

RESEARCH TRIANGLE INSTITUTE

STATUS REPORT #7

STABILITY OF ORGANIC AUDIT MATERIALS AND RESULTS OF SOURCE TEST ANALYSIS AUDITS

by

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EPA Contract No.: 68-02-3767

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NOTICE

This document has been reviewed in accordance with U.S. Environ-mental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

FOREWORD

Source measurement and monitoring efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing data bases needed in developing regulations and to provide means of monitoring compliance with regulations. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has the responsibility for implementation of agency-wide Quality Assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Agency including the Office of Air and Radiation, the Office of Toxic Substances, and the Office of Enforcement.

The need for reliable standards for auditing and documenting the accuracy of source emission measurement of gaseous hydrocarbons, halocarbons, and sulfur compounds is well established. The Quality Assurance Division of EPA's Environmental Monitoring Systems Laboratory has responded to this need through the development of an extensive repository of gaseous compounds. The primary objectives of this ongoing project are (1) to provide accurate gas mixtures to EPA, state/local agencies, or their contractors for performance audits to assess the accuracy of source emission measurements in certain organic chemical manufacturing industries, (2) to verify the vendor's certified analysis of the gas mixtures, (3) to determine the stability of the gas mixtures with time, and (4) to develop new audit materials as requested by EPA. This report describes the current status of this project. Included in the report are (1) a description of the experimental procedures used for the analyses of gas mixtures, (2) a description of the audit procedure, and (3) currently available audit results and stability data.

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ABSTRACT

A repository of 45 gaseous compounds including hydrocarbons, halocarbon, and sulfur species has been established under contract with the U.S. Environmental Protection Agency (USEPA). The main objectives of this on-going project are (1) to provide gas mixtures to EPA, state/local agencies, or their contractors, as performance audits to assess the accuracy of source emission measurements in certain organic chemical manufacturing industries, (2) to corroborate the vendor's certified analysis of the gas mixtures by in-house analysis, (3) to determine the stability of the gas mixtures with time by in-house analysis, and (4) to explore the feasibility of new audit materials as requested by EPA.

Thus far, 31 compounds have been used to conduct 149 different audits. The results of these audits and a description of the experimental procedures used for analyses and available stability data are presented in the status report. Generally the audit results are within 15 percent of the expected values.

Compound stabilities have been determined through multiple analyses of the cylinders containing them. Stability data for up to 7 years is available for many compounds and over 4 years for most compounds. Compounds that are unstable and not suitable for use as an audit material are identified.

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SECTION 1 INTRODUCTION

OBJECTIVES

The need for reliable standards for auditing source emission measurement of gaseous hydrocarbons, halocarbons and sulfur compounds is well established. The Research Triangle Institute (RTI), under contract to the U.S. Environmental Protection Agency (USEPA), has responded to this need through the development of an extensive repository of 39 gaseous compounds. The primary objectives of this ongoing project are (1) to provide accurate gas mixtures to EPA, state/local agencies, or their contractors for performance audits to assess the relative accuracy of source emission measurements in certain organic chemical manufacturing industries, (2) to examine the vendor's certified analysis of the gas mixtures by in-house analysis, (3) to determine the stability of the gas mixtures with time by in-house analysis, and (4) to develop new audit materials, as requested by EPA.

This report describes the current status of this project. Included in the report are (1) a description of the experimental procedures used for initial cylinder analyses and collection of stability data, (2) a description of the audit procedure, and (3) currently available audit results and stability data. Complete details of the study with statistical analyses for ten (10) halocarbons and eight (8) other organics are presented in two journal publications (1,2). Statistical analysis for the remaining compounds will be presented in a future report.

AUDIT MATERIALS CONTAINED IN THE REPOSITORY

Currently, 45 gaseous compounds have been investigated as audit materials. Six of these gaseous compounds have been found to be unstable in cylinders and not suitable as audit materials. The other 39 gaseous compounds in the repository are suitable for conducting performance audits during source testing. The compounds were selected based on the anticipated needs of the Emission Measurement Branch, Office of Air Quality Planning and Standards, USEPA. Table 1 lists the 45 com-

pounds, the concentration ranges, the number of cylinders of each compound currently in the repository, and the cylinder construction material. In Table 1, the audit materials fall into two concentration ranges. The low concentration range between 5 and 50 parts per million (ppm) simulates possible emission standard levels. The high concentration range between 50 and 700 ppm simulates expected source emission levels. The balance gas for all gas mixtures is nitrogen.

TABLE 1. AUDIT MATERIALS CURRENTLY IN THE REPOSITORY

_	Low Concentration Range			High Concentration Range			
	No. of ylinders	Concentration Range (ppm)	Cylinder Construction*	No. of Cylinders	Concentration Range (ppm)	Cylinder Construction*	
Benzene	7	5 - 20	S	11	60 - 400	S	
Ethylene	4	5 - 20	Al	4	300 - 700	AI	
				6	3000 - 20,000	Al	
Propylene	3	5 - 20	Al	3	300 - 700	Al	
Methane/Ethane	-			4	1000 - 6000(M), 200 - 700(E)	, AI	
Propane	4	5 - 20	AI	4	300 - 700	Al	
				4	1000 - 20,000	Al	
Toluene	5	5 - 20	S	4	100 - 700	LS	
Hydrogen Sulfide	7	5 - 50	A1	7	100 - 700	AI	
Meta-Xylene	1	5 - 20	S	2	300 - 700	LS	
Methyