	72	140	66	152
278B ANTHRACENE-D10	80	126	73	137	65	153	58	171
279B BENZO(GHI)PERYLENE-D12	41	245	30	337	19	513	13	771
280B FLUORENE-D10	81	124	75	134	67	148	61	164
281B PHENANTHRENE-D10	84	119	79	127	73	137	67	149
284B PYRENE-D10	73	137	65	154	56	180	48	210
602B 2-NAPHTHYL-D7-AMINE	70	143	62	162	52	193	44	230
603B 2-METHYLPYRIDINE-D7	60	167	50	201	39	256	31	324
604B DIBENZOTHIOPHENE-D8	85	117	81	124	75	134	69	145

Table VI-2 (Continued)

SERIES=LABELLED ANALOGS BY INTERNAL STANDARD

COMPOUND	P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER	P .0001 LOWER	P .0001 UPPER
605B DIBENZOFURAN-D8	84	119	79	127	72	139	66	150
606B N-DODECANE-D26	67	149	59	171	49	204	41	242
607B DIPHENYL-D10-AMINE	81	124	74	135	66	151	59	169
608B DIPHENYL-D10 ETHER	85	115	85	116	82	123	77	129
609B ALPHA-TERPINEOL-D3	49	203	38	261	27	364	20	502
610B STYRENE-2,3,4,5,6-D5	70	143	61	163	52	193	44	228
611B DI-N-BUTYL-D18-AMINE	30	337	17	585	7	1420	2	4238
612B DIPHENYL-D10	81	124	74	136	63	159	52	192
613B P-CYMENE-D14	83	120	78	128	72	140	66	152
617B N-DECANE-D22	70	143	61	163	52	193	44	227
619B N-HEXADECANE-D34	76	132	69	145	61	165	54	186
621B N-EICOSANE-D42	81	123	75	133	68	147	62	162
623B N-TETRACOSANE-D50	74	136	66	151	57	174	50	199
626B N-TRIACONTANE-D62	53	187	43	235	32	316	24	423

Table VI-2 (Continued)

SERIES=COMPOUNDS BY ISOTOPE DILUTION

COMPOUND	P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER	P .0001 LOWER	P .0001 UPPER
301B ACENAPHTHENE	85	115	85	115	83	120	80	125
305B BENZIDINE	63	160	53	189	42	237	34	296
308B 1,2,4-TRICHLOROBENZENE	85	115	85	116	82	122	78	128
309B HEXACHLOROBENZENE	85	115	85	116	82	122	78	128
312B HEXACHLOROETHANE	85	115	82	122	76	131	71	141
318B BIS(2-CHLOROETHYL)ETHER	81	124	75	134	67	148	61	164
320B 2-CHLORONAPHTHALENE	80	125	73	136	65	153	58	171
321A 2,4,6-TRICHLOROPHENOL	85	115	85	115	85	118	81	123
322A P-CHLORO-M-CRESOL	85	115	85	115	85	115	85	115
324A 2-CHLOROPHENOL	85	115	85	116	82	123	78	129
325B 1,2-DICHLOROBENZENE	85	115	84	119	79	127	74	135
326B 1,3-DICHLOROBENZENE	83	121	77	129	71	141	65	154
327B 1,4-DICHLOROBENZENE	81	124	75	133	68	147	62	161
328B 3,3'-DICHLORODENZIDINE	85	115	85	117	81	123	77	130
331A 2,4-DICHLOROPHENOL	84	119	79	127	73	138	67	149
334A 2,4-DIMETHYLPHENOL	84	120	78	127	72	139	67	150
335B 2,4-DINITROTOLUENE	85	115	85	115	83	121	79	127
336B 2,6-DINITROTOLUENE	78	128	71	140	63	160	55	183
337B 1,2-DIPHENYLHYDRAZINE	85	115	84	119	79	126	75	134
339B FLUORANTHENE	84	120	78	127	72	138	67	149
340B 4-CHLOROPHENYL PHENYL ETH	85	117	81	123	76	132	71	142
342B BIS (2-CHLOROISOPROPYL) E	84	118	79	126	73	137	67	148
352B HEXACHLOROBUTADIENE	85	115	84	119	79	127	74	135
353B HEXACHLOROCYCLOPENTADIENE	85	115	85	116	82	123	77	129
354B ISOPHORONE	85	117	81	123	75	133	70	142
355B NAPHTHALENE	85	115	83	121	77	129	73	137
356B NITROBENZENE	85	115	85	115	85	115	85	115
357A 2-NITROPHENOL	85	115	85	117	81	123	77	129
358A 4-NITROPHENOL	78	129	71	142	62	161	55	183
359A 2,4-DINITROPHENOL	85	115	84	119	79	126	75	133
360A 4,6-DINITRO-O-CRESOL	85	118	80	125	74	135	69	145
362B N-NITROSODIPHENYLAMINE	85	116	81	123	74	134	68	148
364A PENTACHLOROPHENOL	85	115	85	117	81	124	77	130
365A PHENOL	82	122	77	130	70	142	65	155
366B BIS (2-ETHYLHEXYL) PHTHAL	85	115	85	118	80	125	76	131
368B DI-N-BUTYL PHTHALATE	85	117	81	123	76	132	71	142
369B DI-N-OCTYL PHTHALATE	85	116	82	123	76	131	71	140
370B DIETHYL PHTHALATE	85	115	83	120	78	128	74	135
371B DIMETHYL PHTHALATE	85	115	83	121	77	129	73	137
372B BENZO(A)ANTHRANCENE	85	116	81	123	76	132	70	142
373B BENZO(A)PYRENE	85	115	85	116	82	122	78	129
374B BENZO(B)FLUORANTHENE	81	124	75	134	67	148	61	164
375B BENZO(K)FLUORANTHENE	42	239	30	329	20	500	13	756
376B CHRYSENE	85	117	81	123	75	133	70	142
377B ACENAPHTHYLENE	80	125	74	135	67	150	60	166
378B ANTHRACENE	80	126	73	136	66	152	60	168
379B BENZO(GHI)PERYLENE	85	117	81	124	75	134	69	145
380B FLUORENE	85	115	84	120	79	127	74	135
381B PHENANTHRENE	85	115	84	119	79	126	75	133
384B PYRENE	85	115	85	118	80	125	76	132
702B BETA NAPHTHYLAMINE	67	150	57	174	47	212	39	256
703B ALPHA PICOLINE	81	124	75	134	67	149	60	165
704B DIBENZOTHIOPHENE	85	116	82	122	77	131	72	140

Table VI-2 (Concluded)

SERIES=COMPOUNDS BY ISOTOPE DILUTION

COMPOUND		P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER	P .0001 LOWER	P .0001 UPPER
705B DIBENZOFURAN		85	115	83	120	78	128	73	136
706B N-DODECANE	C12	80	125	74	136	66	150	60	166
707B DIPHENYLAMINE		79	127	72	139	64	156	57	176
708B DIPHENYLETHER		85	115	85	115	85	116	83	120
709B ALPHA TERPINEOL		78	128	71	140	62	161	54	186
710B STYRENE		83	120	78	129	71	141	65	153
711B DI-N-BUTYL AMINE		.	.	.	.	.	.	.	.
712B BIPHENYL		80	125	74	136	66	152	58	171
713B P-CYMENE		85	115	85	115	83	121	79	127
717B N-DECANE	C10	69	145	60	166	50	198	42	235
719B N-HEXADECANE	C16	85	116	82	122	77	130	72	138
721B N-EICOSANE	C20	76	131	69	144	61	163	54	184
723B N-TETRACOSANE	C24	82	121	77	130	71	142	65	154
726B N-TRIACONTANE	C30	83	120	78	129	71	140	66	152

specifications (in the second and third sections of Table VI-2, for the series labeled compounds by internal standard and compounds by isotope dilution) could be used, allowing one retest for failing compounds, or the .0001 level specifications could be used for a single-round test.

Ongoing Quality Assurance Tests

In each batch of samples processed by the laboratory, one water sample with known composition of 100 µg/L of all compounds is extracted and analyzed. Limits for the recovery of each compound and labeled compounds are given in Table VI-3. The limits are computed from the variance components analysis of extracted samples as

$$\exp[M \pm t(d,\ 1 - \tfrac{p}{2}) \sqrt{S_E^2 + S_A^2 + S_E^2/L + S_A^2/N}\ ] \quad ,$$

where t is as above, and d is the appropriate degrees of freedom, e.g., min (N-L, L-1). The derivation of this formula is given in Appendix K.

The limits are given at individual test levels of p = .05, .01, and .001. For the ongoing quality assurance test of all 154 compounds, the .01 level would be used to achieve an overall 5 percent significance level, assuming one retest is allowed for compounds which fail the first round.

In addition to the one QA sample in each batch, the recovery of labeled compounds is checked for every sample which is analyzed. Limits on the recovery of the labeled compounds in these samples are also obtainable from Table VI-3. Because the analysis of wastewater samples is not necessarily repeatable, an individual test level of p = .001 would be used to give an approximate overall 5 percent level test, with no allowance for retesting of failed compounds.

Table VI-3

ONGOING QUALITY ASSURANCE LIMITS

---------- SERIES=LABELLED ANALOGS BY INTERNAL STANDARD ----------

COMPOUND	P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER
201B ACENAPHTHENE-D10	39	138	30	180	20	270
205B BENZIDINE-D8 (RINGS-D8)	1	1231	0	7167	0	138619
208B 1,2,4-TRICHLOROBENZENE-D3	17	172	10	282	5	592
209B HEXACHLOROBENZENE-13C6	35	216	23	321	13	595
212B HEXACHLOROETHANE-1-13C	7	248	3	563	1	2104
218B BIS(2-CHLOROETHYL)-D8 ETH	34	159	25	222	15	372
220B 2-CHLORONAPHTHALENE-D7	32	149	24	204	15	324
221A 2,4,6-TRICHLOROPHENOL-3,5	45	168	34	226	21	363
222A 4-CHLORO-3-METHYLPHENOL-2	22	199	14	314	7	613
224A 2-CHLOROPHENOL-3,4,5,6-D4	42	137	33	176	23	255
225B 1,2-DICHLOROBENZENE-D4	18	156	11	247	6	494
226B 1,3-DICHLOROBENZENE-D4	15	159	9	260	4	550
227B 1,4-DICHLOROBENZENE-D4	17	156	11	245	6	474
228B 3,3'-DICHLOROBENZIDINE-D6	10	331	5	712	1	2339
231A 2,4-DICHLOROPHENOL-3,5,6-	43	142	34	182	24	260
234A 2,4-DIMETHYLPHENOL-3,5,6-	21	159	14	242	7	449
235B 2,4-DINITROTOLUENE-3,5,6-	29	183	19	275	10	514
236B 2,6-DINITROTOLUENE-D3	43	178	31	250	17	442
237B 1,2-DIPHENYL-D10-HYDRAZIN	35	148	26	200	17	316
239B FLUORANTHENE-D10	39	143	30	187	20	278
240B 4-CHLOROPHENYL PHENYL-D5	38	158	29	212	19	325
242B BIS(2-CHLOROISOPROPYL)ETH	40	129	30	169	20	260
252B HEXACHLORO-1,3-BUTADIENE-	10	215	5	413	2	1103
253B HEXACHLOROCYCLOPENTADIENE	0	642	0	4206	0	86246
254B ISOPHORONE-D8	52	123	44	147	33	193
255B NAPHTHALENE-D8	30	140	22	192	14	305
256B NITROBENZENE-D5	30	157	15	314	2	1982
257A 2-NITROPHENOL-3,4,5,6-D4	45	128	37	158	27	217
258A 4-NITROPHENOL-2,3,5,6-D4	12	345	6	716	2	2221
259A 2,4-DINITROPHENOL-3,5,6-D	27	238	17	378	8	759
260A 4,6-DINITRO-O-CRESOL-D2	40	215	28	307	16	527
262B N-NITROSODIPHENYLAMINE-D6	51	130	40	166	26	256
264A PENTACHLOROPHENOL-13C6	40	184	29	254	18	412
265A PHENOL-2,3,4,5,6-D5	14	237	8	424	3	1000
266B BIS(2-ETHYLHEXYL)PHTHALAT	39	164	28	224	18	364
268B DI-N-BUTYL PHTHALATE-D4	30	149	22	209	13	346
269B DI-N-OCTYL PHTHALATE-D4	17	250	10	433	4	969
270B DIETHYL PHTHALATE-3,4,5,6	10	201	5	375	2	941
271B DIMETHYL PHTHALATE-3,4,5,	2	325	1	923	0	4292
272B BENZO(A)ANTHRACENE-D12	33	220	22	329	12	605
273B BENZO(A)PYRENE-D12	42	149	32	194	21	290
274B BENZO(B)FLUORANTHENE-D12	12	410	5	889	2	2957
275B BENZO(K)FLUORANTHENE-D12	18	363	9	685	4	1786
276B CHRYSENE-D12	33	199	23	290	13	512
277B ACENAPHTHYLENE-D8	42	133	33	168	23	239
278B ANTHRACENE-D10	33	170	23	242	14	419
279B BENZO(GHI)PERYLENE-D12	36	209	25	303	14	529
280B FLUORENE-D10	47	138	38	172	27	238
281B PHENANTHRENE-D10	43	133	34	168	24	241
284B PYRENE-D10	37	147	28	196	18	303
602B 2-NAPHTHYL-D7-AMINE	2	988	0	4064	0	36378
603B 2-METHYLPYRIDINE-D7	11	300	5	608	2	1755
604B DIBENZOTHIOPHENE-D8	49	126	40	156	29	215
605B DIBENZOFURAN-D8	48	130	39	160	28	220
606B N-DODECANE-D26	9	213	5	408	2	1057
607B DIPHENYL-D10-AMINE	32	167	21	249	11	488
608B DIPHENYL-D10 ETHER	38	141	29	186	19	281
609B ALPHA-TERPINEOL-D3	29	217	18	339	9	672
610B STYRENE-2,3,4,5,6-D5	11	190	6	348	2	867
611B DI-N-BUTYL-D18-AMINE	0	3513	0	1828E3	0	542E14
612B DIPHENYL-D10	31	142	17	267	3	1436
613B P-CYMENE-D14	8	235	4	468	2	1286
617B N-DECANE-D22	9	212	5	404	2	1049
619B N-HEXADECANE-D34	37	152	28	202	18	308
621B N-EICOSANE-D42	38	149	29	198	19	306
623B N-TETRACOSANE-D50	35	165	25	229	15	376
626B N-TRIACONTANE-D62	33	189	23	274	13	479

Table VI-3 (Concluded)

SERIES=COMPOUNDS BY ISOTOPE DILUTION

COMPOUND	P .05 LOWER	P .05 UPPER	P .01 LOWER	P .01 UPPER	P .001 LOWER	P .001 UPPER
301B ACENAPHTHENE	80	130	72	144	62	170
305B BENZIDINE	22	346	11	672	4	2051
308B 1,2,4-TRICHLOROBENZENE	85	130	77	144	66	168
309B HEXACHLOROBENZENE	91	123	85	132	76	148
312B HEXACHLOROETHANE	28	620	13	1303	4	4546
318B BIS(2-CHLOROETHYL)ETHER	63	170	50	213	35	306
320B 2-CHLORONAPHTHALENE	52	298	35	442	19	833
321A 2,4,6-TRICHLOROPHENOL	62	189	48	244	32	367
322A P-CHLORO-M-CRESOL	72	137	62	159	49	201
324A 2-CHLOROPHENOL	83	126	76	138	66	159
325B 1,2-DICHLOROBENZENE	79	135	70	152	58	182
326B 1,3-DICHLOROBENZENE	69	182	55	225	40	313
327B 1,4-DICHLOROBENZENE	66	177	53	219	39	300
328B 3,3'-DICHLOROBENZIDINE	75	157	64	185	49	238
331A 2,4-DICHLOROPHENOL	89	126	83	135	75	150
334A 2,4-DIMETHYLPHENOL	70	135	60	156	48	195
335B 2,4-DINITROTOLUENE	82	143	72	164	57	206
336B 2,6-DINITROTOLUENE	80	139	70	159	56	198
337B 1,2-DIPHENYLHYDRAZINE	56	260	40	360	25	587
339B FLUORANTHENE	75	165	64	194	50	248
340B 4-CHLOROPHENYL PHENYL ETH	74	165	63	194	50	247
342B BIS (2-CHLOROISOPROPYL) E	85	131	77	145	65	170
352B HEXACHLOROBUTADIENE	57	216	43	287	28	437
353B HEXACHLOROCYCLOPENTADIENE	79	127	67	148	48	209
354B ISOPHORONE	80	148	70	168	58	204
355B NAPHTHALENE	83	134	75	149	64	174
356B NITROBENZENE	81	136	65	169	36	302
357A 2-NITROPHENOL	83	132	75	145	65	168
358A 4-NITROPHENOL	63	143	51	175	37	244
359A 2,4-DINITROPHENOL	76	127	68	141	58	167
360A 4,6-DINITRO-O-CRESOL	80	128	72	142	61	168
362B N-NITROSODIPHENYLAMINE	67	137	53	173	31	292
364A PENTACHLOROPHENOL	79	134	71	150	60	178
365A PHENOL	71	135	62	154	51	188
366B BIS (2-ETHYLHEXYL) PHTHAL	78	192	64	232	48	310
368B DI-N-BUTYL PHTHALATE	84	149	74	169	60	208
369B DI-N-OCTYL PHTHALATE	84	148	74	166	63	197
370B DIETHYL PHTHALATE	78	186	65	222	50	288
371B DIMETHYL PHTHALATE	79	175	67	207	52	266
372B BENZO(A)ANTHRANCENE	73	149	62	176	48	228
373B BENZO(A)PYRENE	71	171	59	206	44	272
374B BENZO(B)FLUORANTHENE	36	432	20	761	8	1886
375B BENZO(K)FLUORANTHENE	63	131	53	155	41	203
376B CHRYSENE	60	177	48	221	35	306
377B ACENAPHTHYLENE	74	173	61	207	47	272
378B ANTHRACENE	62	162	50	199	37	271
379B BENZO(GHI)PERYLENE	78	146	68	168	54	210
380B FLUORENE	78	135	70	151	59	178
381B PHENANTHRENE	92	119	87	126	80	138
384B PYRENE	81	141	72	159	60	191
702B BETA NAPHTHYLAMINE	22	523	9	1278	2	7248
703B ALPHA PICOLINE	61	143	50	174	37	237
704B DIBENZOTHIOPHENE	80	147	70	168	57	207
705B DIBENZOFURAN	87	133	79	146	68	168
706B N-DODECANE C12	43	285	29	424	16	772
707B DIPHENYLAMINE	65	181	51	231	34	349
708B DIPHENYLETHER	85	131	77	144	66	169
709B ALPHA TERPINEOL	53	182	38	258	19	509
710B STYRENE	61	190	48	244	32	358
711B DI-N-BUTYL AMINE	.	.	.	.	.	.
712B BIPHENYL	74	147	62	176	45	241
713B P-CYMENE	81	131	72	147	60	175
717B N-DECANE C10	28	160	19	237	10	447
719B N-HEXADECANE C16	82	158	71	181	58	223
721B N-EICOSANE C20	60	229	46	301	30	451
723B N-TETRACOSANE C24	86	129	78	142	67	165
726B N-TRIACONTANE C30	69	177	56	215	42	286

Retention Time

Limits are also needed for the retention time and relative retention time for each compound. These limits were obtained from the analysis of retention times for compounds from all 11 study samples. Relative retention time was computed as the ratio of the retention time for each compound to the retention time of the reference compound (~~DETPP~~ 2,2'-DFB for internal standard, the corresponding labeled compound for isotope dilution).

Retention time data received from the laboratories was less reproducible than expected. Past experience with analyses of this type has revealed that laboratories may eliminate the initial isothermal hold, increase the temperature program rate, increase the final temperature or use some combination of these techniques to save analysis time. Although these techniques may work when standard and blanks are being analyzed, separation of complex mixtures often found in samples may be incomplete when they are employed. Because these methods are to be applied to such complex samples, an investigation was made into the actual analytical conditions employed by each laboratory.

When a gas chromatograph is operated isothermally, the retention time of a given compound is an indication of the column temperature, and when a gas chromatograph is operated under temperature programmed conditions, the compounds will elute at predictable intervals. By comparing the retention times of the compounds which elute prior to the end of the initial isothermal hold, and by comparing the intervals at which the conpounds elute during the ramp phase of the temperature program, the actual temperature program employed for a given analysis can be deduced.

Figure VI-1 shows the actual temperature programs used by laboratories in this study. As can be seen, many of the laboratories used programs other than those specified in the method and reinforced in the instructions (superimposed programs have been eliminated to provide clarity). Laboratories A, C, J, K, and O used the proper temperature program. As a result, these data only were used for generation of retention time specifications.

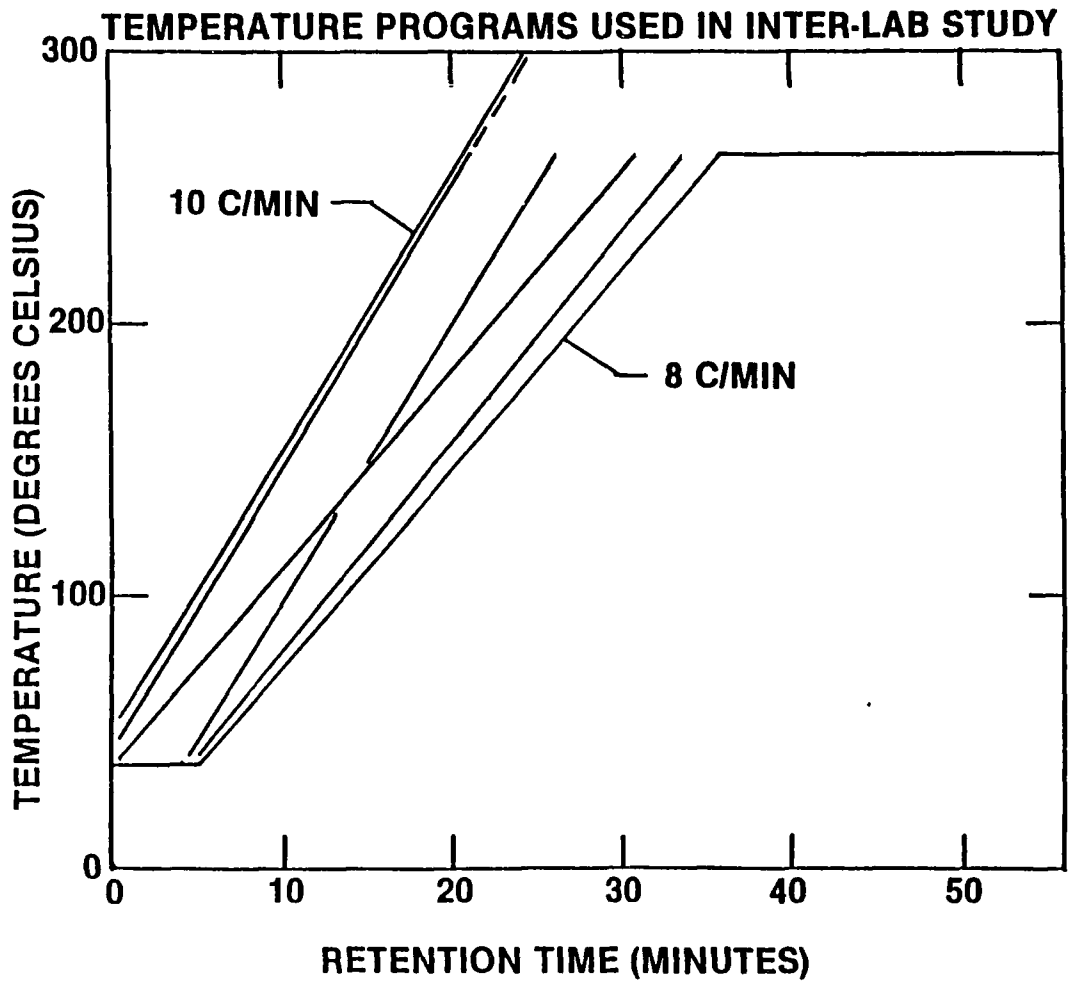

Figure VI-1 COLUMN TEMPERATURE PROGRAMS USED

Before analysis, the relative retention time values were subjected to an outlier analysis similar to that used on the amount values. A QSCREEN at level .001 was used as described in Section IV and Appendix H. Flagged cases had both their retention time and relative retention times set to missing for this analysis. Also only retention times from detected compounds were used. Therefore, for the unlabeled compounds, the BLK entries and most of the EPA entries were missing.

Given in Tables VI-4 and VI-5 are the results for retention times and relative retention times. Nominal-scale analyses were used for these calculations. For these analyses, the mean, standard deviation, coefficient of variation, minimum, and maximum are computed with the standard formulas. The 95 percent confidence limit for the mean of the quantity is computed as

$$\bar{X} \pm t_{n-1}(.975)\, S_n/\sqrt{n} \quad ,$$

where

$$\bar{X} = \sum_{i=1}^{n} X_i/n \quad ,$$

$$S_n = \left(\sum_{i=1}^{n} (X_i - \bar{X})^2/(n-1) \right)^{1/2} \quad ,$$

and t_d is the inverse cumulative distribution function of the t distribution with d degrees of freedom. The prediction limits are given by

$$\bar{X} \pm t_{n-1}(.975)\, S_n \sqrt{1 + \frac{1}{n}} \quad .$$

This is derived analogously to the regression prediction formulas (see, for instance, Draper and Smith, Applied Regression Analysis, p. 30). Also given are the percentage of times the observation fell outside the calculated 95 percent prediction limits.

Table VI-4

RETENTION TIME

COMPOUND	N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
001B ACENAPHTHENE	40	1304	46	3.5	1253	1397	1289	1319	1211	1398	0.0
005B BENZIDINE	30	1856	64	3.4	1805	1967	1832	1880	1724	1988	0.0
008B 1,2,4-TRICHLOROBENZENE	40	958	37	3.9	906	1031	946	970	883	1034	0.0
009B HEXACHLOROBENZENE	39	1522	51	3.4	1472	1626	1506	1539	1418	1627	0.0
012B HEXACHLOROETHANE	32	820	36	4.4	771	882	807	833	746	895	0.0
018B BIS(2-CHLOROETHYL)ETHER	39	704	28	3.9	660	757	695	713	647	761	0.0
020B 2-CHLORONAPHTHALENE	30	1201	41	3.4	1167	1275	1186	1217	1115	1287	0.0
021A 2,4,6-TRICHLOROPHENOL	39	1165	42	3.6	1111	1247	1151	1178	1078	1251	0.0
022A P-CHLORO-M-CPESOL	39	1090	40	3.7	1037	1169	1077	1103	1007	1173	0.0
024A 2-CHLOROPHENOL	38	705	27	3.8	661	754	696	713	649	760	0.0
025B 1,2-DICHLOROBENZENE	40	760	33	4.3	720	825	750	771	693	828	0.0
026B 1,3-DICHLOROBENZENE	39	723	28	3.9	680	779	714	732	666	780	0.0
027B 1,4-DICHLOROBENZENE	40	740	31	4.2	690	790	730	750	676	804	0.0
028B 3,3'-DICHLOROBENZIDINE	39	2090	63	3.0	2049	2221	2070	2111	1960	2220	2.6
031A 2,4-DICHLOROPHENOL	39	947	37	3.9	895	1017	935	959	871	1023	0.0
034A 2,4-DIMETHYLPHENOL	39	923	36	3.9	870	992	911	935	849	998	0.0
035B 2,4-DINITROTOLUENE	40	1344	59	4.4	1265	1452	1325	1363	1222	1465	0.0
036B 2,6-DINITROTOLUENE	39	1297	50	3.8	1232	1374	1281	1314	1195	1400	0.0
037B 1,2-DIPHENYLHYDRAZINE	39	1440	49	3.4	1389	1539	1424	1456	1339	1540	0.0
039B FLUORANTHENE	39	1818	56	3.1	1765	1929	1800	1836	1704	1932	0.0
040B 4-CHLOROPHENYL PHENYL ETH	37	1411	49	3.4	1359	1507	1395	1428	1311	1511	0.0
041B 4-BROMOPHENYL PHENYL ETHE	37	1498	49	3.3	1450	1602	1482	1514	1397	1599	5.4
042B BIS (2-CHLOROISOPROPYL) E	40	799	32	4.0	750	859	789	809	734	864	0.0
052B HEXACHLOROBUTADIENE	39	1006	39	3.8	953	1082	993	1018	927	1085	0.0
053B HEXACHLOROCYCLOPENTADIENE	37	1143	43	3.8	1090	1227	1128	1157	1054	1231	0.0
054B ISOPHORONE	40	889	35	3.9	840	957	878	900	818	959	0.0
055B NAPHTHALENE	40	967	37	3.8	915	1040	955	979	891	1043	0.0
056B NITROBENZENE	16	849	52	6.1	793	906	821	876	735	963	0.0
057A 2-NITROPHENOL	39	899	34	3.8	849	969	888	910	828	969	0.0
058A 4-NITROPHENOL	39	1354	45	3.3	1301	1443	1339	1368	1262	1446	0.0
059A 2,4-DINITROPHENOL	39	1327	46	3.4	1275	1418	1313	1342	1234	1421	0.0
060A 4,6-DINITRO-O-CRESOL	39	1437	49	3.4	1384	1534	1421	1452	1336	1537	0.0
062B N-NITROSODIPHENYLAMINE	16	1464	71	4.8	1388	1537	1426	1502	1309	1619	0.0
064A PENTACHLOROPHENOL	39	1561	52	3.3	1512	1666	1544	1578	1455	1667	0.0
065A PHENOL	40	700	27	3.9	654	750	691	708	644	755	0.0
066B BIS (2-ETHYLHEXYL) PHTHAL	39	2125	64	3.0	2077	2258	2104	2146	1993	2257	2.6
068B DI-N-BUTYL PHTHALATE	39	1723	55	3.2	1678	1837	1705	1741	1609	1836	2.6
069B DI-N-OCTYL PHTHALATE	38	2242	75	3.3	2192	2396	2218	2267	2089	2396	2.6
070B DIETHYL PHTHALATE	39	1413	48	3.4	1363	1510	1398	1429	1314	1513	0.0
071B DIMETHYL PHTHALATE	40	1273	44	3.5	1222	1363	1259	1287	1182	1363	0.0
072B BENZO(A)ANTHRANCENE	39	2088	66	3.2	2042	2223	2066	2109	1952	2223	0.0
073B BENZO(A)PYRENE	40	2353	91	3.9	2240	2544	2324	2382	2167	2539	2.5
074B BENZO(B)FLUORANTHENE	40	2291	79	3.5	2230	2458	2265	2316	2128	2453	2.5
075B BENZO(K)FLUORANTHENE	37	2291	78	3.4	2234	2463	2265	2317	2131	2452	5.4
076B CHRYSENE	39	2085	62	3.0	2044	2214	2065	2106	1958	2213	2.6
077B ACENAPHTHYLENE	32	1247	15	1.2	1216	1261	1242	1253	1216	1278	3.1
078B ANTHRACENE	39	1592	53	3.3	1534	1699	1574	1609	1483	1701	0.0
079B BENZO(GHI)PERYLENE	40	2752	169	6.1	2572	3095	2698	2806	2406	3097	0.0
080B FLUORENE	40	1401	48	3.4	1350	1499	1386	1417	1303	1499	0.0
081B PHENANTHRENE	40	1583	52	3.3	1534	1690	1566	1599	1476	1689	2.5
084B PYRENE	32	1848	72	3.9	1773	1972	1822	1873	1699	1996	0.0
164B 2,2'-DIFLUOROBIPHENYL	54	1163	42	3.6	1112	1249	1152	1174	1078	1248	1.9

Table VI-4 (Continued)

COMPOUND	N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
201B ACENAPHTHENE-D10	50	1298	45	3.5	1247	1392	1285	1311	1207	1389	2.0
205B BENZIDINE-D8 (RINGS-D8)	37	1854	63	3.4	1804	1965	1833	1875	1725	1984	0.0
208B 1,2,4-TRICHLOROBENZENE-D3	49	955	37	3.8	904	1029	945	966	881	1030	0.0
209B HEXACHLOROBENZENE-13C6	49	1521	51	3.3	1472	1625	1506	1535	1418	1624	2.0
212B HEXACHLOROETHANE-1-13C	40	819	35	4.3	770	882	808	830	746	892	0.0
218B BIS(2-CHLOROETHYL)-D8 ETH	30	696	34	4.9	654	750	683	709	624	767	0.0
220B 2-CHLORONAPHTHALENE-D7	49	1185	43	3.6	1134	1273	1173	1197	1098	1272	2.0
221A 2,4,6-TRICHLOROPHENOL-3,5	49	1162	42	3.6	1110	1246	1150	1174	1077	1247	0.0
222A 4-CHLORO-3-METHYLPHENOL-2	47	1086	39	3.6	1037	1170	1075	1098	1008	1165	4.3
224A 2-CHLOROPHENOL-3,4,5,6-D4	48	701	26	3.8	655	752	693	708	647	754	0.0
225B 1,2-DICHLOROBENZENE-D4	40	758	35	4.6	719	823	747	770	686	830	0.0
226B 1,3-DICHLOROBENZENE-D4	50	722	27	3.8	678	777	714	729	666	777	2.0
227B 1,4-DICHLOROBENZENE-D4	50	737	32	4.3	688	788	728	746	673	801	0.0
228B 3,3'-DICHLOROBENZIDINE-D6	49	2088	63	3.0	2048	2220	2070	2106	1960	2216	2.0
231A 2,4-DICHLOROPHENOL-3,5,6-	49	944	36	3.9	892	1016	934	955	870	1018	0.0
234A 2,4-DIMETHYLPHENOL-3,5,6-	49	921	36	3.9	870	992	911	932	848	994	0.0
235B 2,4-DINITROTOLUENE-3,5,6-	40	1359	51	3.8	1305	1451	1343	1375	1255	1464	0.0
236B 2,6-DINITROTOLUENE-D3	39	1283	50	3.9	1230	1373	1267	1299	1181	1386	0.0
237B 1,2-DIPHENYL-D10-HYDRAZIN	49	1433	49	3.4	1385	1535	1419	1447	1334	1531	2.0
239B FLUORANTHENE-D10	50	1813	55	3.0	1763	1926	1798	1829	1702	1924	2.0
240B 4-CHLOROPHENYL PHENYL-D5	49	1406	48	3.4	1357	1506	1393	1420	1310	1503	2.0
242B BIS(2-CHLOROISOPROPYL)ETH	50	788	31	3.9	740	849	779	797	725	851	0.0
252B HEXACHLORO-1,3-BUTADIENE-	49	1005	38	3.8	953	1081	994	1016	927	1083	0.0
253B HEXACHLOROCYCLOPENTADIENE	31	1147	46	4.0	1090	1226	1130	1163	1051	1242	0.0
254B ISOPHORONE-D8	50	881	34	3.8	834	950	871	890	812	949	2.0
255B NAPHTHALENE-D8	50	963	37	3.8	912	1037	953	973	889	1037	0.0
256B NITROBENZENE-D5	20	845	51	6.0	790	903	821	869	736	954	0.0
257A 2-NITROPHENOL-3,4,5,6-D4	50	898	35	3.9	847	968	888	908	828	969	0.0
258A 4-NITROPHENOL-2,3,5,6-D4	49	1349	45	3.4	1298	1442	1336	1362	1257	1441	2.0
259A 2,4-DINITROPHENOL-3,5,6-D	49	1323	45	3.4	1272	1416	1310	1336	1231	1415	2.0
260A 4,6-DINITRO-O-CRESOL-D2	50	1433	48	3.3	1379	1532	1419	1446	1336	1530	6.0
262B N-NITROSODIPHENYLAMINE-D6	30	1447	60	4.2	1387	1536	1424	1469	1322	1572	0.0
264A PENTACHLOROPHENOL-13C6	50	1559	50	3.2	1512	1666	1545	1574	1457	1662	4.0
265A PHENOL-2,3,4,5,6-D5	50	696	27	3.8	653	749	689	704	642	751	0.0
266B BIS(2-ETHYLHEXYL)PHTHALAT	49	2123	64	3.0	2076	2257	2104	2141	1993	2253	2.0
268B DI-N-BUTYL PHTHALATE-D4	50	1719	55	3.2	1677	1835	1703	1734	1608	1830	4.0
269B DI-N-OCTYL PHTHALATE-D4	48	2239	74	3.3	2191	2396	2218	2261	2089	2390	2.1
270B DIETHYL PHTHALATE-3,4,5,6	49	1409	46	3.3	1362	1509	1395	1422	1315	1502	8.2
271B DIMETHYL PHTHALATE-3,4,5,	48	1269	43	3.4	1220	1361	1256	1282	1181	1357	4.2
272B BENZO(A)ANTHRACENE-D12	49	2082	65	3.1	2038	2219	2063	2100	1949	2214	2.0
273B BENZO(A)PYRENE-D12	49	2351	88	3.7	2289	2539	2326	2376	2173	2528	2.0
274B BENZO(B)FLUORANTHENE-D12	49	2281	74	3.3	2229	2451	2259	2302	2130	2432	10.2
275B BENZO(K)FLUORANTHENE-D12	47	2287	77	3.4	2230	2460	2265	2310	2130	2444	8.5
276B CHRYSENE-D12	49	2081	62	3.0	2041	2211	2063	2098	1956	2206	2.0
277B ACENAPHTHYLENE-D8	50	1265	44	3.5	1214	1357	1252	1277	1175	1354	2.0
278B ANTHRACENE-D10	49	1588	52	3.3	1540	1697	1573	1603	1482	1693	2.0
279B BENZO(GHI)PERYLENE-D12	49	2741	166	6.1	2561	3087	2693	2788	2403	3078	2.0
280B FLUORENE-D10	50	1395	47	3.4	1346	1495	1382	1409	1300	1491	2.0
281B PHENANTHRENE-D10	50	1578	51	3.2	1530	1687	1563	1592	1474	1682	2.0
284B PYRENE-D10	40	1844	70	3.8	1772	1969	1821	1866	1699	1988	0.0
301B ACENAPHTHENE	40	1304	46	3.5	1253	1397	1289	1319	1211	1398	0.0
305B BENZIDINE	30	1853	66	3.5	1761	1967	1829	1878	1717	1990	0.0

Table VI-4 (Continued)

COMPOUND	N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
308B 1,2,4-TRICHLOROBENZENE	39	958	37	3.9	906	1031	946	971	882	1035	0.0
309B HEXACHLOROBENZENE	40	1522	50	3.3	1472	1626	1506	1538	1418	1625	2.5
312B HEXACHLOROETHANE	28	823	37	4.4	771	882	809	837	747	899	0.0
318B BIS(2-CHLOROETHYL)ETHER	39	704	28	3.9	660	757	695	713	647	761	0.0
320B 2-CHLORONAPHTHALENE	31	1200	41	3.4	1167	1275	1185	1215	1115	1285	0.0
321A 2,4,6-TRICHLOROPHENOL	37	1165	43	3.7	1111	1247	1151	1180	1077	1254	0.0
322A P-CHLORO-M-CRESOL	35	1091	40	3.6	1037	1169	1078	1105	1009	1173	0.0
324A 2-CHLOROPHENOL	39	705	27	3.8	661	754	696	713	650	759	0.0
325B 1,2-DICHLOROBENZENE	40	760	33	4.3	720	825	750	771	693	828	0.0
326B 1,3-DICHLOROBENZENE	40	724	28	3.9	680	779	715	733	667	782	0.0
327B 1,4-DICHLOROBENZENE	40	740	31	4.2	690	790	730	750	676	804	0.0
328B 3,3'-DICHLOROBENZIDINE	39	2086	61	2.9	2049	2221	2067	2106	1962	2211	15.4
331A 2,4-DICHLOROPHENOL	40	947	36	3.8	895	1017	935	959	872	1021	0.0
334A 2,4-DIMETHYLPHENOL	35	924	38	4.2	870	992	911	937	844	1003	0.0
335B 2,4-DINITROTOLUENE	40	1344	59	4.4	1265	1452	1325	1363	1222	1465	0.0
336B 2,6-DINITROTOLUENE	39	1300	52	4.0	1232	1388	1284	1317	1195	1406	0.0
337B 1,2-DIPHENYLHYDRAZINE	40	1439	49	3.4	1389	1539	1424	1455	1340	1539	2.5
339B FLUORANTHENE	40	1817	55	3.0	1765	1929	1800	1835	1704	1930	0.0
340B 4-CHLOROPHENYL PHENYL ETH	40	1409	48	3.4	1359	1507	1393	1424	1311	1507	2.5
342B BIS (2-CHLOROISOPROPYL) E	40	799	32	4.0	750	859	789	809	734	864	0.0
352B HEXACHLOROBUTADIENE	40	1006	38	3.8	953	1082	994	1018	928	1084	0.0
353B HEXACHLOROCYCLOPENTADIENE	38	1142	43	3.7	1090	1227	1128	1156	1055	1230	0.0
354B ISOPHORONE	40	889	35	3.9	840	957	878	900	818	959	0.0
355B NAPHTHALENE	39	967	38	3.9	915	1040	955	980	890	1044	0.0
356B NITROBENZENE	16	849	52	6.1	793	906	821	876	735	963	0.0
357A 2-NITROPHENOL	40	900	35	3.9	849	969	889	911	828	972	0.0
358A 4-NITROPHENOL	38	1354	46	3.4	1301	1443	1339	1369	1261	1448	0.0
359A 2,4-DINITROPHENOL	38	1325	44	3.3	1275	1418	1311	1340	1234	1416	5.3
360A 4,6-DINITRO-O-CRESOL	36	1435	48	3.3	1384	1534	1419	1451	1336	1534	2.8
362B N-NITROSODIPHENYLAMINE	16	1464	71	4.8	1388	1537	1426	1502	1309	1619	0.0
364A PENTACHLOROPHENOL	40	1561	51	3.3	1512	1666	1544	1577	1456	1665	2.5
365A PHENOL	40	700	27	3.9	654	750	691	708	644	755	0.0
366B BIS (2-ETHYLHEXYL) PHTHAL	36	2124	63	3.0	2085	2258	2102	2145	1994	2254	2.8
368B DI-N-BUTYL PHTHALATE	39	1723	55	3.2	1678	1837	1705	1740	1609	1836	2.6
369B DI-N-OCTYL PHTHALATE	36	2240	81	3.6	2092	2396	2213	2267	2074	2406	0.0
370B DIETHYL PHTHALATE	40	1414	48	3.4	1363	1510	1398	1429	1316	1511	0.0
371B DIMETHYL PHTHALATE	39	1273	45	3.5	1222	1363	1259	1288	1181	1365	0.0
372B BENZO(A)ANTHRANCENE	37	2090	67	3.2	2042	2223	2068	2113	1952	2228	0.0
373B BENZO(A)PYRENE	38	2350	88	3.7	2240	2544	2322	2379	2171	2530	7.9
374B BENZO(B)FLUORANTHENE	38	2293	81	3.5	2230	2458	2266	2319	2126	2459	0.0
375B BENZO(K)FLUORANTHENE	38	2296	81	3.5	2234	2463	2269	2322	2128	2463	2.6
376B CHRYSENE	37	2083	60	2.9	2044	2214	2063	2104	1960	2207	5.4
377B ACENAPHTHYLENE	31	1247	15	1.2	1216	1261	1241	1253	1215	1279	0.0
378B ANTHRACENE	39	1592	53	3.3	1534	1699	1574	1609	1483	1701	0.0
379B BENZO(GHI)PERYLENE	36	2750	166	6.0	2572	3095	2694	2806	2408	3092	2.8
380B FLUORENE	40	1401	48	3.4	1350	1499	1386	1417	1303	1499	0.0
381B PHENANTHRENE	40	1583	52	3.3	1534	1690	1566	1599	1476	1689	2.5
384B PYRENE	30	1852	72	3.9	1775	1972	1826	1879	1704	2001	0.0
502B BETA NAPHTHYLAMINE	38	1371	48	3.5	1320	1466	1355	1387	1272	1470	0.0
503B ALPHA PICOLINE	38	427	8	1.9	415	446	425	430	411	444	2.6
504B DIBENZOTHIOPHENE	31	1564	57	3.7	1509	1665	1543	1585	1446	1683	0.0
505B DIBENZOFURAN	40	1335	46	3.5	1284	1430	1320	1350	1240	1430	0.0

Table VI-4 (Concluded)

COMPOUND		N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
506B N-DODECANE		39	979	37	3.8	928	1055	967	991	903	1055	0.0
507B DIPHENYLAMINE		40	1439	48	3.4	1388	1537	1424	1455	1340	1538	0.0
508B DIPHENYLETHER		39	1216	44	3.6	1164	1304	1202	1230	1126	1306	0.0
509B ALPHA TERPINEOL		39	977	38	3.9	924	1050	964	989	899	1055	0.0
510B STYRENE		40	550	16	3.0	521	580	545	555	517	584	0.0
511B DI-N-BUTYL AMINE		30	733	66	9.0	651	822	708	758	595	871	0.0
512B BIPHENYL		32	1195	48	4.0	1139	1279	1177	1212	1096	1294	0.0
513B P-CYMENE		38	754	30	3.9	708	811	744	763	693	814	0.0
517B N-DECANE	C10	39	719	28	3.9	673	773	710	728	661	777	0.0
519B N-HEXADECANE	C16	39	1471	135	9.2	1354	1720	1427	1515	1194	1748	0.0
521B N-EICOSANE	C20	39	1676	156	9.3	1389	1857	1625	1726	1356	1995	0.0
523B N-TETRACOSANE	C24	39	2024	62	3.1	1962	2151	2003	2044	1896	2151	0.0
526B N-TRIACONTANE	C30	39	2433	109	4.5	2340	2662	2397	2468	2209	2657	2.6
602B 2-NAPHTHYL-D7-AMINE		48	1368	47	3.4	1319	1464	1355	1382	1272	1464	0.0
603B 2-METHYLPYRIDINE-D7		49	417	9	2.3	386	442	415	420	398	437	6.1
604B DIBENZOTHIOPHENE-D8		39	1559	57	3.6	1507	1662	1540	1577	1442	1675	0.0
605B DIBENZOFURAN-D8		49	1331	46	3.5	1281	1428	1318	1345	1237	1425	2.0
606B N-DODECANE-D26		50	953	63	6.6	707	1034	935	971	824	1081	4.0
607B DIPHENYL-D10-AMINE		40	1437	52	3.6	1385	1534	1420	1454	1330	1545	0.0
608B DIPHENYL-D10 ETHER		50	1211	43	3.5	1160	1300	1199	1224	1125	1298	2.0
609B ALPHA-TERPINEOL-D3		50	973	37	3.8	923	1048	962	983	898	1048	0.0
610B STYRENE-2,3,4,5,6-D5		49	546	16	2.8	519	578	541	550	514	577	2.0
611B DI-N-BUTYL-D18-AMINE		20	742	80	10.8	659	832	705	780	570	915	0.0
612B DIPHENYL-D10		30	1205	45	3.7	1163	1275	1189	1222	1112	1298	0.0
613B P-CYMENE-D14		50	742	29	3.9	697	799	734	750	684	801	0.0
617B N-DECANE-D22		50	698	27	3.9	654	751	690	705	643	752	0.0
619B N-HEXADECANE-D34		49	1447	135	9.3	1331	1697	1408	1485	1172	1721	0.0
621B N-EICOSANE-D42		48	1655	151	9.1	1360	1832	1612	1699	1348	1962	0.0
623B N-TETRACOSANE-D50		49	1997	61	3.1	1960	2125	1979	2014	1873	2121	2.0
626B N-TRIACONTANE-D62		49	2384	98	4.1	2308	2597	2356	2412	2184	2584	2.0
702B BETA NAPHTHYLAMINE		39	1371	48	3.5	1320	1466	1355	1386	1273	1469	0.0
703B ALPHA PICOLINE		39	426	10	2.3	392	446	423	430	406	446	2.6
704B DIBENZOTHIOPHENE		32	1564	56	3.6	1509	1665	1543	1584	1447	1680	0.0
705B DIBENZOFURAN		40	1335	46	3.5	1284	1430	1320	1350	1240	1430	0.0
706B N-DODECANE	C12	40	981	38	3.9	928	1055	969	993	903	1058	0.0
707B DIPHENYLAMINE		40	1439	48	3.4	1388	1537	1424	1455	1340	1538	0.0
708B DIPHENYLETHER		40	1216	43	3.6	1164	1304	1202	1230	1127	1305	0.0
709B ALPHA TERPINEOL		39	975	36	3.7	924	1050	963	987	900	1050	2.6
710B STYRENE		37	549	17	3.0	521	580	544	555	515	584	0.0
711B DI-N-BUTYL AMINE		30	733	66	9.0	651	822	708	758	595	871	0.0
712B BIPHENYL		31	1195	49	4.1	1139	1279	1177	1213	1094	1296	0.0
713B P-CYMENE		40	755	30	3.9	708	811	745	764	694	815	0.0
717B N-DECANE	C10	40	720	28	3.9	673	773	711	729	662	778	0.0
719B N-HEXADECANE	C16	40	1469	134	9.1	1354	1720	1426	1512	1195	1744	0.0
721B N-EICOSANE	C20	40	1677	154	9.2	1389	1857	1627	1726	1361	1992	0.0
723B N-TETRACOSANE	C24	39	2025	62	3.1	1984	2151	2005	2046	1897	2153	0.0
726B N-TRIACONTANE	C30	37	2429	106	4.4	2340	2662	2393	2464	2210	2647	2.7

Table VI-5

RELATIVE RETENTION TIME

COMPOUND	N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
001B ACENAPHTHENE	40	1.121	0.005	0.5	1.106	1.129	1.119	1.122	1.110	1.132	2.5
005B BENZIDINE	30	1.590	0.021	1.3	1.553	1.626	1.582	1.598	1.546	1.634	0.0
008B 1,2,4-TRICHLOROBENZENE	40	0.823	0.004	0.5	0.815	0.832	0.822	0.825	0.814	0.833	0.0
009B HEXACHLOROBENZENE	39	1.308	0.009	0.7	1.296	1.326	1.305	1.311	1.289	1.327	0.0
012B HEXACHLOROETHANE	32	0.704	0.007	1.0	0.693	0.714	0.702	0.706	0.690	0.718	0.0
018B BIS(2-CHLOROETHYL)ETHER	39	0.605	0.005	0.9	0.594	0.614	0.603	0.607	0.594	0.616	5.1
020B 2-CHLORONAPHTHALENE	30	1.021	0.003	0.3	1.011	1.025	1.020	1.022	1.015	1.028	6.7
021A 2,4,6-TRICHLOROPHENOL	39	1.001	0.003	0.3	0.995	1.008	1.000	1.002	0.996	1.006	7.7
022A P-CHLORO-M-CRESOL	39	0.937	0.003	0.4	0.930	0.945	0.936	0.938	0.930	0.944	2.6
024A 2-CHLOROPHENOL	38	0.606	0.007	1.1	0.594	0.620	0.603	0.608	0.592	0.619	2.6
025B 1,2-DICHLOROBENZENE	40	0.653	0.010	1.5	0.632	0.665	0.650	0.657	0.633	0.674	5.0
026B 1,3-DICHLOROBENZENE	39	0.623	0.008	1.2	0.608	0.635	0.620	0.625	0.607	0.638	0.0
027B 1,4-DICHLOROBENZENE	40	0.636	0.015	2.3	0.620	0.670	0.631	0.641	0.605	0.666	2.5
028B 3,3'-DICHLOROBENZIDINE	39	1.797	0.026	1.4	1.772	1.849	1.789	1.805	1.745	1.849	0.0
031A 2,4-DICHLOROPHENOL	39	0.814	0.004	0.5	0.805	0.821	0.812	0.815	0.805	0.823	2.6
034A 2,4-DIMETHYLPHENOL	39	0.793	0.005	0.7	0.782	0.805	0.792	0.795	0.783	0.804	5.1
035B 2,4-DINITROTOLUENE	40	1.155	0.028	2.4	1.093	1.180	1.146	1.164	1.097	1.212	2.5
036B 2,6-DINITROTOLUENE	39	1.115	0.024	2.1	1.090	1.177	1.107	1.123	1.066	1.164	5.1
037B 1,2-DIPHENYLHYDRAZINE	39	1.237	0.008	0.7	1.218	1.253	1.235	1.240	1.220	1.255	2.6
039B FLUORANTHENE	39	1.563	0.017	1.1	1.538	1.591	1.558	1.569	1.528	1.598	0.0
040B 4-CHLOROPHENYL PHENYL ETH	37	1.211	0.006	0.5	1.202	1.223	1.208	1.213	1.198	1.224	0.0
041B 4-BROMOPHENYL PHENYL ETHE	37	1.289	0.009	0.7	1.277	1.307	1.286	1.292	1.271	1.307	0.0
042B BIS (2-CHLOROISOPROPYL) E	40	0.687	0.006	0.9	0.674	0.697	0.685	0.689	0.674	0.700	0.0
052B HEXACHLOROBUTADIENE	39	0.864	0.004	0.4	0.857	0.872	0.863	0.866	0.857	0.872	2.6
053B HEXACHLOROCYCLOPENTADIENE	37	0.982	0.002	0.2	0.978	0.987	0.981	0.982	0.977	0.986	2.7
054B ISOPHORONE	40	0.764	0.005	0.7	0.753	0.775	0.762	0.765	0.753	0.774	7.5
055B NAPHTHALENE	40	0.831	0.004	0.5	0.821	0.840	0.830	0.832	0.822	0.840	5.0
056B NITROBENZENE	16	0.719	0.005	0.7	0.713	0.725	0.717	0.722	0.708	0.731	0.0
057A 2-NITROPHENOL	39	0.773	0.005	0.6	0.763	0.784	0.772	0.775	0.764	0.783	5.1
058A 4-NITROPHENOL	39	1.162	0.007	0.6	1.145	1.174	1.160	1.164	1.148	1.176	2.6
059A 2,4-DINITROPHENOL	39	1.141	0.005	0.5	1.132	1.151	1.139	1.143	1.130	1.152	0.0
060A 4,6-DINITRO-O-CRESOL	39	1.234	0.008	0.7	1.215	1.250	1.232	1.237	1.217	1.251	2.6
062B N-NITROSODIPHENYLAMINE	16	1.242	0.008	0.6	1.231	1.252	1.237	1.246	1.224	1.259	0.0
064A PENTACHLOROPHENOL	39	1.342	0.011	0.8	1.329	1.363	1.338	1.345	1.320	1.363	0.0
065A PHENOL	40	0.601	0.007	1.1	0.588	0.616	0.599	0.603	0.587	0.615	2.5
066B BIS (2-ETHYLHEXYL) PHTHAL	39	1.827	0.027	1.5	1.802	1.881	1.818	1.836	1.772	1.882	0.0
068B DI-N-BUTYL PHTHALATE	39	1.481	0.015	1.0	1.465	1.511	1.476	1.486	1.451	1.511	0.0
069B DI-N-OCTYL PHTHALATE	38	1.925	0.028	1.4	1.895	1.984	1.916	1.934	1.868	1.982	5.3
070B DIETHYL PHTHALATE	39	1.214	0.008	0.7	1.196	1.230	1.212	1.217	1.198	1.231	2.6
071B DIMETHYL PHTHALATE	40	1.094	0.005	0.4	1.079	1.101	1.092	1.095	1.084	1.104	5.0
072B BENZO(A)ANTHRANCENE	39	1.795	0.028	1.5	1.765	1.851	1.786	1.804	1.738	1.852	0.0
073B BENZO(A)PYRENE	40	2.023	0.037	1.8	1.944	2.092	2.011	2.034	1.948	2.097	2.5
074B BENZO(B)FLUORANTHENE	40	1.969	0.031	1.6	1.926	2.031	1.959	1.979	1.905	2.032	0.0
075B BENZO(K)FLUORANTHENE	37	1.973	0.031	1.6	1.935	2.035	1.962	1.983	1.909	2.037	0.0
076B CHRYSENE	39	1.793	0.024	1.3	1.770	1.841	1.785	1.800	1.744	1.841	0.0
077B ACENAPHTHYLENE	32	1.090	0.004	0.4	1.076	1.095	1.088	1.091	1.081	1.099	3.1
078B ANTHRACENE	39	1.368	0.011	0.8	1.353	1.391	1.365	1.372	1.346	1.390	2.6
079B BENZO(GHI)PERYLENE	40	2.363	0.086	3.6	2.236	2.488	2.336	2.391	2.187	2.540	0.0
080B FLUORENE	40	1.204	0.007	0.6	1.185	1.218	1.202	1.207	1.189	1.219	2.5
081B PHENANTHRENE	40	1.360	0.012	0.9	1.335	1.383	1.356	1.364	1.336	1.384	2.5
084B PYRENE	32	1.586	0.030	1.9	1.532	1.628	1.575	1.597	1.523	1.649	0.0
164B 2,2'-DIFLUOROBIPHENYL	54	1.000	0.000	0.0	1.000	1.000	1.000	1.000	1.000	1.000	0.0

Table VI-5 (Continued)

COMPOUND	N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
201B ACENAPHTHENE-D10	50	1.116	0.005	0.4	1.101	1.123	1.115	1.117	1.107	1.125	4.0
205B BENZIDINE-D8 (RINGS-D8)	37	1.590	0.020	1.3	1.553	1.625	1.583	1.597	1.549	1.632	0.0
208B 1,2,4-TRICHLOROBENZENE-D3	49	0.821	0.004	0.5	0.813	0.830	0.820	0.823	0.813	0.830	0.0
209B HEXACHLOROBENZENE-13C6	49	1.308	0.010	0.7	1.285	1.326	1.305	1.310	1.288	1.327	2.0
212B HEXACHLOROETHANE-1-13C	40	0.703	0.006	0.9	0.692	0.714	0.701	0.705	0.690	0.717	0.0
218B BIS(2-CHLOROETHYL)-D8 ETH	30	0.596	0.006	1.0	0.587	0.605	0.593	0.598	0.584	0.607	0.0
220B 2-CHLORONAPHTHALENE-D7	49	1.019	0.002	0.2	1.010	1.023	1.018	1.020	1.014	1.024	6.1
221A 2,4,6-TRICHLOROPHENOL-3,5	48	1.000	0.003	0.3	0.995	1.008	0.999	1.000	0.994	1.005	8.3
222A 4-CHLORO-3-METHYLPHENOL-2	46	0.937	0.003	0.3	0.932	0.945	0.936	0.938	0.930	0.943	6.5
224A 2-CHLOROPHENOL-3,4,5,6-D4	47	0.602	0.008	1.3	0.587	0.618	0.600	0.605	0.587	0.618	0.0
225B 1,2-DICHLOROBENZENE-D4	40	0.649	0.009	1.3	0.631	0.662	0.647	0.652	0.632	0.667	2.5
226B 1,3-DICHLOROBENZENE-D4	50	0.620	0.008	1.2	0.607	0.633	0.618	0.623	0.605	0.636	0.0
227B 1,4-DICHLOROBENZENE-D4	50	0.634	0.016	2.5	0.617	0.668	0.629	0.638	0.601	0.666	6.0
228B 3,3'-DICHLOROBENZIDINE-D6	49	1.796	0.025	1.4	1.770	1.848	1.789	1.803	1.744	1.848	2.0
231A 2,4-DICHLOROPHENOL-3,5,6-	48	0.812	0.005	0.6	0.800	0.823	0.811	0.814	0.802	0.822	4.2
234A 2,4-DIMETHYLPHENOL-3,5,6-	49	0.792	0.005	0.7	0.782	0.805	0.791	0.794	0.781	0.803	2.0
235B 2,4-DINITROTOLUENE-3,5,6-	40	1.166	0.007	0.6	1.150	1.179	1.164	1.169	1.152	1.181	2.5
236B 2,6-DINITROTOLUENE-D3	39	1.101	0.005	0.5	1.085	1.109	1.099	1.103	1.090	1.112	5.1
237B 1,2-DIPHENYL-D10-HYDRAZIN	49	1.232	0.008	0.6	1.216	1.247	1.230	1.234	1.216	1.248	2.0
239B FLUORANTHENE-D10	50	1.559	0.018	1.2	1.521	1.587	1.554	1.564	1.522	1.596	2.0
240B 4-CHLOROPHENYL PHENYL-D5	49	1.209	0.007	0.6	1.189	1.222	1.207	1.211	1.194	1.223	2.0
242B BIS(2-CHLOROISOPROPYL)ETH	50	0.678	0.007	1.0	0.665	0.688	0.676	0.679	0.664	0.691	0.0
252B HEXACHLORO-1,3-BUTADIENE-	49	0.864	0.004	0.4	0.857	0.871	0.863	0.865	0.856	0.871	2.0
253B HEXACHLOROCYCLOPENTADIENE	31	0.981	0.002	0.2	0.976	0.987	0.980	0.982	0.976	0.986	3.2
254B ISOPHORONE-D8	50	0.757	0.005	0.6	0.747	0.769	0.756	0.758	0.747	0.767	6.0
255B NAPHTHALENE-D8	50	0.828	0.004	0.5	0.820	0.837	0.827	0.829	0.819	0.836	2.0
256B NITROBENZENE-D5	20	0.716	0.005	0.7	0.710	0.723	0.714	0.719	0.706	0.727	0.0
257A 2-NITROPHENOL-3,4,5,6-D4	49	0.772	0.005	0.7	0.762	0.782	0.771	0.774	0.761	0.783	0.0
258A 4-NITROPHENOL-2,3,5,6-D4	48	1.161	0.007	0.6	1.152	1.176	1.159	1.163	1.147	1.175	2.1
259A 2,4-DINITROPHENOL-3,5,6-D	48	1.138	0.005	0.5	1.130	1.149	1.136	1.140	1.127	1.149	2.1
260A 4,6-DINITRO-O-CRESOL-D2	49	1.233	0.008	0.7	1.213	1.248	1.230	1.235	1.216	1.249	2.0
262B N-NITROSODIPHENYLAMINE-D6	30	1.239	0.007	0.5	1.229	1.251	1.236	1.241	1.225	1.252	0.0
264A PENTACHLOROPHENOL-13C6	49	1.341	0.011	0.8	1.317	1.362	1.338	1.344	1.320	1.363	2.0
265A PHENOL-2,3,4,5,6-D5	50	0.599	0.007	1.2	0.586	0.615	0.597	0.601	0.584	0.613	2.0
266B BIS(2-ETHYLHEXYL)PHTHALAT	49	1.826	0.027	1.5	1.797	1.880	1.818	1.834	1.771	1.880	2.0
268B DI-N-BUTYL PHTHALATE-D4	50	1.478	0.016	1.1	1.447	1.510	1.474	1.483	1.446	1.510	0.0
269B DI-N-OCTYL PHTHALATE-D4	48	1.924	0.028	1.5	1.895	1.983	1.916	1.932	1.867	1.982	4.2
270B DIETHYL PHTHALATE-3,4,5,6	49	1.213	0.008	0.7	1.194	1.228	1.211	1.215	1.197	1.229	2.0
271B DIMETHYL PHTHALATE-3,4,5,	48	1.092	0.005	0.4	1.076	1.100	1.091	1.094	1.083	1.102	2.1
272B BENZO(A)ANTHRACENE-D12	49	1.790	0.027	1.5	1.761	1.845	1.783	1.798	1.735	1.846	0.0
273B BENZO(A)PYRENE-D12	49	2.021	0.033	1.6	1.976	2.085	2.011	2.030	1.954	2.088	0.0
274B BENZO(B)FLUORANTHENE-D12	49	1.964	0.030	1.5	1.924	2.026	1.955	1.973	1.902	2.025	2.0
275B BENZO(K)FLUORANTHENE-D12	47	1.970	0.031	1.6	1.933	2.031	1.960	1.979	1.906	2.033	0.0
276B CHRYSENE-D12	49	1.790	0.023	1.3	1.767	1.837	1.783	1.796	1.743	1.837	2.0
277B ACENAPHTHYLENE-D8	50	1.087	0.004	0.3	1.074	1.093	1.086	1.088	1.080	1.095	4.0
278B ANTHRACENE-D10	49	1.365	0.011	0.8	1.349	1.387	1.362	1.369	1.342	1.388	0.0
279B BENZO(GHI)PERYLENE-D12	49	2.355	0.083	3.5	2.230	2.472	2.332	2.379	2.187	2.524	0.0
280B FLUORENE-D10	50	1.200	0.007	0.6	1.180	1.212	1.198	1.202	1.185	1.214	2.0
281B PHENANTHRENE-D10	50	1.357	0.011	0.8	1.332	1.379	1.353	1.360	1.334	1.380	2.0
284B PYRENE-D10	40	1.584	0.030	1.9	1.535	1.624	1.574	1.593	1.523	1.644	0.0
301B ACENAPHTHENE	40	1.004	0.002	0.2	0.995	1.007	1.003	1.005	0.999	1.009	5.0
305B BENZIDINE	30	1.001	0.000	0.0	1.000	1.002	1.000	1.001	1.000	1.002	0.0

Table VI-5 (Continued)

COMPOUND	N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
308B 1,2,4-TRICHLOROBENZENE	39	1.002	0.001	0.1	0.998	1.005	1.002	1.003	1.000	1.005	7.7
309B HEXACHLOROBENZENE	40	1.000	0.000	0.0	1.000	1.001	1.000	1.000	0.999	1.001	10.0
312B HEXACHLOROETHANE	28	1.000	0.000	0.0	1.000	1.001	1.000	1.000	1.000	1.000	3.6
318B BIS(2-CHLOROETHYL)ETHER	23	1.012	0.002	0.2	1.008	1.018	1.011	1.013	1.007	1.016	4.3
320B 2-CHLORONAPHTHALENE	31	1.002	0.002	0.2	0.994	1.007	1.001	1.003	0.997	1.007	3.2
321A 2,4,6-TRICHLOROPHENOL	37	1.001	0.002	0.2	0.995	1.003	1.000	1.002	0.998	1.004	5.4
322A P-CHLORO-M-CRESOL	35	1.000	0.001	0.1	0.998	1.003	1.000	1.001	0.998	1.003	11.4
324A 2-CHLOROPHENOL	39	1.004	0.003	0.3	0.992	1.007	1.002	1.005	0.997	1.010	7.7
325B 1,2-DICHLOROBENZENE	32	1.001	0.003	0.3	0.993	1.005	1.000	1.003	0.995	1.008	12.5
326B 1,3-DICHLOROBENZENE	40	1.003	0.002	0.2	0.994	1.008	1.002	1.004	0.998	1.008	7.5
327B 1,4-DICHLORODENZENE	40	1.003	0.003	0.3	0.993	1.006	1.002	1.004	0.997	1.009	10.0
328B 3,3'-DICHLOROBENZIDINE	39	1.001	0.000	0.0	1.000	1.001	1.000	1.001	1.000	1.001	5.1
331A 2,4-DICHLOROPHENOL	40	1.001	0.002	0.2	0.991	1.003	1.001	1.002	0.997	1.006	5.0
334A 2,4-DIMETHYLPHENOL	35	1.001	0.001	0.1	1.000	1.003	1.001	1.001	0.999	1.003	8.6
335B 2,4-DINITROTOLUENE	32	1.001	0.001	0.1	1.001	1.003	1.001	1.001	1.000	1.002	3.1
336B 2,6-DINITROTOLUENE	31	1.003	0.001	0.1	1.001	1.004	1.002	1.003	1.001	1.005	3.2
337B 1,2-DIPHENYLHYDRAZINE	40	1.004	0.002	0.2	0.994	1.009	1.003	1.005	0.999	1.009	2.5
339B FLUORANTHENE	40	1.002	0.001	0.1	0.998	1.004	1.002	1.002	1.000	1.004	5.0
340B 4-CHLOROPHENYL PHENYL ETH	40	1.002	0.006	0.6	0.998	1.040	1.001	1.004	0.990	1.015	2.5
342B BIS (2-CHLOROISOPROPYL) E	40	1.013	0.001	0.1	1.011	1.016	1.013	1.013	1.010	1.016	2.5
352B HEXACHLOROBUTADIENE	40	1.000	0.001	0.1	0.999	1.002	1.000	1.001	0.999	1.002	5.0
353B HEXACHLOROCYCLOPENTADIENE	30	1.000	0.000	0.0	1.000	1.002	1.000	1.000	0.999	1.001	3.3
354B ISOPHORONE	40	1.008	0.004	0.4	0.995	1.018	1.006	1.009	0.999	1.017	10.0
355B NAPHTHALENE	39	1.004	0.001	0.1	1.002	1.007	1.003	1.004	1.001	1.006	7.7
356B NITROBENZENE	16	1.004	0.001	0.1	1.002	1.006	1.004	1.005	1.002	1.007	0.0
357A 2-NITROPHENOL	40	1.001	0.004	0.4	0.992	1.005	1.000	1.002	0.994	1.009	12.5
358A 4-NITROPHENOL	38	1.001	0.002	0.2	0.997	1.010	1.001	1.002	0.997	1.006	5.3
359A 2,4-DINITROPHENOL	38	1.002	0.001	0.1	1.001	1.008	1.002	1.003	1.000	1.005	5.3
360A 4,6-DINITRO-O-CRESOL	36	1.001	0.000	0.0	1.001	1.001	1.001	1.001	1.000	1.002	0.0
362B N-NITROSODIPHENYLAMINE	16	1.001	0.000	0.0	1.001	1.002	1.001	1.001	1.000	1.002	6.3
364A PENTACHLOROPHENOL	40	1.000	0.001	0.1	0.995	1.001	1.000	1.000	0.998	1.002	2.5
365A PHENOL	40	1.003	0.004	0.4	0.990	1.010	1.001	1.004	0.995	1.010	7.5
366B BIS (2-ETHYLHEXYL) PHTHAL	36	1.001	0.000	0.0	1.000	1.002	1.000	1.001	1.000	1.002	5.6
368B DI-N-BUTYL PHTHALATE	39	1.001	0.001	0.1	1.001	1.003	1.001	1.002	1.000	1.003	0.0
369B DI-N-OCTYL PHTHALATE	36	1.001	0.000	0.0	1.000	1.001	1.001	1.001	1.000	1.002	0.0
370B DIETHYL PHTHALATE	40	1.001	0.002	0.2	0.996	1.010	1.000	1.002	0.996	1.006	10.0
371B DIMETHYL PHTHALATE	39	1.001	0.002	0.2	0.994	1.003	1.001	1.002	0.998	1.005	5.1
372B BENZO(A)ANTHRACENE	37	1.003	0.002	0.2	0.998	1.009	1.002	1.003	0.999	1.007	5.4
373B BENZO(A)PYRENE	38	1.002	0.001	0.1	0.998	1.004	1.002	1.002	1.000	1.004	2.6
374B BENZO(B)FLUORANTHENE	38	1.003	0.001	0.1	1.000	1.006	1.002	1.003	1.000	1.005	7.9
375B BENZO(K)FLUORANTHENE	38	1.002	0.001	0.1	0.999	1.005	1.001	1.002	1.000	1.004	10.5
376B CHRYSENE	37	1.002	0.001	0.1	1.001	1.006	1.002	1.002	1.000	1.004	5.4
377B ACENAPHTHYLENE	31	1.002	0.001	0.1	1.000	1.004	1.002	1.002	1.000	1.004	9.7
378B ANTHRACENE	39	1.002	0.002	0.2	0.996	1.004	1.001	1.002	0.998	1.006	10.3
379B BENZO(GHI)PERYLENE	36	1.004	0.001	0.1	1.000	1.006	1.003	1.004	1.001	1.006	5.6
380B FLUORENE	40	1.004	0.002	0.2	0.994	1.010	1.003	1.004	0.999	1.008	7.5
381B PHENANTHRENE	40	1.003	0.001	0.1	0.998	1.005	1.002	1.003	1.000	1.005	5.0
384B PYRENE	30	1.002	0.001	0.1	1.001	1.004	1.002	1.002	1.001	1.003	6.7
502B BETA NAPHTHYLAMINE	38	1.178	0.007	0.6	1.165	1.192	1.176	1.181	1.163	1.193	0.0
503B ALPHA PICOLINE	38	0.368	0.015	4.0	0.337	0.387	0.363	0.373	0.338	0.398	5.3
504B DIBENZOTHIOPHENE	31	1.342	0.011	0.8	1.326	1.361	1.338	1.346	1.319	1.364	0.0
505B DIBENZOFURAN	40	1.147	0.006	0.5	1.131	1.157	1.145	1.149	1.135	1.160	2.5

Table VI-5 (Concluded)

COMPOUND		N OF CASES MEASRD	MEAN	STANDARD DEVIATION	COEF OF VARN	MINIMUM	MAXIMUM	LOWER 95PCT CONF_LMT	UPPER 95PCT CONF_LMT	LOWER 95PCT PRED_LMT	UPPER 95PCT PRED_LMT	% OUT OF PRED_LMT
506B N-DODECANE		39	0.843	0.004	0.5	0.835	0.851	0.841	0.844	0.834	0.851	0.0
507B DIPHENYLAMINE		40	1.237	0.008	0.7	1.216	1.252	1.234	1.240	1.220	1.254	2.5
508B DIPHENYLETHER		39	1.045	0.003	0.3	1.035	1.049	1.044	1.046	1.038	1.052	7.7
509B ALPHA TERPINEOL		39	0.839	0.004	0.5	0.831	0.848	0.838	0.841	0.831	0.848	5.1
510B STYRENE		40	0.473	0.009	1.9	0.447	0.491	0.470	0.476	0.454	0.492	2.5
511B DI-N-BUTYL AMINE		30	0.641	0.052	8.2	0.584	0.714	0.621	0.660	0.532	0.749	0.0
512B BIPHENYL		32	1.025	0.002	0.2	1.022	1.030	1.025	1.026	1.022	1.029	9.4
513B P-CYMENE		38	0.649	0.007	1.0	0.637	0.660	0.646	0.651	0.635	0.662	0.0
517B N-DECANE	C10	39	0.619	0.008	1.2	0.605	0.632	0.616	0.621	0.603	0.634	0.0
519B N-HEXADECANE	C16	39	1.265	0.115	9.1	1.198	1.493	1.228	1.302	1.029	1.502	0.0
521B N-EICOSANE	C20	39	1.440	0.121	8.4	1.201	1.529	1.401	1.479	1.192	1.688	0.0
523B N-TETRACOSANE	C24	39	1.740	0.025	1.4	1.696	1.790	1.732	1.748	1.689	1.790	0.0
526B N-TRIACONTANE	C30	39	2.091	0.044	2.1	2.042	2.160	2.076	2.105	2.001	2.180	0.0
602B 2-NAPHTHYL-D7-AMINE		48	1.176	0.007	0.6	1.158	1.188	1.174	1.178	1.163	1.189	2.1
603B 2-METHYLPYRIDINE-D7		49	0.359	0.016	4.6	0.312	0.384	0.354	0.364	0.326	0.393	4.1
604B DIBENZOTHIOPHENE-D8		39	1.337	0.011	0.8	1.312	1.357	1.334	1.341	1.314	1.361	2.6
605B DIBENZOFURAN-D8		49	1.145	0.005	0.5	1.134	1.154	1.143	1.146	1.134	1.155	2.0
606B N-DODECANE-D26		50	0.819	0.044	5.4	0.614	0.878	0.806	0.831	0.730	0.908	4.0
607B DIPHENYL-D10-AMINE		40	1.231	0.009	0.7	1.211	1.246	1.228	1.234	1.213	1.249	2.5
608B DIPHENYL-D10 ETHER		50	1.042	0.003	0.2	1.031	1.046	1.041	1.042	1.036	1.047	4.0
609B ALPHA-TERPINEOL-D3		50	0.836	0.004	0.4	0.828	0.844	0.835	0.837	0.829	0.844	2.0
610B STYRENE-2,3,4,5,6-D5		49	0.469	0.009	2.0	0.443	0.488	0.466	0.472	0.450	0.488	6.1
611B DI-N-BUTYL-D18-AMINE		20	0.653	0.060	9.2	0.591	0.720	0.625	0.681	0.524	0.782	0.0
612B DIPHENYL-D10		30	1.021	0.003	0.2	1.013	1.026	1.020	1.022	1.016	1.027	6.7
613B P-CYMENE-D14		50	0.638	0.007	1.1	0.626	0.651	0.636	0.640	0.624	0.652	0.0
617B N-DECANE-D22		50	0.600	0.007	1.2	0.588	0.617	0.598	0.602	0.585	0.615	2.0
619B N-HEXADECANE-D34		49	1.244	0.115	9.2	1.167	1.473	1.211	1.277	1.010	1.478	0.0
621B N-EICOSANE-D42		48	1.423	0.117	8.2	1.175	1.506	1.389	1.457	1.184	1.662	12.5
623B N-TETRACOSANE-D50		49	1.717	0.023	1.3	1.696	1.763	1.711	1.724	1.671	1.764	0.0
626B N-TRIACONTANE-D62		49	2.050	0.038	1.9	1.998	2.116	2.039	2.060	1.972	2.127	0.0
702B BETA NAPHTHYLAMINE		39	1.001	0.003	0.3	0.994	1.005	1.001	1.002	0.996	1.007	10.3
703B ALPHA PICOLINE		39	1.017	0.006	0.5	1.007	1.033	1.015	1.019	1.006	1.028	7.7
704B DIBENZOTHIOPHENE		32	1.003	0.001	0.1	1.001	1.010	1.002	1.003	1.000	1.006	3.1
705B DIBENZOFURAN		40	1.003	0.002	0.2	0.995	1.005	1.002	1.003	0.998	1.007	7.5
706B N-DODECANE	C12	40	1.018	0.016	1.6	0.963	1.026	1.013	1.023	0.986	1.051	7.5
707B DIPHENYLAMINE		32	1.004	0.002	0.2	0.997	1.006	1.003	1.004	1.000	1.007	3.1
708B DIPHENYLETHER		40	1.003	0.003	0.3	0.993	1.005	1.002	1.004	0.997	1.009	10.0
709B ALPHA TERPINEOL		39	1.003	0.003	0.3	0.998	1.016	1.002	1.004	0.998	1.008	5.1
710B STYRENE		37	1.005	0.002	0.2	1.002	1.009	1.005	1.006	1.002	1.009	10.8
711B DI-N-BUTYL AMINE		16	1.005	0.013	1.3	0.980	1.021	0.999	1.012	0.978	1.033	0.0
712B BIPHENYL		23	1.003	0.001	0.1	1.002	1.009	1.003	1.004	1.001	1.006	4.3
713B P-CYMENE		40	1.015	0.004	0.4	0.993	1.018	1.014	1.016	1.008	1.023	2.5
717B N-DECANE	C10	40	1.030	0.004	0.4	1.021	1.045	1.029	1.031	1.022	1.038	7.5
719B N-HEXADECANE	C16	40	1.016	0.002	0.2	1.011	1.019	1.016	1.017	1.013	1.020	2.5
721B N-EICOSANE	C20	40	1.016	0.003	0.3	1.007	1.023	1.015	1.016	1.010	1.021	10.0
723B N-TETRACOSANE	C24	38	1.013	0.001	0.1	1.011	1.015	1.013	1.014	1.012	1.015	5.3
726B N-TRIACONTANE	C30	37	1.019	0.004	0.4	1.012	1.026	1.018	1.021	1.011	1.028	0.0

VII CONCLUSIONS AND FURTHER WORK

From this study, the conclusion can be drawn that isotope dilution GCMS is the most accurate and precise analytical technique available for the analysis of semi-volatile organic compounds in environmental samples. On average, the precision of analysis is improved by approximately a factor of two, and the accuracy is improved by approximately a factor of three over internal standard techniques.

The interlaboratory validation of Method 1625A represents the most comprehensive evaluation of an isotope dilution GCMS method to date. More than 250,000 pieces of information were collected and evaluated. As a result, the specifications developed should well represent the performance of the method in water and wastewater laboratories. For the most part, the method was tested in laboratories with little or no experience with isotope dilution; the fact that nearly all of these laboratories performed the method with reasonable accuracy and precison is a tribute to the laboratories and to the isotope dilution technique. The current draft of Method 1625B is given in Appendix M of this report, including the limits generated in this study. In a few cases, the limits obtained from the statistical analysis were not found to be practically useful, and no limit was set in those cases.

The limits set in Revision B of Method 1625 reflect all sources of variability, including variability attributable to the number of compounds being tested simultaneously. The specifications are realistic and reflect a 95 percent confidence limit for all tests. With reasonable care, any laboratory practicing these methods should be able to meet every specification in the Method.

A flaw in the study design which should be avoided in subsequent studies was the lack of replicate analyses of the pollutants in water samples. The derivation of quality control limits requires both an interlaboratory component and an intralaboratory component. In this study, the relationship between these components was inferred from labeled compounds data, and assumed to be identical for the pollutants. Although this inference is true within the limits of the measurement technique, a direct measurement of these components would have been more correct.

If funding and time permit, the data collected in this study may be further analyzed to determine if alternate statistical techniques are more appropriate to generation of specifications for retention time, calibration linearity, and other parameters. The response ratio data from the PRR sample could be analyzed to evaluate the use of standardized calibration curves in place of individual laboratory calibrations. The mass spectral data collected need analysis to determine specifications for compound identification, and spectral data from the analysis of decafluorotriphenlyphosphine can be used to determine an interlaboratory spectrum for this reference compound.

Appendix A

31 March 1983 Draft

Method 1625 Revision A
Semivolatile Organic Compounds by Isotope Dilution GCMS

1 Scope and application--this method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GCMS).

1.1 The chemical compounds listed in tables 1 and 2 may be determined in municipal and industrial discharges by this method. The method is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136.1. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.2 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits listed in tables 3 and 4 represent the minimum quantity that can be detected with no interferences present.

1.3 The GCMS portions of this method are for use only by anlysts experienced with GCMS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GCMS should run the performance tests in reference 1 before beginning.

2 Summary of method

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a one liter wastewater sample. The sample is extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques. The extract is dried over sodium sulfate and concentrated to a volume of one mL. An internal standard is added to the extract, and the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by mass spectrometry. The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GCMS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal or external standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the extraction and GCMS systems.

3 Contamination and interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given 8 hr shift, to a maximum of 20). Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450°C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being sampled.

4 Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 2-4.

4.2 The following compounds covered by this method have been tentatively classified as know or suspected human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and β-naphthylamine. Primary standards of these toxic compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5 Apparatus and materials

5.1 Sampling equipment for discrete or composite sampling

5.1.1 Sample bottle, amber glass, 1.1 liters minimum. If amber bottles are not available, samples shall be protected from light. Bottles are detergent water washed, then solvent rinsed or baked at 450°C for one hour minimum before use.

5.1.2 Bottle caps--threaded to fit sample bottles. Caps are lined with Teflon. Aluminum foil may be substituted if the sample is not corrosive.

5.1.3 Compositing equipment--automatic or manual compositing system incorporating glass containers for collection of a minimum 1.1 liters. Sample containers are kept at 0 to 4°C during sampling. Glass or Teflon tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing is thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (6.5) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Continuous liquid-liquid extractor--Teflon or glass connecting joints and stopcocks without lubrication (Hershberg-Wolf Extractor) one liter capacity, Ace Glass 6841-10 or equivalent.

5.3 Drying column--15 to 20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.4 Kuderna-Danish (K-D) apparatus

5.4.1 Concentrator tube--10 mL, graduated (Kontes K-570050-1025 or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.4.2 Evaporative flask--500 mL (Kontes K-570001-0500 or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012).

5.4.3 Snyder column--three-ball macro (Kontes K-503000-0232 or equivalent).

5.4.4 Snyder column--two ball micro (Kontes K-469002-0219 or equivalent).

5.4.5 Boiling chips--approx 10/40 mesh, extracted with methylene chloride and baked at 450°C for one hr minimum.

5.5 Water bath--heated, with concentric ring cover, capable of temperature control (± 2°C), installed in a fume hood.

5.6 Sample vials--amber glass, 2-5 mL with Teflon-lined screw cap.

5.7 Analytical balance--capable of weighing 0.1 mg.

5.8 Gas chromatograph--shall have splitless or on-column injection port; temperature program with 30°C hold; and shall meet all of the performance specifications in section 12.

5.8.1 Column--30 ± 5 m x 0.25 ± 0.02 mm i.d. 5 % phenyl, 95 % methyl silicone bonded phase fused silica capillary column (J & W DB-5 or equivalent).

5.9 Mass spectrometer--electron impact ionization, shall repetitively scan from 35 to 450 amu in one second or less, and shall produce a 70 eV, unit resolution (valleys between m/z 441-443 less than 10 percent of the height of the 441 peak), background corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in table 5 (reference 5). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams. All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.10 Data system--shall collect and record MS data, store mass-intensity data in spectral libraries, process GCMS data, generate reports, and shall calculate and record response factors.

5.10.1 Data acquisition--mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.10.2 Mass spectral libraries--user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GCMS runs for the compounds of interest (7.3).

5.10.3 Data processing--the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GCMS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.10.4 Response factors and multipoint calibrations--the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multi-point calibration curves (section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial and on-going performance shall be computed and maintained (7.8 and 12.8).

6 Reagents and standards

6.1 Sodium hydroxide--reagent grade, 6N in reagent water.

6.2 Sulfuric acid--reagent grade, 6N in reagent water.

6.3 Sodium sulfate--reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g) and conditioned at 450°C for one hour minimum.

6.5 Methylene chloride--distilled in glass (Burdick and Jackson or equivalent).

6.5 Reagent water--water in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard solutions--purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to calculate the concentration of the standard. When not being used, all standards are stored in the dark at -20 to -10°C in screw capped vials with Teflon-lined lids. A mark is placed on the vial at the level of the solution so that

any solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of stock solutions--prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in section 4. The large number of labeled and unlabeled acid, base/neutral, and Appendix C compounds used for combined calibration (section 7) and calibration verification (12.5) require high concentrations (approx 40 µg/mL) when individual stock solutions are prepared so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10-200 µg/mL. Compounds with a reduced MS response are prepared at higher concentration.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 40 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL Teflon-lined vial.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Quality control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check samples indicate a change in concentration.

6.8 Labeled compound spiking solution--from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 µg/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary standard--using stock solutions (6.7), prepare a secondary standard containing all of the compounds in tables 1 and 2 at a concentration of 400 µg/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal standard solution--prepare 2,2'-difluorobiphenyl at a concentration of 10 mg/mL in benzene.
6.11 DFTPP solution--prepare at 50 µg/mL in acetone.
6.12 Solutions for obtaining authentic mass spectra (7.3)--prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries.
6.13 Calibration solutions--combine 0.5 mL of the solution in 6.8 with 25, 50, 125, 250, and 500 µL of the solution in 6.9 and bring to 1.00 mL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100 and 200 µg/mL of the pollutants and a constant nominal 100 µg/mL of the labeled compounds. Spike each solution with 10 µL of the internal standard solution (6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (7.5).
6.14 Performance standard--used for initial (7.10) and on-going (12.8) performance verifications. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 µg/mL.
6.15 Stability of solutions--all standard solutions (6.8-6.13) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within ± 15 percent of the area obtained in the initial analysis of the standard.

7 Calibration
7.1 Using the procedure in section 10, extract and concentrate four 1.0 liter aliquots of reagent water containing one mL each of the performance standard (6.14), and extract and concentrate a one liter aliquot of reagent water containing 0.5 mL of the labeled compound spiking solution (6.8).
7.2 Assemble the GCMS and establish the operating conditions in table 3. Analyze standards per the procedure in section 11 to demonstrate that the analytical system meets the detection limits in tables 3 and 4, and the mass-intensity criteria in table 5 for 50 ng DFTPP.

7.3 Mass spectral libraries--detection and identification of compcunds of interest are dependent upon the spectra stored in user created libraries.

7.3.1 Obtain a mass spectrum of each labeled and unlabeled compound and of the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.3.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.3.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (7.2 and table 5) to normalize it to spectra from other instruments.

7.3.4 The spectrum is edited by saving the 5 most intense mass peaks and all other peaks greater than 10 percent of the base peak. This edited spectrum is stored for reverse search and for compound confirmation.

7.4 Demonstrate that the 20 ng anthracene-d_{10} or phenanthrene-d_{10} produces an area at m/z 188 approx one-tenth that required to exceed the linear range of the system. For a typical instrument, an area of 20,000 to 50,000 is appropriate. The exact value must be determined by experience for each instrument.

7.5 Calibration with isotope dilution--isotope dilution is used when 1) labeled compounds are available, 2) interferences do not preclude its use, and 3) the quantitation mass extracted ion current profile (EICP) area for the compound is in the calibration range. If any of these conditions preclude isotope dilution, internal or external standard methods (7.6 or 7.7) are used.

7.5.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response vs weight ratio (labeled to unlabeled) of the compound

in standard solutions is computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d_5 as the isotopic diluent. Also shown are the ±10 percent error limits (dotted lines). Relative Response (RR) is determined according to the procedures described below. Five data points are employed for calibration.

7.5.2 The relative response of an unlabeled compound to its labeled analog is determined from isotope ratio values calculated from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured for the pure unlabeled compound

R_y = the isotope ratio measured for the labeled compound

R_m = the isotope ratio of an analytical mixture of unlabeled and labeled compounds

The m/z's are selected such that $R_x > R_y$. If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by internal or external standard methods.

7.5.3 Capillary columns usually separate the labeled-unlabeled pair, with the labeled compound eluted first (figure 2). For this case,

R_x = area m_1/z, at the retention time of the unlabeled compound (RT_2)

$R_y = \frac{1}{\text{area } m_2/z}$, at the retention time of the labeled compound (RT_1)

$R_m = \frac{\text{area at } m_1/z \text{ (at } RT_2)}{\text{area at } m_2/z \text{ (at } RT_1)}$, as measured in the mixture of the labeled and unlabeled compounds (figure 2)

and $RR = R_m$.

7.4.4 Special precautions are taken when the labeled-unlabeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the unlabeled compound (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the labeled and unlabeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system

or operator to remove the contributions of the compounds to each other, the equations in 7.5.3 apply. This usually occurs when the height of the valley between the two GC peaks at the the same m/z is less then 10 percent of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is calculated using the following equation:

$$RR = \frac{(R_y - R_m)(R_x + 1)}{(R_m - R_x)(R_y + 1)}$$

R_x is measured as shown in figure 3A

R_y is measured as shown in figure 3B

R_m is measured as shown in figure 3c

For the example, $R_x = \frac{46100}{4780} = 9.64$

$R_y = \frac{2650}{43600} = 0.0608$

$R_m = \frac{49200}{48300} = 1.019$

$RR = 1.107$

7.5.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 µL aliquot of each of the calibration standards (6.13) using the procedure in section 11.

7.5.6 Linearity--if the ratio of relative response to concentration for any compound agrees within 5 percent relative standard deviation over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, a complete calibration curve shall be used for that compound.

7.6 Internal standard calibration--used when criteria for isotope dilution (7.5) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2'-difluorobiphenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (7.10 and 12.8).

7.6.1 Response factors--calibration requires the determination of a response factor (RF) which is defined by the following equation:

$$RF = (A_s C_{is})/(A_{is} C_s)$$

A_s is the area of the characteristic mass for the compound in the daily standard

A_{is} is the area of the characteristic mass for the internal standard

C_{is} is the concentration of the internal standard (μg/mL)

C_s is the concentration of the compound in the daily standard (μg/mL)

7.6.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (6.13); nominally, 10, 20, 50, 100, and 200 μg/mL. The amount of internal standard added to each extract is the same (100 μg/mL) so that C_{is} remains constant. The RF is plotted vs concentration for each compound in the standard (C_s) to produce a calibration curve.

7.6.1.2 Linearity--if the response factor (RF) for any compound agrees within 10 percent relative standard deviation over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.7 External standard calibration--used when interferences preclude use of the isotope dilution and internal standard methods. A master calibration curve is prepared by analyzing a minimum of five concentrations of standards (6.13). Concentration vs peak area is plotted for each compound.

7.7.1 Linearity--if the ratio of response to concentration for any compound agrees within 10 percent relative standard deviation over the 5 point calibration range, an averaged response to concentration ratio may be used for that compound otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.8 Combined calibration--by using calibration solutions containing the labeled and unlabeled compounds and the internal standard (6.13), a single set of analyses can be used to produce calibration curves for the isotope dilution, internal standard, and external standard methods. These curves are

verified each shift (12.5) by analyzing the performance standard (6.14). Recalibration is required only if calibration (12.5) and on-going performance (12.8) criteria cannot be met.

7.9 Polar compound detection--unlabeled benzidine and pentachlorophenol shall be detectable at the 50 µg/mL level (per all criteria in section 13). The 50 µg/mL calibration standard (6.13) can be used to demonstrate this performance.

7.10 Initial intra-laboratory precision and accuracy--as a final step in the calibration procedure, the laboratory shall demonstrate the ability to perform replicate analyses of the compounds to be determined by this method within limits considered normal for these analyses using reagent water as the matrix.

7.10.1 Analyze the four extracts of standards, and the extract of the blank (7.1), adding 10 µL of the internal standard solution (6.10) immediately prior to injection, using the procedure in section 11. Compute the concentration of the unlabeled compounds (tables 1 and 2) by isotope dilution for those compounds which have labeled analogs. Compute the concentration of the unlabeled compounds which have no labeled analogs, and of the labeled compounds, by the internal standard method (7.6). Compute the average percent recovery and the relative standard deviation (coefficient of variation) of percent recovery for all compounds. The average percent recovery shall be 85-115 and the relative standard deviation shall be less than 10 for all compounds by isotope dilution, and the average percent recovery shall be 50-130 and the relative standard deviation shall be less than 35 for all compounds measured by the internal standard method; otherwise, the system variables need to be better controlled and the test repeated until these specifications are met.

8 Quality assurance/quality control (QA/QC)

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. Minimum program requirements consist of an initial demonstration of laboratory performance and analysis of standards and blanks as tests of continued

performance. Specific QA/QC can vary depending on program requirements, but the principles remain the same. Quality is controlled, in part, by restricting the allowable range of a given variable (e.g., GC column temperature) to the limits shown to yield the reproducibility required. Quality is assured by comparing results of analysis of blanks and standards to specifications based on known inter- and intra-laboratory variability for analysis of the compounds of interest or of similar compounds (reference 6).

8.1.1 Intra-laboratory variability of this method is measured using results of four initial analyses of standards in reagent water (7.10.1), and updated with every sample lot. Control limits of ± 3 standard deviations from cumulative data determine acceptable performance. Figure 4 shows an example of such a quality control chart. The laboratory shall maintain these charts to demonstrate the ability to perform acceptable analyses.

8.1.2 Matrix effects are evaluated by comparing the results of analyses of labeled compounds in reagent water to results of analyses of the compounds in samples. Differences in recoveries are attributed to the sample matrix.

8.2 Blanks--before processing samples, a reagent water blank shall be analyzed to demonstrate that the analytical system is interference free. With each sample lot, a blank shall be analyzed to demonstrate freedom from contamination.

8.3 Documentation--laboratory activities shall be documented in log books or on magnetic media. Sample logs connect samples and results; instrument logs record changes which may alter instrument performance; standards logs document preparation and traceability of analytical standards; extraction logs record times, rates, and volumes; QA/QC logs monitor ongoing laboratory performance.

8.4 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The GCMS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analysis of semivolatiles

by this method.

8.5 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

9 Sample collection, preservation, and handling

9.1 Collect samples in glass containers following conventional sampling practices (reference 7). Composite samples are collected in refrigerated glass containers (5.1.3) in accordance with the requirements of the sampling program.

9.2 Maintain samples at 0-4°C from the time of collection until extraction. If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA methods 330.4 and 330.5 may be used to measure residual chlorine (reference 8).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

10 Sample extraction and concentration

10.1 Labeled compound spiking--measure 1.00 ± 0.01 liter of sample into a glass container. For untreated effluents, and samples which are expected to be difficult to extract and/or concentrate, measure an additional 10.0 ± 0.1 mL and dilute to a final volume of 1.00 ± 0.01 liter with reagent water in a glass container.

10.1.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place two 1.00 ± 0.01 liter aliquots of reagent water in glass containers.

10.1.2 Spike 0.5 mL of the labeled compound spiking solution (6.8) into all samples and one reagent water aliquot.

10.1.3 Spike 1.0 mL of the performance standard (6.14) into the remaining reagent water aliquot.

10.1.4 Stir and equilibrate all solutions for 1-2 hr.

10.2 Base/neutral extraction--place 100-150 mL methylene chloride in each continuous extractor and 200-300 in each distilling flask.

10.2.1 Pour the sample(s), blank, and standard aliquots into the extractors. Rinse the glass containers with 50-100 mL

methylene chloride and add to the respective extractor.
10.2.2 Adjust the pH of the waters in the extractors to 12-13 with 6N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1-2 drops of methylene chloride per second will fall from the condenser tip into the water. After 1-2 hours of extraction, test the pH and readjust to 12-13 if required. Extract for 18-24 hours.
10.2.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm anhydrous sodium sulfate. Rinse the distilling flask with 30-50 mL methylene chloride and pour through the drying column. Collect the solution in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction and concentrate per sections 10.4 to 10.6.
10.3 Acid extraction--adjust the pH of the waters in the extractors to 2 or less using 6N sulfuric acid. Charge clean distilling flasks with 300-400 mL methylene chloride. Test and adjust the pH of the waters after the first 1-2 hr of extraction. Extract for 18-24 hours.
10.3.1 Repeat 10.2.3, except label as the acid fraction.
10.4 Concentration--concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes.
10.4.1 Add 1 to 2 clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approx 1 mL methylene chloride through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL methylene chloride. A 5 mL syringe is recommended for this

operation.

10.4.2 For performance standards (7.1 and 12.8) and for blanks (7.1 and 12.7), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for samples.

10.5 Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx 0.2 mL methylene chloride. Adjust the final volume to 1.0 mL.

10.6 Transfer the concentrated extract to a clean screw cap vial. Seal the vial with a Teflon-lined septum, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at -20 to -10°C until ready for analysis.

11 GCMS analysis

11.1 Establish the operating conditions given in tables 3 or 4 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (10.4.2), use the operating conditions in table 3.

11.2 Bring the concentrated extract (10.6) or performance standard (6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (10.6) and bring to the mark with solvent if required.

11.3 Add 10 µL of the internal standard solution (6.10) to the extract immediately prior to analysis to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject 1.0 µL of the standard solution or extract using on-column or splitless injection. Start the GC column initial

isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo-(ghi)perylene or pentachlorophenol peak elutes for the base/neutral or acid fraction, respectively. Return the column to the initial temperature for analysis of the next sample.

12 System performance

12.1 At the beginning of each 8 hr shift during which analyses are performed, system performance and calibration shall be verified. For these tests, analysis of the performance standard (6.14) shall be used to verify all performance criteria with a single analysis. Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples and blanks be analyzed.

12.2 DFTPP spectrum validity--inject 1 µL of the DFTPP solution (6.11) either separately or within a few seconds of injection of the standard analyzed at the beginning of each shift. The criteria in table 5 shall be met.

12.3 Early and late eluted components

12.3.1 Base/nuetral--*N*-nitrosodimethylamine shall be sufficiently resolved from the solvent peak to permit detection, and benzo(ghi)-perylene shall give a mass spectrum which permits detection, both per all criteria in section 13. The retention time of benzo(ghi)-perylene shall be 40-45 minutes.

12.3.2 Acid--phenol shall be sufficiently resolved from the solvent peak to permit detection, and pentachlorophenol shall give a mass spectrum which permits detection, both per all criteria in section 13. The retention time of pentachlorophenol shall be 20-25 minutes.

12.4 GC resolution--the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks.

12.5 Verification of calibration--the response ratios of the labeled/unlabeled pairs shall be within ± 10 percent of their respective points on the original calibration curves (7.5.5). The response factors for the labeled compounds and for the unlabeled compounds having no labeled analog shall be within ± 20 percent of their respective points on the original calibration curves (7.2.1).

12.6 Multiple peaks--each component injected shall give a single, distinct GC peak.

12.7 Laboratory blanks--if any compound of interest (table 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 µg/L (assuming a response factor of 1 relative to the internal standard for compounds not listed in tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

12.8 On-going intra-laboratory precision and accuracy

12.8.1 Analyze the extracted performance standard (10.1.3) prior to analysis of samples from the same lot.

12.8.2 The percent recovery for the unlabeled/labeled pairs shall be 80-120 by isotope dilution. The percent recovery for the labeled compounds and the unlabeled compounds having no labeled analog shall be 40-130 percent by the internal standard method. The result for each compound, measured and recorded as in section 7.8, shall be within ± 3 standard deviations of the result for initial (7.8) and previous on-going data.

12.8.3 Add results which pass the specification in 12.8.2 to initial and previous on-going data. Update QC charts to form a graphic representation of continued laboratory performance (Figure 4).

13 Qualititative determination--accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (12.1). Identification is confirmed when spectra and retention times agree per the following criteria:

13.1 Labeled compounds and unlabeled compounds having no labeled analog:

13.1.1 The signals for all characteristic masses stored in the spectral library (7.3.4) shall be present and shall maximize within the same two consecutive scans.

13.1.2 Either 1) the background corrected EICP areas, or 2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the library.

13.1.3 The relative retention time of the compound shall agree within ± 15 scans or ± 15 seconds (whichever is greater) of the relative retention time in the shift standard (12.1).

13.2 Unlabeled compounds having a labeled analog:

13.2.1 The signals for all characteristic masses stored in the spectral library (7.3.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either 1) the background corrected EICP areas, or 2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the library.

13.2.3 The retention time difference between the labeled/unlabeled pair shall agree within ± 2 scans or ± 2 seconds (whichever is greater) of this difference in the shift standard (12.1).

13.3 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, an experienced spectrometrist is to determine the presence or absence of the compound.

14 Quantitative Determination

14.1 Isotope dilution--by adding a known amount of each labeled compound to every sample prior to extraction, automatic correction for component recovery is accomplished. This is because the unlabeled compound and its labeled analog exhibit the same effects during extraction, concentration, and gas chromatography. Relative response (RR) values for the sample mixtures are used in conjunction with calibration curves (7.5) to determine concentrations directly, so long as the labeled compound spiking levels are constant. For the phenol example given in figure 1, RR would be equal to 1.180. For this RR value, the calibration curve given in figure 1 indicates a concentration of 108 µg/L.

14.2 Internal standard--by adding a constant known amount of internal standard (C_{is}) to every sample extract, the concentration of pollutant (C_o) in the sample is calculated using the

following equation:

$C_o = (A_s C_{is})/(A_{is} V_o RF)$

where V_o is the volume of the original sample in liters and the other terms are as defined in section 7.6.1.

14.3 External standard--compute the concentration from the calibration curve or response/concentration ratio determined from calibration data in section 7.7.

14.4 If the EICP area at the quantitation mass for any compound exceeds the calibration range of the system, the dilute aliquot (10.1) extract is analyzed by isotope dilution; otherwise, the extract is diluted by a factor of 10, 9 µL of internal standard solution (6.10) is added to a 1.0 mL aliquot, and this diluted extract is analyzed by the internal standard method (14.2). Quantify each compound at the highest concentration level within the calibration range.

14.5 Report results for all labeled and unlabeled compounds (tables 1 and 2) found in all blanks, standards, and samples in µg/L to three significant figures.

15 Analysis of complex samples

15.1 Untreated effluents and other samples frequently contain high levels (>1000 µg/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to one mL (10.5); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (10.1) when the sample will not concentrate to 1.0 mL.

15.3 Recovery of internal standard--the EICP area of the internal standard should be within a factor of two of the area in the shift standard (12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred. In this case, use one of the labeled compounds (preferably a polynuclear aromatic hydrocarbon) to compute the concentration of an unlabeled compound with no labeled analog.

15.4 Recovery of labeled compounds--in most samples, labeled compound recoveries should be similar to those from reagent water (12.8). If the recovery of any labeled compound is less than 10 percent of the average, on-going recovery (12.8), the dilute extract (10.1) is analyzed by isotope dilution; otherwise, the extract is diluted and analyzed as in section 14.4. If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the performance standard (12.1) shall be analyzed and calibration verified (12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the extract is handled as in section 14.4.

16 Method performance

16.1 Preliminary method performance data can be found in reference 9.

References

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2. "Working with Carcinogens," DHEW, PHS, CDC, NIOSH, Publication 77-206, (Aug 1977).

3. "OSHA Safety and Health Standards, General Industry" OSHA 2206, 29 CFR 1910 (Jan 1976).

4. "Safety in Acedemic Chemistry Laboratories," ACS Committee on Chemical Safety (1979).

5. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems," J.W. Eichelberger, L.E. Harris, and W.L. Budde, Anal. Chem., 47, 995 (1975).

6. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL, Cincinnati, OH 45268, EPA-600/4-79-019 (March 1979).

7. "Standard Practice for Sampling Water," ASTM Annual Book of Standards, ASTM, Philadelphia, PA, 76 (1980).

8. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL, Cincinnati, OH 45268, EPA 600/4-70-020 (March 1979).

9. Colby, B. N., Beimer, R. G., Rushneck, D. R., and Telliard, W. A., "Isotope Dilution Gas Chromatography-Mass Spectrometry for the Determination of Priority Pollutants in Industrial Effluents." USEPA, Effluent Guidelines Division, Washington DC 20460 (1980).

Table 1

Base/neutral Extractable Compounds

Compound	Storet	CAS Registry	EPA-EGD	NPDES
acenaphthene	34205	83-32-9	001 B	001 B
acenaphthylene	34200	208-96-8	077 B	002 B
anthracene	34220	120-12-7	078 B	003 B
benzidine	39120	92-87-5	005 B	004 B
benzo(a)anthracene	34526	56-55-3	072 B	005 B
benzo(b)fluoranthene	34230	205-99-2	074 B	007 B
benzo(k)fluoranthene	34242	207-08-9	075 B	009 B
benzo(a)pyrene	34247	50-32-8	073 B	006 B
benzo(ghi)perylene	34521	191-24-2	079 B	008 B
biphenyl (Appendix C)	81513	92-52-4	512 B	
bis(2-chloroethyl) ether	34273	111-44-4	018 B	011 B
bis(2-chloroethoxy)methane	34278	111-91-1	043 B	010 B
bis(2-chloroisopropyl) ether	34283	108-60-1	042 B	012 B
bis(2-ethylhexyl) phthalate	39100	117-81-7	066 B	013 B
4-bromophenyl phenyl ether	34636	101-55-3	041 B	014 B
butyl benzyl phthalate	34292	85-68-7	067 B	015 B
n-C10 (Appendix C)	77427	124-18-5	517 B	
n-C12 "	77588	112-40-3	506 B	
n-C14 "	77691	629-59-4	518 B	
n-C16 "	77757	544-76-3	516 B	
n-C18 "	77804	593-45-3	520 B	
n-C20 "	77830	112-95-8	521 B	
n-C22 "	77859	629-97-0	522 B	
n-C24 "	77886	646-31-1	523 B	
n-C26 "	77901	630-01-3	524 B	
n-C28 "		630-02-4	525 B	
n-C30 "		638-68-6	526 B	
2-chloronaphthalene	34581	91-58-7	020 B	016 B
4-chlorophenyl phenyl ether	34641	7005-72-3	040 B	017 B
chrysene	34320	218-01-9	076 B	018 B
p-cymene (Appendix C)	77356	99-87-6	513 B	
dibenzo(a,h)anthracene	34556	53-70-3	082 B	019 B
dibenzofuran (Appendix C)	81302	132-64-9	505 B	
dibenzothiophene (Synfuel)	77639	132-65-0	504 B	
di-n-butylamine (Appendix C)	77300	111-92-2	511 B	
di-n-butyl phthalate	39110	84-74-2	068 B	026 B
1,2-dichlorobenzene	34536	95-50-1	025 B	020 B
1,3-dichlorobenzene	34566	541-73-1	026 B	021 B
1,4-dichlorobenzene	34571	106-46-7	027 B	022 B
3,3'-dichlorobenzidine	34631	91-94-1	028 B	023 B
diethyl phthalate	34336	84-66-2	070 B	024 B
2,4-dimethylphenol	34606	105-67-9	034 A	003 A
dimethyl phthalate	34341	131-11-3	071 B	025 B
2,4-dinitrotoluene	34611	121-14-2	035 B	027 B
2,6-dinitrotoluene	34626	606-20-2	036 B	028 B
dioctyl phthalate	34596	117-84-0	069 B	029 B
diphenylamine (Appendix C)	77579	122-39-4	507 B	
diphenyl ether (Appendix C)	77587	101-84-8	508 B	

Table 1 (continued)

Compound	Storet	CAS Registry	EPA-EGD	NPDES
1,2-diphenylhydrazine	34346	122-66-7	037 B	030 B
fluoranthene	34376	206-44-0	039 B	031 B
fluorene	34381	86-73-7	080 B	032 B
hexachlorobenzene	39700	118-74-1	009 B	033 B
hexachlorobutadiene	34391	87-68-3	052 B	034 B
hexachloroethane	34396	67-72-1	012 B	036 B
hexachlorocyclopentadiene	34386	77-47-4	053 B	035 B
indeno(1,2,3-cd)pyrene	34403	193-39-5	083 B	037 B
isophorone	34408	78-59-1	054 B	038 B
naphthalene	34696	91-20-3	055 B	039 B
β-naphthylamine (Appendix C)	82553	91-59-8	502 B	
nitrobenzene	34447	98-95-3	056 B	040 B
N-nitrosodimethylamine	34438	62-75-9	061 B	041 B
N-nitrosodi-n-proplyamine	34428	621-64-7	063 B	042 B
N-nitrosodiphenylamine	34433	86-30-3	062 B	043 B
phenanthrene	34461	85-01-8	081 B	044 B
phenol	34694	108-95-2	065 A	010 A
α-picoline (Synfuel)	77088	109-06-8	503 B	
pyrene	34469	129-00-0	084 B	045 B
styrene (Appendix C)	77128	100-42-5	510 B	
α-terpineol (Appendix C)	77493	98-55-5	509 B	
1,2,4-trichlorobenzene	34551	120-82-1	008 B	046 B

Table 2

Acid Extractable Compounds

Compound	Storet	CAS Registry	EPA-EGD	NPDES
benzoic acid (Synfuel)	77247	65-85-0	500 A	
4-chloro-3-methylphenol	34452	59-50-7	022 A	008 A
2-chlorophenol	34586	95-57-8	024 A	001 A
2,4-dichlorophenol	34601	120-83-2	031 A	002 A
2,4-dinitrophenol	34616	51-28-5	059 A	005 A
hexanoic acid (Synfuel)	77190	142-62-1	501 A	
2-methyl-4,6-dinitrophenol	34657	534-52-1	060 A	004 A
2-nitrophenol	34591	88-75-5	057 A	006 A
4-nitrophenol	34646	100-02-7	058 A	007 A
pentachlorophenol	39032	87-86-5	064 A	009 A
2,4,6-trichlorophenol	34621	88-06-2	021 A	011 A

Table 3

Gas Chromatography of Base/Neutral Extractable Compounds (1)

Compound	Relative Retention Time (2)	Limit of Detection ng injected (3)
N-nitrosodimethylamine	.26	50
α-picoline	.28	20
styrene	.40	20
p-cymene	.46	20
di-n-butylamine	.51	200
phenol	.58	20
bis(2-chloroethyl) ether	.59	20
1,3-dichlorobenzene	.61	20
1,4-dichlorobenzene	.61	20
1,2-dichlorobenzene	.64	20
bis(2-chloroisopropyl) ether	.67	20
hexachloroethane	.69	20
N-nitroso-di-n-propyl amine	.69	20
nitrobenzene	.70	20
isophorone	.74	50
2,4-dimethylphenol	.77	20
bis(2-chloroethoxy)methane	.78	20
1,2,4-trichlorobenzene	.80	20
naphthalene	.81	20
dodecane	.82	20
hexachlorobutadiene	.84	20
hexachlorocyclopentadiene	.95	20
2,2'-difluorobiphenyl (internal standard)	1.00	20
2-chloronaphthalene	1.01	20
biphenyl	1.03	20
acenaphthalene	1.05	20
dimethyl phthalate	1.05	20
acenaphthene	1.15	20
dibenzofuran	1.18	20
diphenylether	1.06	20
2,6-dinitrotoluene	1.19	20
β-naphthylamine	1.20	20
fluorene	1.23	20
diethyl phthalate	1.23	20
4-chlorophenylphenylether	1.24	20
2,4-dinitrotoluene	1.26	20
1,2-diphenylhydrazine (3)	1.27	20
N-nitrosodiphenylamine (4)	1.26	20
4-bromophenylphenylether	1.31	20
hexachlorobenzene	1.33	20
phenanthrene	1.39	20
anthracene	1.39	20
dibenzothiophene	1.40	20
di-n-butylphthalate	1.51	20
fluoranthene	1.59	20
pyrene	1.62	20
benzidine	1.62	20
2,3,7,8-tetrachlorodibenzo-p-dioxin	-	20
butylbenzylphthalate	1.77	20
chrysene	1.87	20
benzo(a)anthracene	1.87	20
3,3'-dichlorobenzidine	1.87	20
bis(2-ethlyhexyl)phthalate	1.92	20
di-n-octylphthalate	2.13	20

Table 3 (continued)

Compound	RRT	ng
benzo(b)fluoranthene	2.21	20
benzo(k)fluoranthene	2.21	20
benzo(a)pyrene	2.34	20
indeno(1,2,3-cd)pyrene	3.05	50
dibenzo(a,h)anthracene	3.10	50
benzo(g,h,i)perylene	3.26	50

Notes: bonded phase
(1)30 m SE54^fused silica capillary column. conditions: gas velocity 30 cm/sec; temperature program: 5 min at 30°C; 30 -280 at 8°C per min, iso at 280°C until benzo(ghi)perylene elutes
(2) To 2,2'-difluorobiphenyl
(3) This is a minimum level at which the entire analytical system must give recognizable mass spectra (background corrected) and acceptable calibration points.
(4) detected as diphenylamine. If rigorous differentiation between N-nitrosodiphenylamine and diphenylamine is required, EPA Method 607 is to be used.
(5) detected as azobenzene

Table 4

Gas Chromatography of Acid Extractable Compounds (1)

Compound	Relative Retention Time (2)	Limit of Detection ng injected (3)
2-chlorophenol	.59	50
hexanoic acid	.63	200
2-nitrophenol	.75	50
benzoic acid	.83	50
2,4-dichlorophenol	.79	50
2,4,6-trichlorophenol	.97	50
4-chloro-3-methylphenol	.90	50
2,2'-difluorobiphenyl	1.00	20
4-nitrophenol	1.07	50
2,4-dinitrophenol	1.16	200
2-methyl-4,6-dinitrophenol	1.25	200
pentachlorophenol	1.37	100

Notes: bonded phase
(I) 30 m SE54^fused silica capillary column. conditions: gas velocity 30 cm/sec; temperature program: 5 min at 30°C; 30-250°C or until pentachlorophenol elutes
(2) to 2,2'-difluorobiphenyl
(3) This is a minimum level at which the entire analytical system must give recognizable mass spectra (background corrected)and acceptable calibration points.

Table 5

DFTPP Mass-intensity Specifications

Mass	Intensity required
51	30-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	base peak, 100%
199	5-9% of mass 198
275	10-30% of mass 198
441	< mass 443
442	>40% of mass 198
443	17-23% of mass 442

Table 6

Base/Neutral Extractable Compound Characteristic Ions

Compound	labeled analog	Primary m/z
acenaphthene	d10	154/164
acenaphthylene	d8	152/160
anthracene	d10	178/188
benzidine	d8	184/192
benzo(a)anthracene	d12	228/240
benzo(b)fluoranthene	d12	252/264
benzo(k)fluoranthene	d12	252/264
benzo(a)pyrene	d12	252/264
benzo(g,h,i)perylene	d12	276/288
biphenyl	d10	154/164
bis(2-chloroethyl) ether	d8	93/99
bis(2-chloroethoxy)methane	(1)	
bis(2-chloroisopropyl) ether	d12	121/131
bis(2-ethylhexyl) phthalate	d4	149/153
4-bromophenyl phenyl ether	(1)	
butyl benzyl phthalate	(1)	
n-C12	d22	57/66
2-chloronaphthalene	d7	162/169
4-chlorophenyl phenyl ether	d5	204/209
chrysene	d12	228/240
dibenzo(a,h)anthracene	(1)	
dibenzofuran	d8	168/176
dibenzothiophene	d8	184/192
di-n-butylamine	d18	86/96
di-n-butyl phthalate	d4	149/153
1,2-dichlorobenzene	d4	146/152
1,3-dichlorobenzene	d4	146/152
1,4-dichlorobenzene	d4	146/152
3,3'-dichlorobenzidine	d4	252/258
diethyl phthalate	d4	149/153
2,4-dimethylphenol	d3	122/125
2,4-dinitrotoluene	d3	165/167
2,6-dinitrotoluene	d3	165/167
di-n-octyl phthalate	d4	149/153
dimethyl phthalate	d4	163/167
diphenylamine	d6	169/175
diphenyl ether	d10	170/180
1,2-diphenylhydrazine*	d10	77/82
fluoranthene	d10	202/212

Table 6 (continued)

Compound	labeled analog	Primary m/z
fluorene	d10	166/176
hexachlorobenzene	13C6	284/292
hexachlorobutadiene	13C4	225/231
hexachloroethane	13C1	201/204
hexachlorocyclopentadiene	13C1	237/240
indeno(1,2,3-cd)pyrene	(1)	
isophorone	d8	82/88
naphthalene	d8	128/136
β-naphthylamine	d7	143/150
nitrobenzene	d5	77/82
N-nitrosodimethylamine	(1)	
N-nitrosodi-n-propylamine	(1)	
N-nitrosodiphenylamine**	d6	169/175
phenanthrene	d10	178/188
phenol	d5	94/99
α-picoline	d7	93/100
pyrene	d10	202/212
styrene	d5	104/109
α-terpineol	d3	59/62
1,2,4-trichlorobenzene	d3	180/183

*Detected as azobenzene
**Detected as diphenylamine
(1) Compound not available at time of writing

Table 7

Acid Extractable Compound Characteristic Ions

Compound	labeled analog	Primary m/z
benzoic acid	d5	122/127
4-chloro-3-methylphenol	d2	107/109
2-chlorophenol	d4	128/132
2,4-dichlorophenol	d3	162/167
2,4-dinitrophenol	d3	184/187
hexanoic acid	d11	60/63
2-methyl-4,6-dinitrophenol	d2	198/200
2-nitrophenol	d4	139/143
4-nitrophenol	d4	139/143
pentachlorophenol	13C6	266/272
2,4,6-trichlorophenol	d2	196/202

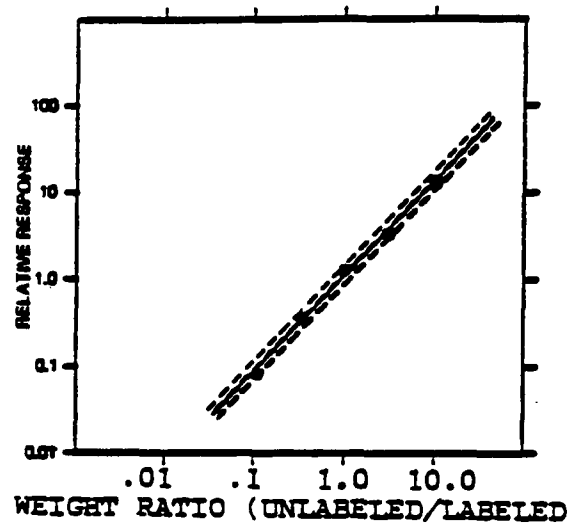

FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ±10 Percent Error Window.

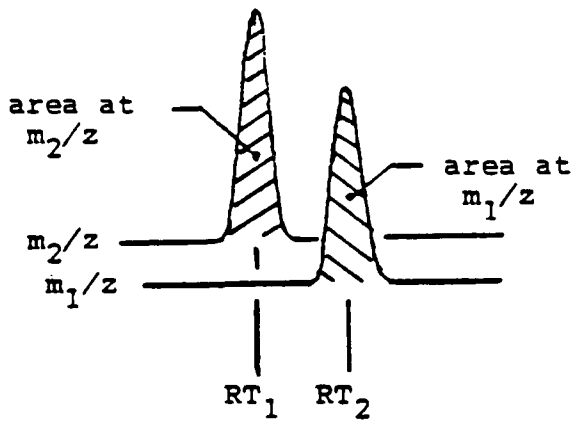

Figure 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled (m_1/z) and Unlabeled (m_2/z) Pairs

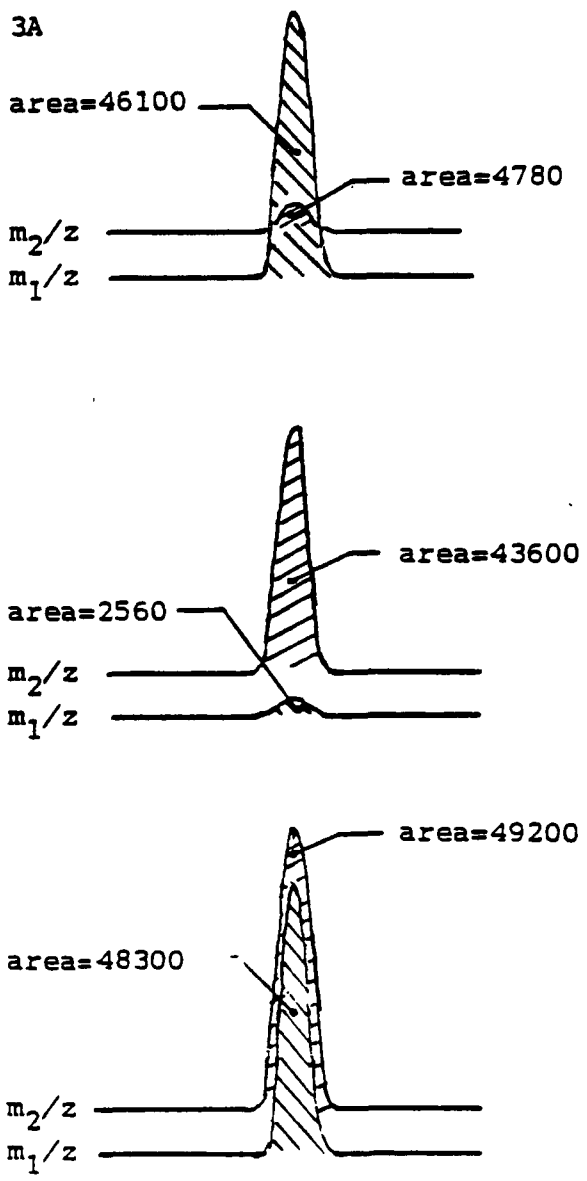

Figure 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabled and Labeled Compounds

Instructions for Preparation and Analysis of Performance Evaluation Samples

1 Overview

In these instructions, numbers in brackets [] refer to paragraph numbers in Method 1625A (March 1983 draft). This Method is the only Method to be used in this performance evaluation.

Performance evaluation consists of three (3) extractions/concentrations and eleven (11) injections. A one liter water sample, a one liter reagent water (method) blank, and a one liter aliquot of reagent water containing a standard (the aqueous standard) are to be extracted and concentrated [10]. The eleven injections are for calibration [7.5], determination of inter-laboratory response ratios (not in the Method), calibration verification [12.5], and analyses of extracts of the blank, aqueous standard, and sample [11], as detailed in table 1.

2 Materials provided--all standards and the water sample have been provided; you are to provide the reagent water for the blank and aqueous standard [10], and decafluorotriphenylphosphine (DFTPP). The five solutions provided are:

Solution identification	Total volume	Purpose	Method Reference
200 µg/mL isotopes	4.3 ± 0.2 mL	calibration (2.5 mL) spiking into waters blank (0.5 mL) standard (0.5 mL) sample (0.5 mL)	[6.8, 6.13] [10.1]
400 µg/mL PP standards	2.0 ± 0.1 mL	calibration (0.95 mL) spiking into water standard (0.25 mL)	[6.9, 6.13] [10.1]
100 µg/mL mixed standards	1.0 ± 0.1 mL	inter-lab response ratios (0.5 mL)	--
10 mg/mL internal standard	1.0 ± 0.1 mL	spiking into extracts	[11.3]
sample	$1.0 \begin{smallmatrix}+0.1\\-0\end{smallmatrix}$ L	performance evaluation	--

The first four solutions listed above are packaged in sealed glass ampuls. Notice that several solutions are used for multiple purposes and that there is little excess. Once an ampul is opened, the solution should be aliquotted as required by these instructions and the Method to preclude changes in concentration caused by evaporation of the solvent. The sample is to be refrigerated (0-4°C) until ready for extraction. The solutions of standards are to be kept in the dark at -20 to -10°C when not in use.

3 Preparations for analysis of performance evaluation samples--in order to successfully analyze the solutions and extracts, it is necessary to obtain a valid spectrum for DFTPP [7.3.3, 12.2, and table 5] and authentic spectra for all labeled (isotopes) and unlabeled (PP standards) compounds. To obtain these authentic spectra, separately inject approx 0.5 µL of the "200 µg/mL isotopes" and approx 0.25 µL of the "400 µg/mL PP standards" [7.3]. Reporting of edited spectra [7.3.4] is required for this performance evaluation (see section 8.1.4 of these instructions). If two compounds coelute, a qualified spectrometrist is to deduce the correct spectrum and/or alternate quantitation mass for each compound.

4 Preparation of standard solutions for calibration and inter-laboratory response ratios.

4.1 Calibration solutions--in each of five (5) cleaned 1.5-3.0 mL

glass vials with Teflon-lined screw caps, place 500 µL of the "200 µg/mL isotopes" and separately add 25, 50, 125, 250 and 500 µL of the "400 µg/mL PP standards." Bring each solution to 1.0 mL total volume with methylene chloride [6.13]. Add 10 µL of the "10 mg/mL internal standard" [6.10] to each vial immediately prior to injection.

4.2 Inter-laboratory response ratio solution--in a cleaned 0.8-3.0 mL glass vial with Teflon-lined screw cap, place 500 µL of the "100 µg/mL mixed standards." Add 5.0 µL of the "10 mg/mL internal standard" immediately prior to injection.

5 Extraction of waters--the flow chart in figure 1 shows the extractions required. Some steps have been omitted from this chart but are detailed in the Method [10]. Continuous extraction only is to be used.

6. Method specifications--all specifications in the Method shall be met with the following exceptions:

6.1 preparation of solutions of standards [6.7-6.9] is not required. All pollutant and labeled standards have been provided and are the only standards to be used.

6.2 determination of initial and on-going recovery and precision [7.1, 7.10, and 12.8] is not to be performed.

6.3 the sample is not "complex" and the section on "Analysis of complex samples" [15] does not apply.

6.4 the specifications for verification of calibration [12.5] need not be met for the "B" shift (injection number 7, table 1); however, differences between the results of this injection and injection number 4 indicate an instability problem with the instrument and can affect the results obtained in injections 8-10. You will be evaluated on all of these results (see section 9 of these instructions).

6.5 the chromatographic conditions [tables 3 and 4] shall supercede the elution time specifications [12.3.1 and 12.3.2]. The column to be used is a 30 meter, 0.25 mm i.d., J & W DB-5, or equivalent. Equivalent means a 30 meter, 0.25 mm i.d., bonded phase fused silica capillary containing 94 percent methyl, 4 percent phenyl, and 1 percent vinyl silicone.

7 Corrections to Method 1625--the additions and corrections in table 2 supercede data in the Method [tables 6 and 7].

8 Reporting

8.1 Data to be reported

8.1.1 hardcopy of mass spectra and mass-intensity lists of DFTPP.

8.1.2 hardcopy of RIC chromatograms from the 11 injections (table 1) normalized on the largest non-solvent peak.

8.1.3 technical data from analysis of the 11 injections as detailed in TAB 1 of "Appendix A Quantitation Reports on Magnetic Tape," and given on the "Performance Evaluation Data Sheet" in figure 2.

8.1.4 hardcopy of library mass-intensity data [7.3.4].

8.2 Formats--data may be reported in one or more of the following formats:

8.2.1 quantitation reports on magnetic tape per the specifications in "Appendix A Quantitation Reports on Magnetic Tape."

8.2.2 hardcopy quantitation reports as given by example in TAB 1 of "Appendix A Quantitation Reports on Magnetic Tape."

8.2.3 "Performance Evaluation Data Sheets" as given in figure 2.

The most desirable forms for data reporting are both magnetic tape and hardcopy quantitation reports. If magnetic tape reporting

only is employed, you assume all risk for illegible and/or non-readable tape. Extensions of time for regeneration of tape shall not be granted. If more than one form of data submission is used, the order of precedence (8.2.1-8.2.3 above) shall be employed for data evaluation; i.e., the magnetic tape data shall be assumed correct; in its absence, the hardcopy shall be assumed correct; in its absence, data sheets (figure 2) shall be assumed correct.

8.3 The compound numbering system given in TAB 2 of "Appendix A Quantitation Reports on Magnetic Tape" is required for all forms of reporting. For data evaluation, compound names shall be ignored. The compound numbering system shall be used as follows:

8.3.1 pollutants with no labeled analog are quantitated by internal or external standard methods [7.6 or 7.7] and reported as three digit numbers with a zero (0) as the first digit for the priority pollutants, and a five (5) as the first digit for the Appendix C and synfuel pollutants.

8.3.2 labeled compounds are quantitated by internal or external standard methods [7.6 or 7.7] and reported as three digit numbers with a two (2) as the first digit for the priority pollutants, and a six (6) as the first digit for the Appendix C and synfuel pollutants.

8.3.3 pollutants quantitated by isotope dilution [7.5] are reported as three digit numbers with a three (3) as the first digit for the priority pollutants, and a seven (7) as the first digit for the Appendix C and synfuel pollutants.

Examples of reporting using these numbers are given in figure 2 and in TAB 1 of "Appendix A Quantitation Reports on Magnetic Tape."

8.4 Deadline-- all data shall be <u>received</u> at the EPA Sample Control Center address given in Section 4 of "Appendix A Quantitation Reports on Magnetic Tape" by 1700 EDST, 22 June 1983. Data received after that hour may result in disqualification of the submitting laboratory.

9 Data evaluation--you are responsible for analyses of only those compounds for which standards have been provided (as given on the data sheets supplied with the standards and sample). Scoring shall be based on the following:

9.1 Completeness--all results for all compounds from all 11 injections, plus RIC chromatograms, DFTPP spectra and lists, and library mass-intensity data.

9.2 Method specifications--all specifications in the Method (other than those specifically excepted in section 6 of these instructions) shall be met.

9.3 Mean concentrations--the compounds (labeled and/or unlabeled) found in the blank, aqueous standard, and sample shall be evaluated as closest to the mean of all submitting laboratories, based on an arithmetic or log-normal distribution (whichever is most appropriate) after removal of outliers. Scoring will be on a compound by compound basis.

10 Questions--concerning these instructions and/or the Method and/or solutions should be addressed to Susan Hancock or Deborah Danforth-Miller at the EPA Sample Control Center (703-557-5040).

Table 1 Injections to be Performed for Performance Evaluation

Shift (1)	Injection number (2)	Solution injected	Report identifier (3)
		DFTPP (4)	
	1	10 µg/mL calibration	(5),(6),CAL,00010,00,C,NA:NA,NA$
	2	20 µg/mL calibration	(5),(6),CAL,00020,00,C,NA:NA,NA$
A	3	50 µg/mL calibration	(5),(6),CAL,00050,00,C,NA:NA,NA$
	4	100 µg/mL calibration	(5),(6),CAL,00100,00,C,NA:NA,NA$
	5	200 µg/mL calibration	(5),(6),CAL,00200,00,C,NA:NA,NA$
	6	100 µg/mL mixed standards	(5),(6),PRR,00100,00,C,NA:NA,NA$
		DFTPP (4)	
	7	100 µg/mL calibration	(5),(6),VER,00100,00,C,NA:NA,NA$
	8	extract of blank	(5),(6),BLK,00000,00,C,1000:1,MM/DD/YY-(7)
B	9	extract of aqueous standard	(5),(6),APS,00000,00,C,1000:1,MM/DD/YY-(7)
	10	extract of sample (acid)	(5),(6),EPA,00000,00,A,1000:1,MM/DD/YY-(7)
	11	extract of sample (b/n)	(5),(6),EPA,00000,00,B,1000:1,MM/DD/YY-(7)

Notes:
(1) All 11 injections must be performed in the order given. The first six injections must be performed within a given eight (8) hour period; the seventh through eleventh injections must be performed within a given eight (8) hour period. The periods can overlap.
(2) All injections must be 1.0 ± 0.2 µL as measured by difference between amount in the syringe before and after injection.
(3) To be used in the "SAMPLE" field in the header of the data file, and on all magnetic tapes, quantitation reports, data sheets, chromatograms, and spectra and lists.
(4) Can be co-injected with the first injection on a given shift if you wish to take the risk that the specifications [table 5] will be met.
(5) Instrument identifier. See section 3.1.3 of Appendix A
(6) Shift on which analysis is performed. See section 3.1.3 of Appendix A
(7) Shift on which extraction is performed. See section 3.1.3 of Appendix A

Table 2

Base/neutral and Acid Extractable Compound Characteristic Masses

Compound	Labeled analog	Primary m/z
bis(2-chloroethoxy)methane		93
bis(2-chloroethyl) ether	d8	93/101
4-bromophenylphenyl ether		248
butylbenzyl phthalate		149
n-C10	d22	57/66
n-C14		57
n-C16	d34	57/66
n-C18		57
n-C20	d42	57/66
n-C22		57
n-C24	d50	57/66
n-C26		57
n-C28		57
n-C30	d62	57/66
p-cymene	d14	114/130
dibenzo(a,h)anthracene		278
2,4-dinitrotoluene	d3	165/168
diphenylamine	d10	169/179
hexachlorocyclopentadiene	13C4	237/241
indeno(1,2,3-cd)pyrene		276
nitrobenzene	d5	123/128
N-nitrosodimethylamine		74
N-nitrosodi-n-propylamine		70
2,4,6-trichlorophenol	d2	196/200

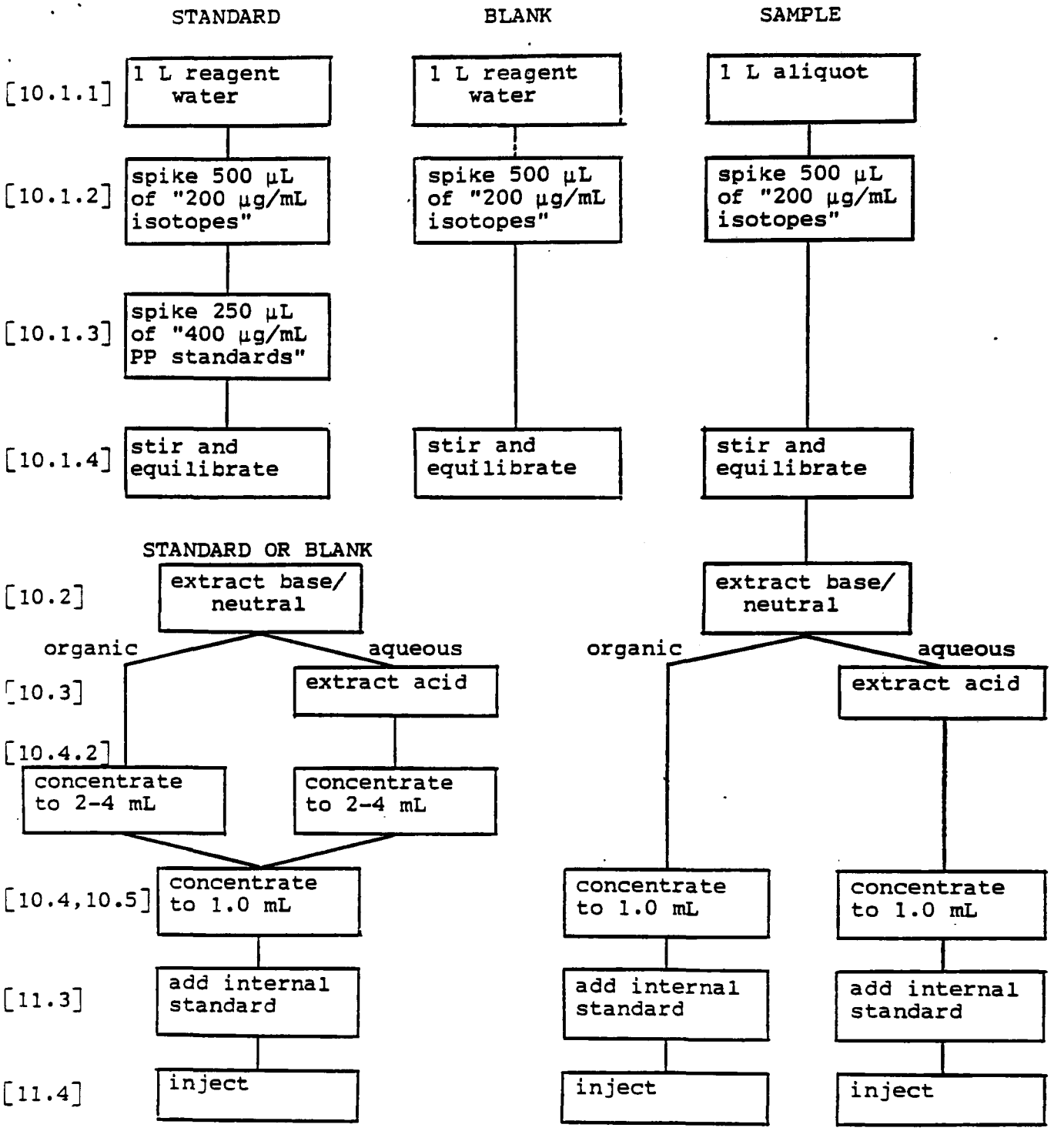

Figure 1 Flow Chart for Extraction/Concentration of Standard, Blank, and Sample for Performance Evaluation by Method 1625A. Numbers in brackets [] refer to section numbers in the Method.

Performance Evaluation Data Sheet Lab [illegible] Identifier [illegible]

EGD Number				Retention time		Rel Ret time		peak	Amount (µg/mL)		Response factor		
Data	Ref	m/z	scan	Data	Library	Data	Library	area	Data	Library	Data	Library	note
164	164	190	1190	20:49	20:49	1.000	1.00	132 000	100	100	1.00	1.00	
061	164	74	367	6:25	6:23	0.308	0.305	70 000	96	100	0.53	0.55	
603	164	100	463	8:06	8:05	0.389	0.388	86 000	102	100	0.65	0.64	
703	603	93	469	8:12	8:11	1.013	1.013	95 000	100	100	1.10	1.10	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)

SAMPLE

Explanation of notes for data fields:

(1) Compound number from Appendix A "Quantitation Reports on Magnetic Tape"
(2) Compound used for quantitation and retention time reference. Compound 164 is 2,2'-difluorobiphenyl.
(3) Quantitation m/z specified in tables 6 and 7 of Method and/or table 2 of these instructions; alternate primary m/z if an interference occurs at primary m/z (see paragraph 3 of the instructions).
(4) Scan number at which the compound elutes in this analysis.
(5) The retention time of the compound in this analysis in either seconds (an integer) or in minutes:seconds, with the colon separating the minutes and seconds.
(6) The reference retention time in the library in seconds or minutes:seconds.
(7) The retention time of the compound divided by the retention time of the reference compound.
(8) The relative retention time (7 above) stored in the library.
(9) The peak area at the quantitation m/z (2 above).
(10) The concentration of the compound detected as computed by internal standard [7.6] or isotope dilution [7.5].
(11) The reference amount in the library.
(12) The response factor for quantitation by the internal standard method [7.6], or the relative response (RR) divided by the concentration for the isotope dilution method [7.5.6].
(13) The response factor or RR/concentration (12 above) stored in the library.
(14) Reference to any notes you may wish to add; e.g., "alternate quantitation mass used."

Figure 2 Sample of Performance Evaluation Data Sheet

A blank Performance Evaluation Data Sheet is also provided; make as many copies of this sheet as you feel you will need. Save the original in case more copies are needed.

Performance Evaluation Data Sheet Lab ____________________ Identifier ____________________

EGD Number				Retention time		Rel Ret time		peak	Amount (µg/mL)		Response factor		
Data	Ref	m/z	scan	Data	Library	Data	Library	area	Data	Library	Data	Library	note

Appendix C

Task Order for Preparation of Performance Evaluation Samples

1 Objective--to prepare and certify priority pollutant calibration standards and samples for performance evaluation of analytical laboratories which respond to EPA's solicitation for priority pollutant analyses using the latest gas chromatography mass spectrometry (GCMS) methods.

2 Standards to be provided--using stable isotopically labeled (labeled) compounds furnished by EPA's Effluent Guidelines Division (EGD) Sample Control Center at Viar & Co (listed in Appendix A, attached), and procured standards from commercial suppliers, prepare solutions of standards to be used by the laboratories for calibration of GCMS instruments.

2.1 Labeled standards

2.1.1 Concentration--nominal concentrations of 200 micrograms per milliliter (µg/mL) in methylene chloride-d_2 and/or benzene-d_6. Prepared by mixing and diluting the government furnished five milligram per milliliter (mg/mL) labeled standards.

2.1.2 Quantity--180-200 mL total, divided into forty (40) each 4.3 ± 0.2 mL aliquots.

2.2 Priority pollutant standards (unlabeled analogs of the compounds in Appendix A, attached)

2.2.1 Concentration--nominal concentrations of 400 µg/mL in methylene chloride and/or benzene. Prepared by mixing and diluting priority pollutant standards.

2.2.2 Quantity--84-90 mL total, divided into forty (40) each two ± 0.1 mL aliquots.

2.3 Mixed labeled and priority pollutant standard

2.3.1 Concentration--nominal concentrations of 100 µg/mL by mixing appropriate volumes of standards in 2.1 and 2.2 and diluting.

2.3.2 Quantity--44-50 mL total, divided into forty (40) each 0.9 ± 0.1 mL aliquots.

2.4 Single lot--sufficient quantities of the solutions in 2.1 and 2.2 shall be prepared to produce all aliquots required in 2.1 through 2.3 from a single lot.

2.4 Internal standard spiking solution--2,2'-difluorobiphenyl

2.4.1 Concentration--ten milligrams per milliliter (mg/mL) in

methylene chloride and/or benzene.

2.4.2 Quantity--fourty (40) each one ± 0.1 mL aliquots

2.5 Packaging

2.5.1 Ampuls--each of the aliquots in 2.1 through 2.4 shall be packaged in a flame sealed, snap top, amber glass ampul.

2.5.2 Sets--a set consisting of one each of the ampuls (2.5.1) of the aliquots in 2.1 through 2.4 (making a total of four per set) shall be packaged in styrofoam (or equivalent) packing material so that shipment by Federal Express (or equivalent air carrier) shall not result in breakage of any ampul.

2.6 Labeling--each of the ampuls (2.5.1) shall be labeled with a unique lot number, and with text as follows:

2.6.1 "200 µg/mL isotopes" to designate the labeled standards (2.1).

2.6.2 "400 µg/mL PP standards" to designate the priority pollutant standards (2.2).

2.6.3 "100 µg/mL mixed standards" to designate the mixed labeled and priority pollutant standard (2.3).

2.6.4 "10 mg/mL internal standard" to designate the internal standard spiking solution (2.4).

2.7 Composition--the true value of the concentrations of the solutions in 2.1 through 2.4 shall be known within ± 5 percent of true value by traceability to source, and verified by analysis.

3 Sample

3.1 Compounds--to be selected from unlabeled analogs of the compounds in Exhibit A, attached. Distribution of these compounds shall be as follows:

3.1.1 Acid--3-4 including phenol and pentachlorophenol

3.1.2 Base/neutral and Appendix C--20-25 including naphthalene, one of the dichlorobenzenes, bis (2-chloroisopropyl) ether, hexachlorobutadiene, diethyl phthalate, 3,3'-dichlorobenzidine, benzo(a)anthracene, chrysene, one of the dinitrotoluenes, styrene, n-triacontane, and dibenzothiophene; and excluding di-n-butyl-, bis(2-ethylhexyl)-, and di-n-octyl phthalates, one of the diphenylamines, and anthracene.

3.1 Concentrations--approximately evenly distributed in the range of 10-200 µg/L in reagent water.
3.2 Quantity--40-50 liters total, divided into 40 each one +0.1, -0 liter aliquots
3.3 Labeling--"water sample" plus a unique lot and serial number.
3.4 Packaging--in cleaned sample bottles with Teflon lined screw cap lids. Leak tested by inverting for one hour minimum with no trace of sample present on lid. Packed in sets as appropriate to fit into containers for shipment to the Sample Control Center or other location as designated by EPA.

4 Documentation
4.1 Deliverables
4.1.1 Two each data sheets listing lot numbers, compounds, concentrations, and solvents for solutions in 2.1 through 2.4, to be shipped with solutions.
4.1.2 One each data sheet listing compounds and concentrations for the sample in section 3, to be held for disposition upon instructions from W A Telliard only.
4.2 In-house records
4.2.1 Traceability--source traceability through documentation shall be maintained for all compounds, mixtures, solutions, lot numbers, and other information for all items in sections 1 through 3.
4.2.2 Preparation--all weights, volumes, dilutions, and other information necessary to prove the final concentrations of the solutions in sections 2 through 3 shall be documented in log-books, on data sheets, or on other forms in an easily understood format.

5 Confidentiality--records of the concentrations and the compounds in the water sample in section 3 shall be maintained in strictest confidence by the contractor and its employees, and/or by anyone else who may gain knowledge of the compounds and/or concentrations from the contractor. If necessary, the contractor shall require signed confidentiality agreements from each of its employees, or others who shall have knowledge of the compounds and concentrations as a result of the

contractor's knowledge. Because release of this information would compromise EPA's objective evaluation of laboratories, the government shall have rights of full restitution for all costs and delays resulting from disclosure of the composition of the performance standard in section 3.

6 Anonymity--the contractor shall not disclose, or make any mark on any ampul (section 2) or sample (section 3) which would identify the contractor as the source of these materials.

7 Automatic qualification of contractor--EPA has selected this contractor based on the contractor's history and performance for the work described herein. Recognizing that this contractor would be one of the laboratories seeking qualification under solicitations requiring analysis of the performance evaluation sample in section 3, and that the contractor will have knowledge of the true values of all solutions and samples in sections 2 through 3, and further that the contractor will certify all solutions and samples by chemical analysis, EPA will deem this contractor to be qualified for work under solicitations associated with analysis of the solutions and samples in sections 2 through 3.

7 Certifications of analysis--as required by EPA, contractor shall submit appropriate certifications for analysis of the standards in section 2, and the sample in section 3.

8 Standards use--to assist the contractor in understanding the concentrations and amounts in section 2, the use of these standards is described below.

8.1 Obtaining authentic mass spectra--the solutions in 2.1 and 2.2 can be used to obtain authentic mass spectra of the labeled compounds and priority pollutants.

8.2 Five point calibration--by combining 0.50 mL of the solution in 2.1 with 25, 50, 125, 250, and 500 µL of the solution in 2.2 and bringing to 1.00 mL total volume, calibration solutions of 10, 20, 50, 100, and 200 µg/mL will be produced.

8.3 The solution in 2.3 is to be used for determination of response ratios.

8.4 Labeled compound spiking--three each 0.5 mL aliquots of the solution in 2.1 will be spiked into a blank, reagent water, and the sample in section 3 to determine contamination, recovery, and ability to analyze samples, respectively.

PURGEABLES/VOLATILES - A

50 µg each component/1 mL methanol-d_4 solution

Components

EPA 3V	MD—1455	Acrylonitrile-d_3
6V	MS—1312	Carbon-^{13}C Tetrachloride
7V	MD—786	Chlorobenzene-d_5
23V	MS—1318	Chloroform-^{13}C
13V	MD—1152	1,1-Dichloroethane-2,2,2-d_3
29V	MD—2201	1,1-Dichloroethylene-d_2
44V	MD—53	Dichloromethane-d_2
32V	MD—2363	1,2-Dichloropropane-d_6
14V	MS—2346	1,1,2-Trichloroethane-1,2-$^{13}C_2$

PURGEABLES/VOLATILES - B

50 µg each component/1 mL methanol-d_4 solution

Components

EPA 4V	MD—6	Benzene-d_6
47V	MS—2313	Bromoform-^{13}C
10V	MD—103	1,2-Dichloroethane-d_4
38V	MD—1756	Ethylbenzene-d_{10}
15V	MD—1416	1,1,2,2-Tetrachloroethane-d_2
86V	MD—351	Toluene-2,3,4,5,6-d_5
11V	MD—1150	1,1,1-Trichloroethane-d_3

PURGEABLES/VOLATILES - C

50 µg each component/1 mL methanol-d_4 solution

Components

EPA 46V	MD—23	Bromomethane-d_3
16V	MD—334	Chloroethane-d_5
45V	MD—324	Chloromethane-d_3
88V	MD—965	Vinyl-d_3 Chloride

PURGEABLES/VOLATILES - D

50 µg each component/1 mL methanol-d_4 solution

Components

EPA 516V	MD—2	Acetone-d_6
48V	MS—2368	Bromodichloromethane-^{13}C
514V	MD—2402	2-Butanone-4,4,4-d_3
51V	MS—2364	Chlorodibromomethane-^{13}C
30V	MD—2526	1,2-Dichloroethylene-1,2-d_2 (cis/trans mixture)
33V	MD—2669	1,3-Dichloropropene-d_4 (cis/trans mixture)
515V	MD—267	Diethyl-d_{10} Ether
85V	MS—2411	Tetrachloroethylene-1,2-$^{13}C_2$
87V	MS—2410	1,1,2-Trichloroethylene-1,2-$^{13}C_2$

Table 1 Volatiles

ACID EXTRACTABLES - 2

5 mg each component/1 mL benzene-d_6 solution

Components

EPA 22A	MD—2355	4-Chloro-3-methylphenol-2,6-d_2
24A	MD—2280	2-Chlorophenol-3,4,5,6-d_4
31A	MD—2281	2,4-Dichlorophenol-3,5,6-d_3
34A	MD—2284	2,4-Dimethylphenol-3,5,6-d_3
60A	MD—2357	4,6-Dinitro-2-methylphenol-3,5-d_2
59A	MD—2285	2,4-Dinitrophenol-3,5,6-d_3
57A	MD—2290	2-Nitrophenol-3,4,5,6-d_4
58A	MD—2356	4-Nitrophenol-2,3,5,6-d_4
64A	MS—2293	Pentachlorophenol-$^{13}C_6$
65A	MD—1502	Phenol-2,3,4,5,6-d_5
21A	MD—2279	2,4,6-Trichlorophenol-3,5-d_2

Table 2 Acids

BASE NEUTRALS - 4.1

5 mg each component/1 mL benzene-d_6 solution

Components

EPA 77B	MD—128	Acenaphthylene-d_8
74B	MD—2360	Benzo(b)fluoranthene-d_{12}
79B	MD—830	Benzo(ghi)perylene-d_{12}
73B	MD—1956	Benzo(a)pyrene-d_{12}
42B	MD—2702	Bis(2-chloroisopropyl)-d_{12} Ether
26B	MD—2405	1,3-Dichlorobenzene-d_4
35B	MD—2407	2,4-Dinitrotoluene-3,5,6-d_3
39B	MD—2361	Fluoranthene-d_{10}
52B	MS—2408	Hexachloro-1,3-butadiene-$^{13}C_4$
53B	MS—2710	Hexachlorocyclopentadiene-1,2,3,4-$^{13}C_4$
81B	MD—120	Phenanthrene-d_{10}

BASE NEUTRALS - 4.2

5 mg each component/1 mL benzene-d_6 solution

Components

EPA 20B	MD—2462	2-Chloronaphthalene-d_7
40B	MD—2312	4-Chlorophenyl Phenyl-d_5 Ether
68B	MD—2310	Di-n-butyl Phthalate-3,4,5,6-d_4
70B	MD—2726	Diethyl Phthalate-3,4,5,6-d_4
69B	MD—2291	Di-n-octyl Phthalate-3,4,5,6-d_4
9B	MS—2354	Hexachlorobenzene-$^{13}C_6$
12B	MS—2406	Hexachloroethane-1-^{13}C
54B	MD—2304	Isophorone-d_8
8B	MD—2706	1,2,4-Trichlorobenzene-3,5,6-d_3

Table 3 Base/neutrals (continued on next page)

BASE NEUTRALS - 6.1

5 mg each component/1 mL benzene-d_6 solution

Components

EPA 72B	MD—364	Benz(a)anthracene-d_{12}
66B	MD—2306	Bis(2-ethylhexyl) Phthalate-3,4,5,6-d_4
25B	MD—1191	1,2-Dichlorobenzene-d_4
27B	MD—1034	1,4-Dichlorobenzene-d_4
71B	MD—2305	Dimethyl Phthalate-3,4,5,6-d_4
36B	MD—2359	2,6-Dinitrotoluene-α,α,α-d_3
56B	MD—27	Nitrobenzene-d_5

BASE NEUTRALS - 6.2

5 mg each component/1 mL benzene-d_6 solution

Components

EPA 1B	MD—42	Acenaphthene-d_{10}
78B	MD—46	Anthracene-d_{10}
75B	MD—2362	Benzo(k)fluoranthene-d_{12}
18B	MD—2479	Bis(2-chloroethyl)-d_8 Ether
76B	MD—402	Chrysene-d_{12}
80B	MD—1298	Fluorene-d_{10}
55B	MD—26	Naphthalene-d_8
84B	MD—363	Pyrene-d_{10}

BASE NEUTRALS - 5

5 mg each component/1 mL benzene-d_6 solution

Components

EPA 5B	MD—2330	Benzidine-d_8 (rings-d_6)
511B	MD—2401	Di-n-butyl-d_{18}-amine
28B	MD—2703	3,3'-Dichlorobenzidine-d_6 (rings-d_6)
507B	MD—2704	Diphenyl-d_{10}-amine
37B	MD—2705	1,2-Diphenyl-d_{10}-hydrazine
503B	MD—2320	2-Methylpyridine-d_7 (α-Picoline)
502B	MD—191	2-Naphthyl-d_7-amine
62B	MD—2311	N-Nitrosodiphenyl-2,2',4,4',6,6'-d_6-amine

Table 3 (continued) Base/neutrals

SEMI-VOLATILES- 1 / APPENDIX C

5 mg each component/1 mL benzene-d_6 solution

Components

EPA	513B	MD—2709	p-Cymene-d_{14}
	517B	MD—960	n-Decane-d_{22}
	505B	MD—2316	Dibenzofuran-d_8
	504B	MD—2315	Dibenzothiophene-d_8
	512B	MD—208	Diphenyl-d_{10}
	508B	MD—373	Diphenyl-d_{10} Ether
	506B	MD—882	n-Dodecane-d_{26}
	521B	MD—1532	n-Eicosane-d_{42}
	519B	MD—821	n-Hexadecane-d_{34}
	510B	MD—126	Styrene-2,3,4,5,6-d_5
	509B	MD—2707	α-Terpineol-d_3
	523B	MD—883	n-Tetracosane-d_{50}
	526B	MD—2708	n-Triacontane-d_{62}

Table 4 Appendix C Semivolatiles

14 Apr 83

Changes to "Task Order for Preparation of Performance Evaluation Samples."

Change #1: Section 2.6.2: change "200 µg/mL..." to "400 µg/mL..."

change #2: In order to differentiate between (1) results of analysis of the "Mixed labeled and priority pollutant standard" (section 2.3 of the task), and (2) the 100 µg/mL calibration solution prepared by mixing appropriate volumes of the "Labeled standards" (section 2.1) and the "Priority pollutant standards" (section 2.2) using the procedure in section 8.2, the contractor shall add one to three of the compounds below to the "Priority pollutant standards" (section 2.2) at a concentration of approximately 100 µg/mL after the appropriate volume of the "Priority pollutant standards" (section 2.2) has been withdrawn for mixing to form the "Mixed labeled and priority pollutant standards" (section 2.3):

Appendix A Quantitation Reports on Magnetic Tape

1. Tape Characteristics

a. Tape - 9 Track; 800/1600 BPI; 600, 1200, or 2400 foot reels
b. Code - ASCII
c. Labels - no internal labels
d. Blocksize - 800 decimal words/block or bytes/block

2. File Characteristics

a. Each quantitation report represents a file on the submitted tape. A tape will contain multiple files. Each of these files must end with a tape mark. The last file on the tape will end with two tape marks.

b. Each line of the quantitation report constitutes a record. Records/lines can be variable in length from one to 80 characters. Each record/line must end with a carriage return (Octal 15 or Hexidecimal OD). A blank line is interpreted as two consecutive carriage returns. A form feed character (Octal 14 or Hexidecimal OC) must be used after the last record in the file to signify the end of all records.

c. Records/lines must be combined into fixed length blocks of 800 bytes in length. Blocks should not include any prefixes or postfixes. Records may span blocks. A file will consist of multiple blocks. Blocks are separated on the tape by inter-record gaps.

3. Data Format

The quantitation report is divided into four basic sections for information reporting. The sections in the order in which they appear within the quantitation report are called:

- Header Section
- Compound List Section
- Techincal Data Section
- Reference Data Section

Each of these sections must be present in each quantitation report submitted on tape. The absence of any one of these sections from a quantitation report is cause for the non acceptance of the quantitation report. TAB 1 provides a sample quantitation report showing these basic sections.

3.1 Header Section

The header section provides descriptive information about the analysis and the conditions under which it was performed. This section at a minimum must contain at least four lines of descriptive information. The four lines that must be present are identified as follows.

- Title Line
- Date/Time Analyzed Line
- Sample Line
- Condition Line

Although the above four lines of information must be present in each header section, there is no actual limit as to the total number of lines that may be present. Laboratories are permitted to use this section to record any additional information deemed necessary to properly identify the analysis. The four lines however must appear in the order specified above, but additional lines may be interspersed between them. The complete specifications for each of these four lines is provided in subsequent paragraphs.

3.1.1 Title Lines

This must be the first line within the header section and as such represents the first line of each quantitation report submitted. Only one title line is permitted within the Header Section. The line must contain the value QUANTITATION REPORT starting in position one (left most position) of the line. Other information may appear after this value on the line but must be separated from the value by at least one space (blank).

3.1.2 Date/Time Analyzed Line

This line must contain the date and time that the analysis was performed. This line must preceed any other date lines within the header section. The format of this line is as follows:

Line Position	Data Element	Format
1-8	Date Analyzed	MM/DD/YY MM = month; 01-12 DD = day; 01-31 YY = year; 83-99
9	Field Delimiter	Space
10-17	Time Analyzed	HH:MM:SS HH = hour; 00-24 MM = minute; 00-59 SS = second; 01-59

3.1.3 Sample Line

The Sample line contains the following data elements. Data elements are recorded on the line in the order specified. Data elements are separate from each other by means of a comma (,). The end of the data element list is signified by a dollar sign ($). The specifications for the sample line are:

Line Position	Data Element	Format
1 - 7	Literal Value	SAMPLE:
8	Field Delimiter	Space
9	Instrument[1]	2 positions; alphanumeric
	Field Delimiter	Comma (,)
	Shift	1 position; alpha Code Meaning G Graveyard D Day S Swing
	Field Delimiter	Comma (,)
	Quan Report Type	3 positions; alphanumeric Code Meaning CAL Calibration PAR Precision and Recovery VER Calibration Verification APS Aqueous Performance Standard EPA EPA Sample STD Standard BLK Blank
	Field Delimiter	Comma (,)
	Sample Number	5 positions; alphanumeric
	Field Delimiter	Comma (,)
	Bottle Number	2 positions; numeric For EPA Samples - Range: 01-99 All others: 00
	Field Delimiter	Comma (,)
	Fraction	1 position; alphanumeric Code Meaning A Acid B Base C Combined acid base/ neutral P Pesticide V Volatile

1. All calibration, precision and recovery, standards and blank quantitation files will be tracked by this instrument number within laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and other initial quantitation files by the laboratory.

Line Position	Data Element	Format
	Field Delimiter	Comma (,)
	Conc/Dilution Factor	11 positions maximum; numeric with colon (:) separating initial and final sample volume NA:NA - used for calibration standards and other runs that are not extracted
	Field Delimiter	Comma (,)
	Date Extracted	MM/DD/YY - X; 10 positions MM - month; 01-12 DD - day; 01-31 YY - year; 83-99 X - shift; G (graveyard) D (day), S (swing) NA - used for calibration standards and other runs that are not extracted
	Dollar Sign ($)	End of data delimiter

3.1.4 Condition Line

The Condition line contains the following data elements. As with the sample line, data elements are recorded on the line in the order indicated. Data elements are separated from each other by means of a comma (,). The end of the data element list is signified by a dollar sign ($). The specifications for this type of line are:

Line Position	Data Element	Format
1 - 7	Literal Value	CONDS.:
8	Field Delimiter	Space
9	Method	5 positions; alphanumeric 1624A or 1625A
	Field Delimiter	Comma (,)
	Column Length	6 positions; alphanumeric expressed in meters ie. 2.5 M or 35 M; Volatile Range 2.8-3.1 M Semi Volatile Range 25-35 M
	Field Delimiter	Comma (,)
	Column Inside Diameter	6 positions; alphanumeric expressed in millimeters ie. 2 mm or .3 mm; Volatile Range: 1-3 mm Semi Volatile Range: - 0.2 - 0.35 mm
	Field Delimiter	Comma (,)
	Column Initial Temperature	7 positions; numeric an at sign (@) is used to separate Hold and Temperature ie Hold @ Temp VolatileTempRange: 25-50°C Semi VolTempRange: 25-35°C
	Field Delimiter	Comma (,)
	Column Temperature Program	10 positions; numeric with a dash (-) separating initial and final temperatures and with an at sign (@) separating temperature program rate ie. 45-250 @ 8

Line Position	Data Element	Format
	Field Delimiter	Comma (,)
	Column Final Temperature	7 positions; numeric with an at sign (@) separating hold and temperature ie. Hold @ Temp
	Field Delimiter	Comma (,)
	Carrier Gas Flow Rate	9 positions; alphanumeric Format: 30ML/M or 30 CM/S; Volatile range: 20–40 ml/min Semi Volatile range: 20–60 cm/sec
	End of Data Delimiter	Dollar sign ($)

3.2 Compound List Section

The compound list section is the second basic section of information appearing on the quantitation report. It identifies the actual compounds that were determined during analysis. This section is made up of two types of lines:

- o Title Line
- o Compound Identification Line

3.2.1 Title Line

The title line must appear first within the Compound List Section. Only one title line may be present in the section. This line is formatted as follows:

Line Position	Data Element	Format
1	Field Delimiter	Space
2-3	Literal Value	NO
	Field Delimiter	Spaces (At Least 2)
	Literal Value	NAME

3.2.2 Compound Identification Line

A compound identification line is included in this section for each compound that was determined during analysis. Compound lines should be shown in the order in which they were determined. Each compound identification line is made up of three data elements specified in the following order within the line:

- o Compound Reference Number
- o EGD Compound Number
- o Compound Name

The compound reference number is a numerical code that establishes the order of compound determination by the GC/MS. The code is used on the Quantitation Report to match up compound identification with compound analysis and reference data appearing in subsequent sections of the report. On each quantitation report this number always starts with 1. The number 1 is always assigned to the first compound that is determined, the number 2 to the second compound is determined and so on.

Each compound identification line will appear in the following format:

Line Position	Data Element	Format
1-3	Compound Reference Number	1 to 3 character number; right justified in field; range 1-250
4-5	Field Delimiter	Spaces (At Least 2)
	EGD Compound Number	3 positions; numeric Range — Meaning 001-129 — Quantitated by internal or external standard 130-199 — Misc., internal standard and surrogate compound 201-299 — Labeled Compound (isotope) Quantitated by internal or external standard

Line Position	Data Element	Format
		Range Meaning 301-399 Quantitated by isotope dilution 501-599 Synfuel specific and Appendix C Comp. quantitated by internal or external standard 601-699 Synfuel specific and Appendix C labeled compounds (isotopes) quantitated by internal or external standard 701-799 Synfuel specific and Appendix C compounds quantitated by isotope dilution.
	Field Delimiter	Spaces (At Least 2)
	Compound Name	70 positions; alphanumeric

3.3 Technical Data Section

The technical data section provides measurement data for each compound that is determined. It is the third section within the quantitation report. This section is made up of two types of lines:

- o Title Line
- o Compound Technical Data Line

3.3.1 Title Line

The title line must appear first within the Technical Data Section. Only one title line may be present in the section. This line is formatted as follows:

Line Position	Data Element	Format
1	Field Delimiter	Space
2-3	Literal Value	NO
	Field Delimiter	Spaces (At Least 2)
	Literal Value	M/E
	Field Delimiter	Spaces (At Least 2)
	Literal Value	SCAN
	Field Delimiter	Spaces (At Least 2)
	Literal Value	TIME
	Field Delimiter	Spaces (At Least 2)
	Literal Value	REF
	Field Delimiter	Spaces (At Least 2)
	Literal Value	RRT
	Free Area	Spaces or other literal values
First non-blank character at or past position 41.	Literal Value	AREA
	Field Delimiter	Spaces (At Least 2)
	Literal Value	AMOUNT
	Free Area	Spaces or other literal values

3.3.2 Compound Technical Data Line

A compound technical data line is included in this section for each compound that is determined. Compound technical data lines are ordered the same as the compound identification lines in the compound list section. The compound reference number is used for this purpose and serves to connect compound identification with the technical data. The compound technical data line at a minimum must contain the following data elements.

- o Compound Reference Number
- o Mass to Charge Ratio
- o Scan Number
- o Retention Time
- o Reference Compound
- o Relative Retention Time
- o Peak Area
- o Amount
- o Unit of Measure

The specific format for this line is as follows:

Line Position	Data Element	Format
1-3	Compound Reference Number	3 positions; numeric; right-justified Range 1-250
	Field Delimiter	Spaces (At Least 1)
	Mass to Charge Ratio (M/Z)	4 positions; numeric; Volatile range: 20-250; Semi Volatile range: 35-450
	Field Delimiter	Spaces (At Least 1)
	Scan Number	5 positions; numeric; range 1-9999
	Field Delimiter	Spaces (At Least 1)
	Retention Time	6 positions; numeric with colon; format; MM:SS
	Field Delimiter	Spaces (At Least 1)
	Reference Compound	3 positions; numeric; range 1-250

Line Position	Data Element	Format
	Field Delimiter	Spaces (At Least 1)
	Relative Retention Time	5 positions; numeric with decimal point and 3 decimal places
	Field Delimiter	Spaces (At Least 1)
	Open Field	Spaces or other field value
First non-blank character at or past position 41.	Peak Area	10 positions; numeric
	Field Delimiter	Spaces (At Least 1)
	Amount	10 positions; numeric with decimal point and 3 decimal places
	Field Delimiter	Spaces (At Least 1)
	Unit of Measure	5 positions; alphanumeric Valid codes: uG/L or uG/ml
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other field values.

3.4 Reference Data Section

The reference data section is the fourth section that must appear on each Quantitation Report. It provides reference and library data about the analysis that was performed. It is made up of two types of lines:

- o Title Line
- o Compound Reference Data Line

3.4.1 Title Line

The title line must appear first within this section. Only one title line is permitted within the section. Subsequent title lines will be deleted if present. The line is formatted as follows:

Line Position	Data Element	Format
1	Field Delimiter	Space
2-3	Literal Value	NO
	Field Delimiter	Spaces (At Least 1)
	Literal Value	RET (L)
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other literal values
First non-blank character at or past position 19.		Spaces (At Least 1)
	Literal Value	RRT (L)
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other literal values
First non-blank character at or past position 43.	Literal Value	AMNT (L)
	Field Delimiter	Spaces (At Least 1)
	Literal Value	R.FAC
	Field Delimiter	Spaces (At Least 1)
	Literal Value	R.FAC (L)
	Open Area	Spaces or other literal values

3.4.2 Compound Reference Data Line

A compound reference data line is included in this section for each compound that is determined. These lines are ordered the same as the compound identification lines in the compound list section. The compound reference number is used for this purpose and serves to connect the compound identification with the reference data. This means that there is a one to one correspondence between the compound identification lines and the reference data lines. The reference data line at a minimum must contain the following data elements.

- Compound Reference Number
- Library Retention Time
- Library Relative Retention Time
- Library Amount
- Response Factor
- Library Response Factor

The specific format for this line is as follows:

Line Position	Data Element	Format
1-3	Compound Reference Number	3 positions; numeric; Range 1-250
	Field Delimiter	Spaces (At Least 1)
	Library Retention Time	6 positions; numeric with colon; format - MM:SS
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other data values
First non-blank character starting at or past position 18.	Library Relative Retention Time	5 positions

Line Position	Data Element	Format
	Field Delimiter	Spaces (At Least 1)
	Open	Spaces or other data values
First non-blank character starting at or past position 41.	Library Amount	9 positions; numeric with decimal point and 2 decimal places
	Field Delimiter	Spaces (At Least 1)
	Response Factor	7 positions; numeric with decimal point and 3 decimal places
	Field Delimiter	Spaces (At Least 1)
	Library Response Factor	7 positions; numeric with decimal point and 3 decimal places.
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other data values.

4. Packaging and Shipping Instructions

Format Requirements:

- Tapes shall be industry standard 9-Track, 800 or 1600 bits per inch with no internal labels.

- Tapes shall contain one or more files. Each file shall end with a tape mark. The last file shall end with two tapemarks. The first file may be preceded with one tapemark.

  See paragraph 2 for Record and Block format descriptions.

Packaging Requirements:

- Each tape reel shall bear a SCC supplied external tape label containing the external tape number, the laboratory name, the tape density, the block size, and the number of files.

- Each tape package shall contain in addition to the tape reel(s) at least one Quantitation Report Magnetic Tape Transmittal Form for each tape reel (see Tab 3 for a sample transmittal form and transmittal form description), all Lab Chronicle Reports associated with the reported samples, and BFB or DFTPP spectra analysis required per shift per machine.

- Tape reels with their associated transmittal forms and chronicle reports shall be packaged in such a way to ensure their safety and integrity. The outside of all packages should be marked with a 'DO NOT X-RAY' label. It will be the laboratory's responsibility to replace any tape, quantitation report data, and accompanying documents damaged during shipping to the Sample Control Center.

Shipping Requirements:

Tape packages shall be shipped either by the U.S. Postal Service or by any carrier with direct delivery.

Shipping address by U.S. Postal Service:

USEPA
Effluent Guidelines Division
Sample Control Center
P.O. Box 1407
Alexandria, VA 22313

Shipping address by other carriers:

USEPA
Effluent Guidelines Division
Sample Control Center
Suite 200
300 N. Lee St.
Alexandria, VA 22313

TAB 1

QUANTITATION REPORT FILE: 12045STD

DATA: 12045STD.TI
05/31/83 10:08:00
SAMPLE: A1,0.CAL=100.00,C,NA:NA,NA$
CONDS.: 16254,30M,0.25MM,5@30,30-280@8,16@280,30C4/S$
FORMULA: INSTRUMENT: A1 WEIGHT: 0.000
SUBMITTED BY: ANALYST: JLP ACCT.NO.:
AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

NO	NAME
1	164 2,2'-DIFLUOROBIPHENYL
2	061 N-NITROSODIMETHYLAMINE
3	603 (D7) A-PICOLINE
4	703 A-PICOLINE
5	610 (D5) STYRENE
6	710 STYRENE
7	265 (D5) PHENOL
8	365 PHENOL
9	617 (D22) N-DECANE
10	218 (D8) BIS(2-CHLOROETHYL)ETHER
11	224 (D4) 2-CHLOROPHENOL
12	324 2-CHLOROPHENOL
13	318 BIS(2-CHLOROETHYL)ETHER
14	717 N-DECANE
15	226 (D4) 1,3 DICHLOROBENZENE
16	326 1,3 DICHLOROBENZENE
17	227 (D4) 1,4 DICHLOROBENZENE
18	327 1,4 DICHLOROBENZENE
19	613 (D14) P-CYMENE
20	713 P-CYMENE
21	225 (D4) 1,2 DICHLOROBENZENE
22	711 DI-N-BUTYLAMINE
23	325 1,2 DICHLOROBENZENE
24	611 (D18) DI-N-BUTYLAMINE
25	242 (D12) BIS(2-CHLOROISOPROPYL)ETHER
26	342 BIS(2-CHLOROISOPROPYL)ETHER
27	312 HEXACHLOROETHANE
28	212 (13C1) HEXACHLOROETHANE
29	063 N-NITROSODI-N-PROPYLAMINE
30	256 (D5) NITROBENZENE
31	356 NITROBENZENE
32	254 (D8)ISOPHORONE
33	354 ISOPHORONE
34	257 (D4) 2-NITROPHENOL
35	357 2-NITROPHENOL
36	234 (D3) 2,4 DIMETHYLPHENOL
37	334 2,4 DIMETHYLPHENOL
38	043 BIS(2-CHLOROETHOXY)METHANE
39	231 (D3) 2,4 DICHLOROPHENOL
40	331 2,4 DICHLOROPHENOL
41	208 (D3) 1,2,4 TRICHLOROBENZENE
42	308 1,2,4 TRICHLOROBENZENE
43	606 (D26) N-DODECANE
44	255 (D8) NAPHTHALENE
45	355 NAPHTHALENE
46	609 (D3) A-TERPINOL

47	709	A-TERPINOL
48	706	N-DODECANE
49	252	(13C4) HEXACHLOROBUTADIENE
50	352	HEXACHLOROBUTADIENE
51	322	P-CHLORO-M-CRESOL
52	222	(D2) P-CHLORO-M-CRESOL
53	253	(13C1) HEXACHLOROCYCLOPENTADIENE
54	353	HEXACHLOROCYCLOPENTADIENE
55	221	(D2) 2,4,6 TRICHLOROPHENOL
56	321	2,4,6 TRICHLOROPHENOL
57	220	(D7) 2-CHLORONAPHTHALENE
58	320	2-CHLORONAPHTHALENE
59	612	(D10) BIPHENYL
60	712	BIPHENYL
61	608	(D10) DIPHENYLETHER
62	708	DIPHENYLETHER
63	277	(D8)ACENAPHTHYLENE
64	377	ACENAPHTHYLENE
65	271	(D4) DIMETHYLPHTHALATE
66	371	DIMETHYLPHTHALATE
67	236	(D3) 2,6 DINITROTOLUENE
68	336	2,6 DINITROTOLUENE
69	201	D10) ACENAPHTHENE
70	301	ACENAPHTHENE
71	259	(D3) 2,4 DINITROPHENOL
72	359	2,4 DINITROPHENOL
73	605	D8) DIBENZOFURAN
74	705	DIBENZOFURAN
75	258	(D4) 4-NITROPHENOL
76	358	4-NITROPHENOL
77	235	(D3) 2,4 DINITROTOLUENE
78	335	2,4 DINITROTOLUENE
79	602	(D7) B-NAPHTHYLAMINE
80	702	B-NAPHTHYLAMINE

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA(HGHT)	AMOUNT		%TOT
1	190	1190	20:49	1	1.000	A BB	132330.	100.000	UG/ML	1.61
2	74	367	6:25	1	0.308	A BV	70180.	99.500	UG/ML	1.60
3	100	463	8:06	1	0.389	A BV	85539.	100.000	UG/ML	1.61
4	93	469	8:12	3	1.013	A BB	95317.	100.000	UG/ML	1.61
5	109	588	10:17	1	0.494	A BB	119666.	100.000	UG/ML	1.61
6	104	591	10:21	5	1.005	A VB	144340.	102.500	UG/ML	1.65
7	99	733	12:50	1	0.616	A BV	191732.	100.000	UG/ML	1.61
8	94	735	12:52	7	1.003	A BV	136305.	100.100	UG/ML	1.61
9	66	735	12:52	1	0.618	A VV	245693.	100.000	UG/ML	1.25
10	101	735	12:52	1	0.618	A BV	29087.	100.000	UG/ML	1.25
11	132	739	12:56	1	0.621	A BB	74696.	100.000	UG/ML	1.61
12	128	742	12:59	11	1.004	A BB	95399.	100.200	UG/ML	1.61
13	93	742	12:59	10	1.010	A VB	114471.	99.750	UG/ML	1.24
14	57	756	13:14	9	1.029	A VV	126009.	100.500	UG/ML	1.25
15	152	760	13:18	1	0.639	A BB	50905.	100.000	UG/ML	1.25
16	146	762	13:20	15	1.003	A BB	91219.	100.750	UG/ML	1.25
17	152	769	13:27	1	0.646	A BB	52591.	100.000	UG/ML	1.25
18	146	772	13:31	17	1.004	A BB	98554.	99.250	UG/ML	1.24
19	130	779	13:38	1	0.655	A BB	206174.	100.000	UG/ML	1.25
20	119	791	13:51	19	1.015	A BB	214925.	99.750	UG/ML	1.24
21	152	801	14:01	1	0.673	A BB	50244.	100.000	UG/ML	1.25
22	86	804	14:04	24	1.000	A BV	11385.	100.250	UG/ML	1.25
23	146	804	14:04	21	1.004	A BB	92511.	100.000	UG/ML	1.25
24	96	804	14:04	1	0.676	A BB	1099.	100.000	UG/ML	1.25
25	131	822	14:23	1	0.691	A BV	25865.	100.000	UG/ML	1.25

26	121	832	14:34	25	1.012	A	BV	24739.	99.250	UG/ML	1.24
27	201	854	14:57	28	1.000	A	BB	27447.	100.000	UG/ML	1.61
28	204	854	14:57	1	0.718	A	BB	12281.	100.000	UG/ML	1.61
29	70	861	15:04	1	0.724	A	VV	90102.	99.500	UG/ML	1.60
30	128	873	15:17	1	0.734	A	BB	33737.	100.000	UG/ML	1.61
31	123	877	15:21	30	1.005	A	BB	43919.	102.000	UG/ML	1.64
32	88	919	16:05	1	0.772	A	VB	119964.	100.000	UG/ML	1.61
33	82	926	16:12	32	1.008	A	VV	92654.	99.000	UG/ML	1.59
34	143	931	16:18	1	0.782	A	BB	26177.	100.000	UG/ML	1.61
35	139	934	16:21	34	1.003	A	BV	38114.	100.200	UG/ML	1.61
36	125	955	16:43	1	0.803	A	BB	52780.	100.000	UG/ML	1.25
37	122	956	16:44	36	1.001	A	BV	99027.	100.000	UG/ML	1.25
38	93	971	17:00	1	0.816	A	BV	130312.	99.250	UG/ML	1.24
39	167	979	17:08	1	0.823	A	BB	35165.	100.000	UG/ML	1.61
40	162	981	17:10	39	1.002	A	BV	70600.	99.800	UG/ML	1.60
41	183	988	17:17	1	0.830	A	BB	66187.	100.000	UG/ML	1.61
42	180	990	17:19	41	1.002	A	BB	69057.	99.000	UG/ML	1.59
43	66	990	17:19	1	0.832	A	VV	173929.	100.000	UG/ML	1.25
44	136	997	17:27	1	0.838	A	BB	232175.	100.000	UG/ML	1.61
45	128	1000	17:30	44	1.003	A	BB	280748.	100.500	UG/ML	1.61
46	62	1006	17:36	1	0.845	A	VB	69136.	100.000	UG/ML	1.61
47	59	1009	17:39	46	1.003	A	BB	100849.	99.500	UG/ML	1.60
48	57	1012	17:43	43	1.022	A	VV	158423.	99.750	UG/ML	1.24
49	231	1037	18:09	1	0.871	A	BB	22826.	100.000	UG/ML	1.61
50	225	1037	18:09	49	1.000	A	BB	37916.	101.000	UG/ML	1.62
51	107	1117	19:33	52	1.000	A	BV	129831.	100.300	UG/ML	1.61
52	109	1117	19:33	1	0.939	A	BV	99414.	100.000	UG/ML	1.61
53	241	1171	20:30	1	0.984	A	BB	23908.	100.000	UG/ML	1.61
54	237	1171	20:30	53	1.000	A	BB	28030.	101.000	UG/ML	1.62
55	200	1190	20:49	1	1.000	A	BB	37779.	100.000	UG/ML	1.61
56	196	1191	20:51	55	1.001	A	BB	30623.	100.100	UG/ML	1.61
57	169	1214	21:15	1	1.020	A	BB	114393.	100.000	UG/ML	1.25
58	162	1217	21:18	57	1.002	A	BB	215477.	100.750	UG/ML	1.25
59	164	1217	21:18	1	1.023	A	BB	214588.	100.000	UG/ML	1.25
60	154	1221	21:22	59	1.003	A	BB	208044.	101.500	UG/ML	1.26
61	180	1238	21:40	1	1.040	A	BB	80639.	100.000	UG/ML	1.61
62	170	1244	21:46	61	1.005	A	BB	104115.	101.000	UG/ML	1.62
63	160	1293	22:38	1	1.087	A	BB	166440.	100.000	UG/ML	1.25
64	152	1296	22:41	63	1.002	A	BB	225824.	99.500	UG/ML	1.24
65	167	1296	22:41	1	1.089	A	VV	161184.	100.000	UG/ML	1.25
66	163	1298	22:43	65	1.002	A	VV	185673.	99.750	UG/ML	1.24
67	167	1308	22:53	1	1.099	A	VB	27562.	100.000	UG/ML	1.25
68	165	1311	22:57	67	1.002	A	BB	33165.	100.250	UG/ML	1.25
69	164	1326	23:12	1	1.114	A	BB	117288.	100.000	UG/ML	1.25
70	154	1332	23:19	69	1.005	A	BB	160368.	101.000	UG/ML	1.26
71	187	1352	23:40	1	1.136	A	BB	8463.	100.000	UG/ML	1.61
72	184	1354	23:42	71	1.001	A	BB	9632.	99.900	UG/ML	1.60
73	176	1359	23:47	1	1.142	A	BB	176268.	100.000	UG/ML	1.25
74	168	1363	23:51	73	1.003	A	BV	213270.	100.750	UG/ML	1.25
75	143	1378	24:07	1	1.158	A	VB	27783.	100.000	UG/ML	1.61
76	139	1379	24:08	75	1.001	A	BV	35052.	99.900	UG/ML	1.60
77	168	1385	24:14	1	1.164	A	BB	42897.	100.000	UG/ML	1.25
78	165	1387	24:16	77	1.001	A	VB	47189.	99.500	UG/ML	1.24
79	150	1396	24:26	1	1.173	A	BV	82896.	100.000	UG/ML	1.61
80	143	1400	24:30	79	1.003	A	BV	170206.	99.000	UG/ML	1.59

NO	RET(L)	RATIO	RRT(L)	RATIO	AMNT	AMNT(L)	R.FAC	R.FAC(L)	RATIO
01	20:49	1.00	1.000	1.00	100.00	100.00	1.000	1.000	1.00
02	6:25	1.00	0.308	1.00	99.50	99.50	0.533	0.533	1.00
03	8:06	1.00	0.389	1.00	100.00	100.00	0.646	0.646	1.00
04	8:12	1.00	0.392	2.58	100.00	100.00	1.114	1.114	1.00

05	10:17	1.00	0.494	1.00	100.00	100.00	0.904	0.904	1.00
06	10:21	1.00	0.497	2.02	102.50	102.50	1.177	1.177	1.00
07	12:50	1.00	0.614	1.00	100.00	100.00	1.449	1.449	1.00
08	12:52	1.00	0.618	1.62	100.10	100.10	0.710	0.710	1.00
09	12:52	1.00	0.618	1.00	100.00	100.00	1.857	1.857	1.00
10	12:52	1.00	0.618	1.00	100.00	100.00	0.220	0.220	1.00
11	12:56	1.00	0.621	1.00	100.00	100.00	0.564	0.564	1.00
12	12:59	1.00	0.623	1.61	100.20	100.20	1.275	1.275	1.00
13	12:59	1.00	0.624	1.62	99.75	99.75	3.945	3.945	1.00
14	13:14	1.00	0.635	1.62	100.50	100.50	0.510	0.510	1.00
15	13:18	1.00	0.639	1.00	100.00	100.00	0.385	0.385	1.00
16	13:20	1.00	0.640	1.57	100.75	100.75	1.779	1.779	1.00
17	13:27	1.00	0.646	1.00	100.00	100.00	0.397	0.397	1.00
18	13:31	1.00	0.649	1.55	99.25	99.25	1.888	1.888	1.00
19	13:39	1.00	0.655	1.00	100.00	100.00	1.558	1.558	1.00
20	13:51	1.00	0.665	1.53	99.75	99.75	1.045	1.045	1.00
21	14:01	1.00	0.673	1.00	100.00	100.00	0.380	0.380	1.00
22	14:04	1.00	0.676	1.49	100.25	100.25	10.333	10.333	1.00
23	14:04	1.00	0.676	1.48	100.00	100.00	1.841	1.841	1.00
24	14:04	1.00	0.676	1.00	100.00	100.00	0.008	0.008	1.00
25	14:23	1.00	0.691	1.00	100.00	100.00	0.195	0.195	1.00
26	14:34	1.00	0.699	1.45	99.25	99.25	0.964	0.964	1.00
27	14:57	1.00	0.718	1.39	100.00	100.00	2.235	2.235	1.00
28	14:57	1.00	0.718	1.00	100.00	100.00	0.093	0.093	1.00
29	15:04	1.00	0.724	1.00	99.50	99.50	0.684	0.684	1.00
30	15:17	1.00	0.734	1.00	100.00	100.00	0.255	0.255	1.00
31	15:21	1.00	0.737	1.36	102.00	102.00	1.276	1.276	1.00
32	16:05	1.00	0.772	1.00	100.00	100.00	0.907	0.907	1.00
33	16:12	1.00	0.778	1.30	99.00	99.00	0.780	0.780	1.00
34	16:18	1.00	0.782	1.00	100.00	100.00	0.198	0.198	1.00
35	16:21	1.00	0.795	1.28	100.20	100.20	1.453	1.453	1.00
36	16:43	1.00	0.803	1.00	100.00	100.00	0.399	0.399	1.00
37	16:44	1.00	0.803	1.25	100.00	100.00	1.876	1.876	1.00
38	17:00	1.00	0.816	1.00	99.25	99.25	0.992	0.992	1.00
39	17:08	1.00	0.823	1.00	100.00	100.00	0.266	0.266	1.00
40	17:10	1.00	0.823	1.22	99.80	99.80	2.012	2.012	1.00
41	17:17	1.00	0.830	1.00	100.00	100.00	0.500	0.500	1.00
42	17:19	1.00	0.832	1.20	99.00	99.00	1.054	1.054	1.00
43	17:19	1.00	0.832	1.00	100.00	100.00	1.314	1.314	1.00
44	17:27	1.00	0.838	1.00	100.00	100.00	1.755	1.755	1.00
45	17:30	1.00	0.840	1.19	100.50	100.50	1.203	1.203	1.00
46	17:36	1.00	0.845	1.00	100.00	100.00	0.522	0.522	1.00
47	17:39	1.00	0.848	1.18	99.50	99.50	1.466	1.466	1.00
48	17:43	1.00	0.850	1.20	99.75	99.75	0.913	0.913	1.00
49	18:09	1.00	0.871	1.00	100.00	100.00	0.172	0.172	1.00
50	18:09	1.00	0.871	1.15	101.00	101.00	1.645	1.645	1.00
51	19:33	1.00	0.939	1.07	100.30	100.30	1.302	1.302	1.00
52	19:33	1.00	0.939	1.00	100.00	100.00	0.751	0.751	1.00
53	20:30	1.00	0.984	1.00	100.00	100.00	0.181	0.181	1.00
54	20:30	1.00	0.984	1.02	101.00	101.00	1.161	1.161	1.00
55	20:49	1.00	1.000	1.00	100.00	100.00	0.285	0.285	1.00
56	20:51	1.00	1.001	1.00	100.10	100.10	0.810	0.810	1.00
57	21:15	1.00	1.020	1.00	100.00	100.00	0.864	0.864	1.00
58	21:18	1.00	1.023	0.98	100.75	100.75	1.870	1.870	1.00
59	21:18	1.00	1.023	1.00	100.00	100.00	1.622	1.622	1.00
60	21:22	1.00	1.025	0.98	101.50	101.50	0.955	0.955	1.00
61	21:40	1.00	1.040	1.00	100.00	100.00	0.609	0.609	1.00
62	21:46	1.00	1.045	0.96	101.00	101.00	1.278	1.278	1.00
63	22:38	1.00	1.087	1.00	100.00	100.00	1.258	1.258	1.00
64	22:41	1.00	1.087	0.92	99.50	99.50	1.364	1.364	1.00
65	22:41	1.00	1.089	1.00	100.00	100.00	1.218	1.218	1.00

66	22:43	1.00	1.091	0.92	99.75	99.75	1.155	1.155	1.00
67	22:53	1.00	1.099	1.00	100.00	100.00	0.208	0.208	1.00
68	22:57	1.00	1.102	0.91	100.25	100.25	1.200	1.200	1.00
69	23:12	1.00	1.114	1.00	100.00	100.00	0.886	0.886	1.00
70	23:19	1.00	1.119	0.90	101.00	101.00	1.354	1.354	1.00
71	23:40	1.00	1.136	1.00	100.00	100.00	0.064	0.064	1.00
72	23:42	1.00	1.138	0.88	99.90	99.90	1.139	1.139	1.00
73	23:47	1.00	1.142	1.00	100.00	100.00	1.332	1.332	1.00
74	23:51	1.00	1.145	0.88	100.75	100.75	1.201	1.201	1.00
75	24:07	1.00	1.159	1.00	100.00	100.00	0.210	0.210	1.00
76	24:08	1.00	1.159	0.86	99.90	99.90	1.263	1.263	1.00
77	24:14	1.00	1.164	1.00	100.00	100.00	0.324	0.324	1.00
78	24:16	1.00	1.165	0.86	99.50	99.50	1.106	1.106	1.00
79	24:26	1.00	1.173	1.00	100.00	100.00	0.626	0.626	1.00
80	24:30	1.00	1.176	0.85	99.00	99.00	2.074	2.074	1.00

QUANTITATION REPORT FILE: 12045STD

DATA: 12045STD.TI
05/31/83 10:08:00
SAMPLE: A1,D,CAL,100.00,C,NA:NA,NAS
CONDS.: 1625A,30M,0.25MM,5@30.30-280@8.16@280,30CM/SS
FORMULA: INSTRUMENT: A1 WEIGHT: 0.000
SUBMITTED BY: ANALYST: JLP ACCT.NO.:
AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

NO	NAME
1	164 2,2'-DIFLUOROBIPHENYL
2	619 (D34) N-HEXADECANE
3	280 (D10)FLUORENE
4	340 FLUORENE
5	719 N-HEXADECANE
6	240 (D5)4-CHLOROPHENYLPHENYLETHER
7	340 4-CHLOROPHENYLPHENYLETHER
8	270 (D4) DIETHYLPHTHALATE
9	370 DIETHYLPHTHALATE
10	607 (D10)DIPHENYLAMINE
11	237 (D10) DIPHENYLHYDRAZINE
12	260 (D2) 4,6 DINITROCRESOL
13	360 4,6 DINITRO-C-CRESOL
14	262 (D6) N-NITROSODIPHENYLAMINE
15	362 N-NITROSODIPHENYLAMINE
16	707 DIPHENYLAMINE
17	337 1,2 DIPHENYLHYDRAZINE
18	041 4-BROMOPHENYLPHENYLETHER
19	309 HEXACHLOROBENZENE
20	209 (13C6) HEXACHLOROBENZENE
21	604 (D8) DIBENZOTHIOPHENE
22	704 DIBENZOTHIOPHENE
23	364 PENTACHLOROPHENOL
24	264 (13C6) PENTACHLOROPHENOL
25	281 D10 PHENANTHRENE
26	381 PHENANTHRENE
27	278 (D10) ANTHRACENE
28	378 ANTHRACENE
29	621 (D42) N-EICOSANE
30	268 (D4) DI-N-BUTYL PHTHALATE
31	368 DI-N-BUTYL PHTHALATE
32	721 N-EICOSANE
33	239 (D10) FLUORANTHENE
34	339 FLUORANTHENE
35	205 (D8) BENZIDINE
36	305 BENZIDINE
37	284 (D10) PYRENE
38	384 PYRENE
39	623 (D50) N-TETRACOSANE
40	067 BUTYLBENZYLPHTHALATE
41	723 N-TETRACOSANE
42	276 (D12) CHRYSENE
43	272 (D12)BENZO(A)ANTHRACENE
44	376 CHRYSENE
45	372 BENZO(A)ANTHRACENE
46	228 (D6) 3,3' DICHLOROBENZIDINE

47	328	3,3' DICHLORORENZIDINE
48	266	(A4) BIS(2-ETHYLHEXYL)PHTHALATE
49	366	BIS(2-ETHYLHEXYL)PHTHALATE
50	269	(D4) DI-N-OCTYLPHTHALATE
51	369	DI-N-OCTYLPHTHALATE
52	274	(D12) BENZO(B)FLUORANTHENE
53	374	BENZO(B)FLUORANTHENE
54	275	(D12) BENZO(K)FLUORANTHENE
55	375	BENZO(K)FLUORANTHENE
56	273	(D12) BENZO(A)PYRENE
57	373	BENZO(A)PYRENE
58	626	(D62) N-TRIACONTANE
59	726	N-TRIACONTANE
60	083	INDENOPYRENE
61	082	DIBENZO(A,H)ANTHRACENE
62	279	(D12)BENZO(G,H,I)PERYLENE
63	379	BENZO(G,H,I)PERYLENE

NO	M/E	SCAN	TIME	PEF	RRT	METH	AREA(HGHT)	AMOUNT		%TOT
1	190	1190	20:49	1	1.000	A BB	132330.	100.000	UG/ML	1.25
2	66	1404	24:34	1	1.180	A BV	174336.	100.000	UG/ML	1.25
3	176	1422	24:53	1	1.195	A BV	128723.	100.000	UG/ML	1.61
4	166	1428	24:59	3	1.004	A BB	191185.	100.000	UG/ML	1.61
5	57	1428	24:59	2	1.017	A BV	217537.	100.250	UG/ML	1.25
6	209	1431	25:03	1	1.203	A VB	59235.	100.000	UG/ML	1.25
7	204	1434	25:06	6	1.002	A VB	57010.	102.000	UG/ML	1.27
8	153	1437	25:09	1	1.208	A VV	202942.	100.000	UG/ML	1.25
9	149	1439	25:11	8	1.001	A VV	237539.	100.000	UG/ML	1.25
10	179	1457	25:30	1	1.224	A VV	124214.	100.000	UG/ML	1.61
11	82	1459	25:32	1	1.226	A BB	380511.	100.000	UG/ML	1.61
12	200	1461	25:34	1	1.228	A BB	12760.	100.000	UG/ML	1.61
13	198	1463	25:36	12	1.001	A VB	16713.	100.100	UG/ML	1.61
14	175	1464	25:37	1	1.230	A VV	151118.	100.000	UG/ML	1.61
15	169	1465	25:38	14	1.001	A VV	294521.	101.000	UG/ML	1.62
16	169	1465	25:38	10	1.005	A VV	294061.	100.000	UG/ML	1.61
17	77	1465	25:38	11	1.004	A VV	466838.	100.000	UG/ML	1.61
18	248	1523	26:39	1	1.280	A BB	41779.	100.500	UG/ML	1.25
19	284	1546	27:03	20	1.000	A VB	39699.	100.000	UG/ML	1.61
20	292	1546	27:03	1	1.299	A BB	29095.	100.000	UG/ML	1.61
21	192	1579	27:38	1	1.327	A BV	183008.	100.000	UG/ML	1.25
22	184	1583	27:42	21	1.003	A BB	211801.	100.000	UG/ML	1.25
23	266	1585	27:44	24	1.000	A BB	22404.	100.400	UG/ML	1.61
24	272	1585	27:44	1	1.332	A BB	18341.	100.000	UG/ML	1.61
25	188	1602	28:02	1	1.346	A VV	196845.	100.000	UG/ML	1.25
26	178	1607	28:07	25	1.003	A VV	245017.	105.000	UG/ML	1.31
27	188	1612	28:13	1	1.355	A VV	192533.	100.000	UG/ML	1.25
28	178	1616	28:17	27	1.002	A VB	243085.	101.500	UG/ML	1.26
29	66	1735	30:22	1	1.458	A VV	169801.	100.000	UG/ML	1.25
30	153	1738	30:25	1	1.461	A VV	270559.	100.000	UG/ML	1.25
31	149	1740	30:27	30	1.001	A VB	376900.	99.000	UG/ML	1.23
32	57	1759	30:47	29	1.014	A VV	225089.	100.000	UG/ML	1.25
33	212	1825	31:56	1	1.534	A VB	135111.	100.000	UG/ML	1.61
34	202	1829	32:00	33	1.002	A VV	147718.	99.500	UG/ML	1.60
35	192	1862	32:35	1	1.565	A BB	36193.	100.000	UG/ML	1.25
36	184	1862	32:35	35	1.000	A BV	32639.	100.000	UG/ML	1.25
37	212	1865	32:38	1	1.567	A VV	121888.	100.000	UG/ML	1.61
38	202	1869	32:42	37	1.002	A VV	148070.	101.000	UG/ML	1.62
39	66	2008	35:08	1	1.687	A VV	126632.	100.000	UG/ML	1.25
40	149	2013	35:14	1	1.692	A BB	12593.	100.250	UG/ML	1.25
41	57	2034	35:36	39	1.013	A VB	141715.	100.250	UG/ML	1.25
42	240	2088	36:32	1	1.755	A BV	52320.	100.000	UG/ML	1.25

43	240	2088	36:32	1	1.755	A BV	52380.	100.000	UG/ML	1.25
44	228	2093	36:38	42	1.002	A BV	64884.	99.500	UG/ML	1.24
45	228	2093	36:38	43	1.002	A BV	65018.	100.700	UG/ML	1.25
46	258	2097	36:42	1	1.762	A BB	11593.	100.000	UG/ML	1.25
47	252	2098	36:43	46	1.000	A BV	9796.	99.500	UG/ML	1.24
48	153	2131	37:18	1	1.791	A BB	48827.	100.000	UG/ML	1.25
49	149	2133	37:20	48	1.001	A BV	111563.	200.000	UG/ML	2.49
50	153	2265	39:38	1	1.903	A BV	57834.	100.000	UG/ML	1.61
51	149	2267	39:40	50	1.001	A BB	91503.	100.000	UG/ML	1.61
52	264	2321	40:37	1	1.950	A BV	34614.	100.000	UG/ML	1.25
53	252	2330	40:46	52	1.004	A BV	40688.	100.000	UG/ML	1.25
54	264	2331	40:48	1	1.959	A VV	39434.	100.000	UG/ML	1.25
55	252	2336	40:53	54	1.002	A VV	44592.	100.750	UG/ML	1.25
56	264	2408	42:08	1	2.024	A BV	26250.	100.000	UG/ML	1.25
57	252	2415	42:16	56	1.003	A VV	34427.	99.750	UG/ML	1.24
58	66	2460	43:03	1	2.067	A BV	42364.	100.000	UG/ML	1.25
59	57	2526	44:12	58	1.027	A BV	45153.	100.000	UG/ML	1.25
60	276	2831	49:33	1	2.379	A VV	17673.	100.000	UG/ML	1.25
61	278	2851	49:54	1	2.396	A BV	19679.	100.750	UG/ML	1.25
62	298	2935	51:22	1	2.466	QEDT	12284.	100.000	UG/ML	1.25
63	276	2953	51:41	62	1.006	A BV	18144.	100.000	UG/ML	1.25

NO	RET(L)	RATIO	RRT(L)	RATIO	AMNT	AMNT(L)	R.FAC	R.FAC(L)	RATIO
1	20:49	1.00	1.000	1.00	100.00	100.00	1.000	1.000	1.00
2	24:34	1.00	1.180	1.00	100.00	100.00	1.317	1.317	1.00
3	24:53	1.00	1.195	1.00	100.00	100.00	0.973	0.973	1.00
4	24:59	1.00	1.200	0.84	100.00	100.00	1.485	1.485	1.00
5	24:59	1.00	1.200	0.85	100.25	100.25	1.245	1.245	1.00
6	25:03	1.00	1.203	1.00	100.00	100.00	0.448	0.448	1.00
7	25:06	1.00	1.205	0.83	102.00	102.00	0.944	0.944	1.00
8	25:09	1.00	1.208	1.00	100.00	100.00	1.534	1.534	1.00
9	25:11	1.00	1.208	0.83	100.00	100.00	1.170	1.170	1.00
10	25:30	1.00	1.224	1.00	100.00	100.00	0.939	0.939	1.00
11	25:32	1.00	1.226	1.00	100.00	100.00	2.875	2.875	1.00
12	25:34	1.00	1.229	1.00	100.00	100.00	0.096	0.096	1.00
13	25:36	1.00	1.229	0.81	100.10	100.10	1.308	1.308	1.00
14	25:37	1.00	1.230	1.00	100.00	100.00	1.142	1.142	1.00
15	25:38	1.00	1.231	0.81	101.00	101.00	1.930	1.930	1.00
16	25:38	1.00	1.234	0.81	100.00	100.00	2.367	2.367	1.00
17	25:38	1.00	1.231	0.82	100.00	100.00	1.227	1.227	1.00
18	26:39	1.00	1.280	1.00	100.50	100.50	0.314	0.314	1.00
19	27:03	1.00	1.299	0.77	100.00	100.00	1.364	1.364	1.00
20	27:03	1.00	1.299	1.00	100.00	100.00	0.220	0.220	1.00
21	27:38	1.00	1.327	1.00	100.00	100.00	1.383	1.383	1.00
22	27:42	1.00	1.330	0.75	100.00	100.00	1.157	1.157	1.00
23	27:44	1.00	1.332	0.75	100.40	100.40	1.217	1.217	1.00
24	27:44	1.00	1.332	1.00	100.00	100.00	0.139	0.139	1.00
25	28:02	1.00	1.346	1.00	100.00	100.00	1.488	1.488	1.00
26	28:07	1.00	1.350	0.74	105.00	105.00	1.185	1.185	1.00
27	28:13	1.00	1.355	1.00	100.00	100.00	1.455	1.455	1.00
28	28:17	1.00	1.358	0.74	101.50	101.50	1.244	1.244	1.00
29	30:22	1.00	1.458	1.00	100.00	100.00	1.283	1.283	1.00
30	30:25	1.00	1.461	1.00	100.00	100.00	2.045	2.045	1.00
31	30:27	1.00	1.462	0.68	99.00	99.00	1.407	1.407	1.00
32	30:47	1.00	1.478	0.69	100.00	100.00	1.326	1.326	1.00
33	31:56	1.00	1.534	1.00	100.00	100.00	1.021	1.021	1.00
34	32:00	1.00	1.534	0.65	99.50	99.50	1.099	1.099	1.00
35	32:35	1.00	1.565	1.00	100.00	100.00	0.274	0.274	1.00
36	32:35	1.00	1.561	0.64	100.00	100.00	0.902	0.902	1.00
37	32:38	1.00	1.567	1.00	100.00	100.00	0.921	0.921	1.00
38	32:42	1.00	1.571	0.64	101.00	101.00	1.203	1.203	1.00

39	35:08	1.00	1.687	1.00	100.00	100.00	0.957	0.957	1.00
40	35:14	1.00	1.692	1.00	100.25	100.25	0.095	0.095	1.00
41	35:36	1.00	1.709	0.59	100.25	100.25	1.116	1.116	1.00
42	36:32	1.00	1.752	1.00	100.00	100.00	0.395	0.395	1.00
43	36:32	1.00	1.758	1.00	100.00	100.00	0.396	0.396	1.00
44	36:38	1.00	1.755	0.57	99.50	99.50	1.246	1.246	1.00
45	36:38	1.00	1.758	0.57	100.70	100.70	1.233	1.233	1.00
46	36:42	1.00	1.762	1.00	100.00	100.00	0.088	0.088	1.00
47	36:43	1.00	1.763	0.57	99.50	99.50	0.849	0.849	1.00
48	37:18	1.00	1.791	1.00	100.00	100.00	0.369	0.369	1.00
49	37:20	1.00	1.792	0.56	200.00	200.00	1.142	1.142	1.00
50	39:38	1.00	1.903	1.00	100.00	100.00	0.437	0.437	1.00
51	39:40	1.00	1.905	0.53	100.00	100.00	1.409	1.409	1.00
52	40:37	1.00	1.950	1.00	100.00	100.00	0.262	0.262	1.00
53	40:46	1.00	1.950	0.51	100.00	100.00	1.176	1.176	1.00
54	40:48	1.00	1.959	1.00	100.00	100.00	0.298	0.298	1.00
55	40:53	1.00	1.963	0.51	100.75	100.75	1.122	1.122	1.00
56	42:08	1.00	2.024	1.00	100.00	100.00	0.198	0.198	1.00
57	42:16	1.00	2.029	0.49	99.75	99.75	1.315	1.315	1.00
58	43:03	1.00	2.067	1.00	100.00	100.00	0.320	0.320	1.00
59	44:12	1.00	2.123	0.48	100.00	100.00	1.066	1.066	1.00
60	49:33	1.00	2.379	1.00	100.00	100.00	0.134	0.134	1.00
61	49:54	1.00	2.396	1.00	100.75	100.75	0.148	0.148	1.00
62	51:22	1.00	2.466	1.00	100.00	100.00	0.093	0.093	1.00
63	51:41	1.00	2.481	0.41	100.00	100.00	1.477	1.477	1.00

TAB 2

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
001	B	ACENAPHTHENE	P
002	V	ACROLEIN	P
003	V	ACRYLONITRILE	P
004	V	BENZENE	P
005	B	BENZIDINE	P
006	V	CARBON TETRACHLORIDE	P
007	V	CHLOROBENZENE	P
008	B	1,2,4-TRICHLOROBENZENE	P
009	B	HEXACHLOROBENZENE	P
010	V	1,2-DICHLOROETHANE	P
011	V	1,1,1-TRICHLOROETHANE	P
012	B	HEXACHLOROETHANE	P
013	V	1,1-DICHLOROETHANE	P
014	V	1,1,2-TRICHLOROETHANE	P
015	V	1,1,2,2-TETRACHLOROETHANE	P
016	V	CHLOROETHANE	P
017	V	BIS (CHLOROMETHYL) ETHER (NR)	P
018	B	BIS(2-CHLOROETHYL)ETHER	P
019	V	2-CHLOROETHYLVINYL ETHER	P
020	B	2-CHLORONAPHTHALENE	P
021	A	2,4,6-TRICHLOROPHENOL	P
022	A	P-CHLORO-M-CRESOL	P
023	V	CHLOROFORM	P
024	A	2-CHLOROPHENOL	P
025	B	1,2-DICHLOROBENZENE	P
026	B	1,3-DICHLOROBENZENE	P
027	B	1,4-DICHLOROBENZENE	P
028	B	3,3'-DICHLOROBENZIDINE	P
029	V	1,1-DICHLOROETHYLENE	P
030	V	1,2-TRANS-DICHLOROETHYLENE	P
031	A	2,4-DICHLOROPHENOL	P
032	V	1,2-DICHLOROPROPANE	P
033	V	1,3-DICHLOROPROPYLENE	P
034	A	2,4-DIMETHYLPHENOL	P
035	B	2,4-DINITROTOLUENE	P
036	B	2,6-DINITROTOLUENE	P
037	B	1,2-DIPHENYLHYDRAZINE	P
038	V	ETHYLBENZENE	P
039	B	FLORANTHENE	P
040	B	4-CHLOROPHENYL PHENYL ETHER	P
041	B	4-BROMOPHENYL PHENYL ETHER	P
042	B	BIS (2-CHLOROISOPROPYL) ETHER	P
043	B	BIS (2-CHLOROETHOXY) METHANE	P
044	V	METHYLENE CHLORIDE	P
045	V	METHYL CHLORIDE	P
046	V	METHYL BROMIDE	P
047	V	BROMOFORM	P
048	V	DICHLOROBROMOMETHANE	P
049	V	TRICHLOROFLUOROMETHANE (NR)	P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
050	V	DICHLORODIFLUOROMETHANE (NR)	P
051	V	CHLORODIBROMOMETHANE	P
052	B	HEXACHLOROBUTADIENE	P
053	B	HEXACHLOROCYCLOPENTADIENE	P
054	B	ISOPHORONE	P
055	B	NAPHTHALENE	P
056	B	NITROBENZENE	P
057	A	2-NITROPHENOL	P
058	A	4-NITROPHENOL	P
059	A	2,4-DINITROPHENOL	P
060	A	4,6-DINITRO-O-CRESOL	P
061	B	N-NITROSODIMETHYLAMINE	P
062	B	N-NITROSODIPHENYLAMINE	P
063	B	N-NITROSODI-N-PROPYLAMINE	P
064	A	PENTACHLOROPHENOL	P
065	A	PHENOL	P
066	B	BIS (2-ETHYLHEXYL) PHTHALATE	P
067	B	BUTYL BENZYL PHATHALATE	P
068	B	DI-N-BUTYL PHTHALATE	P
069	B	DI-N-OCTYL PHTHALATE	P
070	B	DIETHYL PHTHALATE	P
071	B	DIMETHYL PHTHALATE	P
072	B	BENZO(A)ANTHRACENE	P
073	B	BENZO(A)PYRENE	P
074	B	BENZO(B)FLUORANTHENE	P
075	B	BENZO(K)FLUORANTHENE	P
076	B	CHRYSENE	P
077	B	ACENAPHTHYLENE	P
078	B	ANTHRACENE	P
079	B	BENZO(GHI)PERYLENE	P
080	B	FLUORENE	P
081	B	PHENANTHRENE	P
082	B	DIBENZO(A,H)ANTHRACENE	P
083	B	INDENO(1,2,3-CD)PYRENE	P
084	B	PYRENE	P
085	V	TETRACHLOROETHYLENE	P
086	V	TOLUENE	P
087	V	TRICHLOROETHYLENE	P
088	V	VINYL CHLORIDE	P
089	P	ALDRIN	P
090	P	DIELDRIN	P
091	P	CHLORDANE	P
092	P	4,4'-DDT	P
093	P	4,4'-DDE	P
094	P	4,4'-DDD	P
095	P	ALPHA-ENDOSULFAN	P
096	P	BETA-ENDOSULFAN	P
097	P	ENDOSULFAN SULFATE	P
098	P	ENDRIN	P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
099	P	ENDRIN ALDEHYDE	P
100	P	HEPTACHLOR	P
101	P	HEPTACHLOR EPOXIDE	P
102	P	ALPHA-BHC	P
103	P	BETA-BHC	P
104	P	GAMMA-BHC	P
105	P	DELTA-BHC	P
106	P	PCB-1242	P
107	P	PCB-1254	P
108	P	PCB-1221	P
109	P	PCB-1232	P
110	P	PCB-1248	P
111	P	PCB-1260	P
112	P	PCB-1016	P
113	P	TOXAPHENE	P
129	B	2,3,7,8-TCDD	P
130	V	XYLENES	P
150	A	PHENOL-D6	S
151	A	PENTAFLUOROPHENOL	S
152	V	PENTAFLOROBENZENE	S
153	A	TRIFLUORO-M-CRESOL	S
154	V	2,2-DIFLUOROTETRACHLOROETHANE	S
155	B	2-FLUOROBIPHENYL	S
156	B	1-FLUORONAPHTHALENE	S
157	A	2-FLOUROPHENOL	S
158	B	2-FLUORONAPHTHALENE	S
159	B	PYRIDINE-D5	S
160	B	ANILINE-D5	S
161	B	NAPHTHALENE-D8	S
162	V	TOLUENE-D8	S
163	B	NITROBENZENE-D5	S
164	B	2,2'-DIFLUOROBIPHENYL	I
165	V	BENZENE-D6	S
166	B	DECAFLUOROBIPHENYL	S
167	V	M-DIFLUOROBENZENE	S
168	V	METHYLENE CHLORIDE-D2	S
169	V	1,1,2,2-TETRACHLOROETHANE-D2	S
170	V	ETHYLBENZENE-D10	S
172	V	1,2 DICHLOROETHANE-D4	S
173	V	2,2 DICHLOPROPANE-D6	S
174	V	CHLOROBENZENE-D5	S
175	B	1,2 DICHLOROBENZENE-D4	S
176	B	CHRYSENE D12	S
177	B	FLUORENE D10	S
178	A	2-NITROPHENOL D4	S
179	B	DI-N-BUTYL-PHTHALATE-D4	S
180	B	4-FLUOROANILINE	S
181	V	BROMOCHLOROMETHANE	I
182	V	2-BROMO-1-CHLOROPROPANE	I

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
183	V	1,4-DICHLOROBUTANE	I
201	B	ACENAPHTHENE-010	D
202	V		D
203	V	ACRYLONITRILE-03	D
204	V	BENZENE-06	D
205	B	BENZIDINE-08 (MIGS-08)	D
206	V	CARBON-13C TETRACHLORIDE	D
207	V	CHLOROBENZENE-05	D
208	B	1,2,4-TRICHLOROBENZENE-03	D
209	B	HEXACHLOROBENZENE-13C6	D
210	V	1,2-DICHLOROETHANE-04	D
211	V	1,1,1-TRICHLOROETHANE-03	D
212	B	HEXACHLOROETHANE-1-13C	D
213	V	1,1-DICHLOROETHANE-2,2,2-03	D
214	V	1,1,2-TRICHLOROETHANE-13C2	D
215	V	1,1,2,2-TETRACHLOROETHANE-02	D
216	V	CHLOROETHANE-05	D
217	V		D
218	B	BIS(2-CHLOROETHYL)-08 ETHER	D
219	V		D
220	B	2-CHLORONAPHTHALENE-07	D
221	A	2,4,6-TRICHLOROPHENOL-3,5-02	D
222	A	4-CHLORO-3-METHYLPHENOL-2,6-02	D
223	V	CHLOROFORM-13C	D
224	A	2-CHLOROPHENOL-3,4,5,6-04	D
225	B	1,2-DICHLOROBENZENE-04	D
226	B	1,3-DICHLOROBENZENE-04	D
227	B	1,4-DICHLOROBENZENE-04	D
228	B	3,3'-DICHLOROBENZIDINE-06	D
229	V	1,1-DICHLOROETHYLENE-02	D
230	V	1,2-DICHLOROETHYLENE-02	D
231	A	2,4-DICHLOROPHENOL-3,5,6-03	D
232	V	1,2-DICHLOROPROPANE-04	D
233	V	1,3-DICHLOROPROPYLENE-1,2-02	D
234	A	2,4-DIMETHYLPHENOL-3,5,6-03	D
235	B	2,4-DINITROTOLUENE-3,5,6-03	D
236	B	2,6-DINITROTOLUENE-03	D
237	B	1,2-DIPHENYL-010-HYDRAZINE	D
238	V	ETHYLBENZENE-010	D
239	B	FLUORANTHENE-010	D
240	B	4-CHLOROPHENYL PHENYL-05 ETHER	D
241	B		D
242	B	BIS(2-CHLOROISOPROPYL)ETHER012	D
243	B		D
244	V	METHYLENE CHLORIDE-02	D
245	V	CHLOROMETHANE-03	D
246	V	BROMOMETHANE-03	D
247	V	BROMOFORM-13C	D
248	V	BROMODICHLOROMETHANE-13C	D

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
249	V		D
250	V		D
251	V	CHLORODIBROMOMETHANE-13C	D
252	B	HEXACHLORO-1,3-BUTADIENE-13C4	D
253	B	HEXACHLOROCYCLOPENTADIENE-13C	D
254	B	ISOPHORONE-D8	D
255	B	NAPHTHALENE-D8	D
256	B	NITROBENZENE-D5	D
257	A	2-NITROPHENOL-3,4,5,6-D4	D
258	A	4-NITROPHENOL-2,3,5,6-D4	D
259	A	2,4-DINITROPHENOL-3,5,6-D3	D
260	A	4,6-DINITRO-O-CRESOL-D2	D
261	B		D
262	B	N-NITROSODIPHENYLAMINE-D6	D
263	B		D
264	A	PENTACHLOROPHENOL-13C6	D
265	A	PHENOL-2,3,4,5,6-D5	D
266	B	BIS(2-ETHYLHEXYL)PHTHALATE-D4	D
267	B		D
268	B	DI-N-BUTYL PHTHALATE-D4	D
269	B	DI-N-OCTYL PHTHALATE-D4	D
270	B	DIETHYL PHTHALATE-3,4,5,6-D4	D
271	B	DIMETHYL PHTHALATE-3,4,5,6-D4	D
272	B	BENZO(A)ANTHRACENE-D12	D
273	B	BENZO(A)PYRENE-D12	D
274	B	BENZO(B)FLUORANTHENE-D12	D
275	B	BENZO(K)FLUORANTHENE-D12	D
276	B	CHRYSENE-D12	D
277	B	ACENAPHTHYLENE-D8	D
278	B	ANTHRACENE-D10	D
279	B	BENZO(GHI)PERYLENE-D12	D
280	B	FLUORENE-D10	D
281	B	PHENANTHRENE-D10	D
282	B		D
283	B		D
284	B	PYRENE-D10	D
285	V	TETRACHLOROETHYLENE-13C2	D
286	V	TOLUENE-2,3,4,5,6-D5	D
287	V	1,1,2-TRICHLOROETHYLENE-13C2	D
288	V	VINYL-D3 CHLORIDE	D
289	P		D
290	P		D
291	P		D
292	P		D
293	P		D
294	P		D
295	P		D
296	P		D
297	P		D

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
298	P		D
299	P		D
301	B	ACENAPHTHENE	P
302	V		P
303	V	ACRYLONITRILE	P
304	V	BENZENE	P
305	B	BENZIDINE	P
306	V	CARBON TETRACHLORIDE	P
307	V	CHLOROBENZENE	P
308	B	1,2,4-TRICHLOROBENZENE	P
309	B	HEXACHLOROBENZENE	P
310	V	1,2-DICHLOROETHANE	P
311	V	1,1,1-TRICHLOROETHANE	P
312	B	HEXACHLOROETHANE	P
313	V	1,1-DICHLOROETHANE	P
314	V	1,1,2-TRICHLOROETHANE	P
315	V	1,1,2,2-TETRACHLOROETHANE	P
316	V	CHLOROETHANE	P
317	V		P
318	B	BIS(2-CHLOROETHYL)ETHER	P
319	V		P
320	B	2-CHLORONAPHTHALENE	P
321	A	2,4,6-TRICHLOROPHENOL	P
322	A	P-CHLORO-M-CRESOL	P
323	V	CHLOROFORM	P
324	A	2-CHLOROPHENOL	P
325	B	1,2-DICHLOROBENZENE	P
326	B	1,3-DICHLOROBENZENE	P
327	B	1,4-DICHLOROBENZENE	P
328	B	3,3'-DICHLOROBENZIDINE	P
329	V	1,1-DICHLOROETHYLENE	P
330	V	1,2-DICHLOROETHYLENE	P
331	A	2,4-DICHLOROPHENOL	P
332	V	1,2-DICHLOROPROPANE	P
333	V	1,3-DICHLOROPROPYLENE	P
334	A	2,4-DIMETHYLPHENOL	P
335	B	2,4-DINITROTOLUENE	P
336	B	2,6-DINITROTOLUENE	P
337	B	1,2-DIPHENYLHYDRAZINE	P
338	V	ETHYLBENZENE	P
339	B	FLORANTHENE	P
340	B	4-CHLOROPHENYL PHENYL ETHER	P
341	B		P
342	B	BIS (2-CHLOROISOPROPYL) ETHER	P
343	B		P
344	V	METHYLENE CHLORIDE	P
345	V	METHYL CHLORIDE	P
346	V	METHYL BROMIDE	P
347	V	BROMOFORM	P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
348	V	DICHLOROBROMOMETHANE	P
349	V		P
350	V		P
351	V	CHLORODIBROMOMETHANE	P
352	B	HEXACHLOROBUTADIENE	P
353	B	HEXACHLOROCYCLOPENTADIENE	P
354	B	ISOPHORONE	P
355	B	NAPHTHALENE	P
356	B	NITROBENZENE	P
357	A	2-NITROPHENOL	P
358	A	4-NITROPHENOL	P
359	A	2,4-DINITROPHENOL	P
360	A	4,6-DINITRO-O-CRESOL	P
361	B		P
362	B	N-NITROSODIPHENYLAMINE	P
363	B		P
364	A	PENTACHLOROPHENOL	P
365	A	PHENOL	P
366	B	BIS (2-ETHYLHEXYL) PHTHALATE	P
367	B		P
368	B	DI-N-BUTYL PHTHALATE	P
369	B	DI-N-OCTYL PHTHALATE	P
370	B	DIETHYL PHTHALATE	P
371	B	DIMETHYL PHTHALATE	P
372	B	BENZO(A)ANTHRACENE	P
373	B	BENZO(A)PYRENE	P
374	B	BENZO(B)FLUORANTHENE	P
375	B	BENZO(K)FLUORANTHENE	P
376	B	CHRYSENE	P
377	B	ACENAPHTHYLENE	P
378	B	ANTHRACENE	P
379	B	BENZO(GHI)PERYLENE	P
380	B	FLUORENE	P
381	B	PHENANTHRENE	P
382	B		P
383	B		P
384	B	PYRENE	P
385	V	TETRACHLOROETHYLENE	P
386	V	TOLUENE	P
387	V	TRICHLOROETHYLENE	P
388	V	VINYL CHLORIDE	P
389	P		P
390	P		P
391	P		P
392	P		P
393	P		P
394	P		P
395	P		P
396	P		P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND		COMPOUND TYPE
397	P			P
398	P			P
399	P			P
500	A	BENZOIC ACID		P
501	A	HEXANOIC ACID		P
502	B	BETA NAPHTHYLAMINE		P
503	B	ALPHA PICOLINE		P
504	B	DIBENZOTHIOPHENE		P
505	B	DIBENZOFURAN		P
506	B	N-DODECANE		P
507	B	DIPHENYLAMINE		P
508	B	DIPHENYLETHER		P
509	B	ALPHA TERPINEOL		P
510	B	STYRENE		P
511	B	DI-N-BUTYL AMINE		P
512	B	BIPHENYL		P
513	B	P-CYMENE		P
514	V	METHYL ETHYL KETONE		P
515	V	DIETHYL ETHER		P
516	V	ACETONE		P
517	B	N-DECANE	C10	P
518	B	N-TETRADECANE	C14	P
519	B	N-HEXADECANE	C16	P
520	B	N-OCTADECANE	C18	P
521	B	N-EICOSANE	C20	P
522	B	N-DOCOSANE	C22	P
523	B	N-TETRACOSANE	C24	P
524	B	N-HEXACOSANE	C26	P
525	B	N-OCTACOSANE	C28	P
526	B	N-TRIACONTANE	C30	P
600	A	BENZOIC-D5 ACID		D
601	A	HEXANOIC-D11 ACID		D
602	B	2-NAPHTHYL-D7-AMINE		D
603	B	2-METHYLPYRIDINE-D7		D
604	B	DIBENZOTHIOPHENE-D8		D
605	B	DIBENZOFURAN-D8		D
606	B	N-DODECANE-D26		D
607	B	DIPHENYL-D10-AMINE		D
608	B	DIPHENYL-D10 ETHER		D
609	B	ALPHA-TERPINEOL-D3		D
610	B	STYRENE-2,3,4,5,6-D5		D
611	B	DI-N-BUTYL-D18-AMINE		D
612	B	DIPHENYL-D10		D
613	B	P-CYMENE-D14		D
614	V	2-BUTANONE-4,4,4-D3 (MEK)		D
615	V	DIETHYL-D10 ETHER		D
616	V	ACETONE-D6		D
617	B	N-DECANE-D22		D
618	B			D

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND		COMPOUND TYPE
619	B	N-HEXADECANE-D34		D
620	B			D
621	B	N-EICOSANE-D42		D
622	B			D
623	B	N-TETRACOSANE-D50		D
624	B			D
625	B			D
626	B	N-TRIACONTANE-D62		D
700	A	BENZOIC ACID		P
701	A	HEXANOIC ACID		P
702	B	BETA NAPHTHYLAMINE		P
703	B	ALPHA PICOLINE		P
704	B	DIBENZOTHIOPHENE		P
705	B	DIBENZOFURAN		P
706	B	N-DODECANE	C12	P
707	B	DIPHENYLAMINE		P
708	B	DIPHENYLETHER		P
709	B	ALPHA TERPINEOL		P
710	B	STYRENE		P
711	B	DI-N-BUTYL AMINE		P
712	B	BIPHENYL		P
713	B	P-CYMENE		P
714	V	METHYL ETHYL KETONE		P
715	V	DIETHYL ETHER		P
716	V	ACETONE		P
717	B	N-DECANE	C10	P
718	B			P
719	B	N-HEXADECANE	C16	P
720	B			P
721	B	N-EICOSANE	C20	P
722	B			P
723	B	N-TETRACOSANE	C24	P
724	B			P
725	B			P
726	B	N-TRIACONTANE	C30	P

427 RECORDS PRINTED

TAB 3 - Quantitation Report Magnetic Tape Transmittal Form Description

The main purpose of the tape transmittal form is to ensure the complete and correct data processing of a tape volume (reel). Depending upon the number of files per volume at least one tape transmittal form must accompany each tape volume sent to the Sample Control Center. Field descriptions are as follows.

Laboratory	:	The laboratory name.
Return Tape To	:	The address to which the tape volume is to be returned after processing by the SCC.
External Tape #	:	The tape number on the SCC supplied external tape label.
Tape Density	:	Either 800 or 1600 bpi.
Block Size	:	The number of bytes per block.
Number of Files	:	The number of Quantitation report files on the tape.
Contact Person and Phone Number	:	Who to contact at the laboratory, regarding any difficulties in the processing of the tape volume, and their phone number including area code.
File Position	:	The relative file position of the Quantitation Report File on the tape volume. The first file on the tape has a relative file position of 1. The second file on the tape has a relative file position of 2.

EPA Sample Number : The 5 digit sample number assigned by the SCC. N/A if not applicable.

Type : The 3 position EGLD code signifying the sample type. Required.

Fraction : 1 position code:

A = Acid
B = Base/Neutral
C = Combined Acid Base/Neutral
V = Volatile
P = Pesticide

N/A if not applicable.

Conc/Dilu : The concentration or dilution ratio of the sample fraction before analysis. N/A if not applicable.

Date Analyzed : The date of analysis.

USEPA Effluent Guidelines Division
Quantitation Report Magnetic Tape Transmittal Form

Revision: A
Date: 5May83

Laboratory: _____
Return Tape To: _____
_____
_____
_____

External Tape #: _____
Tape Density (BPI): _____
Block Size: _____
Number of Files: _____

Contact Person and Phone Number: _____ (____) ____ - _____

File Position	EPA Sample #	Type	Fraction	Conc/Dilu	Date Analyzed

The data recorded on this tape have been verified and are true and complete.

Date: _____ Analyst: _____ QA: _____

Quantitation Report Magnetic Tape Additional Files Transmittal Form

This form is only to be used when there are more than 14 quantitation report files on a tape and it must be used with a Quantitation Report Magnetic Tape Transmittal Form. As many Additional Files Transmittal Forms can be used to accommodate all of the files on the tape. The field definitions are identical to those on the Quantitation Report Magnetic Tape Transmittal Form.

Appendix E

EGD Data Elements

Following is a brief description of each data element which is to be stored in the EGD data base. The originating source for the data element is also given. The complete specification for each data element and an example of its use are given on a separate page following this summary.

Amount – The quantitative measurement of the compound determined by GC or GC/MS analysis. The amount is computed for the compound using the referenced internal standard or isotopic diluent and is multiplied by the concentration or dilution factor to yield final solution concentrations in ug/L.

Amount (Library) – The reference amount in the standard and the amount on which quantitation is based.

Bottle Number – A numeric code which uniquely identifies the bottles used for a particular sample.

Carrier Gas Flow Rate – The volumetric (volume/time) rate of flow of the carrier gas in the gas chromatograph, or the linear gas velocity (distance/time) when a capillary column is used.

Column Final Hold – The final temperature of the gas chromatograph column and the length of time that it was held.

Column Initial Hold – The initial temperature of the gas chromatograph column and the length of time that it was held.

Column Inside Diameter – The internal diameter of the gas chromatograph column.

Column Length – The length of the gas chromatograph column.

Column Temperature Program – The change in column temperature with respect to time giving the initial and final column temperatures.

Compound Comment Code – A coded value for any optional text that may be associated with each compound.

Compound Name – The name of the compound determined. The compound name corresponds to the EGD compound number, as given in the "USEPA Effluent Guidelines Division Compound Code Listing."

Compound Number – A numerical code which uniquely identifies each unique chemical compound, as given in the "Effluent Guidelines Division Compound Code Listing."

Compound Order Number – A numerical code that establishes the order of compound determination by the GC/MS. The code is used on the Quantitation Report to match segments of the compound data within the report.

Compound Type – A coded value which identifies a chemical compound as a priority pollutant (P), or surrogate (S), internal standard (I), or isotopic dilutent (D).

Concentration/Dilution Factor – The ratio of the volume of sample extracted or diluted to the volume analyzed.

Date Analyzed – The date that the sample fraction was analyzed by the laboratory.

Date Extracted – The date that the laboratory extracted the sample for analysis.

Date Sampled – The date the sample was taken by the field sampler.

Episode Comment Code – A coded value for comments associated with an episode.

Episode Number – The SCC assigned identification code with designates the specific sampling trip.

Fraction – A coded value which designates the compound as either an acid, base/neutral, volatile, pesticide or dioxin.

Fraction Comment Code – A coded value for any optional text that may be associated with each fraction.

Industrial Category Code – The classification of the industrial processes performed by the plant where a sample was taken.

Instrument – A coded value assigned by the laboratory that uniquely identifies each GC/MS instrument within a laboratory. All Calibration, Precision and Recovery, Standards and Blank Quantitation files will be tracked by this instrument number within Laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and other initial QA runs by the laboratory.

Laboratory – A numerical code used to identify the specific laboratory where the sample was analyzed.

Mass to Charge Ratio – Designates the quantitation ion. Abreviated as M/Z or M/E.

Method – A coded value which uniquely identifies the method protocol that was followed during analysis.

Peak Area – The area beneath the peak of a mass chromatogram. The peak area is proportional to the amount of the detected compound at an observed mass to charge ratio. It is used to compute the concentration of the compound present in the sample.

PH Level – The negative logarithm of the effective hydrogen ion concentration as expressed in grain equivalents per liter.

Plant Code – A numerical code used to distinguish specific plants which have been sampled.

Proprietary Indicator – A coded value which designates whether or not the analysis data from a sample is proprietary. Also indicates that confidentiality papers have been signed.

Quantitation Report Type – A coded value that uniquely identifies the particular type of quantitation report that is being submitted.

Reference Compound – A numeric code that is used as a pointer to the internal standard or isotopic diluent within a quantitation report.

Relative Retention Time – The quotient of the retention time of a compound divided by its internal standard or isotopic diluent.

Relative Retention Time (Library) – The relative retention time stored in the library. The value is based on the analysis of a standard containing both compounds.

Response Factor – The ratio between the response for the sample and a response for a standard under identical analytical conditions.

Response Factor (Library) – The response factor stored in the library. The value is determined from analysis of a standard.

Retention Time – The time it takes the identified compound to elute from the gas chromatograph.

Retention Time (Library) – The known time it takes an identified compound to elute from the gas chromatograph. The time is determined from analysis of a standard.

Sample Comment Code – A coded value for any optional text that may be associated with each sample.

Sample Number – The SCC assigned identification code which identifies the individual samples.

Sample Point (Site) – The specific point within an industrial wastestream where a sample was taken.

Sample Point Flow – The flow rate at the point at which the sample was taken. Value is recorded from a flow meter or other flow measuring device.

Sample Type – A coded value which describes the type of sample.

Scan Number – Gives the scan at which the compound was detected by the mass spectrometer.

Shift – The scheduled period of operation of the GC/MS instrument. Operation is divided into three shifts/day.

Time Analyzed – The time that the sample fraction was analyzed by the laboratory.

Unit of Measure – The unit of measurement for the amount.

The following chart shows the source of each data element.

SUMMARY OF DATA SOURCES FOR COLLECTION OF ORGANIC PRIORITY POLLUTANT DATA

	COLLECTION SOURCE			
DATA FIELD	SAMTRAC[1]	TR[2] LC	LAB[3] TAPE	GENERATED
Amount			X	
Amount (Library)			X	
Bottle Number			X	
Carrier Gas Flow Rate			X	
Column Final Hold			X	
Column Initial Hold			X	
Column Inside Diameter			X	
Column Length			X	
Column Temperature Program			X	
Compound Comment Code		X		
Compound Name				X
Compound Number			X	
Compound Reference Number			X	
Compound Type				X
Concentration/Dilution Factor			X	
Date Analyzed			X	
Date Extracted			X	
Date Sampled		X		
Episode Comment Codes		X		
Episode Number	X			
Fraction			X	
Fraction Comment Code		X		
Industrial Category Code	X			
Instrument			X	
Laboratory	X			

(1) SAMTRAC - Computerized logistics system at the Sample Control Center.

(2) TR LC - Traffic Reports and Lab Chronicles.

(3) Quantitation Reports on magnetic tape received from analytical laboratories.

SUMMARY OF DATA SOURCES FOR COLLECTION OF ORGANIC PRIORITY POLLUTANT DATA

	COLLECTION SOURCE			
DATA FIELD	SAMTRAC[1]	TR[2] LC	LAB[3] TAPE	GENERATED
Mass to Charge Ratio			X	
Method			X	
Peak Area			X	
PH Level		X		
Plant Code	X			
Proprietary Indicator		X		
Quantitation Report Type			X	
Reference Compound			X	
Relative Retention Time			X	
Relative Retention Time (Library)			X	
Response Factor			X	
Response Factor (Library)			X	
Retention Time			X	
Retention Time (Library)			X	
Sample Comment Code		X		
Sample Number	X	X	X	
Sample Point (Site)		X		
Sample Point Flow		X		
Sample Type			X	
Scan Number			X	
Shift			X	
Time Analyzed			X	
Unit of Measure			X	

(1) SAMTRAC - Computerized logistics system at the Sample Control Center.

(2) TR LC - Traffic Reports and Lab Chronicles.

(3) Quantitation Reports on magnetic tape received from analytical laboratories.

NOTES ON TYPE/LENGTH DESCRIPTION

The Type/Length Description for each EGD Data Element represents how each data field is stored internally in the computer or how each data field is represented on the quantitation report.

TYPES:

Z	-	Numeric data only - leading zeroes not printed.
9	-	Numeric data only - zeroes printed.
X	-	Alpha numeric data.
V	-	Implied decimal point.
.	-	Explicit decimal point.

LENGTH:

(N) Where N is a positive integer value from 1 to 255, gives the number of data positions allocated internally by the computer to store this portion of the data field.

EXAMPLES:

Example 1. 9(7)V9(3)

9	-	Numeric data.
(7)+(3)	-	10 data positions allocated.
V	-	Implied decimal point after 3rd position from the right.

Can also be expressed as 9999999V999.

The number 1,130.31 would be stored internally under this Type/Length description as:

'0001130310'

NOTES ON TYPE/LENGTH DESCRIPTION (CONT.)

The computer program would also know that there is a decimal point implied between '0001130' and '310'.

Example 2. X(6)

X	-	Alphanumeric data.
(6)	-	6 data positions allocated.

Can also be expressed as XXXX.

The field 'EPA1' would be stored internally or printed as:

'EPA1'

Example 3. ZZZZZZ9.999

Z	-	Numeric data - zeroes not printed.
9	-	Numeric data - zeroes printed.
.	-	Explicit decimial point printed.

6 (Z's) + 4 (9's) + 1 (.) = 11 data positions allowed.

The field '0000023010' would be printed as:

' 23.010'

ELEMENT NAME: AMOUNT

Definition: The quantitative measurement of the compound determined by GC or GC/MS analysis. The amount is computed for the compound using the referenced internal standard or isotopic diluent and is multiplied by the concentration or dilution factor to yield final solution concentration in ug/L.

Input	**Type/Length**
Quantitation Report	ZZZZZ9.999
As Stored Internally	9(7)V9(3)

Unit of Measure

Ug/L

Edit Criteria:

Range: 10.000-999,999.999 ug/L

Examples:

Volatiles: Concentration (AMOUNT) is reported on quantitation report in ug/l; if sample is diluted to bring a pollutant within the analytical range of the instrument, the concentration is multiplied by the dilution factor. For example, a concentration of 60 ug/l from analysis of a sample which has been diluted 1:10 results in a final concentration of 600 ug/l.

Semi-volatiles: Concentration (AMOUNT) is reported on quantitation report in ug/ml; sample is assumed concentrated by a factor of 1000 (concentration factor 1000:1), based on extraction of 1.00 liter of sample and a final extract volume of 1.0 ml. If extract is diluted to bring the concentration of a pollutant within calibration range of the instrument, the concentration factor is reduced by the amount of the dilution. For example, if the extract is diluted 1:10, the concentration factor becomes 100:1 (1000:10 = 100:1).

ELEMENT NAME: AMOUNT (LIBRARY)

Definition: The reference amount in the standard and the amount on which quantitation is based.

Input	**Type/Length**
Quantitation Report	ZZZZZ9.99
As Stored Internally	9(7)V9(3)

Unit of Measure

The amount is reported as a pure number but must always be accompanied by a UNIT. See UNIT.

Edit Criteria:

Range: 1.000 - 1000.000 ug/L

Examples: See AMOUNT.

ELEMENT NAME: BOTTLE NUMBER

Definition: A numeric code which uniquely identifies the bottles used for a particular sample. Used as a suffix to the SAMPLE NUMBER.

Input	**Type/Length**
Quantitation Report	X(2)
As Stored Internally	X(2)

Unit of Measure

Each.

Edit Criteria:

Range 01-99.

Example: See SAMPLE NUMBER.

ELEMENT NAME: CARRIER GAS FLOW RATE

Definition: The volumetric (volume/time) rate of flow of the carrier gas in the gas chromatograph for packed columns, or the linear gas velocity (distance/time) for capillary columns.

Input	**Type/Length**
Quantitation Report	X(9)
As Stored Internally	X(9)

Unit of Measure

Volatiles (packed column): ML/Min
Semi-volatiles (capillary column): CM/Sec

Edit Criteria:

Ranges: Volatiles: 20-40 mL/min; Semi Volatiles: 20-60 cm/sec; Dioxin: 20-60 cm/sec

ELEMENT NAME: COLUMN FINAL HOLD

Definition: The final temperature of the gas chromatograph column and the length of time that it was held.

Input	**Type/Length**
Quantitation Report	X(7)
As Stored Internally	X(7)

Unit of Measure

Time: minutes
Temperature: degrees Celsius
Units are understood and not reported.

Edit Criteria:

Format: Hold @ temperature ie XXX@XXX

Example: 15 @ 280 means that the column was held for 15 minutes.

ELEMENT NAME: COLUMN INITIAL HOLD

Definition: The initial temperature of the gas chromatograph column and the length of time that it was held.

Input	**Type/Length**
Quantitation Report	X(7)
As Stored Internally	X(7)

Unit of Measure

Time: minutes
Temperature: degrees Celsius
Units are understood and not reported.

Edit Criteria:

1. Format: Hold @ Temp ie XXX@XXX

2. Temperature Ranges: Volatiles: 25-50^{o}C; Semi Volatiles: 25-35^{o}C

Example: See COLUMN FINAL HOLD.

ELEMENT NAME: COLUMN INSIDE DIAMETER

Definition: The internal diameter of the gas chromatograph column.

Input	**Type/Length**
Quantitation Report	X(6)
As Stored Internally	X(6)

Unit of Measure

Millimeter (MM)

Edit Criteria:

Ranges: Volatiles: 1-3mm; Semi-Volatiles: 0.2-0.35 mm; Dioxin: 0.2-0.35 mm

ELEMENT NAME: COLUMN LENGTH

Definition: The length of the gas chromatograph column.

Input	**Type/Length**
Quantitation Report	X(6)
As Stored Internally	X(6)

Unit of Measure

Meters (M)

Edit Criteria:

Ranges: Volatiles: 2.8-3.1 m; Semi-Volatiles: 25-35 m; Dioxin: 25-65 m

ELEMENT NAME: COLUMN TEMPERATURE PROGRAM

Definition: The change in column temperature with respect to time giving the initial and final column temperatures.

Input	**Type/Length**
Quantitation Report	X(10)
As Stored Internally	X(10)

Unit of Measure

Initial temperature: degrees Celsius
Final temperature: degrees Celsius
Rate: degrees Celsius per minute
Units are understood and not reported.

Edit Criteria:

1. Format: Initial Temp - Final Temp @ Temp Program rate ie XXX-XXX @ XX

2. Range: 1.5-8.5^{o}C/min

Examples:

45-220 @ 8
30-280 @ 8

ELEMENT NAME: COMPOUND COMMENT CODE

Definition: A coded value for any optional text that may be associated with each compound.

Input	**Type/Length**
Traffic Report	X(4)
Laboratory Chronicles	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the compound comment code table. Range C001-C999.

See attached Compound Comment Code Table for valid codes.

I S O T O P E D I L U T I O N

COMPOUND LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
C001	COMBINATION OF 2 PCB'S
C002	DATA FROM B/N FRACTION
C003	BELOW VALID CALIBR.RANGE:NOTE CONC. FACTOR
C004	ACID ANALYZED IN B/N FRACTION
C005	NATURALLY OCCUR. CPD. INADVERT. SPIKED IN
C006	QUANTITATED BY ISOTOPE DILUTION
C007	1:5000 DILUTION
C008	55.4 UG FOUND IN BLANK
C009	5.92 UG FOUND IN BLANK
C010	52.8 UG FOUND IN BLANK
C011	33.0 UG FOUND IN BLANK
C012	5.1 UG FOUND IN BLANK
C013	11.8 UG FOUND IN BLANK
C014	13.1 UG FOUND IN BLANK
C015	SEVERE INTERFERENCES
C016	INTERFERENCES
C017	5.0 UG FOUND IN BLANK
C018	3.02 UG FOUND IN BLANK
C019	8.24 UG FOUND IN BLANK
C020	COMMON LAB CONTAMINANT(METHYLENE CHLORIDE)
C021	SLIGHT EMULSION PRESENT IN ACID CPDS.
C022	UNCONFIRMED
C023	4PPB FOUND IN BLANK
C024	8PPB FOUND IN BLANK
C025	5PPB FOUND IN BLANK
C026	USED M/2 86
C027	225,227 DATA COMBIN COMPLX INTEGRATION
C028	DISC ERROR-DATA LOST FROM FILE
C029	272,276 DATA AVG'D
C030	278,281 DATA AVG'D
C031	USED M/2 144
C032	PEAK OVERLAP, USE 2ND ION
C033	USED CUMM. RF AVG
C034	ACTUALLY SPIKED AT 10 PPB; RESULTS NORMALIZED TO 20
C035	ACTUAL SPIKE 50 PPB; RESULTS NORMALIZED TO 100
C036	218,265 INTEGRATION PROBLEM-OVERLAP
C037	16 PPM FOUND IN FIELD BLANK-11084 (EP 806)
C038	POOR INTEGRATION IN REGION OF 218,225,227
C039	29 PPH FOUND IN TRIP BLANK - EP 803
C040	1/5000 DILUTIONS AFTER SUBTRACT 12 PCENT AS BKGD
C041	BUTYL BENZYL PHTHALATE SEEN AT 13 PPB
C042	BUTYL BENZYL PHTHALATE SEEN AT 11 PPB
C043	BUTYL BENZYL PHTHALATE SEEN AT 8 PPB
C044	2.5 PPB FOUND IN LAB BLANK
C045	2.7 PPB FOUND IN LAB BLANK
C046	2.1 PPB FOUND IN LAB BLANK
C047	NOT MEASURED
C048	BACKGROUND OVERLAP PREVENTS INTEGRATION
C049	HIGH CONTAMINATION CAUSED POOR CHARACTERIZATION
C050	NOT MEASURED DUE TO FILE ERROR

I S O T O P E D I L U T I O N

COMPOUND LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
C051	JUST AT DETECTION LIMIT
C052	PEAK OVERLAP, POOR INTEGRATION
C053	POOR COMPUTER INTEGRATION
C054	PNA'S AT BKGD LEVEL, GEN. DET'N LIM. BETWN 2-3
C055	RECOVERY NOT QUANTIFIABLE, HEAVY PHENOLIC OVERLAP
C056	HEAVY OVERLAP MAY BE IN EFFECT
C057	1/100 DILUTION
C058	ISOTOPES COULDN'T BE USED FOR QUANTIFICATION
C059	POOR INTEGRAT. OF LBLED COMDS CAUSED BY SATUR. PEAKS
C060	POOR INTEGRATION IN REGION
C061	ACTUAL SPIKE 80-NORMALIZED TO 100
C062	AVERAGE OF COMPOUNDS 272 AND 276
C063	AVERAGE OF COMPOUNDS 278 AND 281
C064	SPIKED WITH TWO ACID SURROGATES
C065	ACTUAL SPIKE 50-NORMALIZED TO 100
C066	DETECTION LIMIT APPROX. 50 UG/L
C067	AVERAGE OF COMPOUNDS 225 AND 227

ELEMENT NAME: COMPOUND NAME

Definition: The name of the compound determined. The compound name corresponds to the EGD compound number.

Input	**Type/Length**
Quantitation Report	X(30)
As Stored Internally	X(30)

Unit of Measure

N/A

Edit Criteria:

See attached EGLD Compound Table for valid names.

Examples:

BROMOFORM
1,2-DICHLORBENZENE-D4

ELEMENT NAME: COMPOUND NUMBER

Definition: A numerical code which uniquely identifies each unique chemical compound.

Input	**Type/Length**
Quantitation Report	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

001-129 = Priority Pollutants quantitated by internal or external standard.
130-199 = Miscellaneous surrogates and internal standards.
200-299 = Priority Pollutant labeled compounds (isotopes) quantitated by internal or external standard.
300-399 = Priority Pollutants quantitated by isotope dilution.
400-429 = Labeled compounds (isotopes) quantitated by internal or external standard.
500-599 = Syn Fuel specific and Appendix C compounds quantitated by internal or external standard.
600-699 = Syn Fuel specific and Appendix C labeled compounds (isotopes) quantitated by internal or external standard.
700-799 = Syn Fuel specific and Appendix C compounds quantitated by isotope dilution.
800-829 = Pollutants 100-129 quantitated by isotope dilution.

See attached EGLD Compount Table.

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
001	B	ACENAPHTHENE	P
002	V	ACROLEIN	P
003	V	ACRYLONITRILE	P
004	V	BENZENE	P
005	B	BENZIDINE	P
006	V	CARBON TETRACHLORIDE	P
007	V	CHLOROBENZENE	P
008	B	1,2,4-TRICHLOROBENZENE	P
009	B	HEXACHLOROBENZENE	P
010	V	1,2-DICHLOROETHANE	P
011	V	1,1,1-TRICHLOROETHANE	P
012	B	HEXACHLOROETHANE	P
013	V	1,1-DICHLOROETHANE	P
014	V	1,1,2-TRICHLOROETHANE	P
015	V	1,1,2,2-TETRACHLOROETHANE	P
016	V	CHLOROETHANE	P
017	V	BIS (CHLOROMETHYL) ETHER (NR)	P
018	B	BIS(2-CHLOROETHYL)ETHER	P
019	V	2-CHLOROETHYLVINYL ETHER	P
020	B	2-CHLORONAPHTHALENE	P
021	A	2,4,6-TRICHLOROPHENOL	P
022	A	4-CHLORO-3-METHYLPHENOL	P
023	V	CHLOROFORM	P
024	A	2-CHLOROPHENOL	P
025	B	1,2-DICHLOROBENZENE	P
026	B	1,3-DICHLOROBENZENE	P
027	B	1,4-DICHLOROBENZENE	P
028	B	3,3'-DICHLOROBENZIDINE	P
029	V	1,1-DICHLOROETHENE	P
030	V	TRANS-1,2-DICHLOROETHENE	P
031	A	2,4-DICHLOROPHENOL	P
032	V	1,2-DICHLOROPROPANE	P
033	V	T-1,3-DICHLOROPROPENE	P
034	A	2,4-DIMETHYLPHENOL	P
035	B	2,4-DINITROTOLUENE	P
036	B	2,6-DINITROTOLUENE	P
037	B	1,2-DIPHENYLHYDRAZINE	P
038	V	ETHYLBENZENE	P
039	B	FLUORANTHENE	P
040	B	4-CHLOROPHENYL PHENYL ETHER	P
041	B	4-BROMOPHENYL PHENYL ETHER	P
042	B	BIS (2-CHLOROISOPROPYL) ETHER	P
043	B	BIS (2-CHLOROETHOXY) METHANE	P
044	V	METHYLENE CHLORIDE	P
045	V	CHLOROMETHANE	P
046	V	BROMOMETHANE	P
047	V	BROMOFORM	P
048	V	BROMODICHLOROMETHANE	P
049	V	TRICHLOROFLUOROMETHANE (NR)	P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
099	P	ENDRIN ALDEHYDE	P
100	P	HEPTACHLOR	P
101	P	HEPTACHLOR EPOXIDE	P
102	P	ALPHA-BHC	P
103	P	BETA-BHC	P
104	P	GAMMA-BHC	P
105	P	DELTA-BHC	P
106	P	PCB-1242	P
107	P	PCB-1254	P
108	P	PCB-1221	P
109	P	PCB-1232	P
110	P	PCB-1248	P
111	P	PCB-1260	P
112	P	PCB-1016	P
113	P	TOXAPHENE	P
129	D	2,3,7,8-TCDD	P
130	V	XYLENES	P
150	A	PHENOL-D6	S
151	A	PENTAFLUOROPHENOL	S
152	V	PENTAFLOUROBENZENE	S
153	A	TRIFLUORO-M-CRESOL	S
154	V	2,2-DIFLUOROTETRACHLOROETHANE	S
155	B	2-FLUOROBIPHENYL	S
156	B	1-FLUORONAPHTHALENE	S
157	A	2-FLOUROPHENOL	S
158	B	2-FLUORONAPHTHALENE	S
159	B	PYRIDINE-D5	S
160	B	ANILINE-D5	S
161	B	NAPHTHALENE-D8	S
162	V	TOLUENE-D8	S
163	B	NITROBENZENE-D5	S
164	B	2,2'-DIFLUOROBIPHENYL	I
165	V	BENZENE-D6	S
166	B	DECAFLUOROBIPHENYL	S
167	V	M-DIFLUOROBENZENE	S
168	V	METHYLENE CHLORIDE-D2	S
169	V	1,1,2,2-TETRACHLOROETHANE-D2	S
170	V	ETHYLBENZENE-D10	S
172	V	1,2 DICHLOROETHANE-D4	S
173	V	2,2 DICHLOPROPANE-D6	S
174	V	CHLOROBENZENE-D5	S
175	B	1,2 DICHLOROBENZENE-D4	S
176	B	CHRYSENE D12	S
177	B	FLUORENE D10	S
178	A	2-NITROPHENOL D4	S
179	B	DI-N-BUTYL-PHTHALATE-D4	S
180	B	4-FLUOROANILINE	S
181	V	BROMOCHLOROMETHANE	I
182	V	2-BROMO-1-CHLOROPROPANE	I

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
050	V	DICHLORODIFLUOROMETHANE (NR)	P
051	V	DIBROMOCHLOROMETHANE	P
052	B	HEXACHLORO-1,3-BUTADIENE	P
053	B	HEXACHLOROCYCLOPENTADIENE	P
054	B	ISOPHORONE	P
055	B	NAPHTHALENE	P
056	B	NITROBENZENE	P
057	A	2-NITROPHENOL	P
058	A	4-NITROPHENOL	P
059	A	2,4-DINITROPHENOL	P
060	A	2-METHYL-4,6-DINITROPHENOL	P
061	B	N-NITROSODIMETHYLAMINE	P
062	B	N-NITROSODIPHENYLAMINE	P
063	B	N-NITROSODI-N-PROPYLAMINE	P
064	A	PENTACHLOROPHENOL	P
065	A	PHENOL	P
066	B	BIS (2-ETHYLHEXYL) PHTHALATE	P
067	B	BUTYL BENZYL PHTHALATE	P
068	B	DI-N-BUTYL PHTHALATE	P
069	B	DI-N-OCTYL PHTHALATE	P
070	B	DIETHYL PHTHALATE	P
071	B	DIMETHYL PHTHALATE	P
072	B	BENZO(A)ANTHRACENE	P
073	B	BENZO(A)PYRENE	P
074	B	BENZO(B)FLUORANTHENE	P
075	B	BENZO(K)FLUORANTHENE	P
076	B	CHRYSENE	P
077	B	ACENAPHTHYLENE	P
078	B	ANTHRACENE	P
079	B	BENZO(GHI)PERYLENE	P
080	B	FLUORENE	P
081	B	PHENANTHRENE	P
082	B	DIBENZO(A,H)ANTHRACENE	P
083	B	INDENO(1,2,3-CD)PYRENE	P
084	B	PYRENE	P
085	V	TETRACHLOROETHENE	P
086	V	TOLUENE	P
087	V	TRICHLOROETHENE	P
088	V	VINYL CHLORIDE	P
089	P	ALDRIN	P
090	P	DIELDRIN	P
091	P	CHLORDANE	P
092	P	4,4'-DDT	P
093	P	4,4'-DDE	P
094	P	4,4'-DDD	P
095	P	ALPHA-ENDOSULFAN	P
096	P	BETA-ENDOSULFAN	P
097	P	ENDOSULFAN SULFATE	P
098	P	ENDRIN	P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
248	V	BROMODICHLOROMETHANE-13C	D
249	V		D
250	V		D
251	V	DIBROMOCHLOROMETHANE-13C	D
252	B	HEXACHLORO-1,3-BUTADIENE-13C4	D
253	B	HEXACHLOROCYCLOPENTADIENE-13C4	D
254	B	ISOPHORONE-D8	D
255	B	NAPHTHALENE-D8	D
256	B	NITROBENZENE-D5	D
257	A	2-NITROPHENOL-3,4,5,6-D4	D
258	A	4-NITROPHENOL-2,3,5,6-D4	D
259	A	2,4-DINITROPHENOL-3,5,6-D3	D
260	A	2-METHYL-4,6-DINITROPHENOL-D2	D
261	B		D
262	B	N-NITROSODIPHENYLAMINE-D6	D
263	B		D
264	A	PENTACHLOROPHENOL-13C6	D
265	A	PHENOL-2,3,4,5,6-D5	D
266	B	BIS(2-ETHYLHEXYL)PHTHALATE-D4	D
267	B		D
268	B	DI-N-BUTYL PHTHALATE-D4	D
269	B	DI-N-OCTYL PHTHALATE-D4	D
270	B	DIETHYL PHTHALATE-3,4,5,6-D4	D
271	B	DIMETHYL PHTHALATE-3,4,5,6-D4	D
272	B	BENZO(A)ANTHRACENE-D12	D
273	B	BENZO(A)PYRENE-D12	D
274	B	BENZO(B)FLUORANTHENE-D12	D
275	B	BENZO(K)FLUORANTHENE-D12	D
276	B	CHRYSENE-D12	D
277	B	ACENAPHTHYLENE-D8	D
278	B	ANTHRACENE-D10	D
279	B	BENZO(GHI)PERYLENE-D12	D
280	B	FLUORENE-D10	D
281	B	PHENANTHRENE-D10	D
282	B		D
283	B		D
284	B	PYRENE-D10	D
285	V	TETRACHLOROETHENE-1,2-13C2	D
286	V	TOLUENE-2,3,4,5,6-D5	D
287	V	TRICHLOROETHENE-13C2	D
288	V	VINYL CHLORIDE-D3	D
289	P		D
290	P		D
291	P		D
292	P		D
293	P		D
294	P		D
295	P		D
296	P		D

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
183	V	1,4-DICHLOROBUTANE	I
184	D	2,3,7,8-TCDD-37CL4	I
201	B	ACENAPHTHENE-D10	D
202	V		D
203	V	ACRYLONITRILE-D3	D
204	V	BENZENE-D6	D
205	B	BENZIDINE (RINGS-D8)	D
206	V	CARBON TETRACHLORIDE-13C	D
207	V	CHLOROBENZENE-D5	D
208	B	1,2,4-TRICHLOROBENZENE-D3	D
209	B	HEXACHLOROBENZENE-13C6	D
210	V	1,2-DICHLOROETHANE-D4	D
211	V	1,1,1-TRICHLOROETHANE-D3	D
212	B	HEXACHLOROETHANE-1-13C	D
213	V	1,1-DICHLOROETHANE-2,2,2-D3	D
214	V	1,1,2-TRICHLOROETHANE-13C2	D
215	V	1,1,2,2-TETRACHLOROETHANE-D2	D
216	V	CHLOROETHANE-D5	D
217	V		D
218	B	BIS(2-CHLOROETHYL)ETHER-D8	D
219	V		D
220	B	2-CHLORONAPHTHALENE-D7	D
221	A	2,4,6-TRICHLOROPHENOL-3,5-D2	D
222	A	4-CHLORO-3-METHYLPHENOL-2,6-D2	D
223	V	CHLOROFORM-13C	D
224	A	2-CHLOROPHENOL-3,4,5,6-D4	D
225	B	1,2-DICHLOROBENZENE-D4	D
226	B	1,3-DICHLOROBENZENE-D4	D
227	B	1,4-DICHLOROBENZENE-D4	D
228	B	3,3'-DICHLOROBENZIDINE-D6	D
229	V	1,1-DICHLOROETHENE-D2	D
230	V	TRANS-1,2-DICHLOROETHENE-D2	D
231	A	2,4-DICHLOROPHENOL-3,5,6-D3	D
232	V	1,2-DICHLOROPROPANE-D6	D
233	V	T-1,3-DICHLOROPROPENE-1,2-D2	D
234	A	2,4-DIMETHYLPHENOL-3,5,6-D3	D
235	B	2,4-DINITROTOLUENE-3,5,6-D3	D
236	B	2,6-DINITROTOLUENE-A,A,A-D3	D
237	B	1,2-DIPHENYL-D10-HYDRAZINE	D
238	V	ETHYLBENZENE-D10	D
239	B	FLUORANTHENE-D10	D
240	B	4-CHLOROPHENYL PHENYL-D5 ETHER	D
241	B		D
242	B	BIS(2-CHLOROISOPROPYL)ETHERD12	D
243	B		D
244	V	METHYLENE CHLORIDE-D2	D
245	V	CHLOROMETHANE-D3	D
246	V	BROMOMETHANE-D3	D
247	V	BROMOFORM-13C	D

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
347	V	BROMOFORM	P
348	V	BROMODICHLOROMETHANE	P
349	V		P
350	V		P
351	V	DIBROMOCHLOROMETHANE	P
352	B	HEXACHLORO-1,3-BUTADIENE	P
353	B	HEXACHLOROCYCLOPENTADIENE	P
354	B	ISOPHORONE	P
355	B	NAPHTHALENE	P
356	B	NITROBENZENE	P
357	A	2-NITROPHENOL	P
358	A	4-NITROPHENOL	P
359	A	2,4-DINITROPHENOL	P
360	A	2-METHYL-4,6-DINITROPHENOL	P
361	B		P
362	B	N-NITROSODIPHENYLAMINE	P
363	B		P
364	A	PENTACHLOROPHENOL	P
365	A	PHENOL	P
366	B	BIS (2-ETHYLHEXYL) PHTHALATE	P
367	B		P
368	B	DI-N-BUTYL PHTHALATE	P
369	B	DI-N-OCTYL PHTHALATE	P
370	B	DIETHYL PHTHALATE	P
371	B	DIMETHYL PHTHALATE	P
372	B	BENZO(A)ANTHRACENE	P
373	B	BENZO(A)PYRENE	P
374	B	BENZO(B)FLUORANTHENE	P
375	B	BENZO(K)FLUORANTHENE	P
376	B	CHRYSENE	P
377	B	ACENAPHTHYLENE	P
378	B	ANTHRACENE	P
379	B	BENZO(GHI)PERYLENE	P
380	B	FLUORENE	P
381	B	PHENANTHRENE	P
382	B		P
383	B		P
384	B	PYRENE	P
385	V	TETRACHLOROETHENE	P
386	V	TOLUENE	P
387	V	TRICHLOROETHENE	P
388	V	VINYL CHLORIDE	P
389	P		P
390	P		P
391	P		P
392	P		P
393	P		P
394	P		P
395	P		P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
297	P		D
298	P		D
299	P		D
301	B	ACENAPHTHENE	P
302	V		P
303	V	ACRYLONITRILE	P
304	V	BENZENE	P
305	B	BENZIDINE	P
306	V	CARBON TETRACHLORIDE	P
307	V	CHLOROBENZENE	P
308	B	1,2,4-TRICHLOROBENZENE	P
309	B	HEXACHLOROBENZENE	P
310	V	1,2-DICHLOROETHANE	P
311	V	1,1,1-TRICHLOROETHANE	P
312	B	HEXACHLOROETHANE	P
313	V	1,1-DICHLOROETHANE	P
314	V	1,1,2-TRICHLOROETHANE	P
315	V	1,1,2,2-TETRACHLOROETHANE	P
316	V	CHLOROETHANE	P
317	V		P
318	B	BIS(2-CHLOROETHYL)ETHER	P
319	V		P
320	B	2-CHLORONAPHTHALENE	P
321	A	2,4,6-TRICHLOROPHENOL	P
322	A	4-CHLORO-3-METHYLPHENOL	P
323	V	CHLOROFORM	P
324	A	2-CHLOROPHENOL	P
325	B	1,2-DICHLOROBENZENE	P
326	B	1,3-DICHLOROBENZENE	P
327	B	1,4-DICHLOROBENZENE	P
328	B	3,3'-DICHLOROBENZIDINE	P
329	V	1,1-DICHLOROETHENE	P
330	V	TRANS-1,2-DICHLOROETHENE	P
331	A	2,4-DICHLOROPHENOL	P
332	V	1,2-DICHLOROPROPANE	P
333	V	T-1,3-DICHLOROPROPENE	P
334	A	2,4-DIMETHYLPHENOL	P
335	B	2,4-DINITROTOLUENE	P
336	B	2,6-DINITROTOLUENE	P
337	B	1,2-DIPHENYLHYDRAZINE	P
338	V	ETHYLBENZENE	P
339	B	FLUORANTHENE	P
340	B	4-CHLOROPHENYL PHENYL ETHER	P
341	B		P
342	B	BIS (2-CHLOROISOPROPYL) ETHER	P
343	B		P
344	V	METHYLENE CHLORIDE	P
345	V	CHLOROMETHANE	P
346	V	BROMOMETHANE	P

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
396	P		P
397	P		P
398	P		P
399	P		P
429	D	2,3,7,8-TCDD-13C12	D
500	A	BENZOIC ACID	P
501	A	HEXANOIC ACID	P
502	B	2-NAPHTHYLAMINE	P
503	B	2-METHYLPYRIDINE	P
504	B	DIBENZOTHIOPHENE	P
505	B	DIBENZOFURAN	P
506	B	N-DODECANE (N-C12)	P
507	B	DIPHENYLAMINE	P
508	B	DIPHENYL ETHER	P
509	B	ALPHA-TERPINEOL	P
510	B	STYRENE	P
511	B	DI-N-BUTYL AMINE	P
512	B	BIPHENYL	P
513	B	P-CYMENE	P
514	V	2-BUTANONE (MEK)	P
515	V	DIETHYL ETHER	P
516	V	ACETONE	P
517	B	N-DECANE (N-C10)	P
518	B	N-TETRADECANE (N-C14)	P
519	B	N-HEXADECANE (N-C16)	P
520	B	N-OCTADECANE (N-C18)	P
521	B	N-EICOSANE (N-C20)	P
522	B	N-DOCOSANE (N-C22)	P
523	B	N-TETRACOSANE (N-C24)	P
524	B	N-HEXACOSANE (N-C26)	P
525	B	N-OCTACOSANE (N-C28)	P
526	B	N-TRIACONTANE (N-C30)	P
600	A	BENZOIC-D5 ACID	D
601	A	HEXANOIC ACID-D11	D
602	B	2-NAPHTHYLAMINE-D7	D
603	B	2-METHYLPYRIDINE-D7	D
604	B	DIBENZOTHIOPHENE-D8	D
605	B	DIBENZOFURAN-D8	D
606	B	N-DODECANE-D26 (N-C12)	D
607	B	DIPHENYLAMINE-D10	D
608	B	DIPHENYL ETHER-D10	D
609	B	ALPHA-TERPINEOL-D3	D
610	B	STYRENE-2,3,4,5,6-D5	D
611	B	DI-N-BUTYL AMINE-D18	D
612	B	BIPHENYL-D10	D
613	B	P-CYMENE-D14	D
614	V	2-BUTANONE-4,4,4-D3 (MEK)	D
615	V	DIETHYL ETHER-D10	D
616	V	ACETONE-D6	D

USEPA EFFLUENT GUIDELINES DIVISION

COMPOUND CODE LISTING

EGLD COMPOUND NUMBER	FRACTION	COMPOUND	COMPOUND TYPE
617	B	N-DECANE-D22 (N-C10)	D
618	B		D
619	B	N-HEXADECANE-D34 (N-C16)	D
620	B		D
621	B	N-EICOSANE-D42 (N-C20)	D
622	B		D
623	B	N-TETRACOSANE-D50 (N-C24)	D
624	B		D
625	B		D
626	B	N-TRIACONTANE-D62 (N-C30)	D
700	A	BENZOIC ACID	P
701	A	HEXANOIC ACID	P
702	B	2-NAPHTHYLAMINE	P
703	B	2-METHYLPYRIDINE	P
704	B	DIBENZOTHIOPHENE	P
705	B	DIBENZOFURAN	P
706	B	N-DODECANE (N-C12)	P
707	B	DIPHENYLAMINE	P
708	B	DIPHENYL ETHER	P
709	B	ALPHA-TERPINEOL	P
710	B	STYRENE	P
711	B	DI-N-BUTYL AMINE	P
712	B	BIPHENYL	P
713	B	P-CYMENE	P
714	V	2-BUTANONE (MEK)	P
715	V	DIETHYL ETHER	P
716	V	ACETONE	P
717	B	N-DECANE (N-C10)	P
718	B		P
719	B	N-HEXADECANE (N-C16)	P
720	B		P
721	B	N-EICOSANE (N-C20)	P
722	B		P
723	B	N-TETRACOSANE (N-C24)	P
724	B		P
725	B		P
726	B	N-TRIACONTANE (N-C30)	P
829	D	2,3,7,8-TCDD	P

430 RECORDS PRINTED

ELEMENT NAME: COMPOUND ORDER NUMBER

Definition: A numerical code that establishes the order of compound determination by the GC/MS. The code is used on the Quantitation Report to match the segments of compound data within the report.

Input	**Type/Length**
Quantitation Report	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Range: 001-250

ELEMENT NAME: COMPOUND TYPE

Definition: A coded value which identifies a chemical compound as a priority pollutant or surrogate.

Input	**Type/Length**
Generated Based on Compound Number	X(1)
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

CODE	VALUE
D	Isotopic Diluent
I	Internal Standard
P	Priority Pollutant
S	Surrogate

ELEMENT NAME: DATE EXTRACTED

Definition: The date that the laboratory extracted the sample for analysis.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: MM/DD/YY, where MM is the month; DD is the day; and YY is the last two digits of the Gregarian calendar year.

Example: 07/15/83 is July 15, 1983.

ELEMENT NAME: DATE ANALYZED

Definition: The date that the sample fraction was analyzed by the laboratory.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: MM/DD/YY, where MM is the month; DD is the day; and YY is the last two digits of the Gregarian calendar year.

Example: 07/15/83 is July 15, 1983.

ELEMENT NAME: DATE SAMPLED

Definition: The date the sample was taken by the field sampler.

Input	**Type/Length**
Traffic Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: MM/DD/YY, where MM is the month; DD is the day; and YY is the last two digits of the Gregarian calendar year.

Example: 07/15/83 is July 15, 1983.

ELEMENT NAME: EPISODE COMMENT CODE

Definition: A coded value for comments associated with an episode.

Input	**Type/Length**
Traffic Reports	X(4)
Laboratory Chronicles	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Episode Comment Code Table. Range E001-E999.

See attached Episode Comment Code Table for a list of valid codes.

ELEMENT NAME: EPISODE NUMBER

Definition: The SCC assigned identification code designating the sampling trip.

Input	**Type/Length**
SAMTRAC	9(4)
As Stored Internally	9(4)

Unit of Measure

N/A

Edit Criteria:

1. Numeric

2. Greater than 0119.

ELEMENT NAME: FRACTION

Definition: A coded value which designates the compound as either an acid, base/neutral, volatile, pesticide or dioxin.

Input	**Type/Length**
Priority Pollutant Data Sheet	X(1)
QA/QC Sheet	
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

CODE	VALUE
A	Acid Compound
B	Base/Neutral Compound
C	Combined Acid Base/Neutral
D	Dioxin
P	Pesticide Compound
V	Volatile Compound

ELEMENT NAME: FRACTION COMMENT CODE

Definition: A coded value for any optional text that may be associated with each fraction.

Input	**Type/Length**
Traffic Report/Lab Chronicles	X(4)
Priority Pollutant Data Sheet	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Fraction Comment Code Table. Range F001-F999.

See attached Fraction Comment Code Table for a list of valid codes.

I S O T O P E D I L U T I O N

FRACTION LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
F001	ENTIRE FRACTION NOT DETECTED
F002	ENTIRE FRACTION NOT REQUIRED
F003	ENTIRE FRACTION NOT ANALYZED
F004	BAD EMULSION-SPL. CENTRIFUGED AFTER EACH EXTRACT WASH.
F005	SAMPLE CENTRIFUGED AFTER 3RD ORGANIC WASH
F006	SAMPLE WASHES WERE CENTRIFUGED AFTER EACH WASH
F007	BAD EMULSION DURING ORGANIC WASHES
F008	BAD EMULSION
F009	EMULSION PRESENT
F010	SAMPLES RECEIVED AT 17 DEGREES CENTIGRADE
F011	SAMPLE RECEIVED AT 19 DEGREES CENTIGRADE
F012	SLIGHT EMULSION PRESENT
F013	EMULSION PRESENT - SAMPLES CENTRIFUGED
F014	SPL. SPIKED WITH 100UG B/N & A STABLE LABELLED CPDS.
F015	1:50 DILUT. & 5UL. 12037 INT. STD.+5UL LBLD. VOA(80PPM)
F016	UNPRESERVED
F017	1/50 DILUT. & 5UL. INT. STD.+5UL. LABELLED VOA(80 PPM)
F018	SPIKED WITH 5UL. EACH VOA INT. STD. & LBLD. VOA(80PPM)
F019	DILUTED 10 TIMES FOR ANALYSIS
F020	SPKD. 2/5UL. EACH VOA INT. STD.+5UL LBLD. VOA +20UL.MTX.
F021	MATRIX DUPLICATE(REPLICATE)
F022	DILUTED 1:100
F023	100 ML. OF SPL. SPKD. W/100UG. B/N&A STBL. LBLD. CPDS.
F024	SPKD. W/5UL. EACH VOA INT. STD. & LABELLED VOA
F025	MATRIX
F026	DILUTED 1:10
F027	DILUTED 1:50
F028	DILUTED 1:20
F029	SPKD. W/5UL. EA. VOA INT.STD. & LBLD. VOA+20UL. MTX DUP.
F030	LBLD., APPEND C, & SYNFL SPKS ADDED(75ML. NAOH BASE REQ)
F031	DILUTED 1:40
F032	DILUTED 1:25
F033	DILUTED 1:500
F034	DILUTED 1:1000
F035	1L+100ML. EXT.:SPIKED W/100UG A&B/N STBL. LBLD CPDS.
F036	MATRIX SPKD. W/100UG STABLE LBLD. & UNLBLD. A&B/N CPDS.
F037	SPKD. W/20PPM VOA INT. STD.+80PPM LBLD. VOA, DIL. 1:10
F038	SPKD. W/20PPM VOA INT. STD.+80PPM LBLD. VOA, DIL. 1:5000
F039	SPKD. W/20PPM VOA INT. STD.+80PPM LBLD. VOA
F040	DILUTED 1/5
F041	SPKD. W/5UL, VOA INT. STD.(20PPM)&LBLD(80PPM), DIL. 1/10
F042	ACID ANALYZED IN B/N FRACTION
F043	100 UG LABELLED B/N, A, PHTHALATE
F044	500 UG UNLABELLED B/N, A, PHTHALATE
F045	100 UG UNLABELLED B/N, A, PHTHALATE
F046	24 HOUR COMPOSITE
F047	REPLICATE
F048	22 HOUR COMPOSITE
F049	100 UG/L LABELLED B/N ADDED
F050	100 UG/L LABELLED A ADDED

I S O T O P E D I L U T I O N

FRACTION LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
F051	100 UG/L UNLABELLED B/N ADDED
F052	100 UG/L UNLABELLED A ADDED
F053	MATRIX SPIKE
F054	FINAL VOLUME 4 ML, DILUTED 10,000X
F055	FINAL VOLUME 2.8 ML, DILUTED 10,000X
F056	FINAL VOLUME 95 ML, DILUTED 1000X
F057	FINAL VOLUME 14.3 ML, DILUTED 1000X
F058	FINAL VOLUME 13.8 ML, DILUTED 1000X
F059	FINAL VOLUME 6.4 ML, DILUTED 10,000X
F060	FINAL VOLUME 8.5 ML, DILUTED 10,000X
F061	FINAL VOLUME 10.9 ML, DILUTED 1000X
F062	FINAL VOLUME 10.2 ML, DILUTED 1000X
F063	DILUTED 1:2000
F064	DILUTED 1:4000
F065	DILUTED 1:30
F066	DILUTED 1:60
F067	CONC 1000:1; A & B/N INJEC ON CAP.
F068	SAMPLES ORIG SPIKED W/ COCKTAILS
F069	SAMPLE PARTIALLY SPKD W/ COCKTAILS
F070	ANALYZED IN TRIPLICATE
F071	MANUAL EXTRACTION
F072	GOOD RECOVERY-SAMPLES NEUTRAL UPON RECEIPT
F073	EMULSION PROBLEMS WITH EXTRCTABLES
F074	SHAKE OUT USED FOR EXTRACTION
F075	MODERATE EMULSION
F076	ONE VIAL ARRIVED BROKEN, BUT HAD DUPLICATE
F077	PRESERVED
F078	PRESERVED WITH 3 DROPS SODIUM THIOSULFATE EACH BOTTLE
F079	HEAVY EMULSION-B/N AND A EXTRAS W/CONT EXTR THEN XS SOLV
F080	SAMPLE HAD TO BE DILUTED TWICE
F081	LABLE SPIKE INCREASED FROM 50 TO 200 PPB
F082	SAMPLE RERUN NOV. 4, 1982
F083	EXTRACTS COMBINED FOR INJECTION
F084	VOA'S NOT RUN SECOND TIME-NONE DETECTED

ELEMENT NAME: INDUSTRIAL CATEGORY CODE

Definition: The classification of the industrial processes performed by the plant where a sample was taken.

Input	**Type/Length**
SAMTRAC	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Industrial Category Code Table.

See attached Industrial Category Code Table for a list of codes.

I S O T O P E D I L U T I O N

INDUSTRIAL CATEGORY CODE TABLE

CODE	DESCRIPTION
100	SOAPS + DETERG.
110	ADHESIVES
120	LEATHER TANNING
130	POTWS
200	TEXTILES
210	GUM + WOOD
220	PULP + PAPER
230	TIMBER
240	PRINTING + PUB
250	PAINT + INK
300	ORGANICS
310	PESTICIDES
320	PHARMACEUTICALS
330	CARBON BLACK
340	RUBBER
350	PLASTICS + SYN
370	MINERAL MINING
400	COAL MINING
410	ORE MINING
420	PAVING + ROOF
430	STEAM ELECTRIC
440	PETROLEUM REF.
450	OIL + GAS
500	IRON + STEEL
510	FOUNDRIES
520	ELECTROPLATING
530	NONFERROUS MET.
531	NONFRS.MTL.PH1
532	NONFRS.MTL.PH2
540	BATTERIES
550	PLASTICS
560	COIL COATING
570	COPPER
580	PORC + ENAMEL
590	ALUMINUM FORM
600	PHOTOGRAPHIC
700	INORGANIC CHEMS
701	INORG.CHEM.1
702	INORG.CHEM.2
710	MECH PRODUCTS
720	ELEC + ELECTRON
730	EXPLOSIVES
740	AUTO + OTHER
750	PHOSPHATES
760	SHIPBUILDING
770	LANDFILL
780	MISC.ENVIRON
790	FRUITS + VEG.
800	SYN. FUELS
810	METAL.FINISHING

I S O T O P E D I L U T I O N

INDUSTRIAL CATEGORY CODE TABLE

CODE	DESCRIPTION
820	OSW
830	NURP
840	FERTILIZERS
850	OWPO
860	ENFORCEMENT
870	NONFERROUS FORMING
880	WATER SUPPLY

ELEMENT NAME: INSTRUMENT

Definition: A coded value assigned by the laboratory that uniquely identifies each GC/MS instrument within a laboratory. All Calibration, Precision and Recovery, Standards and Blank Quantitation files will be tracked by this instrument member within Laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and other initial quantitation runs by the laboratory.

Input	**Type/Length**
Quantitation Report	X(2)
As Stored Internally	X(2)

Unit of Measure

N/A

Edit Criteria:

Range: 01-99, AA-ZZ or any two character combination.

ELEMENT NAME: LABORATORY

Definition: A numerical code used to identify the specific laboratory where the sample was analyzed.

Input	**Type/Length**
SAMTRAC	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the SAMTRAC Laboratory Code Table.

See attached Laboratory Code Table for a list of valid codes.

I S O T O P E D I L U T I O N

LABORATORY CODE TABLE

CODE	DESCRIPTION
100	ERCO
110	SPECTRIX
120	FOREMOST
130	RADIAN(SAC)
140	S3
150	WCTS
160	ARL
170	RADIAN(AUS)
180	TAC
190	RALTECH
200	MONSANTO
210	EMS
220	MCCRONE
230	CARB-LEX
240	OTHER
250	MRI
260	VARC
270	BATTELLE
280	BARRINGE
290	TRW
300	JACOBS P
310	REG IV
320	REG V
330	REG VII
340	REG VIII
350	ACUREX
360	ENVIRO
370	STI
380	BCL-OSW
390	EMSL-OSW
400	SRI
410	IT-ENVI.
420	VERSAR
430	CENTEC
440	ARTHUR D. LITTLE
450	GSRI
460	ESE
470	SHELL
480	MIDWEST RESEARCH INSTITUTE
490	USEPA REGION 2
500	U.S. TESTING

ELEMENT NAME: MASS TO CHARGE RATIO

Definition: Designates the quantitation ion. Abbreviated as M/Z, or M/E.

Input	**Type/Length**
Quantitation Report	ZZZ9
As Stored Internally	9(4)

Unit of Measure

N/A

Edit Criteria:

Ranges: Volatiles: 20-250; Semi-Volatiles: 35-450.

ELEMENT NAME: METHOD

Definition: A coded value which uniquely identifies the method protocol that was followed during analysis.

Input	**Type/Length**
Quantitation Report	X(5)
As Stored Internally	X(5)

Unit of Measure

N/A

Edit Criteria:

Acceptable Codes:

1624A
1625A
613
613E
713

ELEMENT NAME: PEAK AREA

Definition: The area beneath the peak of a mass chromatogram. The peak area is proportional to the amount of the detected compound at an observed mass to charge ratio. It is used to compute the concentration of the compound present in the sample.

Input	**Type/Length**
Quantitation Report	ZZZZZZZZZ9.
As Stored Internally	9(10)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: PH LEVEL

Definition: The negative logarithm of the effective hydrogen ion concentration as expressed in grain equivalents per liter.

Input	**Type/Length**
Traffic Report	Z9.999
As Stored Internally	9(2)V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: PLANT CODE

Definition: A numeric code used to distinguish specific industrial plants which have been sampled.

Input	**Type/Length**
SAMTRAC	9(4)
As Stored Internally	9(4)

Unit of Measure

N/A

Edit Criteria:

Must be a four digit number.

Comment:

1. Plant ID's are unique within the isotope dilution program.

2. The episode number for the first occurrence of a plant visit is used as the plant's identification number.

3. See attached Plant Code Table for a list of current codes.

I S O T O P E D I L U T I O N

PLANT-ID CODE TABLE

CODE	DESCRIPTION		
0389	47A	K	TN
0523	50	TR	NJ
0709	HOLSTON ARMY AMMO PL	KINGSPORT	TN
0711	FT SNELLING	FT SNELLING	MN
0712	FT LEWIS	DUPONT	WA
0713	MORGANTOWN TECH CNTR	MORGANTOWN	WV
0727	POPE AND TALBOT	EAU CLAIRE	WI
0760	SHELL OIL	KENAI	AK
0761	AMOCO	KENAI	AK
0766	GULF OF MEXICO	NEW ORLEANS	LA
0769	GENERAL ELECTRIC	SCHENECTADY	NY
0770	ARCO	PRUDHOE BAY	AK
0788	SEATTLE CSO	SEATTLE	WA
0793	ST. PAUL CSO	ST. PAUL	MN
0801	PROVIDENCE CSO	PROVIDENCE	RI
0806	ST. LOUIS CSO	ST. LOUIS	MO
0821	PLANT #2	MH	NC
0846	PLANT #3	W	WV
0848	PLANT #4	DP	TX
0852	PLANT #5	GF	WV
0868	HUNTINGTON ALLOYS	BURNAUGH	KY
0928	PLANT #6	B	NJ
0929	PLANT #7	NM	WV
0931	BALL CORPORATION	GREENVILLE	TN
0932	PLANT #9	A	TX
0933	PLANT #10	P	LA
0934	BRUSH WELLMAN	ELMORE	OH
0950	PLANT #11	GC	IL
0951	PLANT #12	C	TX

ELEMENT NAME: PROPRIETARY INDICATOR

Definition: A coded value which designates whether or not the analysis data from a sample is proprietary. Also indicates that confidentiality papers have been signed.

Input	**Type/Length**
Traffic Report	X(1)
Lab Chronicles	
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

CODE	VALUE
P	Proprietary
N	Not Proprietary

ELEMENT NAME: QUANTITATION REPORT TYPE

Definition: A coded value that uniquely identifies the particular type of quantitation report that is being submitted.

Input	**Type/Length**
Quantitation Report	X(3)
As Stored Internally	X(3)

Unit of Measure

N/A

Edit Criteria:

CODE	VALUE
APS	Aqueous Performance Standard
BLK	Blank
CAL	Calibration
EPA	EPA Sample
PAR	Precision and Recovery
STD	Standard
VER	Calibration Verification

ELEMENT NAME: REFERENCE COMPOUND

Definition: A numeric code that is used as a pointer to the internal standard or isotopic diluent within a quantitation report.

Input	**Type/Length**
Quantitation Report	ZZ9
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Range: 001-250

ELEMENT NAME: RELATIVE RETENTION TIME

Definition: The quotient of the retention time of a compound divided by its internal standard or isotopic diluent.

Input	**Type/Length**
Quantitation Report	Z9.999
As Stored Internally	99V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: RELATIVE RETENTION TIME (LIBRARY)

Definition: The relative retention time stored in the library. The value is based on the analysis of a standard containing both compounds.

Input	**Type/Length**
Quantitation Report	Z9.999
As Stored Internally	9(2)V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: RESPONSE FACTOR

Definition: The ratio between the response for the sample and a response for a standard under identical analytical conditions. Computed per the following equation:

$$RF = \frac{A_S C_{IS}}{A_{IS} C_S}$$

where

A_S is the PEAK AREA for the compound from analysis of a standard.
A_{IS} is the PEAK AREA for the internal standard.
C_{IS} is the concentration of the internal standard.
C_S is the concentration of the compound.

Input	**Type/Length**
Quantitation Report	ZZZ9.99
As Stored Internally	9(4)V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: RESPONSE FACTOR (LIBRARY)

Definition: The response factor stored in the library. The value is determined from analysis of a standard.

Input	**Type/Length**
Quantitation Report	ZZZ9.999
As Stored Internally	9(4)V9(3)

Unit of Measure

N/A

Edit Criteria:

Example: See RESPONSE FACTOR.

ELEMENT NAME: RETENTION TIME

Definition: The time it takes the identified compound to elute from the gas chromatograph.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: HH:MM:SS
MM:SS
SS

Where HH is hours; MM is minutes; SS is seconds.

ELEMENT NAME: RETENTION TIME (LIBRARY)

Definition: The known time it takes an identified compound to elute from the gas chromatograph. The time is determined from analysis of a standard.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: HH:MM:SS
MM:SS
SS

Where HH is hours; MM is minutes; SS is seconds.

ELEMENT NAME: SAMPLE COMMENT CODE

Definition: A coded value for any optional text that may be associated with each sample.

Input	**Type/Length**
Traffic Reports	X(4)
Lab Chronicles	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Sample Comment code table. Range S001-S999

See attached Sample Comment Code Table for a list of valid codes.

I S O T O P E D I L U T I O N

SAMPLE LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
S001	SAMPLE ANALYZED IN DUPLICATE
S002	PESTICIDES ANALYZED IN B/N FRACTION-REGULAR
S003	PESTICIDES ANALYZED IN B/N FRACTION-REGULAR QA
S004	VAT AREA TO INFLUENT TO TREATMENT PLANT
S005	A20 AREA INFLUENT TO TREATMENT PLANT
S006	TREATED WASTE
S007	DECANT TANK(EFFLUENT)
S008	GASIFIER SLUICE H20
S009	CYCLONE QUENCH
S010	SCRUBBER H20
S011	COAL PILE RUNOFF
S012	MANHOLE #4
S013	MANHOLE #5
S014	MANHOLE #6
S015	SOUR WATER STRIPPER
S016	BIO UNIT EFFLUENT
S017	FILTER EFFLUENT
S018	STILLING BASIN
S019	OIL/WATER SEP. EFFLUENT
S020	SAND FILTER
S021	CARBON FILTER EFFLUENT
S022	LAB WASTE
S023	VENTURI SCRUBBER DAY 1
S024	VENTURI SCRUBBER DAY 2
S025	VENTURI SCRUBBER DAY 3
S026	DIRECT COOLER DAY 1
S027	DIRECT COOLER DAY 2
S028	DIRECT COOLER DAY 3
S029	SOURCE WATER
S030	MAKE-UP WATER
S031	DECANTER LIQUOR
S032	INLET-FREE WATER KNOCKOUT #4
S033	OUTLET FLOTATION CELL-DISCHARGE
S034	FREE WATER KNOCKOUT
S035	OVERBOARD DISCHARGE
S036	PLATFORM EC33A
S037	PLATFORM EC14CF
S038	PLATFORM V39D(V22D)
S039	PLATFORM V119D
S040	PLATFORM SMI6A
S041	PLATFORM SMI105A(SMI106A)
S042	PLATFORM EI120CF
S043	PLATFORM SMI208B
S044	PLATFORM EI296B
S045	PLATFORM V225A(V247A0
S046	PLATFORM SMI23BAUX
S047	PLATFORM SMI130B
S048	PLATFORM EI18CF
S049	PLATFORM EI57A-E
S050	PLATFORM EI238E

I S O T O P E D I L U T I O N

SAMPLE LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
S051	PLATFORM SS107S-94
S052	PLATFORM SS107S-93
S053	PLATFORM SS219A
S054	PLATFORM ST177
S055	PLATFORM BM2C-AM
S056	PLATFORM BM2C-PM
S057	PLATFORM BDCCF5-AM
S058	PLATFORM BDCCF5-PM
S059	PLATFORM GIBDB600
S060	PLATFORM SP62A
S061	PLATFORM WD70I
S062	PLATFORM WD105C
S063	PLATFORM ST135
S064	PLATFORM WD90A
S065	PLATFORM SP24/27
S066	PLATFORM SP62A
S067	PLATFORM SP65A
S068	PLATFORM WD45E-AM
S069	PLATFORM WD45E-PM
S070	QUENCH RECYCLE LIQUOR
S071	DS 1-13 WATER AND GAS PRODUCTION
S072	FS 1 TRAIN B TREATER OUTLET
S073	S-1 RAW INFLUENT
S074	S-2 ROUTE FILTER EFFLUENT
S075	S-3 DEPHENOLIZED EFFLUENT
S076	S-4 FREE NH3 STILL EFFLUENT
S077	S-5 FIXED NH3 EFFLUENT
S078	S-6 BIOLOGICAL TREATMENT INFLUENT
S079	S-7 BIOLOGICAL TREATMENT EFFLUENT
S080	S-8 FILTER EFFLUENT
S081	S-9 ACTIVATED CARBON EFFLUENT
S082	S-10 RESIN COLUMN EFFLUENT
S083	BATCH TEST1 BIOLOGICAL TREATMENT EFFLUENT
S084	BACKGROUND WATER
S085	ALL FRACTIONS 24 HOUR COMPOSITE
S086	ALL FRACTIONS COMPOSITE
S087	100 UG/L LABELLED B/N AND A ADDED
S088	LAB RECEIVED SAMPLE 4 DAYS AFTER COLLECTION
S089	AVG VALUE ASSN TO INDIST ISOMERIC PEAKS
S090	LANDER DW BACKGROUND/STORM WATER RUNOFF
S091	EXTRACTABLE ORGANICS - 24 HOUR COMPOSITE
S092	MICHIGAN DRY WEATHER BACK/STORM WATER RUNOFF
S093	CITY WATER/TAP WATER
S094	LAND WW BACK/SEWERAGE/STORM WATER RO
S095	SEWERAGE/STORM WATER RUNOFF
S096	LANDER CSO/SEWERAGE/STORM WATER RO
S097	LANDER 1ST FLUSH SEWERAGE/STORM WATER RO
S098	LANDER RUNOFF/STORM WATER RUNOFF
S099	MICH WW BACK/SEWERAGE/STORM WATER RO
S100	MICH CS FLOW/STORM WATER RUNOFF

I S O T O P E D I L U T I O N

SAMPLE LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
S101	MICHIGAN CSO/STORM WATER RUNOFF
S102	MICHIGAN FIRST FLUSH/STORM WATER RUNOFF
S103	MICH RUNOFF/STORM WATER RUNOFF
S104	PRECIPITATION/STORM WATER RUNOFF
S105	EUS DW COMP
S106	PHA DW COMP
S107	1 VOA VIAL FOR 19:00 WAS BROKEN
S108	PHN STORM DW COMP
S109	TAP WATER
S110	E AND A DWC
S111	E AND E
S112	1 HOUR COMPOSITE-EXTRACTABLE ORGANICS
S113	WHEN ISOS NOT DISCREET, AVE VALUE ASSIGNED TO SPECIES
S114	E AND A WW BACKGROUND
S115	E AND A CS FL
S116	E AND A CSO
S117	E AND A FIRST FLOW
S118	E AND A RUNOFF
S119	E AND E WW BACKGROUND
S120	E AND E CS FLOW
S121	E AND E CSO
S122	E AND E FIRST FLOW
S123	E AND E RUNOFF
S124	EXTRACTABLE ORGANICS - 8 HOUR COMPOSITE
S125	BCH DW COMP
S126	POOR CHROMATOGRAPHIC RESULTS WITH DIRTY SAMPLE
S127	EUS WASTE WATER BACKGROUND
S128	EUS COMBINED SEWER FLOW
S129	EXTRACTABLE ORGANICS - 1.5 HR. COMPOSITE
S130	EXTRACTABLE ORGANICS - .25 HR. COMPOSITE
S131	EUS CSO
S132	EUS FIRST FLOW
S133	EUS RUNOFF
S134	EXTRACTABLE ORGANICS - .50 HR. COMPOSITE
S135	EUS PRECIPIT
S136	BCH WASTE WATER BACKGROUND
S137	BCH COMBINED SEWER FLOW
S138	BCH CSO
S139	BCH FIRST FLOW
S140	BCH RUNOFF
S141	PIE WASTE WATER BACKGROUND
S142	PIE CS FLOW
S143	PIE CSO
S144	PIE FIRST FLOW
S145	PIE RUNOFF
S146	EQUALIZATION POND-EFFLUENT TO PREAERATION
S147	CLARIFIER EFF. AT CHLORINE CONTACT-CHAMBER INF.
S148	RAW WASTE INFLUENT TO EQUALIZATION
S149	DIOXIN 1 LITER SAMPLE
S150	DIOXIN 10 LITER SAMPLE

I S O T O P E D I L U T I O N

SAMPLE LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
S151	SURGE BASIN EFF. AFTER NEUTRALIZATION
S152	SECONDARY CLARIFIER GRAB
S153	NEUTRALIZATION SURGE TANK EFFLUENT
S154	EQUALIZATION EFF. TO AERATION
S155	SEC. CLARIFIER EFF. TO PRESSURE FILTER
S156	PRESSURE FILTER EFF. TO RIVER
S157	ALKALINE SEWER RAW WASTE GRAB
S158	CLARIFIER EFFLUENT GRAB
S159	ACID SEWER BASIN PUMP TO NEUT.
S160	PRIMARY CLARIFIER INFLUENT
S161	SECONDARY CLARIFIER EFFLUENT
S162	RAW WASTE TO CLARIFIER THICKENER
S163	REACTOR CLARIFIER EFFLUENT TO AERATION
S164	SEC. CLARIFIER TO PRESSURE FILTERS
S165	FINAL PRESSURE TO FILTER EFFLUENT
S166	SOURCE CITY WATER
S167	BATTERY CAN RINSE WASTEWATER
S168	SURFACE TREATMENT RINSE WASTEWATER
S169	OLEFIN UNIT #2 API SEPARATOR EFFLUENT
S170	NEW SURGE TANK EFF. TO AERATION BASIN
S171	SEC. CLARIFIER EFF. TO POLISHING BASIN WEST CLARIFIER
S172	FINAL EFFLUENT FROM POLISHING POND
S173	FINAL CLARIFIER EFFLUENT
S174	18 INCH HEADER INF. PIPE TO EQUALIZATION EG1
S175	EQUALIZATION EC1 TO UNOX
S176	S1B CLARIFIER EFF. TO S2A AND S2B CLARIFIERS
S177	SOURCE WELL WATER
S178	BILLET WASHING AFTER VACUUM CASTING
S179	BILLET WASHING AFTER SINTERING
S180	SAWING/GRINDING COOLANT/LUBRICANT 2C
S181	BE NITRIC ACID PICKLING BATH
S182	BE NITRIC ACID PICKLING RINSE DAY 1
S183	BE NITRIC ACID PICKLING RINSE DAY 2
S184	BE SAWING/GRINDING COOLANT
S185	BE QUALITY INSPECTION WATER
S186	HOT ROLLING BE NI CONTACT COOLING WATER
S187	PROCESS WATER DAY 1
S188	NUMBER 6 LAGOON EFFLUENT DAY 1
S189	PROCESS WATER DAY 2
S190	PROCESS WATER DUP DAY 2
S191	NUMBER 6 LAGOON EFFLUENT DAY 2
S192	NUMBER 6 LAGOON EFFLUENT DAY 3
S193	STEAM STRIPPER INFLUENT GRAB
S194	STEAM STRIPPER EFFLUENT GRAB
S195	INFLUENT TO EQUALIZATION BASIN EC-1
S196	INFLUENT TO AERATION UNITS AB-1
S197	EFFLUENT FROM CLARIFIER S1-A
S198	FINAL EFFLUENT FROM CLARIFIER S2-A
S199	INFLUENT TO STEAM STRIPPER
S200	EFFLUENT FROM STEAM STRIPPER

I S O T O P E D I L U T I O N

SAMPLE LEVEL COMMENT CODE TABLE

CODE	DESCRIPTION
S201	OILY WASTEWATER TREATMENT INF-DAY1
S202	OILY WASTEWATER TREATMENT EFF-DAY 1
S203	OILY WASTEWATER TREATMENT INF-DAY 2
S204	OILY WASTEWATER TREATMENT EFF-DAY 2
S205	VACUUM MELTING STEAM CONDENSATE
S206	EXTRUSION PRESS HEAT TREATMENT CONTACT COOLING H20
S207	OILY WASTEWATER TREATMENT INF-DAY 3
S208	OILY WASTEWATER TREATMENT EFF-DAY 3
S209	PICKLING RINSEWATER TREATMENT INF-DAY 3
S210	INF. TO WWTP API SEPARATOR (APII)
S211	NEW SURGE BASIN EFFLUENT TO AERATION (NSBE)
S212	SECONDARY CLARIFIER EFFLUENT TO POLISHING BASIN (SCE)
S213	FINAL EFFLUENT FROM POLISHING POND (FNE)
S214	SLUDGE RECYCLE GRAB
S215	RECYCLE SLUDGE FROM S1A AND S1B CLARIFIERS
S216	TANK 99 SKIMMER EFFLUENT TO EQUALIZATION
S217	FINAL CLARIFIER EFFLUENT AT 1330 HOURS
S218	SOUTH PLANT WIER BOX-SECONDARY CLARIFIER EFF.
S219	SOUTH PLANT FEED SPLITTER BOX-INF. TO AERATION
S220	EAST SIDE PLANT SECONDARY CLARIFIER EFFLUENT
S221	EAST SIDE PLANT TK1715 OVERFLOW TO AERATION
S222	EAST SIDE PLANT WEST PLANT NEUTRALIZATION SUMP
S223	OP-1 PLANT SECONDARY CLARIFIER EFFLUENT
S224	OP-1 PLANT WASTE H20 TK1721 OVERFLOW INF TO AERA.
S225	OP-1 PLANT TRICKLING FILTER INF. TK1722 OVERFLOW
S226	NEUTRALIZATION BASIN FLAME TO NORTH POND-RAW WASTE
S227	ACTIVATED CARBON FINAL EFFLUENT IN MONITOR BLDG.

ELEMENT NAME: SAMPLE NUMBER

Definition: The SCC assigned identification code which identifies the individual samples. For calibration and performance standards, is used to indicate the nominal concentration of the standard.

Input	**Type/Length**
Quantitation Report	ZZ999
As Stored Internally	9(5)

Unit of Measure

N/A

Edit Criteria:

a. Must be a five digit number.

b. Range: 00001-99999

Examples: 00100 accompanied by a QUANTITATION REPORT TYPE of VER would define a Calibration Verification standard at a nominal concentration of 100 ug/mL (or 100 ug/L for volatiles).

ELEMENT NAME: SAMPLE POINT (SITE)

Definition: The specific point within an industrial wastestream where a sample was taken.

Input	**Type/Length**
Traffic Report	X(1)
Lab Chronicles	
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Sample Site Table.

See attached Sample Site Table for a valid list.

I S O T O P E D I L U T I O N

SITE DESCRIPTION TABLE

CODE	DESCRIPTION
A	(SUP)-RAW WATER (SUPPLY WATER)
B	(PRO)-IN-LINE PROCESS (PROCESS)
C	(INF)-UNTREATED EFFLUENT (RAW WASTE WATER)
D	(EFF)-TREATED EFFLUENT
E	(RUN)-RUNOFF
F	(PRI)-PRIMARY EFFLUENT
G	(INT)-INTERMEDIATE POINT
H	(OTH)-OTHER
I	(IN1)-INTERMEDIATE POINT 1
J	(IN2)-INTERMEDIATE POINT 2
K	(IN3)-INTERMEDIATE POINT 3
L	(IN4)-INTERMEDIATE POINT 4
M	(IN5)-INTERMEDIATE POINT 5

ELEMENT NAME: SAMPLE POINT FLOW

Definition: The flow rate at the point at which the sample was taken. Value is recorded from a flow meter or other flow measuring device.

Input	**Type/Length**
Traffic Report	X(5)
As Stored Internally	X(5)

Unit of Measure

Per 1,000 gallons/day.

Edit Criteria:

ELEMENT NAME: SAMPLE TYPE

Definition: A coded value which describes the type of sample.

Input	**Type/Length**
Traffic Report	X(2)
As Stored Internally	X(2)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Sample Type Code Table.

See attached table for valid codes.

I S O T O P E D I L U T I O N

SAMPLE TYPE CODE TABLE

CODE	DESCRIPTION
AD	ADDITIONAL OR MISCELLANEOUS DATA
CB	COMPOSITE BLANK
EP	EPA SAMPLE
MB	METHOD BLANK
MD	MATRIX REPLICATE(DUPLICATE)
ME	METHOD SPIKE
MS	MATRIX SPIKE
Q	UNSPIKED FRACTION
Q1	SPIKED FRACTION 1
Q2	SPIKED FRACTION 2
R	REGULAR SAMPLE
RB	REAGENT BLANK
RQ	REGULAR AND QA SAMPLE
TB	VOA TRIP BLANK

ELEMENT NAME: SCAN NUMBER

Definition: Gives the scan at which the compound was detected by the mass spectrometer.

Input	**Type/Length**
Quantitation Report	ZZ999
As Stored Internally	9(5)

Unit of Measure

N/A

Edit Criteria:

Range: 00001-99999

ELEMENT NAME: SHIFT

Definition: The scheduled period of operation of the GC/MS instrument. Operation is divided into three shifts/day.

Input	**Type/Length**
Quantitation Report	X(1)
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Code	Meaning
G	Graveyard (0000-0759; Midnight to 8 AM)
D	Day (0800 - 1559; 8 AM to 4 PM)
S	Swing (1600 - 1159; 4 PM to Midnight)

ELEMENT NAME: TIME ANALYZED

Definition: The time thtat the sample fraction was analyzed by the laboratory.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: HH:MM:SS

Appendix F

EVALUATION OF PRR SAMPLE

In doing calculations for calibration linearity and ongoing calibration/verification testing, the variability of analysis results on calibration standards is needed. The CAL 100, VER, and PRR samples are all prepared from standards at 100 µg/mL in organic solvent. The PRR sample was prepared at the central laboratory by mixing solutions of the pollutants and labeled compounds; the 100 µg/mL calibration solution (used for both calibration and calibration verification) was prepared by each laboratory by mixing the same volumes of the same solutions. Because this mixing process was performed in different locations and at different times, the possibility existed that the operations were not performed identically and that results might not be equivalent. Because the PRR sample adds considerably to the number of observations in the procedure, it was decided to include the PRR if it was first checked for bias relative to the CAL 100 and VER samples.

Bias was assessed by performing an analysis of variance of the measured amounts of each compound. A two-way layout was used by laboratory and sample (CAL 100 and VER were assigned the same sample identifier for this test, to be contrasted with PRR). The analysis was conducted on the logarithms of the amounts. The hypothesis tested was that the average amount measured in the PRR sample was equal to that measured in the CAL 100 and VER sample, for each laboratory. Significant results at the .05 level were found in only about 5 percent of the cases. This was likely due to random chance, and no bias was judged to be present in the PRR sample.

Appendix G

LABORATORY EXTREMAL RANK SCREENING

The extreme rank sum test for outliers is a nonparametric analysis of a two-way layout ("objects" x "judges") to decide whether any of the objects has a different mean response from the other objects. This method, proposed by Youden (1963) and discussed by Thompson and Willke (1963), proceeds by calculating the sum, across judges, of the ranking of the set of objects for each judge. If all the objects are equivalent, then the ranking for each judge will be random. Under this null hypothesis, Thompson and Willke present asymptotic significance formulas and small-sample simulation results. For use in this study, the "objects" are the laboratories, the "judges" are the samples, and the quantity of interest is the absolute deviation of the measured amount for each laboratory from the median concentration across all laboratories for the sample. This procedure then tests whether any single laboratory has results that are on the average farther from the common median result than other laboratories' results (in either direction). Since there are usually 7 to 11 laboratories (objects) in the comparison for each compound and 8 to 10 samples (judges), the asymptotic formulas for the significance points were used. At a significance level α (α = .05 in this study), if $(\alpha J!)/2I \leq 1$ (where J is the number of judges and I is the number of objects), the null hypothesis is rejected for any object with rank sum outside of the interval (J + R, IJ - R), where

$$R = I\left(\frac{\alpha J!}{2I}\right)^{\left(\frac{1}{J}\right)} - \frac{J + 1}{2} \quad .$$

If $\alpha J!/2I > 1$, then the interval is

$$\frac{J(I + 1)}{2} \pm Q,$$

where

$$Q = \sqrt{\frac{I(I + 1)J}{12}}\, N[(1-\alpha)^{1/I}] \quad ,$$

N is the inverse cumulative distribution of a standard normal random variable, and $N[(1 - \alpha)^{1/I}]$ is used to obtain an approximate $(1 - \alpha)$th quantile point of the maximum of I normal variates.

In this study, only large values of the absolute deviation are of interest, so only the right-hand limit was tested, and the significance point was adjusted appropriately.

where tabled values are given. In preliminary runs with these two methods QSCREEN found many more points than FSCREEN when both were used at level $\alpha = .01$. To evaluate the source of this difference, several simulation runs were performed to test the methods. One thousand sets each of 5, 10, and 15 standard normal variates were tested with each method at $\alpha = .01$ and $\alpha = .05$, and the mean proportion of rejected points was computed. The results of the simulation are presented in Table H-1.

Table H-1

SIMULATION RESULTS FOR OUTLIER SCREENING METHODS

$\alpha = .01$

Set Size	Number of Sets	Mean Proportion Rejected QSCREEN	FSCREEN
5	1000	0.0000*	0.0022 ± 0.0007
10	1000	0.0149 ± 0.0012	0.0017 ± 0.0004
15	1000	0.0192 ± 0.0013	0.0009 ± 0.0002

$\alpha = .05$

Set Size	Number of Sets	Mean Proportion Rejected QSCREEN	FSCREEN
5	1000	0.0076 ± 0.0012	0.0104 ± 0.0014
10	1000	0.0422 ± 0.0020	0.0076 ± 0.0009
15	1000	0.0517 ± 0.0022	0.0040 ± 0.0005

* The interquantile range (IQR) used in QSCREEN is computed by SAS using a weighted linear combination of adjacent order statistics. For $N < 8$ this includes the extreme points in the scale calculation, hence only very extreme points are rejected by QSCREEN for very small N.

Appendix H

OUTLIER SCREENING METHODS

Two methods of screening individual data values were used in this study to screen across the set of laboratory results for each compound and sample type. The first method, a robust quantile screening method (QSCREEN), was suggested in Hoaglin, Mosteller, and Tukey, Understanding Robust and Exploratory Data Analysis, pp. 30-39. QSCREEN (α) estimates the $(1 - \alpha/2)$th and $\alpha/2$th percentiles of its data by

$$M \pm \frac{N(1-\alpha)}{2\,N(.75)} \cdot IQR \quad ,$$

where M is the median, IQR is the interquantile range (75th percentile - 25th percentile), and N is the inverse distribution function of the normal distribution. Points outside this range are rejected.

The second method, called Ferguson's method (FSCREEN) and based on the sample kurtosis, is described in the "Standard Practice for Dealing with Outlying Observations" 1982 Annual Book of ASTM Standards. FSCREEN (α) computes

$$b_2 = n \sum_{i=1}^{n} (X_i - \bar{X})^4 \Big/ \left(\sum_{i=1}^{n} (X_i - \bar{X})^2 \right)^2$$

and compares it to tabled percentiles of the sample kurtosis. If the tabled value is exceeded at the α level, the farthest point from the mean is dropped. Then b_2 is recomputed on the remaining points, and this procedure is iterated to convergence.

The levels (.001 for QSCREEN, .01 for FSCREEN) used for these screenings were chosen for several reasons. QSCREEN can be adjusted to any desired α level, whereas FSCREEN can be used only at $\alpha = .01$ and $\alpha = .05$,

Therefore, QSCREEN is seen to be performing at very close to its nominal level on normal data, but FSCREEN does not find as many points as it should according to its level. Because extensive checking failed to reveal any problems with the implementation, it can only be suggested that the same problem may exist in the cited tables of significance levels.

So that both methods would perform in practice at approximately the same power, QSCREEN was used for the actual screening at level $\alpha = .001$ and FSCREEN at level $\alpha = .01$. Approximately 2 percent of the actual data was identified as outliers by one or both of these methods.

Appendix I

ESTIMATION OF VARIANCE COMPONENTS

For the purpose of calculating quality control limits from the data in this study, the variance components model assumes that the logarithm of the measured amount X_{lm} measured by laboratory l and replicate m can be written as

$$\log(X_{lm}) = \mu + E_l + A_{lm} \quad ,$$

for $l = 1, \ldots, L$ laboratories and $m = 1, \ldots, n_l$ replicate measurements at laboratory l. μ is the (fixed) average response; E_l is the (random) interlaboratory effect with mean 0 and variance $\sigma^2_{E_2}$; A_{lm} is the (random) intralaboratory effect, with mean 0 and variance σ^2_A.

The variance components analysis was performed by the maximum likelihood method using BMDP3V to estimate the inter- and intralaboratory variance components of the logarithms of the measured amounts. For the start-up and ongoing limits, the replicates used were the BLK, APS, and EPA samples (SAMGRP = WTR), using only the labeled compound results from the BLK and EPA samples.* For the calibration verification limits, the replicates used were the CAL 100, VER, and PRR samples (SAMGRP = CAL). Table I-1 gives the results of the variance components analysis for each sample type and compound series (1 = compounds by internal standard, 2 = labeled analogues by internal standard, 3 = compounds by isotope dilution). For each compound, the total number of observations, the total number of laboratories, the logarithmic mean M (labeled "MU"), the square roots of the variance components S_E (interlaboratory) and S_A (intralaboratory), and the percentage of the total variance due to interlaboratory variation $100 \times S_E^2/(S_E^2 + S_A^2)$ are given.

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* No unlabeled compounds were included in the BLK sample, and the unlabeled compounds in the EPA sample were at varying amounts and only a few compounds were actually present.

Because intralaboratory replicates were not available for series 1 and series 3 compounds for the WTR samples, the total variance $S_E^2 + S_A^2$ for these cases was estimated by the variance among the available measurements (one per lab on the APS sample), and then decomposed into the components according to the ratio of inter- and intralaboratory variance found for the WTR series 2 compounds.

Table I-1

RESULTS OF VARIANCE COMPONENTS ANALYSIS

SERIES=1 SAMGRP=CAL

COMPOUND	TOTAL OBS	TOTAL LABS	MU	S_E	S_A	% VAR DUE TO LAB
001B ACENAPHTHENE	36	13	4.56	0.07	0.08	43.10
005B BENZIDINE	33	11	4.59	0.27	0.45	25.93
008B 1,2,4-TRICHLOROBENZENE	34	12	4.59	0.08	0.11	36.94
009B HEXACHLOROBENZENE	37	13	4.65	0.07	0.21	9.69
012B HEXACHLOROETHANE	31	11	4.61	0.13	0.16	38.19
018B BIS(2-CHLOROETHYL)ETHER	37	13	4.61	0.07	0.18	12.21
020B 2-CHLORONAPHTHALENE	26	9	4.53	0.08	0.14	23.15
021A 2,4,6-TRICHLOROPHENOL	31	12	4.61	0.09	0.12	34.92
022A P-CHLORO-M-CRESOL	29	11	4.50	0.05	0.06	41.02
024A 2-CHLOROPHENOL	37	13	4.57	0.06	0.13	15.94
025B 1,2-DICHLOROBENZENE	37	13	4.60	0.04	0.17	6.22
026B 1,3-DICHLOROBENZENE	37	13	4.56	0.08	0.12	29.45
027B 1,4-DICHLOROBENZENE	35	13	4.58	0.06	0.12	17.82
028B 3,3'-DICHLOROBENZIDINE	32	11	4.66	0.19	0.27	32.35
031A 2,4-DICHLOROPHENOL	37	13	4.62	0.00	0.13	0.00
034A 2,4-DIMETHYLPHENOL	33	12	4.50	0.05	0.08	22.87
035B 2,4-DINITROTOLUENE	36	13	4.65	0.00	0.20	0.00
036B 2,6-DINITROTOLUENE	38	13	4.58	0.13	0.20	27.85
037B 1,2-DIPHENYLHYDRAZINE	30	11	4.61	0.00	0.13	0.00
039B FLUORANTHENE	33	12	4.60	0.07	0.18	14.55
040B 4-CHLOROPHENYL PHENYL ETH	36	13	4.58	0.04	0.14	6.37
041B 4-BROMOPHENYL PHENYL ETHE	25	13	4.65	0.09	0.13	31.13
042B BIS (2-CHLOROISOPROPYL) E	29	10	4.65	0.04	0.17	6.18
052B HEXACHLOROBUTADIENE	34	13	4.60	0.06	0.11	21.03
053B HEXACHLOROCYCLOPENTADIENE	35	13	4.65	0.03	0.14	3.89
054B ISOPHORONE	35	13	4.57	0.14	0.15	46.23
055B NAPHTHALENE	37	13	4.54	0.09	0.14	27.71
056B NITROBENZENE	17	6	4.63	0.06	0.11	20.04
057A 2-NITROPHENOL	35	13	4.60	0.06	0.12	20.83
058A 4-NITROPHENOL	28	10	4.69	0.13	0.28	17.55
059A 2,4-DINITROPHENOL	37	13	4.80	0.19	0.21	44.21
060A 4,6-DINITRO-O-CRESOL	33	12	4.66	0.10	0.19	21.54
062B N-NITROSODIPHENYLAMINE	11	4	4.71	0.05	0.12	14.99
064A PENTACHLOROPHENOL	34	12	4.73	0.09	0.22	14.32
065A PHENOL	37	13	4.56	0.07	0.15	18.63
066B BIS (2-ETHYLHEXYL) PHTHAL	35	12	4.66	0.17	0.24	34.27
068B DI-N-BUTYL PHTHALATE	34	12	4.60	0.10	0.15	30.80
069B DI-N-OCTYL PHTHALATE	36	13	4.60	0.17	0.32	22.68
070B DIETHYL PHTHALATE	36	13	4.58	0.05	0.18	7.44
071B DIMETHYL PHTHALATE	38	13	4.60	0.14	0.18	39.10
072B BENZO(A)ANTHRANCENE	34	12	4.63	0.09	0.27	9.29
073B BENZO(A)PYRENE	34	12	4.47	0.22	0.49	16.40
074B BENZO(B)FLUORANTHENE	31	11	4.62	0.23	0.41	23.59
075B BENZO(K)FLUORANTHENE	35	12	4.46	0.30	0.45	30.21
076B CHRYSENE	36	13	4.60	0.12	0.42	8.00
077B ACENAPHTHYLENE	28	11	4.57	0.06	0.08	42.26
078B ANTHRACENE	38	13	4.57	0.11	0.20	21.74
079B BENZO(GHI)PERYLENE	31	11	4.56	0.03	0.42	0.45
080B FLUORENE	38	13	4.58	0.10	0.17	24.75
081B PHENANTHRENE	38	13	4.60	0.12	0.20	26.35
084B PYRENE	31	11	4.57	0.10	0.19	22.98
502B BETA NAPHTHYLAMINE	30	11	4.60	0.15	0.26	24.95
503B ALPHA PICOLINE	31	11	4.59	0.09	0.21	15.78
504B DIBENZOTHIOPHENE	34	12	4.60	0.10	0.16	25.68
505B DIBENZOFURAN	37	13	4.60	0.03	0.15	3.13
506B N-DODECANE	34	12	4.64	0.10	0.21	17.95
507B DIPHENYLAMINE	31	11	4.62	0.00	0.17	0.00
508B DIPHENYLETHER	33	12	4.55	0.14	0.17	41.62
509B ALPHA TERPINEOL	35	12	4.58	0.15	0.16	44.31
510B STYRENE	34	12	4.59	0.07	0.18	13.80
511B DI-N-BUTYL AMINE	8	3	4.81	0.24	0.46	21.30
512B BIPHENYL	34	12	4.59	0.07	0.11	29.40
513B P-CYMENE	36	13	4.63	0.04	0.16	5.96
517B N-DECANE C10	36	12	4.62	0.13	0.25	19.46
519B N-HEXADECANE C16	38	13	4.58	0.08	0.19	14.77
521B N-EICOSANE C20	38	13	4.61	0.20	0.20	49.53
523B N-TETRACOSANE C24	36	12	4.66	0.11	0.19	25.88
526B N-TRIACONTANE C30	32	12	4.67	0.25	0.28	44.90

Table I-1 (Continued)

SERIES=1 SAMGRP=WTR

COMPOUND	TOTAL OBS	TOTAL LABS	MU	S_E	S_A	% VAR DUE TO LAB
001B ACENAPHTHENE	.	12	4.29	0.11	0.14	39.55
005B BENZIDINE	.	8	3.03	0.92	0.80	57.01
008B 1,2,4-TRICHLOROBENZENE	.	11	4.04	0.35	0.36	47.61
009B HEXACHLOROBENZENE	.	12	4.42	0.13	0.21	28.45
012B HEXACHLOROETHANE	.	10	3.44	0.78	0.72	53.69
018B BIS(2-CHLOROETHYL)ETHER	.	12	4.39	0.18	0.13	66.44
020B 2-CHLORONAPHTHALENE	.	9	3.74	0.43	0.44	48.87
021A 2,4,6-TRICHLOROPHENOL	.	10	4.48	0.10	0.13	39.72
022A P-CHLORO-M-CRESOL	.	10	4.33	0.05	0.35	2.02
024A 2-CHLOROPHENOL	.	12	4.38	0.22	0.14	72.02
025B 1,2-DICHLOROBENZENE	.	11	4.03	0.29	0.19	68.46
026B 1,3-DICHLOROBENZENE	.	12	3.88	0.37	0.35	53.40
027B 1,4-DICHLOROBENZENE	.	12	3.92	0.33	0.31	52.55
028B 3,3'-DICHLOROBENZIDINE	.	9	3.83	0.65	0.47	66.05
031A 2,4-DICHLOROPHENOL	.	12	4.44	0.17	0.13	65.33
034A 2,4-DIMETHYLPHENOL	.	11	3.92	0.52	0.20	86.37
035B 2,4-DINITROTOLUENE	.	12	4.48	0.19	0.11	72.61
036B 2,6-DINITROTOLUENE	.	12	4.46	0.12	0.22	23.82
037B 1,2-DIPHENYLHYDRAZINE	.	10	4.41	0.24	0.20	57.26
039B FLUORANTHENE	.	10	4.30	0.21	0.20	53.15
040B 4-CHLOROPHENYL PHENYL ETH	.	12	4.35	0.13	0.21	27.58
041B 4-BROMOPHENYL PHENYL ETHE	.	12	4.39	.	.	.
042B BIS (2-CHLOROISOPROPYL) E	.	9	4.31	0.12	0.10	57.29
052B HEXACHLOROBUTADIENE	.	12	3.83	0.55	0.46	58.39
053B HEXACHLOROCYCLOPENTADIENE	.	10	2.23	1.59	0.88	76.40
054B ISOPHORONE	.	11	4.39	0.19	0.18	52.24
055B NAPHTHALENE	.	12	4.14	0.24	0.25	47.48
056B NITROBENZENE	.	5	4.35	0.13	0.10	64.65
057A 2-NITROPHENOL	.	12	4.40	0.19	0.13	66.21
058A 4-NITROPHENOL	.	9	4.29	0.22	0.43	21.24
059A 2,4-DINITROPHENOL	.	12	4.51	0.32	0.27	58.76
060A 4,6-DINITRO-O-CRESOL	.	11	4.63	0.40	0.43	47.09
062B N-NITROSODIPHENYLAMINE	.	4	4.57	0.10	0.31	9.35
064A PENTACHLOROPHENOL	.	11	4.35	0.24	0.24	51.25
065A PHENOL	.	12	4.34	0.00	0.33	0.00
066B BIS (2-ETHYLHEXYL) PHTHAL	.	9	4.47	0.20	0.11	74.49
068B DI-N-BUTYL PHTHALATE	.	11	4.19	0.36	0.17	81.78
069B DI-N-OCTYL PHTHALATE	.	11	4.34	0.21	0.11	78.01
070B DIETHYL PHTHALATE	.	11	3.90	0.43	0.49	43.72
071B DIMETHYL PHTHALATE	.	12	3.52	0.71	0.66	53.22
072B BENZO(A)ANTHRANCENE	.	9	4.37	0.21	0.12	76.50
073B BENZO(A)PYRENE	.	10	4.39	0.24	0.13	75.95
074B BENZO(B)FLUORANTHENE	.	10	4.00	0.59	0.71	41.07
075B BENZO(K)FLUORANTHENE	.	11	3.74	0.84	0.76	54.83
076B CHRYSENE	.	10	4.34	0.13	0.17	37.25
077B ACENAPHTHYLENE	.	10	4.34	0.12	0.11	51.03
078B ANTHRACENE	.	12	4.25	0.17	0.18	46.18
079B BENZO(GHI)PERYLENE	.	9	4.39	0.34	0.23	69.35
080B FLUORENE	.	12	4.38	0.07	0.17	14.31
081B PHENANTHRENE	.	11	4.30	0.09	0.18	20.42
084B PYRENE	.	10	4.19	0.37	0.25	67.66
502B BETA NAPHTHYLAMINE	.	9	3.69	1.29	0.29	95.09
503B ALPHA PICOLINE	.	9	4.11	0.28	0.39	33.77
504B DIBENZOTHIOPHENE	.	11	4.32	0.11	0.17	32.39
505B DIBENZOFURAN	.	12	4.37	0.13	0.17	38.41
506B N-DODECANE	.	11	3.86	0.89	0.62	66.93
507B DIPHENYLAMINE	.	10	4.33	0.20	0.18	54.56
508B DIPHENYLETHER	.	11	4.23	0.12	0.13	46.45
509B ALPHA TERPINEOL	.	11	4.40	0.12	0.08	68.35
510B STYRENE	.	11	3.75	0.41	0.30	64.12
511B DI-N-BUTYL AMINE	.	2	1.45	0.00	0.75	0.00
512B BIPHENYL	.	11	4.26	0.10	0.26	11.92
513B P-CYMENE	.	11	3.84	0.38	0.30	60.31
517B N-DECANE C10	.	11	3.03	1.02	0.96	52.90
519B N-HEXADECANE C16	.	10	4.33	0.14	0.18	38.26
521B N-EICOSANE C20	.	12	4.41	0.17	0.14	58.01
523B N-TETRACOSANE C24	.	10	4.46	0.21	0.11	77.54
526B N-TRIACONTANE C30	.	10	4.40	0.23	0.16	68.80

Table I-1 (Continued)

SERIES=2 SAMGRP=CAL

COMPOUND	TOTAL OBS	TOTAL LABS	MU	S_E	S_A	% VAR DUE TO LAB
201B ACENAPHTHENE-D10	33	12	4.60	0.00	0.07	0.00
205B BENZIDINE-D8 (RINGS-D8)	31	11	4.74	0.00	0.52	0.00
208B 1,2,4-TRICHLOROBENZENE-D3	30	12	4.63	0.00	0.10	0.00
209B HEXACHLOROBENZENE-13C6	31	11	4.67	0.03	0.20	1.91
212B HEXACHLOROETHANE-1-13C	27	10	4.62	0.00	0.15	0.00
218B BIS(2-CHLOROETHYL)-D8 ETH	29	10	4.59	0.03	0.14	4.16
220B 2-CHLORONAPHTHALENE-D7	35	13	4.59	0.00	0.07	0.00
221A 2,4,6-TRICHLOROPHENOL-3,5	27	10	4.60	0.08	0.07	56.79
222A 4-CHLORO-3-METHYLPHENOL-2	35	13	4.62	0.00	0.08	0.00
224A 2-CHLOROPHENOL-3,4,5,6-D4	34	12	4.60	0.00	0.12	0.00
225B 1,2-DICHLOROBENZENE-D4	31	12	4.62	0.00	0.10	0.00
226B 1,3-DICHLOROBENZENE-D4	33	12	4.60	0.00	0.14	0.00
227B 1,4-DICHLOROBENZENE-D4	33	13	4.62	0.00	0.09	0.00
228B 3,3'-DICHLOROBENZIDINE-D6	31	11	4.68	0.14	0.36	12.72
231A 2,4-DICHLOROPHENOL-3,5,6-	35	13	4.63	0.00	0.10	0.00
234A 2,4-DIMETHYLPHENOL-3,5,6-	37	13	4.59	0.03	0.12	5.20
235B 2,4-DINITROTOLUENE-3,5,6-	27	10	4.64	0.00	0.12	0.00
236B 2,6-DINITROTOLUENE-D3	23	9	4.59	0.00	0.19	0.00
237B 1,2-DIPHENYL-D10-HYDRAZIN	33	12	4.61	0.02	0.12	2.45
239B FLUORANTHENE-D10	34	13	4.67	0.00	0.16	0.00
240B 4-CHLOROPHENYL PHENYL-D5	36	13	4.60	0.04	0.12	7.97
242B BIS(2-CHLOROISOPROPYL)ETH	29	10	4.63	0.00	0.17	0.00
252B HEXACHLORO-1,3-BUTADIENE-	31	12	4.61	0.00	0.08	0.00
253B HEXACHLOROCYCLOPENTADIENE	28	10	4.74	0.00	0.15	0.00
254B ISOPHORONE-D8	36	13	4.62	0.00	0.14	0.00
255B NAPHTHALENE-D8	35	13	4.59	0.00	0.07	0.00
256B NITROBENZENE-D5	14	5	4.60	0.00	0.12	0.00
257A 2-NITROPHENOL-3,4,5,6-D4	36	13	4.62	0.04	0.10	14.68
258A 4-NITROPHENOL-2,3,5,6-D4	30	11	4.77	0.21	0.22	48.77
259A 2,4-DINITROPHENOL-3,5,6-D	34	12	4.73	0.06	0.20	9.10
260A 4,6-DINITRO-O-CRESOL-D2	33	12	4.69	0.05	0.12	13.65
262B N-NITROSODIPHENYLAMINE-D6	22	8	4.59	0.00	0.10	0.00
264A PENTACHLOROPHENOL-13C6	34	12	4.68	0.04	0.18	4.33
265A PHENOL-2,3,4,5,6-D5	36	13	4.61	0.01	0.16	0.74
266B BIS(2-ETHYLHEXYL)PHTHALAT	31	11	4.64	0.16	0.17	44.70
268B DI-N-BUTYL PHTHALATE-D4	32	12	4.61	0.05	0.13	11.51
269B DI-N-OCTYL PHTHALATE-D4	35	13	4.66	0.10	0.33	8.01
270B DIETHYL PHTHALATE-3,4,5,6	36	13	4.62	0.00	0.16	0.00
271B DIMETHYL PHTHALATE-3,4,5,	37	13	4.59	0.03	0.15	4.27
272B BENZO(A)ANTHRACENE-D12	32	12	4.65	0.09	0.26	11.35
273B BENZO(A)PYRENE-D12	34	12	4.56	0.17	0.44	13.25
274B BENZO(B)FLUORANTHENE-D12	32	11	4.59	0.14	0.41	10.49
275B BENZO(K)FLUORANTHENE-D12	35	12	4.57	0.00	0.43	0.00
276B CHRYSENE-D12	34	12	4.62	0.07	0.30	5.62
277B ACENAPHTHYLENE-D8	35	13	4.60	0.00	0.09	0.00
278B ANTHRACENE-D10	29	11	4.58	0.00	0.11	0.00
279B BENZO(GHI)PERYLENE-D12	34	12	4.54	0.00	0.43	0.00
280B FLUORENE-D10	35	13	4.61	0.00	0.10	0.00
281B PHENANTHRENE-D10	33	12	4.63	0.06	0.08	34.99
284B PYRENE-D10	31	12	4.70	0.17	0.15	54.73
602B 2-NAPHTHYL-D7-AMINE	30	11	4.73	0.15	0.17	44.20
603B 2-METHYLPYRIDINE-D7	34	12	4.61	0.00	0.25	0.00
604B DIBENZOTHIOPHENE-D8	27	11	4.61	0.04	0.07	26.94
605B DIBENZOFURAN-D8	33	12	4.60	0.00	0.09	0.00
606B N-DODECANE-D26	39	13	4.62	0.05	0.19	6.38
607B DIPHENYL-D10-AMINE	24	9	4.63	0.03	0.10	7.44
608B DIPHENYL-D10 ETHER	31	12	4.62	0.00	0.05	0.00
609B ALPHA-TERPINEOL-D3	33	11	4.68	0.05	0.34	1.87
610B STYRENE-2,3,4,5,6-D5	34	12	4.59	0.09	0.17	21.82
611B DI-N-BUTYL-D18-AMINE	12	4	4.71	0.00	0.53	0.00
612B DIPHENYL-D10	12	4	4.59	0.10	0.09	56.59
613B P-CYMENE-D14	34	13	4.60	0.00	0.09	0.00
617B N-DECANE-D22	36	13	4.69	0.10	0.17	24.32
619B N-HEXADECANE-D34	37	13	4.60	0.00	0.13	0.00
621B N-EICOSANE-D42	31	11	4.61	0.07	0.10	30.17
623B N-TETRACOSANE-D50	35	12	4.67	0.10	0.15	33.57
626B N-TRIACONTANE-D62	33	12	4.61	0.23	0.30	37.27

Table I-1 (Continued)

SERIES=2 SAMGRP=WTR

COMPOUND	TOTAL OBS	TOTAL LABS	MU	S_E	S_A	% VAR DUE TO LAB
201B ACENAPHTHENE-D10	33	11	4.29	0.17	0.22	39.55
205B BENZIDINE-D8 (RINGS-D8)	21	8	3.45	1.12	0.97	57.01
208B 1,2,4-TRICHLOROBENZENE-D3	32	11	3.98	0.35	0.37	47.61
209B HEXACHLOROBENZENE-13C6	29	10	4.46	0.21	0.33	28.45
212B HEXACHLOROETHANE-1-13C	26	9	3.71	0.55	0.51	53.69
218B BIS(2-CHLOROETHYL)-D8 ETH	30	10	4.30	0.27	0.19	66.44
220B 2-CHLORONAPHTHALENE-D7	36	12	4.24	0.24	0.24	48.87
221A 2,4,6-TRICHLOROPHENOL-3,5	24	9	4.47	0.17	0.21	39.72
222A 4-CHLORO-3-METHYLPHENOL-2	33	12	4.19	0.07	0.49	2.02
224A 2-CHLOROPHENOL-3,4,5,6-D4	30	11	4.33	0.22	0.13	72.02
225B 1,2-DICHLOROBENZENE-D4	32	11	3.96	0.39	0.26	68.46
226B 1,3-DICHLOROBENZENE-D4	32	11	3.89	0.37	0.35	53.40
227B 1,4-DICHLOROBENZENE-D4	35	12	3.96	0.35	0.33	52.55
228B 3,3'-DICHLOROBENZIDINE-D6	30	10	4.04	0.61	0.44	66.05
231A 2,4-DICHLOROPHENOL-3,5,6-	33	12	4.36	0.21	0.15	65.33
234A 2,4-DIMETHYLPHENOL-3,5,6-	34	12	4.05	0.42	0.17	86.37
235B 2,4-DINITROTOLUENE-3,5,6-	28	10	4.28	0.34	0.21	72.61
236B 2,6-DINITROTOLUENE-D3	23	8	4.48	0.14	0.25	23.82
237B 1,2-DIPHENYL-D10-HYDRAZIN	33	11	4.28	0.24	0.20	57.26
239B FLUORANTHENE-D10	34	12	4.31	0.21	0.20	53.15
240B 4-CHLOROPHENYL PHENYL-D5	36	12	4.36	0.17	0.27	27.58
242B BIS(2-CHLOROISOPROPYL)ETH	27	9	4.27	0.19	0.16	57.29
252B HEXACHLORO-1,3-BUTADIENE-	32	11	3.82	0.51	0.43	58.39
253B HEXACHLOROCYCLOPENTADIENE	20	9	2.33	1.50	0.83	76.40
254B ISOPHORONE-D8	32	11	4.39	0.13	0.13	52.24
255B NAPHTHALENE-D8	35	12	4.17	0.23	0.25	47.48
256B NITROBENZENE-D5	12	4	4.23	0.19	0.14	64.65
257A 2-NITROPHENOL-3,4,5,6-D4	33	12	4.33	0.19	0.13	66.21
258A 4-NITROPHENOL-2,3,5,6-D4	29	10	4.17	0.33	0.64	21.24
259A 2,4-DINITROPHENOL-3,5,6-D	31	11	4.38	0.36	0.31	58.76
260A 4,6-DINITRO-O-CRESOL-D2	30	11	4.53	0.25	0.27	47.09
262B N-NITROSODIPHENYLAMINE-D6	20	7	4.40	0.06	0.18	9.35
264A PENTACHLOROPHENOL-13C6	30	11	4.46	0.24	0.23	51.25
265A PHENOL-2,3,4,5,6-D5	33	12	4.05	0.00	0.63	0.00
266B BIS(2-ETHYLHEXYL)PHTHALAT	28	10	4.38	0.26	0.15	74.42
268B DI-N-BUTYL PHTHALATE-D4	32	11	4.21	0.31	0.15	81.78
269B DI-N-OCTYL PHTHALATE-D4	34	12	4.19	0.52	0.27	78.01
270B DIETHYL PHTHALATE-3,4,5,6	34	12	3.78	0.45	0.51	43.72
271B DIMETHYL PHTHALATE-3,4,5,	35	12	3.24	0.82	0.77	53.22
272B BENZO(A)ANTHRACENE-D12	31	11	4.44	0.36	0.20	76.50
273B BENZO(A)PYRENE-D12	30	11	4.37	0.24	0.13	75.95
274B BENZO(B)FLUORANTHENE-D12	28	10	4.24	0.49	0.58	41.07
275B BENZO(K)FLUORANTHENE-D12	31	11	4.39	0.48	0.44	54.83
276B CHRYSENE-D12	30	11	4.40	0.24	0.31	37.25
277B ACENAPHTHYLENE-D8	36	12	4.31	0.18	0.18	51.03
278B ANTHRACENE-D10	30	10	4.32	0.24	0.26	46.18
279B BENZO(GHI)PERYLENE-D12	30	11	4.46	0.32	0.21	69.35
280B FLUORENE-D10	34	12	4.39	0.09	0.22	14.31
281B PHENANTHRENE-D10	33	11	4.32	0.11	0.22	20.42
284B PYRENE-D10	31	11	4.30	0.24	0.17	67.66
602B 2-NAPHTHYL-D7-AMINE	24	10	3.66	1.33	0.30	95.09
603B 2-METHYLPYRIDINE-D7	32	11	4.04	0.42	0.59	33.77
604B DIBENZOTHIOPHENE-D8	28	10	4.36	0.12	0.17	32.39
605B DIBENZOFURAN-D8	32	11	4.37	0.13	0.17	38.41
606B N-DODECANE-D26	35	12	3.79	0.57	0.40	66.93
607B DIPHENYL-D10-AMINE	24	8	4.29	0.25	0.23	54.56
608B DIPHENYL-D10 ETHER	32	11	4.30	0.19	0.21	46.45
609B ALPHA-TERPINEOL-D3	30	10	4.37	0.36	0.24	68.35
610B STYRENE-2,3,4,5,6-D5	32	11	3.81	0.50	0.37	64.12
611B DI-N-BUTYL-D18-AMINE	8	3	3.38	0.00	1.05	0.00
612B DIPHENYL-D10	12	4	4.20	0.08	0.21	11.92
613B P-CYMENE-D14	35	12	3.79	0.57	0.46	60.31
617B N-DECANE-D22	34	12	3.78	0.51	0.48	52.90
619B N-HEXADECANE-D34	32	12	4.32	0.19	0.24	38.26
621B N-EICOSANE-D42	33	11	4.32	0.23	0.19	58.01
623B N-TETRACOSANE-D50	31	11	4.32	0.30	0.16	77.54
626B N-TRIACONTANE-D62	31	11	4.37	0.32	0.21	68.80

Table I-1 (Continued)

SERIES=3 SAMGRP=CAL

COMPOUND	TOTAL OBS	TOTAL LABS	MU	S_E	S_A	% VAR DUE TO LAB
301B ACENAPHTHENE	36	12	4.58	0.04	0.05	41.17
305B BENZIDINE	31	11	4.45	0.15	0.22	30.03
308B 1,2,4-TRICHLOROBENZENE	33	11	4.58	0.02	0.05	11.53
309B HEXACHLOROBENZENE	32	11	4.60	0.00	0.05	0.00
312B HEXACHLOROETHANE	26	9	4.56	0.15	0.07	82.53
318B BIS(2-CHLOROETHYL)ETHER	30	10	4.58	0.06	0.10	28.49
320B 2-CHLORONAPHTHALENE	26	9	4.52	0.13	0.11	61.25
321A 2,4,6-TRICHLOROPHENOL	33	11	4.60	0.09	0.04	79.85
322A P-CHLORO-M-CRESOL	31	11	4.52	0.07	0.03	85.61
324A 2-CHLOROPHENOL	36	12	4.58	0.07	0.05	63.20
325B 1,2-DICHLOROBENZENE	32	12	4.58	0.02	0.06	10.34
326B 1,3-DICHLOROBENZENE	34	12	4.62	0.03	0.09	10.16
327B 1,4-DICHLOROBENZENE	37	13	4.59	0.04	0.10	14.17
328B 3,3'-DICHLOROBENZIDINE	32	11	4.61	0.06	0.05	52.28
331A 2,4-DICHLOROPHENOL	37	13	4.58	0.03	0.09	12.21
334A 2,4-DIMETHYLPHENOL	35	12	4.54	0.04	0.09	17.37
335B 2,4-DINITROTOLUENE	29	10	4.58	0.08	0.05	74.02
336B 2,6-DINITROTOLUENE	24	9	4.56	0.03	0.12	7.74
337B 1,2-DIPHENYLHYDRAZINE	35	12	4.59	0.07	0.06	55.97
339B FLUORANTHENE	39	13	4.58	0.04	0.09	14.01
340B 4-CHLOROPHENYL PHENYL ETH	38	13	4.59	0.01	0.08	3.20
342B BIS (2-CHLOROISOPROPYL) E	30	10	4.61	0.05	0.08	24.65
352B HEXACHLOROBUTADIENE	34	12	4.58	0.00	0.06	0.00
353B HEXACHLOROCYCLOPENTADIENE	29	10	4.63	0.07	0.05	62.33
354B ISOPHORONE	33	12	4.59	0.05	0.07	27.27
355B NAPHTHALENE	39	13	4.59	0.03	0.07	17.16
356B NITROBENZENE	15	5	4.62	0.05	0.01	93.28
357A 2-NITROPHENOL	39	13	4.61	0.04	0.06	32.25
358A 4-NITROPHENOL	27	10	4.56	0.04	0.12	11.40
359A 2,4-DINITROPHENOL	36	12	4.60	0.08	0.06	63.31
360A 4,6-DINITRO-O-CRESOL	33	11	4.56	0.07	0.08	41.42
362B N-NITROSODIPHENYLAMINE	18	6	4.58	0.07	0.07	49.64
364A PENTACHLOROPHENOL	36	12	4.58	0.06	0.06	54.61
365A PHENOL	39	13	4.55	0.07	0.10	34.27
366B BIS (2-ETHYLHEXYL) PHTHAL	39	13	4.59	0.02	0.06	7.93
368B DI-N-BUTYL PHTHALATE	36	13	4.61	0.02	0.07	6.56
369B DI-N-OCTYL PHTHALATE	39	13	4.60	0.00	0.07	0.37
370B DIETHYL PHTHALATE	37	13	4.59	0.00	0.07	0.00
371B DIMETHYL PHTHALATE	38	13	4.60	0.08	0.07	56.58
372B BENZO(A)ANTHRANCENE	31	11	4.58	0.06	0.07	44.49
373B BENZO(A)PYRENE	34	12	4.61	0.04	0.05	36.22
374B BENZO(B)FLUORANTHENE	32	11	4.57	0.08	0.10	35.86
375B BENZO(K)FLUORANTHENE	32	12	4.70	0.20	0.42	18.62
376B CHRYSENE	37	13	4.59	0.05	0.08	30.07
377B ACENAPHTHYLENE	35	12	4.56	0.00	0.11	0.00
378B ANTHRACENE	35	12	4.58	0.04	0.11	10.71
379B BENZO(GHI)PERYLENE	28	10	4.62	0.06	0.07	38.29
380B FLUORENE	38	13	4.53	0.11	0.06	73.61
381B PHENANTHRENE	39	13	4.60	0.04	0.06	25.52
384B PYRENE	33	12	4.56	0.08	0.06	64.10
702B BETA NAPHTHYLAMINE	31	11	4.56	0.02	0.19	0.93
703B ALPHA PICOLINE	28	10	4.62	0.03	0.10	9.83
704B DIBENZOTHIOPHENE	35	12	4.60	0.04	0.07	23.65
705B DIBENZOFURAN	32	11	4.59	0.00	0.06	0.00
706B N-DODECANE C12	36	12	4.59	0.05	0.11	16.07
707B DIPHENYLAMINE	27	9	4.60	0.08	0.11	32.43
708B DIPHENYLETHER	33	11	4.59	0.06	0.04	67.95
709B ALPHA TERPINEOL	20	7	4.51	0.00	0.11	0.00
710B STYRENE	31	11	4.61	0.04	0.09	20.02
711B DI-N-BUTYL AMINE	6	2	4.79	0.13	0.26	20.39
712B BIPHENYL	24	8	4.61	0.09	0.10	43.31
713B P-CYMENE	31	12	4.60	0.06	0.05	61.07
717B N-DECANE C10	32	11	4.52	0.09	0.18	19.31
719B N-HEXADECANE C16	37	13	4.58	0.09	0.07	60.10
721B N-EICOSANE C20	39	13	4.62	0.06	0.13	16.43
723B N-TETRACOSANE C24	39	13	4.61	0.00	0.09	0.00
726B N-TRIACONTANE C30	38	13	4.63	0.13	0.09	68.20

Table I-1 (Concluded)

SERIES=3 SAMGRP=WTR

COMPOUND	TOTAL OBS	TOTAL LABS	MU	S_E	S_A	% VAR DUE TO LAB
301B ACENAPHTHENE	.	10	4.63	0.06	0.08	39.55
305B BENZIDINE	.	8	4.47	0.42	0.36	57.01
308B 1,2,4-TRICHLOROBENZENE	.	9	4.66	0.06	0.06	47.61
309B HEXACHLOROBENZENE	.	9	4.66	0.03	0.05	28.45
312B HEXACHLOROETHANE	.	8	4.88	0.45	0.42	53.69
318B BIS(2-CHLOROETHYL)ETHER	.	9	4.64	0.17	0.12	66.44
320B 2-CHLORONAPHTHALENE	.	9	4.83	0.25	0.26	48.87
321A 2,4,6-TRICHLOROPHENOL	.	9	4.69	0.14	0.18	39.72
322A P-CHLORO-M-CRESOL	.	9	4.60	0.02	0.13	2.02
324A 2-CHLOROPHENOL	.	10	4.63	0.07	0.05	72.02
325B 1,2-DICHLOROBENZENE	.	10	4.64	0.09	0.06	68.46
326B 1,3-DICHLOROBENZENE	.	10	4.72	0.15	0.14	53.40
327B 1,4-DICHLOROBENZENE	.	11	4.68	0.15	0.15	52.55
328B 3,3'-DICHLOROBENZIDINE	.	10	4.69	0.13	0.09	66.05
331A 2,4-DICHLOROPHENOL	.	12	4.66	0.06	0.04	65.33
334A 2,4-DIMETHYLPHENOL	.	10	4.57	0.13	0.05	86.37
335B 2,4-DINITROTOLUENE	.	8	4.69	0.10	0.06	72.61
336B 2,6-DINITROTOLUENE	.	8	4.66	0.05	0.10	23.82
337B 1,2-DIPHENYLHYDRAZINE	.	11	4.79	0.25	0.22	57.26
339B FLUORANTHENE	.	11	4.71	0.12	0.11	53.15
340B 4-CHLOROPHENYL PHENYL ETH	.	12	4.71	0.09	0.15	27.58
342B BIS (2-CHLOROISOPROPYL) E	.	9	4.66	0.07	0.06	57.29
352B HEXACHLOROBUTADIENE	.	11	4.71	0.22	0.18	58.39
353B HEXACHLOROCYCLOPENTADIENE	.	5	4.60	0.07	0.04	76.40
354B ISOPHORONE	.	11	4.69	0.10	0.09	52.24
355B NAPHTHALENE	.	11	4.66	0.07	0.08	47.48
356B NITROBENZENE	.	4	4.65	0.06	0.04	64.65
357A 2-NITROPHENOL	.	12	4.65	0.08	0.06	66.21
358A 4-NITROPHENOL	.	8	4.55	0.08	0.15	21.24
359A 2,4-DINITROPHENOL	.	11	4.58	0.09	0.07	58.76
360A 4,6-DINITRO-O-CRESOL	.	9	4.62	0.07	0.07	47.09
362B N-NITROSODIPHENYLAMINE	.	5	4.56	0.04	0.11	9.35
364A PENTACHLOROPHENOL	.	11	4.63	0.08	0.08	51.25
365A PHENOL	.	12	4.58	0.00	0.14	0.00
366B BIS (2-ETHYLHEXYL) PHTHAL	.	11	4.81	0.17	0.10	74.49
368B DI-N-BUTYL PHTHALATE	.	9	4.72	0.11	0.05	81.78
369B DI-N-OCTYL PHTHALATE	.	12	4.71	0.11	0.06	78.01
370B DIETHYL PHTHALATE	.	12	4.79	0.13	0.14	43.72
371B DIMETHYL PHTHALATE	.	11	4.77	0.12	0.12	53.22
372B BENZO(A)ANTHRANCENE	.	9	4.65	0.13	0.07	76.50
373B BENZO(A)PYRENE	.	11	4.70	0.16	0.09	75.95
374B BENZO(B)FLUORANTHENE	.	9	4.83	0.33	0.39	41.07
375B BENZO(K)FLUORANTHENE	.	9	4.51	0.11	0.10	54.83
376B CHRYSENE	.	12	4.64	0.14	0.19	37.25
377B ACENAPHTHYLENE	.	11	4.73	0.13	0.13	51.03
378B ANTHRACENE	.	11	4.61	0.14	0.15	46.18
379B BENZO(GHI)PERYLENE	.	9	4.67	0.11	0.07	69.33
380B FLUORENE	.	12	4.63	0.05	0.11	14.31
381B PHENANTHRENE	.	10	4.65	0.02	0.05	20.42
384B PYRENE	.	10	4.67	0.10	0.07	67.66
702B BETA NAPHTHYLAMINE	.	6	4.69	0.55	0.13	95.08
703B ALPHA PICOLINE	.	9	4.54	0.10	0.14	33.77
704B DIBENZOTHIOPHENE	.	10	4.69	0.07	0.11	32.39
705B DIBENZOFURAN	.	10	4.67	0.06	0.07	38.41
706B N-DODECANE C12	.	11	4.71	0.33	0.23	66.93
707B DIPHENYLAMINE	.	8	4.69	0.15	0.14	54.56
708B DIPHENYLETHER	.	9	4.66	0.06	0.07	46.45
709B ALPHA TERPINEOL	.	6	4.59	0.18	0.12	68.35
710B STYRENE	.	10	4.68	0.19	0.14	64.12
711B DI-N-BUTYL AMINE	.	2	3.46	0.00	2.93	0.00
712B BIPHENYL	.	7	4.65	0.05	0.12	11.92
713B P-CYMENE	.	9	4.63	0.08	0.06	60.31
717B N-DECANE C10	.	9	4.21	0.26	0.24	52.90
719B N-HEXADECANE C16	.	11	4.73	0.09	0.11	38.26
721B N-EICOSANE C20	.	12	4.76	0.22	0.19	58.01
723B N-TETRACOSANE C24	.	9	4.66	0.07	0.04	77.54
726B N-TRIACONTANE C30	.	12	4.70	0.17	0.12	68.80

Appendix J

BINOMIAL CALCULATIONS FOR MULTIPLE TESTS

Because of the large number of compounds that may be tested in Method 1625A, the individual compound test criteria probability levels are determined in the start-up and continuing QA/QC tests to account for the simultaneous testing of multiple compounds. In other EPA method validation studies, the compound-specific performance specifications have usually been determined using a 5 percent probability level. However, for Method 1625, if the individual test level is left at .05, the chance that at least one test will fail approaches certainty as the number of tests increases. In particular, for the start-up test on Method 1625A, there are over 150 compounds, each tested for precision and accuracy, for a total of over 300 tests. If each item is tested at the .05 level, the odds are about 1 in 5 million* of all tests being passed, even if the equipment is perfect, due to random variation, assuming tests are passed or failed independently.

Two factors can be adjusted to account for this effect: the rejection level for the test can be made smaller, and a retest can be allowed for those items that failed the first round. Table J-1 presents the probabilities associated with various possibilities. Assume that N tests are performed in the first round, each with individual test level p. In the calculations that follow, it is assumed that the results for each individual test are independent.

The probability of failure for one or more items on the first round is

$$1 - (1-p)^N \quad .$$

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* $.95^{300} \approx 2 \times 10^{-7}$

Table J-1

PROBABILITY OF FAILING QUALITY CONTROL TESTS

Number of Tests	Individual Test Level	Probability Fail Round 1	Probability Fail Round 2
10	0.050	0.401	0.025
	0.020	0.183	0.004
	0.010	0.096	0.001
	0.001	0.010	0.000
50	0.050	0.923	0.118
	0.020	0.636	0.020
	0.010	0.395	0.005
	0.001	0.049	0.000
60	0.050	0.954	0.139
	0.020	0.702	0.024
	0.010	0.453	0.006
	0.001	0.058	0.000
120	0.050	0.998	0.259
	0.020	0.911	0.047
	0.010	0.701	0.012
	0.001	0.113	0.000
150	0.050	1.000	0.313
	0.020	0.952	0.058
	0.010	0.779	0.015
	0.001	0.139	0.000
300	0.050	1.000	0.528
	0.020	0.998	0.113
	0.010	0.951	0.030
	0.001	0.259	0.000

The average probability of failure in the second round is obtained by averaging the probabilities of failure given K failures in the first round, i.e.,

$$P(\text{fail in round 2}) = 1 - \sum_{K=0}^{N} \binom{N}{K} p^K (1-p)^{N-K} (1-p)^K \quad .$$

As the table demonstrates, even for small values of N, there is a significant probability of failure in the first round with .05 level tests, though the probability of failure on the second round is quite small. For 300 tests, even with the retest allowed, the overall probability of failure is over 50 percent. Dropping the test level to .01 decreases the second-round failure probability to under 5 percent, and therefore this would be the recommended procedure in situations with more than a few tests.

In order to avoid the second round of tests, smaller test levels are necessary. For instance, for 50 tests, if the test levels are set at .001, the chance of failing on one or more test criteria on the first round is reduced to less than 5 percent. This would allow a test procedure which can be performed in one round of testing. For more than 50 tests, even smaller rejecton levels would be necessary. For instance, an individual level of .0001 would achieve 5 percent overall for up to 500 tests.

In considering a two-round test, it would be useful to calculate limits on the number of failures in the first round of testing, such that if the analyst observes this many failures or more, he will not waste time with a second round of testing, but instead proceed to correcting and recalibrating his instrumentation. These limits would not be considered part of the actual test procedure, but instead could be considered as cost/benefit guidelines for the analyst in deciding whether to attempt the second round of testing.

A reasonable way to calculate such a limit involves a retrospective test of the hypothesis that the test failures are not actually occurring at the specified level p, and suggests that the operator abandon the second round if a binomial test with K failures out of N tries rejects the level p at significance level .05.

If this many failures are seen in the first round, it is highly likely that there is a problem with the instrument, and the chances of passing the second round are probably low. (Note this does not imply the converse, since if there are problems with only a few compounds, only a few failures might be seen in the first round and the problem compounds will only be detected on the second round of testing.) This cutoff number depends on both the number of initial tests N and the individual level p. The recommended value of K for each current EPA analytical method is given in Table J-2.

Table J-2

FIRST-ROUND CUTOFFS FOR TWO-ROUND TESTING

Method	Number of Compounds	Number of Start-up Items	Individual p Level	Cutoff for[1] Start-up	Cutoff for[2] Ongoing
601	28	56	.05	7	4
602	7	14	.05	3	2
602/605	2	4	.05	2	2
606	6	12	.05	3	2
607	3	6	.05	2	2
608	24	48	.05	6	4
609	4	8	.05	3	2
610	16	32	.05	5	3
611	5	10	.05	3	2
612	9	18	.05	4	3
613	1	2	.05	2	1
624	31	62	.05	7	5
625 A	12	24	.05	4	3
625 B/N	48	96	.05	9	6
1624	60	120	.05	11	7
1625A	154	308	.01	7	5

[1] N = number of start-up items

[2] N = number of compounds

Appendix K

DERIVATION OF QUALITY CONTROL LIMITS FOR ACCURACY

If we observe a test series $X_1, \ldots X_N$ independently drawn from a normal distribution with unknown mean μ and unknown variance σ^2, the mean and variance can be estimated by

$$\bar{X} = \frac{1}{N} \sum_{i=1}^{N} X_i$$

$$S^2 = \frac{1}{N-1} \sum_{i=1}^{N} (X_i - \bar{X})^2 \quad .$$

A 100(1-p) percent confidence interval for a single independent future observation X from the same distribution ("prediction interval") can be constructed by noting that $X - \bar{X}$ has mean zero and variance $(1 + \frac{1}{N})$, hence

$$X \quad (\bar{X} - t_{N-1}(1 - \tfrac{p}{2})\sqrt{(1 + \tfrac{1}{N})}S,\ \bar{X} + t_{N-1}(1 - \tfrac{p}{2})\sqrt{(1 + \tfrac{1}{N})}S)$$

with probability exactly 1-p, where t_{N-1} is the inverse cumulative t distribution with N-1 degrees of freedom. All of the quality control limits formulas for accuracy used in this report are extensions of this concept.

Known Mean

If μ is known, the interval can be replaced by

$$X \quad (\mu - t_{N-1}(1 - \tfrac{p}{2})S,\ \mu + t_{N-1}(1 - \tfrac{p}{2})S)$$

with probability 1-p.

Lognormal Data

If instead of X and the X_i being distributed normally, they have a lognormal distribution, with logarithmic mean μ and logarithmic variance σ^2, the limit can be derived by letting $Y_i = \log (X_i)$ and computing $\bar{Y}$ and S_Y^2, the analogous estimates of μ and σ^2. Because of the monotonicity of the log transform, the prediction interval for the future value of $Y = \log (X)$ can be exponentiated to obtain

$$X \in (\exp[\bar{Y} - t_{N-1}(1 - \tfrac{p}{2})\sqrt{(1 + \tfrac{1}{N})}S_Y],\ \exp[\bar{Y} + t_{N-1}(1 - \tfrac{p}{2})\sqrt{(1 + \tfrac{1}{N})}S_Y])$$

with probability 1-p.

Average of Lognormal Values

Because the start-up test is to be based on the arithmetic average of four observations, we consider the case where we are interested in a prediction interval for the average of n future values $\bar{X}_n$ when the data are drawn from a lognormal distribution, with parameters μ and σ^2. Even though $\bar{X}_n$ will have neither a normal nor a lognormal distribution, for small values of n the distribution will be very similar to a lognormal distribution. (See for instance the EPA Development Document for Electroplating, Appendix E.) Therefore, we let

$$Y = \log (\bar{X}_n)$$

and derive a prediction interval for Y which can then be exponentiated to produce a prediction interval for $\bar{X}_n$.

By standard properties of the lognormal distribution and averaging, $\bar{X}_n$ has mean

$$m = \exp(\mu + \tfrac{1}{2}\sigma^2)$$

and variance

$$m^2\eta^2/n \quad ,$$

where

$$\eta^2 = \exp(\sigma^2) - 1 \quad .$$

By the delta method (see for instance Rao, p 388) applied to $f(x) = \log(x)$, $Y = \log(\bar{X}_n)$ will have mean

$$f(m) + f'(m)\, E(\bar{X}_n - m) + \frac{1}{2} f''(m)\, E(\bar{X}_n - m)^2 + \ldots$$

$$\simeq \mu + \frac{1}{2}\sigma^2 - \frac{1}{2}\frac{\eta^2}{n} \quad .$$

Similarly expanding $f^2(X)$ around $X = m$, subtracting the square of the mean, and dropping higher order terms shows that Y will have variance

$$\frac{1}{2}\ 2f'(m)^2\, E(\bar{X}_n - m)^2 + \ldots$$

$$= \frac{1}{m^2} \cdot \frac{m^2 \eta^2}{n} + \ldots$$

$$\simeq \frac{\eta^2}{n} \quad .$$

Since μ and σ^2 are estimated by $\bar{Y}_N$ and S_Y^2 as before, we have that

$$Y - (\bar{Y}_N + \frac{1}{2}S_Y^2 - \frac{1}{2}\eta_Y^2/n)$$

(where $\eta_Y^2 = \exp(S_Y^2) - 1$) has asymptotic mean zero and variance approximately

$$\frac{\eta^2}{n} + \frac{\sigma^2}{N} + \frac{1}{4}(1 - \frac{1}{n})^2 \frac{2\sigma^4}{(N-1)} \quad ,$$

using the fact that $\eta^2 = \exp(\sigma^2) - 1 \quad \sigma^2$ for small σ^2 to combine the S_Y^2 and η_Y^2 terms and noting that S_Y^2 and $\bar{Y}_N$ are independent.

Therefore, an approximate 100(1-p) percent confidence interval for $\bar{X}_n$ can be computed as

$$(\exp[(\bar{Y}_N + \frac{1}{2} S_Y^2 - \frac{1}{2}\eta_Y^2/n) - t_{N-1}(1 - \frac{p}{2}) \cdot \tilde{S}] \quad ,$$

$$\exp[(\bar{Y}_N + \frac{1}{2} S_Y^2 - \frac{1}{2}\eta_Y^2/n) + t_{N-1}(1 - \frac{p}{2}) \cdot \tilde{S}]) \quad ,$$

where

$$\tilde{S}^2 = \eta_Y^2/n + S_Y^2 \frac{1}{N} + \frac{1}{2}(1-\frac{1}{n})^2 \frac{S_Y^4}{N-I} \quad .$$

Variance Components

If the data are drawn from a hierarchical variance structure

$$X_{ij} = \mu + \alpha_i + \varepsilon_{ij} \qquad \begin{matrix} i = 1, \ldots, I \\ j = 1, \ldots, J \end{matrix}$$

where $\alpha_i \sim N(0, \sigma_\alpha^2)$ and $\varepsilon_{ij} \sim N(0, \sigma_\varepsilon^2)$ and are independent, the estimates M, S_α^2, and S_ε^2 of the parameters μ_α^2, and α_ε^2, respectively are obtainable through a variance components analysis (i.e. the maximum likelihood variance components analysis computed by BMDP program 3V). The asymptotic variance of M will be

$$\sigma_\alpha^2/I + \sigma_\varepsilon^2/IJ \quad ,$$

$I(J-1)S_\varepsilon^2/\sigma_\varepsilon^2$ will have a chi-squared distribution with I(J-1) degrees of freedom, and $(I-1)S_\alpha^2/\sigma_\alpha^2$ can be approximated by a chi-squared distribution with I-1 degrees of freedom.

Since the difference X-M has mean 0 and asymptotic variance $\sigma_\alpha^2 + \sigma_\varepsilon^2 + \sigma_\alpha^2/I + \sigma_\varepsilon^2/IJ$, an approximate 100(1-p) percent prediction interval is given by

$$(M - t_d(1 - \tfrac{p}{2})\, S,\ M + t_d(1 - \tfrac{p}{2})\, S)$$

where $S = \sqrt{S_\alpha^2 + S_\varepsilon^2 + S_\alpha^2/I + S_\varepsilon^2/IJ}$.

Because S_α^2 and S_ε^2 have different degrees of freedom, the choice of d = min(I(J-1), I-1) gives the conservatively widest t-interval, and ensures coverage probability of at least 1-p.

Applications

Combinations of these techniques yield the prediction intervals for each test series, as described below.

The limits for the arithmetic average ($\bar{X}_n$) of the four startup amounts are obtained by combining the "average of lognormal" and variance components ideas above to give

$$\exp[(m + \tfrac{1}{2}S_A^2 - \tfrac{1}{2}\eta_A^2 1n) \pm t(d, 1-p/2)\sqrt{(S_E^2 + \eta_A^2/n + S_E^2/L + S_A^2/N + (1-1/n)^2 S_A^4/(2(N-L))}]$$

where M, S_A, and S_E are as above, $\eta_A^2 = \exp(S_A^2) - 1$, n is the number of replicates in the start-up test (i.e., 4), N is the number of measurements in the study, L is the number of laboratories in the study, and t(d, 1-p/2) is the appropriate two-sided t value for test level p, based on d degrees of freedom. In order to produce conservative intervals, the degrees of freedom used was the minimum of those appropriate to either of the variances appearing in the formula, i.e., min(N-L, L-1). (For the WTR series 1 and 3 calculations, L-1 was used.)

The ongoing calibration verification limits are obtained from the analysis of the CAL type samples as

$$\exp[1n(100) \pm t(d, 1 - \tfrac{p}{2})S_A] \quad ,$$

where d = N-L, the degrees of freedom in the estimation of S_A, using the lognormal and known-mean concepts.

The ongoing QA/QC limits are obtained from the analysis of the WTR type samples

$$\exp[M \pm t(d, 1-p/2)\sqrt{S_E^2 + S_A^2 + S_E^2/L + S_A^2/N)}] \quad ,$$

where d = min(N-L, L-1) using the lognormal and variance components concepts.

Appendix L

DERIVATION OF QUALITY CONTROL LIMITS FOR PRECISION

Since the start-up precision test for this method is to be based on the standard deviation of the amounts measured in the four start-up samples, we need to determine the distribution of

$$S = \frac{1}{n-1} \sum_{i=1}^{n} (X_i - \bar{X}_n)^2 \quad ,$$

where the X_i are distributed a lognormally, with logarithmic mean μ and logarithmic variance σ^2. As S does not appear to have any common distributional form, a simulation was performed to estimate the percentiles, as described below. The distribution of S depends intrinsically upon the logarithmic variance σ^2, but μ can be removed from consideration by noting that $S' = S/\exp(\mu)$ can be considered to come from a lognormal distribution with parameters 0 and σ^2, by scale translation of the lognormal. Finally, for a range of σ^2 values, the upper quantiles of S' were determined by simulation. The results are shown in Table L-1 as $Q(1-p, \sigma)$ for p = .05 and .01. The simulations were performed with SAS, using 10,000 replicates, and the quantiles were estimated with PROC UNIVARIATE, definition 4.*

An approximate 100(1-p)th percentile of S, then would be estimated by $\exp(\bar{Y}_N)\ Q(1-p, S_Y)$, where $\bar{Y}_N$ and S_Y^2 are the estimates of μ and σ^2 based on the logarithms of the data, as discussed in Appendix K. In order to correct

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* Let $X_{(1)} \leq \ldots \leq X_{(n)}$ be the ordered observation. For the t^{th} percentile, where q = t/100, let (n+1)q = j + g, where j is the integer part and g the fractional part of (n+1)q. Then the t^{th} percentile by definition 4 is the weighted average of adjacent order statistics aimed at $X_{(q[n+1])}$, i.e. $(1-g)X_{(j)} + gX_{(j+1)}$, where $X_{(n+1)}$ taken to be $X_{(n)}$. See the SAS User's Guide: Basics, p. 579.

for the effect of the use of the estimated S_Y^2 in the place of σ^2 in Q, a correction term of

$$K(1\text{-}p,\ d) = \sqrt{\frac{F_{n-1,\ d}(1\text{-}p)}{C_{n-1}(1\text{-}p)/(n\text{-}1)}}$$

was suggested, where F and C are the inverse cumulative distributions of the F and chi-squared distributions, respectively. This correction represents the ratio between the percentiles of F-limits and chi-squared limits for a standard deviation in the ordinary (nonlogarithmic) situation, and should be approximately correct for use in this situation.*

The precision limit on the standard deviation was then calculated as

$$\exp(M)\ Q(1\text{-}p,\ S_A)\ K(1\text{-}p,\ d) \quad ,$$

where Q is the quantile function at 1-p of S_A tabulated above, linearly interpolated; K is the approximate correction factor for the estimation of S_A; and d is the degrees of freedom in the estimate of S_A, e.g., N-L.

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* In a nominal-scale analysis from $N(\mu,\ \sigma^2)$, the test with σ^2 known is to compare s/σ with a chi-square limit: $\sqrt{C_{n-1}(1\text{-}p)/n\text{-}1}$. If α^2 is unknown, the tests is an F-test comparing S/S_x with $\sqrt{F_{n-1,d}(1\text{-}p)}$. The ratio of these two limits is the difference due to estimating σ^2 in the nominal-scale case, and should be approximately appropriate in the situation of interest.

Table L-1

PERCENTILES OF THE STANDARD DEVIATION
OF FOUR OBSERVATIONS FROM LN(0, σ^2)

Logarithmic Std(σ)	95th Percentile	99th Percentile
.02	0.0319	0.0385
.04	0.0639	0.0774
.06	0.0963	0.1166
.08	0.1290	0.1559
.10	0.1623	0.1694
.15	0.2476	0.3032
.20	0.3397	0.4186
.25	0.4362	0.5474
.30	0.5410	0.7010
.35	0.6595	0.8728
.40	0.7888	1.0673
.45	0.9336	1.2835
.50	1.0906	1.4559
.60	1.4559	2.1796
.70	1.9180	3.0131
.80	2.5033	4.1054
.90	3.2186	5.5222
1.00	4.1196	7.4720
1.10	5.2213	10.0262
1.20	6.6588	13.3723
.		

Appendix M

Method 1625 Revision B

Semivolatile Organic Compounds by Isotope Dilution GCMS

1 Scope and application

1.1 This method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GCMS).

1.2 The chemical compounds listed in tables 1 and 2 may be determined in municipal and industrial discharges by this method. The method is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136.1. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits listed in tables 3 and 4 represent the minimum quantity that can be detected with no interferences present.

1.4 The GCMS portions of this method are for use only by analysts experienced with GCMS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GCMS should run the performance tests in reference 1 before beginning.

2 Summary of method

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a one liter wastewater sample. The sample is extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques. The extract is dried over sodium sulfate and concentrated to a volume of one mL. An internal standard is added to the extract, and the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by a mass spectrometer (MS). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GCMS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal or external standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the extraction and GCMS systems.

3 Contamination and interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials shall be demonstrated to be free from interfernces under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given 8 hr shift, to a maximum of 20). Specific selection of

reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450 °C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being samples.

4 Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 2-4.

4.2 The following compounds covered by this method have been tentatively classified as know or suspected human or mammalian carcinogens: benzo(a)anthracene, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and B-naphthylamine. Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5 Apparatus and materials

5.1 Sampling equipment for discrete or composite sampling.

5.1.1 Sample bottle, amber glass, 1.1 liters minimum. If amber bottles are not available, samples shall be protected from light. Bottles are detergent water washed, then solvent rinsed or baked at 450 C for one hour minimum before use.

5.1.2 Bottle caps--threaded to fit sample bottles. Caps are lined with Teflon. Aluminum foil may be substituted if the sample in not corrosive. Liners are detergent water washed, then reagent water (section 6.5) and solvent rinsed, and baked at approx 200 *x*C for one hour minimum before use.

5.1.3 Compositing equipment--automatic or manual compositing system incorporating glass containers for collection of a mimimum 1.1 liters. Sample containers are kept at 0 to 4 *x*C during sampling. Glass or Teflon tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing is thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (section 6.5) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Continuous liquid-liquid extractor--Teflon or glass connecting joints and stopcocks without lubrication (Hershberg-Wolf Extractor) one liter capacity, Ace Glass 6841-10, or equivalent.

5.3 Drying column--15 to 20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.4 Kuderna-Danish (K-D) apparatus

5.4.1 Concentrator tube--10mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.4.2 Evaporation flask--500 mL (Kontes K-570001-0500, or

equivalent), attached to concentrator tube with springs (Kontes K-662750-0012).

5.4.3 Snyder column--three ball macro (Kontes K-503000-0232, or equivalent).

5.4.4 Snyder column--two ball micro (Kontes K-469002-0219, or equivalent).

5.4.5 Boiling chips--approx 10/40 mesh, extracted with methylene chloride and baked at 450 xC for one hr minimum.

5.5 Water bath--heated, with concentric ring cover, capable of temperature control (q 2 xC), installed in a fume hood.

5.6 Sample vials--amber glass, 2-5 mL with Teflon-lined screw cap.

5.7 Analytical balance--capable of weighing 0.1 mg.

5.8 Gas chromatograph--shall have splitless or on-column injection port for capillary column, temperature program with 30 xC hold, and shall meet all of the performance specifications in section 12.

5.8.1 Column--30 q 5 m x 0.25 q 0.02 mm i.d. 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J & W DB-5, or equivalent).

5.9 Mass spectrometer--70 eV electron impact ionization, shall repetitively scan from 35 to 450 amu in 0.95 to 1.00 second, and shall produce a unit resolution (valleys between m/z 441-442 less than 10 percent of the height of the 441 peak), background corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in table 5 (reference 5). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams. All portions of

the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.10 Data system--shall collect and record MS data, store mass-intensity data in spectral libraries, process GCMS data, generate reports, and shall compute and record response factors.

5.10.1 Data acquisition--mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.10.2 Mass spectral libraries--user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GCMS runs for the compounds of interest (section 7.2).

5.10.3 Data processing--the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GCMS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.10.4 Response factors and multipoint calibrations--the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multi-point calibration curves (section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial (section 8.2) and on-going (section 12.7) performance shall be computed and maintained.

6 Reagents and standards

6.1 Sodium hydroxide--reagent grade, 6N in reagent water.

6.2 Sulfuric acid--reagent grade, 6N in reagent water.

6.3 Sodium sulfate--reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g) and conditioned at 450 *x*C for one hour minimum.

6.4 Methylene chloride--distilled in glass (Burdick and Jackson, or equivalent).

6.5 Reagent water--water in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard solutions--purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at -20 to -10 *x*C in screw-capped vials with Teflon-lined lids. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of stock solutions--prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in section 4. The large number of labeled and unlabeled acid, base/neutral, and Appendix C compounds used for combined calibration (section 7) and calibration verification (12.5) require high concentrations (approx 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10-200

fg/mL. Compounds with a reduced MS response may be prepared at higher concentrations.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Qaulity control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check samples indicates a change in concentration.

6.8 Labeled compound spiking solution--from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 fg/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary standard--using stock solutions (section 6.7), prepare a secondary standard containing all of the compounds in tables 1 and 2 at a concentration of 400 fg/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal standard solution--prepare 2,2'-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene.

6.11 DFTPP solution--prepare at 50 fg/mL in acetone.

6.12 Solutions for obtaining authentic mass spectra (section 7.2)--prepare mixtures of compounds at concentrations which will

assure authentic spectra are obtained for storage in libraries.

6.13 Calibration solutions--combine 0.5 mL of the solution in section 6.8 with 25, 50, 125, 250, and 500 fL of the solution in section 6.9 and bring to 1.00 mL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100 and 200 fg/mL of the pollutants and a constant nominal 100 fg/mL of the labeled compounds. Spike each solution with 10 fL of the internal standard solution (section 6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (section 7.4).

6.14 Precision and recovery standard--used for determination of initial (section 8.2) and on-going (section 12.7) precision and recovery. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 fg/mL.

6.15 Stability of solutions--all standard solutions (sections 6.8 - 6.14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within q 15 percent of the area obtained in the initial analysis of the standard.

7 Calibration

7.1 Assemble the GCMS and establish the operating conditions in table 3. Analyze standards per the procedure in section 11 to demonstrate that the analytical system meets the detection limits in tables 3 and 4, and the mass-intensity criteria in table 5 for 50 ng DFTPP.

7.2 Mass spectral libraries--detection and identification of compounds of interest are dependent upon spectra stored in user

created libraries.

7.2.1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.2.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (section 7.1 and table 5) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak. This edited spectrum is stored for reverse search and for compound confirmation.

7.3 Analytical range--demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (section 15.4). The 20 fg/mL calibration

standard (section 6.13) can be used to demonstrate this performance.

7.3.1 Polar compound detection--demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 fg/mL level (per all criteria in section 13). The 50 fg/mL calibration standard (section 6.13) can be used to demonstrate this performance.

7.4 Calibration with isotope dilution--isotope dilution is used when 1) labeled compounds are available, 2) interferences do not preclude its use, and 3) the quantitation mass extracted ion current profile (EICP) area for the compound is in the calibration range. If any of these conditions preclude isotope dilution, internal or external standard methods (section 7.5 or 7.6) are used.

7.4.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d5 as the isotopic diluent. Also shown are the ± 10 percent error limits (dotted lines). Relative Response (RR) is determined according to the procedures described below. A minimum of five data points are employed for calibration.

7.4.2 The relative response of a pollutant to its labeled analog is determined from isotope ratio values computed from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured for the pure pollutant.

R_y = the isotope ratio measured for the labeled compound.

R_m = the isotope ratio of an analytical mixture of pollutant and

labeled compounds.

The m/z's are selected such that $R_x > R_y$. If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by internal or external standard methods.

7.4.3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (figure 2). For this case,

R_x = [area m_1/z]/1, at the retention time of the pollutant (RT_2).

R_y = 1/[area m_2/z], at the retention time of the labeled compound RT_1)

R_m = [area at m_1/z (at RT_2)]/[area at m_2/z (at RT_1)], as measured in the mixture of the pollutant and labeled compounds (figure 2), and RR = R_m.

7.4.4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in section 7.4.3 apply. This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10 percent of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is computed using the following equation:

$RR = (R_y - R_m)(R_x + 1)/(R_m - R_x)(R_y + 1)$, where R_x is measured as shown in figure 3A, R_y is measured as shown in figure

3B, and R_m is measured as shown in figure 3C. For the example, R_x = 46100/47800 = 9.644, R_y = 2650/43600 = 0.0608, R_m = 49200/48300 = 1.019, and RR = 1.114.

7.4.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 fL aliquot of each of the calibration standards (section 6.13) using the procedure in section 11. Compute the RR at each concentration.

7.4.6 Linearity--if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard--used when criteria for isotope dilution (section 7.4) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2'-difluorobiphenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (sections 8.4 and 12.7.4).

7.5.1 Response factors--calibration requires the determination of response factors (RF) which are defined by the following equation:

$RF = (A_s \times C_{is})/(A_{is} \times C_s)$, where

A_s is the area of the characteristic mass for the compound in the daily standard

A_{is} is the area of the characteristic mass for the internal standard

C_{is} is the concentration of the internal standard (fg/mL)

C_s is the concentration of the compound in the daily standard

(fg/mL)

7.5.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (section 6.13); nominally, 10, 20, 50, 100, and 200 fg/mL. The amount of internal standard added to each extract is the same (100 fg/mL) so that C_{is} remains constant. The RF is plotted vs concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.1.2 Linearity--if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 External standard calibration--used when interferences preclude use of the isotope dilution and internal standard. methods. A master calibration curve is prepared by analyzing a minimum of five concentrations of standards (section 6.13). Concentration vs peak area is plotted for each compound.

7.7.1 Linearity--if the ratio of response to concentration for any compound is constant (less than 60 percent coefficient of variation) over the 5 point calibration range, an averaged response to concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.8 Combined calibration--by using calibration solutions (section 6.13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution, internal standard, and external standard methods. These curves are verified each shift

(section 12.5) by analyzing the 100 fg/mL calibration standard (section 6.13). Recalibration is required only if calibration verification (section 12.5) criteria cannot be met.

8 Quality assurance/quality control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method

performance within acceptable limits (section 15).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (section 6.14) that the analysis system is in control. These procedures are described in sections 12.1, 12.5, and 12.7.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in section 8.4.

8.2 Initial precision and accuracy--to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 Extract, concentrate, and analyze two sets of four one-liter aliquots (8 aliquots total) of the precision and recovery standard (section 6.14) according to the procedure in section 10.

8.2.2 Using results of the first set of four analyses, compute the average recovery (X) in *f*g/mL and the standard deviation of the recovery (s) in *f*g/mL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy in table 8. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 8 present a substantial probability that one or more will fail the acceptance

criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and X for only those compounds which failed the test of the first set of four analyses (section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for these compounds. In this event, correct the problem and repeat the entire test (section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Analyze each sample according to the method beginning in section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7.5).

8.3.3 Compare the labeled compound recovery for each compound with the corresponding limits in table 8. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample is complex and is to be diluted and reanalyzed per section 15.4.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in section 8.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express

the accuracy assessment as a percent recovery interval from P - $2s_p$ to P + $2s_p$. For example, if P = 90% and s_p = 10%, the accuracy interval is expressed as 70 - 110%. Update the accuracy assessment for each compound on a regular basis (e.g. after each 5 - 10 new accuracy measurements).

8.5 Blanks--reagent water blanks are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a blank with each sample lot (samples started through the extraction process on the same 8 hr shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (section 6.14) to demonstrate freedom from contamination.

8.5.2 If any of the compounds of interest (tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 fg/L (assuming a response factor of 1 relative to the internal standard for compounds not listed in tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (section 7), calibration verification (section 12.5), and for initial (section 8.2) and on-going (section 12.7) precision and recovery should be identical, so that the most precise results will be obtained. The GCMS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of semi-volatiles by this method.

8.7 Depending on specific program requirements, field replicates

may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

9 Sample collection, preservation, and handling

9.1 Collect samples in glass containers following conventional sampling practices (reference 7). Composite samples are collected in refrigerated glass containers (section 5.1.3) in accordance with the requirements of the sampling program.

9.2 Maintain samples at 0-4 xC from the time of collection until extraction. If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA methods 330.4 and 330.5 may be used to measure residual chlorine (reference 8).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

10 Sample extraction and concentration (See figure 4)

10.1 Labeled compound spiking--measure 1.00 q 0.01 liter of sample into a glass container. For untreated effluents, and samples which are expected to be difficult to extract and/or concentrate, measure an additional 10.0 q 0.1 mL and dilute to a final volume of 1.00 q 0.01 liter with reagent water in a glass container.

10.1.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1.00 q 0.01 liter aliquots of reagent water in glass containers.

10.1.2 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into all samples and one reagent water aliquot.

10.1.3 Spike 1.0 mL of the precision and recovery standard

(section 6.14) into the two remaining reagent water aliquots.

10.1.4 Stir and equilibrate all solutions for 1-2 hr.

10.2 Base/neutral extraction--place 100-150 mL methylene chloride in each continuous extractor and 200-300 in each distilling flask.

10.2.1 Pour the sample(s), blank, and standard aliquots into the extractors. Rinse the glass containers with 50-100 mL methylene chloride and add to the respective extractor.

10.2.2 Adjust the pH of the waters in the extractors to 12-13 with 6N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1-2 drops of methylene chloride per second will fall from the condensor tip into the water. After 1-2 hours of extraction, test the pH and readjust to 12-13 if required. Extract for 18-24 hours.

10.2.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm anhydrous sodium sulfate: Rinse the distilling flask with 30-50 mL of methylene chloride and pour through the drying column. Collect the solution in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction, and concentrate per sections 10.4 to 10.5.

10.3 Acid extraction--adjust the pH of the waters in the extractors to 2 or less using 6N sulfuric acid. Charge clean distilling flasks with 300-400 mL of methylene chloride. Test and adjust the pH of the waters after the first 1-2 hr of extraction. Extract for 18-24 hours.

10.3.1 Repeat section 10.2.3, except label as the acid fraction.

10.4 Concentration--concentrate the extracts in separate 500 mL

K-D flasks equipped with 10 mL concentrator tubes.

10.4.1 Add 1 to 2 clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approx one mL of methylene chloride through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lowers joint into the concentrator tube with 1-2 mL of methylene chloride. A 5-mL syringe is recommended for this operation.

10.4.2 For performance standards (sections 8.2 and 12.7) and for blanks (section 8.5), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for samples.

10.5 Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its

lower joint into the concentrator tube with approx Ø.2 mL of methylene chloride. Adjust the final volume to 1.Ø mL.

1Ø.6 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at -2Ø to -1Ø *x*C until ready for analysis.

11 GCMS analysis

11.1 Establish the operating conditions given in tables 3 or 4 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (section 1Ø.4.2), use the operating conditions in table 3.

11.2 Bring the concentrated extract (section 1Ø.6) or standard (sections 6.13-6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (sections 6.6 and 1Ø.6) and bring to the mark with solvent if required.

11.3 Add the internal standard solution (section 6.1Ø) to the extract (use 1.Ø *f*L of solution per Ø.1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject a volume of the standard solution or extract such that 1ØØ ng of the internal standard will be injected, using on-column or splitless injection. For 1 mL extracts, this volume will be 1.Ø *f*L. Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo (ghi) perylene or pentachlorophenol peak elutes for the base/neutral or acid fraction, respectively. Return the column to the initial temperature for analysis of the next

sample.

12 System and laboratory performance

12.1 At the beginning of each 8 hr shift during which analyses are performed, GCMS system performance and calibration are verified for all pollutants and labeled compounds. For these tests, analysis of the 100 fg/mL calibration standard (section 6.13) shall be used to verify all performance criteria. Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

12.2 DFTPP spectrum validity--inject 1 fL of the DFTPP solution (section 6.11) either separately or within a few seconds of injection of the standard (section 12.1) analyzed at the beginning of each shift. The criteria in table 5 shall be met.

12.3 Retention times--the absolute retention time of 2,2'-difluorobiphenyl shall be within the range of 1078 to 1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in tables 3 and 4.

12.4 GC resolution--the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks.

12.5 Calibration verification--compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These

concentrations are computed based on the calibration data determined in section 7.

12.5.1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in table 8. If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed. If, however, any compound fails, the measurement system is not performing properly for that compound. In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (section 12.1), or recalibrate (section 7).

12.6 Multiple peaks--each compound injected shall give a single, distinct GC peak.

12.7 On-going precision and accuracy.

12.7.1 Analyze the extract of one of the pair of precision and recovery standards (section 10.1.3) prior to analysis of samples from the same lot.

12.7.2 Compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method.

12.7.3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in table 8. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 8 present a substantial probability that one or more will fail when all compounds are analyzed. To determine if the extraction/ concentration system is out of control or if the failure is caused by probability, proceed as follows:

12.7.3.1 Analyze the second aliquot of the pair of precision and recovery standards (section 10.1.3).

12.7.3.2 Compute the concentration of only those pollutants or labeled compounds that failed the previous test (section 12.7.3). If these compounds now pass, the extraction/concentration processes are in control and analysis of blanks and samples may proceed. If, however, any of the same compounds fail again, the extraction/ concentration processes are not being performed properly for these compounds. In this event, correct the problem, re-extract the sample lot (section 10) and repeat the on-going precision and recovery test (section 12.7).

12.7.4 Add results which pass the specifications in section 12.7.2 to initial and previous on-going data. Update QC charts to form a graphic representation of continued laboratory performance (Figure 5). Develop a statement of laboratory accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from $R - 2s_r$ to $R + 2s_r$. For example, if R = 95% and s_r = 5%, the accuracy is 85 - 105%.

13 Qualitative determination

13.1 Qualitative determination is accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (section 12.1) and with data stored in the

spectral libraries (section 7.2.4). Identification is confirmed when spectra and retention times agree per the criteria below.

13.2 Labeled compounds and pollutants having no labeled analog:

13.2.1 The signals for all characteristic masses stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (Ø.5 to 2 times) for all masses stored in the library.

13.2.3 The retention time relative to the nearest eluted internal standard shall be within q 15 scans or q 15 seconds, whichever is greater.

13.3 Pollutants having a labeled analog:

13.3.1 The signals for all characteristic masses stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.3.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.3.3 The retention time difference between the pollutant and its labeled analog shall agree within q 6 scans or q 6 seconds (whichever is greater) of this difference in the shift standard (section 12.1).

13.4 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, an experienced spectrometrist (section 1.4) is to

determine the presence or absence of the compound.

14 Quantitative determination

14.1 Isotope dilution--by adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography. Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the phenol example given in figure 1 (section 7.4.1), RR would be equal to 1.114. For this RR value, the phenol calibration curve given in figure 1 indicates a concentration of 10.8 fg/mL in the sample extract (C_{ex}).

14.2 Internal standard--compute the concentration in the extract using the response factor determined from calibration data (section 7.5) and the following equation:

C_{ex} (fg/mL) = (A_s x C_{is})/(A_{is} x RF)

where C_{ex} is the concentration of the compound in the extract, and the other terms are as defined in section 7.5.1.

14.3 External standard--compute the concentration in the extract (C_{ex}) from the calibration curve or calibration factor determined from data in section 7.7.

14.5 The concentration of the pollutant in water is computed using the volumes of the original water sample (section 10.1) and the final extract volume (section 10.5), as follows:

Concentration in water (fg/L) = (C_{ex} x V_{ex})/V_s

where V_{ex} is the extract volume in mL, and V_s is the sample

volume in liters.

14.4 If the EICP area at the quantitation mass for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (section 10.1) is analyzed by isotope dilution; otherwise, the extract is diluted by a factor of 10, 9 fL of internal standard solution (section 6.10) are added to a 1.0 mL aliquot, and this diluted extract is analyzed by the internal standard method (section 14.2). Quantify each compound at the highest concentration level within the calibration range.

14.5 Report results for all pollutants and labeled compounds (tables 1 and 2) found in all standards, blanks, and samples, in fg/L, to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (section 14.4) and the labeled compound recovery is within the normal range for the method (section 15.4).

15 Analysis of complex samples

15.1 Untreated effluents and other samples frequently contain high levels (>1000 fg/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to one mL (section 10.5); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (section 10.1) when the sample will not concentrate to 1.0 mL. If a dilute aliquot was not extracted, and the sample holding time (section 9.3) has not been exceeded, dilute an aliquot of the sample with reagent water and re-extract (section 10.1); otherwise, dilute the extract (section 14.4) and analyze by the internal standard method (section 14.2).

15.3 Recovery of internal standard--the EICP area of the internal standard should be within a factor of two of the area in the shift standard (section 12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred. In this case, use one of the labeled compounds (preferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog.

15.4 Recovery of labeled compounds--in most samples, labeled compound recoveries will be similar to those from reagent water (section 12.7). If the labeled compound recovery is outside the limits given in table 8, the dilute extract (section 10.1) is analyzed as in section 14.4. If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the 100 fg/mL calibration standard (section 12.1) shall be analyzed and calibration verified (section 12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

16 Method performance

16.1 Interlaboratory performance for this method is detailed in references 9 and 10.

References

1. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories" USEPA, EMSL Cincinnati, Ohio 45268, EPA-600/4-80-025 (April 1980).

2. "Working with Carcinogens," DHEW, PHS, CDC, NIOSH, Publication 77-206, (Aug 1977).

3. "OSHA Safety and Health Standards, General Industry" OSHA 2206, 29 CFR 1910 (Jan 1976).

4. "Safety in Academic Chemistry Laboratories," ACS Committee on Chemical Safety (1979).

5. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems," J.W. Eichelberger, L.E. Harris, and W.L. Budde, Anal. Chem., 47, 955 (1975).

6. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL, Cincinnati, OH 45268, EPA-600/4-79-019 (March 1979).

7. "Standard Practice for Sampling Water," ASTM Annual Book of Standards, ASTM, Philadelphia, PA, 76 (1980).

8. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL, Cincinnati, OH 45268, EPA 600/4-70-020 (March 1979).

9. Colby, B.N., Beimer, R.G., Rushneck, D.R., and Telliard, W.A., "Isotope Dilution Gas Chromatography-Mass Spectrometry for the Determination of Priority Pollutants in Industrial Effluents." USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980).

10. "Inter-laboratory Validation of US Environmental Protection Agency Method 1625," USEPA, Effluent Guidelines Division, Washington, DC 20460 (June 15, 1984).

Table 1

Base/Neutral Extractable Compounds

Compound	Storet	CAS Registry	EPA-EGD	NPDES
acenaphthene	34205	83-32-9	001 B	001 B
acenaphthylene	34200	208-96-8	077 B	002 B
anthracene	34220	120-12-7	078 B	003 B
benzidine	39120	92-87-5	005 B	004 B
benzo(a)anthracene	34526	56-55-3	072 B	005 B
benzo(b)fluoranthene	34230	205-99-2	074 B	007 B
benzo(k)fluoranthene	34242	207-08-9	075 B	009 B
benzo(a)pyrene	34247	50-32-8	073 B	006 B
benzo(ghi)perylene	34521	191-24-2	079 B	008 B
biphenyl (Appendix C)	81513	92-52-4	512 B	
bis(2-chloroethyl) ether	34273	111-44-4	018 B	011 B
bis(2-chloroethoxy)methane	34278	111-91-1	043 B	010 B
bis(2-chloroisopropyl) ether	34283	108-60-1	042 B	012 B
bis(2-ethylhexyl) phthalate	39100	117-81-7	066 B	013 B
4-bromophenyl phenyl ether	34636	101-55-3	041 B	014 B
butyl benzyl phthalate	34292	85-68-7	067 B	015 B
n-C10 (Appendix C)	77427	124-18-5	517 B	
n-C12 (Appendix C)	77588	112-40-2	506 B	
n-C14 (Appendix C)	77691	629-59-4	518 B	
n-C16 (Appendix C)	77757	544-76-3	519 B	
n-C18 (Appendix C)	77804	593-45-3	520 B	
n-C20 (Appendix C)	77830	112-95-8	521 B	
n-C22 (Appendix C)	77859	629-97-0	522 B	
n-C24 (Appendix C)	77886	646-31-1	523 B	
n-C26 (Appendix C)	77901	630-01-3	524 B	
n-C28 (Appendix C)	78116	630-02-4	525 B	

n-C30 (Appendix C)	78117	638-68-6	526 B	
carbazole (4c)	77571	86-74-8	528 B	
2-chloronaphthalene	34581	91-58-7	020 B	016
4-chlorophenyl phenyl ether	34641	7005-72-3	040 B	017
chrysene	34320	218-01-9	076 B	018
p-cymene (Appendix C)	77356	99-87-6	513 B	
dibenzo(a,h)anthracene	34556	53-70-3	082 B	019
dibenzofuran (Appendix C)	81302	132-64-9	505 B	
dibenzothiophene (Synfuel)	77639	132-65-0	504 B	
di-n-butyl phthalate	39110	84-74-2	068 B	026
1,2-dichlorobenzene	34536	95-50-1	025 B	020
1,3-dichlorobenzene	34566	541-73-1	026 B	021
1,4-dichlorobenzene	34571	106-46-7	027 B	022
3,3'-dichlorobenzidine	34631	91-94-1	028 B	023
diethyl phthalate	34336	84-66-2	070 B	024
2,4-dimethylphenol	34606	105-67-9	034 A	003
dimethyl phthalate	34341	131-11-3	071 B	025
2,4-dinitrotoluene	34611	121-14-2	035 B	027
2,6-dinitrotoluene	34626	606-20-2	036 B	029
di-n-octyl phthalate	34596	117-84-0	069 B	029
diphenylamine (Appendix C)	77579	122-39-4	507 B	
diphenyl ether (Appendix C)	77587	101-84-8	508 B	
1,2-diphenylhydrazine	34346	122-66-7	037 B	030
fluoranthene	34376	206-44-0	039 B	031
fluorene	34381	86-73-7	080 B	032
hexachlorobenzene	39700	118-74-1	009 B	033
hexachlorobutadiene	34391	87-68-3	052 B	034
hexachloroethane	34396	67-72-1	012 B	036
hexachlorocyclopentadiene	34386	77-47-4	053 B	035

ideno(1,2,3-cd)pyrene	34403	193-39-5	083 B	037 B
isophorone	34408	78-59-1	054 B	038 B
naphthalene	34696	91-20-3	055 B	039 B
a-naphthylamine (Appendix C)	82553	91-59-8	502 B	
nitrobenzene	34447	98-95-3	056 B	040 B
N-nitrosodimethylamine	34438	62-75-9	061 B	041 B
N-nitrosodi-n-proplyamine	34428	621-64-7	063 B	042 B
N-nitrosodiphenylamine	34433	86-30-3	062 B	043 B
phenanthrene	34461	85-01-8	081 B	044 B
phenol	34694	108-95-2	065 A	010 A
'-picoline (Synfuel)	77088	109-06-8	503 B	
pyrene	34469	129-00-0	084 B	045 B
styrene (Appendix C)	77128	100-42-5	510 B	
'-terpineol (Appendix C)	77493	98-55-5	509 B	
1,2,3-trichlorobenzene (4c)	77613	87-61-6	529 B	
1,2,4-trichlorobenzene	34551	120-82-1	008 B	046 B

Table 2

Acid Extractable Compounds

Compound	Storet	CAS Registry	EPA-EGD	NPI
4-chloro-3-methylphenol	34452	59-50-7	Ø22 A	ØØ8
2-chlorophenol	34586	95-57-8	Ø24 A	ØØ1
2,4-dichlorophenol	346Ø1	12Ø-83-2	Ø31 A	ØØ2
2,4-dinitrophenol	34616	51-28-5	Ø59 A	ØØ5
2-methyl-4,6-dinitrophenol	34657	534-52-1	Ø6Ø A	ØØ4
2-nitrophenol	34591	88-75-5	Ø57 A	ØØ6
4-nitrophenol	34646	1ØØ-Ø2-7	Ø58 A	ØØ7
pentachlorophenol	39Ø32	87-86-5	Ø64 A	ØØ9
2,3,6-trichlorophenol (4c)	77688	93-37-55	53Ø A	
2,4,5-trichlorophenol (4c)		95-95-4	531 A	
2,4,6-trichlorophenol	34621	88-Ø6-2	Ø21 A	Ø11

Table 3

Gas Chromatography of Base/neutral Extractable Compounds

EGD No.	Compound	Retention time Mean (sec)	EGD Ref	Relative	Detection limit (fg/L) (note 1)
164	2,2'-difluorobiphenyl (int std)	1163	164	1.000 - 1.000	10
061	N-nitrosodimethylamine	385	164	ns	50
603	alpha picoline-d7	417	164	0.326 - 0.393	50
703	alpha picoline	426	603	1.006 - 1.028	50
610	styrene-d5	546	164	0.450 - 0.488	10
710	styrene	549	610	1.002 - 1.009	10
613	p-cymene-d14	742	164	0.624 - 0.652	10
713	p-cymene	755	613	1.008 - 1.023	10
265	phenol-d5	696	164	0.584 - 0.613	10
365	phenol	700	265	0.995 - 1.010	10
218	bis(2-chloroethyl) ether-d8	696	164	0.584 - 0.607	10
318	bis(2-chloroethyl) ether	704	218	1.007 - 1.016	10
617	n-decane-d22	698	164	0.585 - 0.615	10
717	n-decane	720	617	1.022 - 1.038	10
226	1,3-dichlorobenzene-d4	722	164	0.605 - 0.636	10
326	1,3-dichlorobenzene	724	226	0.998 - 1.008	10
227	1,4-dichlorobenzene-d4	737	164	0.601 - 0.666	10
327	1,4-dichlorobenzene	740	227	0.997 - 1.009	10
225	1,2-dichlorobenzene-d4	758	164	0.632 - 0.667	10
325	1,2-dichlorobenzene	760	225	0.995 - 1.008	10
242	bis(2-chloroisopropyl) ether-d12	788	164	0.664 - 0.691	10
342	bis(2-chloroisopropyl) ether	799	242	1.010 - 1.016	10
212	hexachloroethane-13C	819	164	0.690 - 0.717	10
312	hexachloroethane	823	212	0.999 - 1.001	10

Ø63	N-nitrosodi-n-propylamine	83Ø	164	ns	2Ø
256	nitrobenzene-d5	845	164	Ø.7Ø6 - Ø.727	1Ø
356	nitrobenzene	849	256	1.ØØ2 - 1.ØØ7	1Ø
254	isophorone-d8	881	164	Ø.747 - Ø.767	1Ø
354	isophorone	889	254	Ø.999 - 1.Ø17	1Ø
234	2,4-dimethylphenol	921	164	Ø.781 - Ø.8Ø3	1Ø
334	2,4-dimethylphenol	924	234	Ø.999 - 1.ØØ3	1Ø
Ø43	bis(2-chloroethoxy) methane	939	164	ns	1Ø
2Ø8	1,2,4-trichlorobenzene-d3	955	164	Ø.813 - Ø.83Ø	1Ø
3Ø8	1,2,4-trichlorobenzene	958	2Ø8	1.ØØØ - 1.ØØ5	1Ø
255	naphthalene-d8	963	164	Ø.819 - Ø.836	1Ø
355	naphthalene	967	255	1.ØØ1 - 1.ØØ6	1Ø
6Ø9	alpha-terpineol-d3	973	164	Ø.829 - Ø.844	1Ø
7Ø9	alpha-terpineol	975	6Ø9	Ø.998 - 1.ØØ8	1Ø
6Ø6	n-dodecane-d26	953	164	Ø.73Ø - Ø.9Ø8	1Ø
7Ø6	n-dodecane	981	6Ø6	Ø.986 - 1.Ø51	1Ø
529	1,2,3-trichlorobenzene	1ØØ3	164	ns	1Ø
252	hexachlorobutadiene-13C4	1ØØ5	164	Ø.856 - Ø.871	1Ø
352	hexachlorobutadiene	1ØØ6	252	Ø.999 - 1.ØØ2	1Ø
253	hexachlorocyclopentadiene-13C4	1147	164	Ø.976 - Ø.986	1Ø
353	hexachlorocyclopentadiene	1142	253	Ø.999 - 1.ØØ1	1Ø
22Ø	2-chloronaphthalene-d7	1185	164	1.Ø14 - 1.Ø24	1Ø
32Ø	2-chloronaphthalene	12ØØ	22Ø	Ø.997 - 1.ØØ7	1Ø
518	n-tetradecane	12Ø3	164	ns	1Ø
612	biphenyl-d1Ø	1205	164	1.Ø16 - 1.Ø27	1Ø
712	biphenyl	1195	612	1.ØØ1 - 1.ØØ6	1Ø
6Ø8	diphenyl ether-d1Ø	1211	164	1.Ø36 - 1.Ø47	1Ø
7Ø8	diphenyl ether	1216	6Ø8	Ø.997 - 1.ØØ9	1Ø
277	acenaphthylene-d8	1265	164	1.Ø8Ø - 1.Ø95	1Ø

377	acenaphthylene	1247	277	1.000 - 1.004	10
271	dimethyl phthalate-d4	1269	164	1.083 - 1.102	10
371	dimethyl phthalate	1273	271	0.998 - 1.005	10
236	2,6-dinitrotoluene-d3	1283	164	1.090 - 1.112	10
336	2,6-dinitrotoluene	1300	236	1.001 - 1.005	10
201	acenaphthene-d10	1298	164	1.107 - 1.125	10
301	acenaphthene	1304	201	0.999 - 1.009	10
605	dibenzofuran-d8	1331	164	1.134 - 1.155	10
705	dibenzofuran	1335	605	0.998 - 1.007	10
602	beta-naphthylamine-d7	1368	164	1.163 - 1.189	50
702	beta-naphthylamine	1371	602	0.996 - 1.007	50
280	fluorene-d10	1395	164	1.185 - 1.214	10
380	fluorene	1401	281	0.999 - 1.008	10
240	4-chlorophenyl phenyl ether-d5	1406	164	1.194 - 1.223	10
340	4-chlorophenyl phenyl ether	1409	240	0.990 - 1.015	10
270	diethyl phthalate-d4	1409	164	1.197 - 1.229	10
370	diethyl phthalate	1414	270	0.996 - 1.006	10
619	n-hexadecane-d34	1447	164	1.010 - 1.478	10
719	n-hexadecane	1469	619	1.013 - 1.020	10
235	2,4-dinitrotoluene-d3	1359	164	1.152 - 1.181	10
335	2,4-dinitrotoluene	1344	235	1.000 - 1.002	10
237	1,2-diphenylhydrazine-d8	1433	164	1.216 - 1.248	20
337	1,2-diphenylhydrazine (note 2)	1439	237	0.999 - 1.009	20
607	diphenylamine-d10	1437	164	1.213 - 1.249	20
707	diphenylamine	1439	607	1.000 - 1.007	20
262	N-nitrosodiphenylamine-d6	1447	164	1.225 - 1.252	20
362	N-nitrosodiphenylamine (note 3)	1464	262	1.000 - 1.002	20
041	4-bromophenyl phenyl ether	1498	164	1.271 - 1.307	10
209	hexachlorobenzene-13C6	1521	164	1.288 - 1.327	10

309	hexachlorobenzene	1522	209	0.999 - 1.001	10
281	phenanthrene-d10	1578	164	1.334 - 1.380	10
520	n-octadecane	1580	164	ns	10
381	phenanthrene	1583	281	1.000 - 1.005	10
278	anthracene-d10	1588	164	1.342 - 1.388	10
378	anthracene	1592	278	0.998 - 1.006	10
604	dibenzothiophene-d8	1559	164	1.314 - 1.361	10
704	dibenzothiophene	1564	604	1.000 - 1.006	10
528	carbazole	1650	164	ns	20
621	n-eicosane-d42	1655	164	1.184 - 1.662	10
721	n-eicosane	1677	621	1.010 - 1.021	10
268	di-n-butyl phthalate-d4	1719	164	1.446 - 1.510	10
368	di-n-butyl phthalate	1723	268	1.000 - 1.003	10
239	fluoranthene-d10	1813	164	1.522 - 1.596	10
339	fluoranthene	1817	239	1.000 - 1.004	10
284	pyrene-d10	1844	164	1.523 - 1.644	10
384	pyrene	1852	284	1.001 - 1.003	10
205	benzidine-d8	1854	164	1.549 - 1.632	50
305	benzidine	1853	205	1.000 - 1.002	50
522	n-docosane	1889	164	ns	10
623	n-tetracosane-d50	1997	164	1.671 - 1.764	10
723	n-tetracosane	2025	612	1.012 - 1.015	10
067	butylbenzyl phthalate	2060	164	ns	10
276	chrysene-d12	2081	164	1.743 - 1.837	10
376	chrysene	2083	276	1.000 - 1.004	10
272	benzo(a)anthracene-d12	2082	164	1.735 - 1.846	10
372	benzo(a)anthracene	2090	272	0.999 - 1.007	10
228	3,3'-dichlorobenzidine-d6	2088	164	1.744 - 1.848	50
328	3,3'-dichlorobenzidine	2086	228	1.000 - 1.001	50

266	bis(2-ethylhexyl) phthalate-d4	2123	164	1.771 - 1.880	10
366	bis(2-ethylhexyl) phthalate	2124	266	1.000 - 1.002	10
525	n-hexacosane	2147	164	ns	10
269	di-n-octyl phthalate-d4	2239	164	1.867 - 1.982	10
369	di-n-octyl phthalate	2240	269	1.000 - 1.002	10
525	n-octacosane	2272	164	ns	10
274	benzo(b)fluoranthene-d12	2281	164	1.902 - 2.025	10
374	benzo(b)fluoranthene	2293	274	1.000 - 1.005	10
275	benzo(k)fluoranthene-d12	2287	164	1.906 - 2.033	10
374	benzo(k)fluoranthene	2293	275	1.000 - 1.005	10
273	benzo(a)pyrene-d12	2351	164	1.954 - 2.088	10
373	benzo(a)pyrene	2350	273	1.000 - 1.004	10
626	n-triacontane-d62	2384	164	1.972 - 2.127	10
726	n-triacontane	2429	626	1.011 - 1.028	10
083	indeno(1,2,3-cd)pyrene	2650	164	ns	20
082	dibenzo(a,h)anthracene	2660	164	ns	20
279	benzo(ghi)perylene-d12	2741	164	2.187 - 2.524	20
379	benzo(ghi)perylene	2750	279	1.001 - 1.006	20

note 1: This is a minimum level at which the entire GCMS system must give recognizable mass spectra (background corrected) and acceptable calibration points.

note 2: detected as azobenzene

note 3: detected as diphenylamine

ns = specification not available at time of release of method

Column: 30 q 2 m x 0.25 q 0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary

Temperature program: 5 min at 30 xC; 30 - 280 xC at 8 xC per min; isothermal at 280 xC until benzo(ghi)perylene elutes

Gas velocity: 30 q 5 cm/sec

Table 4

Gas Chromatography of Acid Extractable Compounds

EGD No.	Compound	Retention time: Mean (sec)	Retention time: EGD Ref	Retention time: Relative	Detection limit (fg/L) (note 1)
164	2,2'-difluorobiphenyl (int std)	1163	164	1.000 - 1.000	10
224	2-chlorophenol-d4	701	164	0.587 - 0.618	10
324	2-chlorophenol	705	224	0.997 - 1.010	10
257	2-nitrophenol-d4	898	164	0.761 - 0.783	20
357	2-nitrophenol	900	257	0.994 - 1.009	20
231	2,4-dichlorophenol-d3	944	164	0.802 - 0.822	10
331	2,4-dichlorophenol	947	231	0.997 - 1.006	10
222	4-chloro-3-methylphenol-d2	1086	164	0.930 - 0.943	10
322	4-chloro-3-methylphenol	1091	222	0.998 - 1.003	10
221	2,4,6-trichlorophenol-d2	1162	164	0.994 - 1.005	10
321	2,4,6-trichlorophenol	1165	221	0.998 - 1.004	10
531	2,4,5-trichlorophenol	1170	164	ns	10
530	2,3,6-trichlorophenol	1195	164	ns	10
259	2,4-dinitrophenol-d3	1323	164	1.127 - 1.149	50
359	2,4-dinitrophenol	1325	259	1.000 - 1.005	50
258	4-nitrophenol-d4	1349	164	1.147 - 1.175	50
358	4-nitrophenol	1354	258	0.997 - 1.006	50
260	2-methyl-4,6-dinitrophenol-d2	1433	164	1.216 - 1.249	20
360	2-methyl-4,6-dinitrophenol	1435	260	1.000 - 1.002	20
264	pentachlorophenol-13C6	1559	164	1.320 - 1.363	50
364	pentachlorophenol	1561	264	0.998 - 1.002	50

note 1: This is a minimum level at which the entire GCMS system must give recognizable mass spectra (background corrected) and acceptable

calibration points

ns = specification not available at time of release of method

Column: 30 q 2 m x 0.25 q 0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary

Temperature program: 5 min at 30 xC; 30 - 250 xC or until pentachlorophenol elutes

Gas velocity: 30 q 5 cm/sec

Table 5

DFTPP Mass-intensity Specifications

Mass	Intensity required
51	30 - 80 percent of mass 198
68	less than 2 percent of mass 69
70	less than 2 percent of mass 69
127	30 - 60 percent of mass 198
197	less than 1 percent of mass 198
199	5 - 9 percent of mass 198
275	10 - 30 percent of mass 198
441	less than mass 443
442	40 - 100 percent of mass 198
443	17 - 23 percent of mass 442

Table 6

Base/neutral Extractable Compound Characteristic Masses

Compound	Labeled analog	Primary m/z
acenaphthene	d10	154/164
acenaphthylene	d8	152/160
anthracene	d10	178/188
benzidine	d8	184/192
benzo(a)anthracene	d12	228/240
benzo(b)fluoranthene	d12	252/264
benzo(k)fluoranthene	d12	252/264
benzo(a)pyrene	d12	252/264
benzo(ghi)perylene	d12	276/288
biphenyl	d10	154/164
bis(2-chloroethyl) ether	d8	93/101
bis(2-chloroethoxy)methane		93
bis(2-chloroisopropyl) ether	d12	121/131
bis(2-ethylhexyl) phthalate	d4	149/153
4-bromophenyl phenyl ether		248
butyl benzyl phthalate		149
n-C10	d22	55/66
n-C12	d26	55/66
n-C14		55
n-C16	d34	55/66
n-C18		55
n-C20	d42	55/66
n-C22		55
n-C24	d50	55/66

n-C26		55
n-C28		55
n-C30	d62	55/66
carbazole	d8	167/175
2-chloronaphthalene	d7	162/169
4-chlorophenyl phenyl ether	d5	204/209
chrysene	d12	228/240
p-cymene	d14	114/130
dibenzo(a,h)anthracene		278
dibenzofuran	d8	168/176
dibenzothiophene	d8	184/192
di-n-butyl phthalate	d4	149/153
1,2-dichlorobenzene	d4	146/152
1,3-dichlorobenzene	d4	146/152
1,4-dichlorobenzene	d4	146/152
3,3'-dichlorobenzidine	d6	252/258
diethyl phthalate	d4	149/153
2,4-dimenthylphenol	d3	122/125
dimethyl phthalate	d4	163/167
2,4-dinitrotoluene	d3	164/168
2,6-dinitrotoluene	d3	165/167
di-n-octyl phthalate	d4	149/153
diphenylamine	d10	169/179
diphenyl ether	d10	170/180
1,2-diphenylhydrazine*	d10	77/82
fluoranthene	d10	202/212
fluorene	d10	166/176
hexachlorobenzene	$^{13}C6$	284/292
hexachlorobutadiene	$^{13}C4$	225/231

hexachloroethane	^{13}C	201/204
hexachlorocyclopentadiene	$^{13}C4$	237/241
ideno(1,2,3-cd)pyrene		276
isophorone	d8	82/88
naphthalene	d8	128/136
B-naphthylamine	d7	143/150
nitrobenzene	d5	128/128
N-nitrosodimethylamine		74
N-nitrosodi-n-proplyamine		70
N-nitrosodiphenylamine**	d6	169/175
phenanthrene	d10	178/188
phenol	d5	94/71
a-picoline	d7	93/100
pyrene	d10	202/212
styrene	d5	104/109
a-terpineol	d3	59/62
1,2,3-trichlorobenzene	d3	180/183
1,2,4-trichlorobenzene	d3	180/183

*detected as azobenzene

**detected as diphenylamine

Table 7

Acid Extractable Compound Characteristic Masses

Compound	Labeled analog	Primary m/z
4-chloro-3-methylphenol	d2	107/109
2-chlorophenol	d4	128/132
2,4-dichlorophenol	d3	162/167
2,4-dinitrophenol	d3	184/187
2-methyl-4,6-dinitrophenol	d2	198/200
2-nitrophenol	d4	139/143
4-nitrophenol	d4	139/143
pentachlorophenol	$^{13}C6$	266/272
2,3,6-trichlorophenol	d2	196/200
2,4,5-trichlorophenol	d2	196/200
2,4,6-trichlorophenol	d2	196/200

Table 8

Acceptance Criteria for Performance Tests

EGD No.	Compound	Acceptance criteria: Initial precision and accuracy Section 8.2.3 (fg/L) s	X	labeled compound recovery Sec 8.3 and 14.2 P (%)	calibration verification Sec 12.5 (fg/mL)	On-go accur Sec 1 R (fg
301	acenaphthene	21	79 - 134		80 - 125	72 -
201	acenaphthene-d10	38	38 - 147	20 - 270	71 - 141	30 -
377	acenaphthylene	38	69 - 186		60 - 166	61 -
277	acenaphthylene-d8	31	39 - 146	23 - 239	66 - 152	33 -
378	anthracene	41	58 - 174		60 - 168	50 -
278	anthracene-d10	49	31 - 194	14 - 419	58 - 171	23 -
305	benzidine	119	16 - 518		34 - 296	11 -
205	benzidine-d8	269	ns - ns	ns - ns	ns - ns	ns -
372	benzo(a)anthracene	20	65 - 168		70 - 142	62 -
272	benzo(a)anthracene-d12	41	25 - 298	12 - 605	28 - 357	22 -
374	benzo(b)fluoranthene	183	32 - 545		61 - 164	20 -
274	benzo(b)fluoranthene-d12	168	11 - 577	ns - ns	14 - ns	ns -
375	benzo(k)fluoranthene	26	59 - 143		13 - ns	53 -
275	benzo(k)fluoranthene-d12	114	15 - 514	ns - ns	13 - ns	ns -
373	benzo(a)pyrene	26	62 - 195		78 - 129	59 -
273	benzo(a)pyrene-d12	24	35 - 181	21 - 290	12 - ns	32 -
379	benzo(ghi)perylene	21	72 - 160		69 - 145	58 -
279	benzo(ghi)perylene-d12	45	29 - 268	14 - 529	13 - ns	25 -
712	biphenyl (Appendix C)	41	75 - 148		58 - 171	62 -

612 biphenyl-d10	43	28 - 165	ns - ns	52 - 192	17 - 267
318 bis(2-chloroethyl) ether	34	55 - 196		61 - 164	50 - 213
218 bis(2-chloroethyl) ether-d8	33	29 - 196	15 - 372	52 - 194	25 - 222
043 bis(2-chloroethoxy)methane*	27	43 - 153		44 - 228	39 - 166
342 bis(2-chloroisopropyl) ether	17	81 - 138		67 - 148	77 - 145
242 bis(2-chloroisopropyl)ether-d12	27	35 - 149	20 - 260	44 - 229	30 - 169
366 bis(2-ethylhexyl) phthalate	31	69 - 220		76 - 131	64 - 232
266 bis(2-ethylhexyl) phthalate-d4	29	32 - 205	18 - 364	43 - 232	28 - 224
041 4-bromophenyl phenyl ether*	44	44 - 140		52 - 193	35 - 172
067 butyl benzyl phthalate*	31	19 - 233		22 - 450	35 - 170
717 n-C10 (Appendix C)	51	24 - 195		42 - 235	19 - 237
617 n-C10-d22	70	ns - 298	ns - ns	44 - 227	ns - 504
706 n-C12 (Appendix C)	74	35 - 369		60 - 166	29 - 424
606 n-C12-d26	53	ns - 331	ns - ns	41 - 242	ns - 408
518 n-C14 (Appendix C)*	109	ns - 985		37 - 268	ns - ns
719 n-C16 (Appendix C)	33	80 - 162		72 - 138	71 - 181
619 n-C16-d34	46	37 - 162	18 - 308	54 - 186	28 - 202
520 n-C18 (Appendix C)*	39	42 - 131		40 - 249	35 - 167
721 n-C20 (Appendix C)	59	53 - 263		54 - 184	46 - 301
621 n-C20-d42	34	34 - 172	19 - 306	62 - 162	29 - 198
522 n-C22 (Appendix C)*	31	45 - 152		40 - 249	39 - 195
723 n-C24 (Appendix C)	11	80 - 139		65 - 154	78 - 142
623 n-C24-d50	28	27 - 211	15 - 376	50 - 199	25 - 229
524 n-C26 (Appendix C)*	35	35 - 193		26 - 392	31 - 212
525 n-C28 (Appendix C)*	35	35 - 193		26 - 392	31 - 212
726 n-C30 (Appendix C)	32	61 - 200		66 - 152	56 - 215
626 n-C30-d62	41	27 - 242	13 - 479	24 - 423	23 - 274
528 carbazole (4c)*	38	36 - 165		44 - 227	31 - 188
320 2-chloronaphthalene	100	46 - 357		58 - 171	35 - 442

220	2-chloronaphthalene-d7	41	30 - 168	15 - 324	72 - 139	24 -
322	4-chloro-3-methylphenol	37	76 - 131		85 - 115	62 -
222	4-chloro-3-methylphenol-d2	111	30 - 174	ns - 613	68 - 147	14 -
324	2-chlorophenol	13	79 - 135		78 - 129	76 -
224	2-chlorophenol-d4	24	36 - 162	23 - 255	55 - 180	33 -
340	4-chlorophenyl phenyl ether	42	75 - 166		71 - 142	63 -
240	4-chlorophenyl phenyl ether-d5	52	40 - 161	19 - 325	57 - 175	29 -
376	chrysene	51	59 - 186		70 - 142	48 -
276	chrysene-d12	69	33 - 219	13 - 512	24 - 411	23 -
713	p-cymene (Appendix C)	18	76 - 140		79 - 127	72 -
613	p-cymene-d14	67	ns - 359	ns - ns	66 - 152	ns -
082	dibenzo(a,h)anthracene*	55	23 - 299		13 - 761	19 -
705	dibenzofuran (Appendix C)	20	85 - 136		73 - 136	79 -
605	dibenzofuran-d8	31	47 - 136	28 - 220	66 - 150	39 -
704	dibenzothiophene (Synfuel)	31	79 - 150		72 - 140	70 -
604	dibenzothiophene-d8	31	48 - 130	29 - 215	69 - 145	40 -
368	di-n-butyl phthalate	15	76 - 165		71 - 142	74 -
268	di-n-butyl phthalate-d4	23	23 - 195	13 - 346	52 - 192	22 -
325	1,2-dichlorobenzene	17	73 - 146		74 - 135	70 -
225	1,2-dichlorobenzene-d4	35	14 - 212	ns - 494	61 - 164	11 -
326	1,3-dichlorobenzene	43	63 - 201		65 - 154	55 -
226	1,3-dichlorobenzene-d4	48	13 - 203	ns - 550	52 - 192	ns -
327	1,4-dichlorobenzene	42	61 - 194		62 - 161	53 -
227	1,4-dichlorobenzene-d4	48	15 - 193	ns - 474	65 - 153	11 -
328	3,3'-dichlorobenzidine	26	68 - 174		77 - 130	64 -
228	3,3'-dichlorobenzidine-d6	80	ns - 562	ns - ns	18 - 558	ns -
331	2,4-dichlorophenol	12	85 - 131		67 - 149	83 -
231	2,4-dichlorophenol-d3	28	38 - 164	24 - 260	64 - 157	34 -
370	diethyl phthalate	44	75 - 196		74 - 135	65 -

270	diethyl phthalate-d4	78	ns - 260	ns - ns	47 - 211	ns - ns
334	2,4-dimethylphenol	13	62 - 153		67 - 150	60 - 156
234	2,4-dimethylphenol-d3	22	15 - 228	ns - 449	58 - 172	14 - 242
371	dimethyl phthalate	36	74 - 188		73 - 137	67 - 207
271	dimethyl phthalate-d4	108	ns - 640	ns - ns	50 - 201	ns - ns
359	2,4-dinitrophenol	18	72 - 134		75 - 133	68 - 141
259	2,4-dinitrophenol-d3	66	22 - 308	ns - ns	39 - 256	17 - 378
335	2,4-dinitrotoluene	18	75 - 158		79 - 127	72 - 164
235	2,4-dinitrotoluene-d3	37	22 - 245	10 - 514	53 - 187	19 - 275
336	2,6-dinitrotoluene	30	80 - 141		55 - 183	70 - 159
236	2,6-dinitrotoluene-d3	59	44 - 184	17 - 442	36 - 278	31 - 250
369	di-n-octyl phthalate	16	77 - 161		71 - 140	74 - 166
269	di-n-octyl phthalate-d4	46	12 - 383	ns - ns	21 - 467	10 - 433
707	diphenylamine (Appendix C)	45	58 - 205		57 - 176	51 - 231
607	diphenylamine-d10	42	27 - 206	11 - 488	59 - 169	21 - 249
708	diphenyl ether (Appendix C)	19	82 - 136		83 - 120	77 - 144
608	diphenyl ether-d10	37	36 - 155	19 - 281	77 - 129	29 - 186
337	1,2-diphenylhydrazine	73	49 - 308		75 - 134	40 - 360
237	1,2-diphenylhydrazine-d10	35	31 - 173	17 - 316	58 - 174	26 - 200
339	fluoranthene	33	71 - 177		67 - 149	64 - 194
239	fluoranthene-d10	35	36 - 161	20 - 278	47 - 215	30 - 187
380	fluorene	29	81 - 132		74 - 135	70 - 151
280	fluorene-d10	43	51 - 131	27 - 238	61 - 164	38 - 171
309	hexachlorobenzene	16	90 - 124		78 - 128	85 - 132
209	hexachlorobenzene-13C6	81	36 - 228	13 - 595	38 - 265	23 - 321
352	hexachlorobutadiene	56	51 - 251		74 - 135	43 - 287
252	hexachlorobutadiene-13C4	63	ns - 316	ns - ns	68 - 148	ns - 413
312	hexachloroethane	227	21 - ns		71 - 141	13 - ns
212	hexachloroethane-13C1	77	ns - 400	ns - ns	47 - 212	ns - 563

353	hexachlorocyclopentadiene	15	69	- 144			77 - 129	67 -
253	hexachlorocyclopentadiene-13C4	60	ns	- ns	ns	- ns	47 - 211	ns -
083	ideno(1,2,3-cd)pyrene*	55	23	- 299			13 - 761	19 -
354	isophorone	25	76	- 156			70 - 142	70 -
254	isophorone-d8	23	49	- 133	33	- 193	52 - 194	44 -
360	2-methyl-4,6-dinitrophenol	19	77	- 133			69 - 145	72 -
260	2-methyl-4,6-dinitrophenol-d2	64	36	- 247	16	- 527	56 - 177	28 -
355	naphthalene	20	80	- 139			73 - 137	75 -
255	naphthalene-d8	39	28	- 157	14	- 305	71 - 141	22 -
702	a-naphthylamine (Appendix C)	49	10	- ns			39 - 256	ns -
602	a-naphthylamine-d7	33	ns	- ns	ns	- ns	44 - 230	ns -
356	nitrobenzene	25	69	- 161			85 - 115	65 -
256	nitrobenzene-d5	28	18	- 265	ns	- ns	46 - 219	15 -
357	2-nitrophenol	15	78	- 140			77 - 129	75 -
257	2-nitrophenol-d4	23	41	- 145	27	- 217	61 - 163	37 -
358	4-nitrophenol	42	62	- 146			55 - 183	51 -
258	4-nitrophenol-d4	188	14	- 398	ns	- ns	35 - 287	ns -
061	N-nitrosodimethylamine*	198	21	- 472			40 - 249	12 -
063	N-nitrosodi-n-proplyamine*	198	21	- 472			40 - 249	12 -
362	N-nitrosodiphenylamine	45	65	- 142			68 - 148	53 -
262	N-nitrosodiphenylamine-d6	37	54	- 126	26	- 256	59 - 170	40 -
364	pentachlorophenol	21	76	- 140			77 - 130	71 -
264	pentachlorophenol-13C6	49	37	- 212	18	- 412	42 - 237	29 -
381	phenanthrene	13	93	- 119			75 - 133	87 -
281	phenanthrene-d10	40	45	- 130	24	- 241	67 - 149	34 -
365	phenol	36	77	- 127			65 - 155	62 -
265	phenol-d5	161	21	- 210	ns	- ns	48 - 208	ns -
703	'-picoline (Synfuel)	38	59	- 149			60 - 165	50 -
603	'-picoline-d7	138	11	- 380	ns	- ns	31 - 324	ns -

384	pyrene	19	76 - 152		76 - 132	72 - 159
284	pyrene-d10	29	32 - 176	18 - 303	48 - 210	28 - 196
710	styrene (Appendix C)	42	53 - 221		65 - 153	48 - 244
610	styrene-d5	49	ns - 281	ns - ns	44 - 228	ns - 348
709	'-terpineol (Appendix C)	44	42 - 234		54 - 186	38 - 258
609	'-terpineol-d3	48	22 - 292	ns - 672	20 - 502	18 - 339
529	1,2,3-trichlorobenzene (4c)*	69	15 - 229		60 - 167	11 - 297
308	1,2,4-trichlorobenzene	19	82 - 136		78 - 128	77 - 144
208	1,2,4-trichlorobenzene-d3	57	15 - 212	ns - 592	61 - 163	10 - 282
530	2,3,6-trichlorophenol (4c)*	30	58 - 137		56 - 180	51 - 153
531	2,4,5-trichlorophenol (4c)*	30	58 - 137		56 - 180	51 - 153
321	2,4,6-trichlorophenol	57	59 - 205		81 - 123	48 - 244
221	2,4,6-trichlorophenol-d2	47	43 - 183	21 - 363	69 - 144	34 - 226

*measured by internal standard; specification derived from related compound.

d = detected; result must be greater than zero.

ns = no specification; limit is outside the range that can be measured reliably.

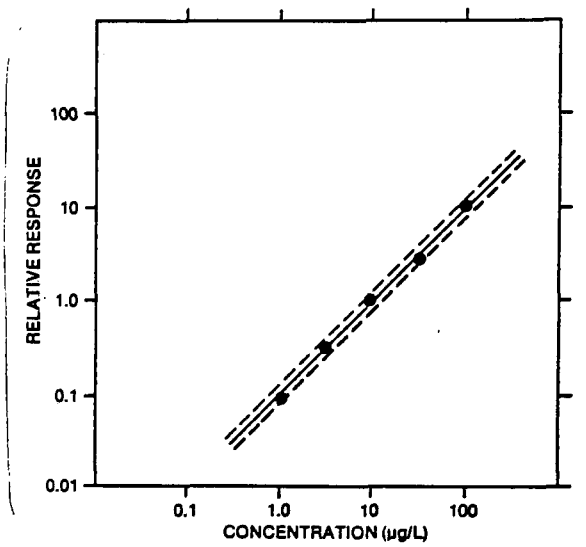

FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ± 10 Percent Error Window.

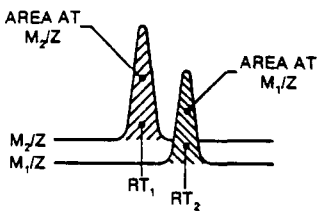

FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled (m_1/z) and Unlabeled (m_2/z) Pairs.

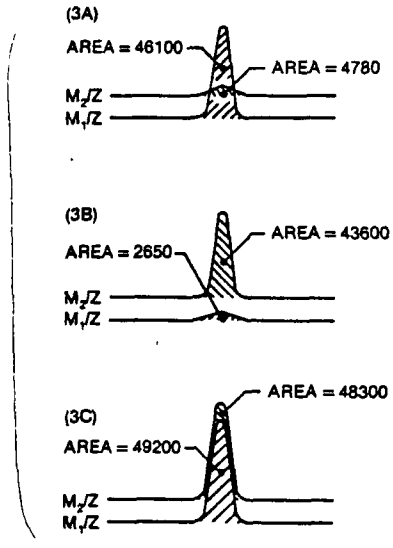

FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.

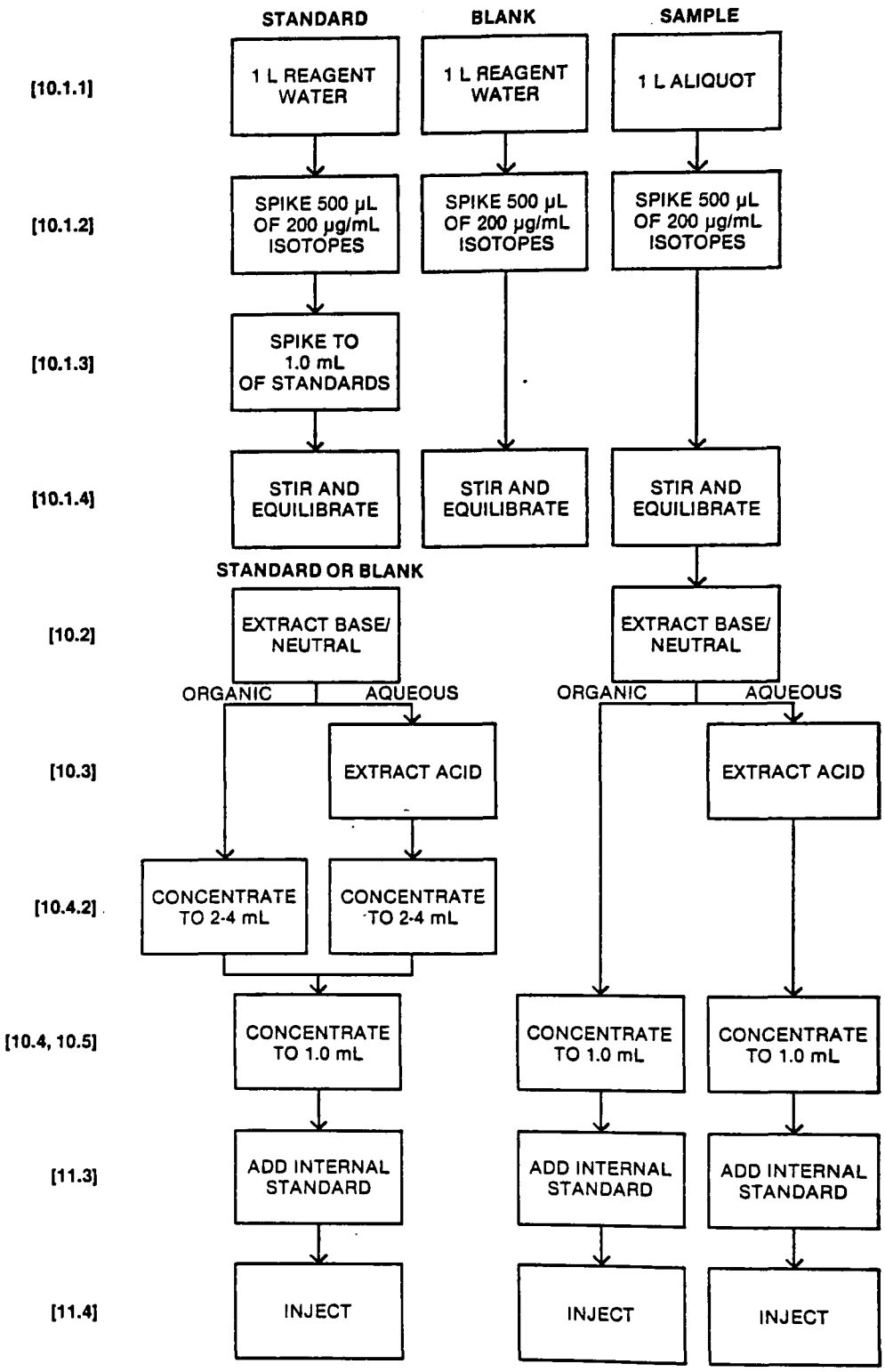

FIGURE 4 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

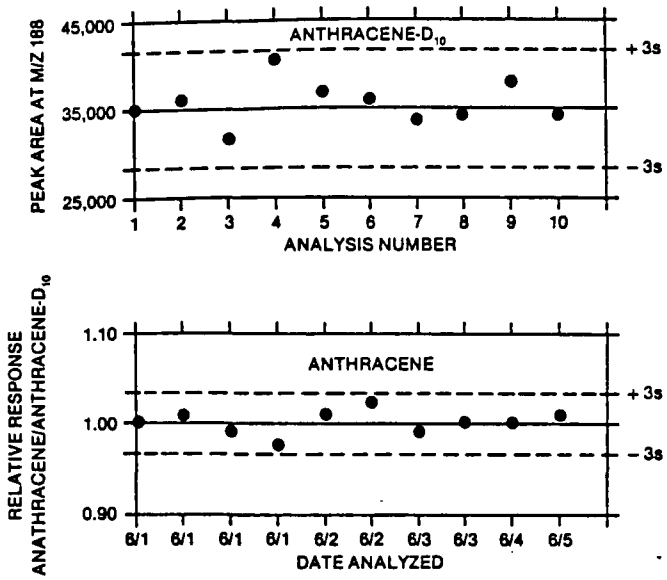

FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-d_{10} (lower graph) Plotted as a Function of Time or Analysis Number.

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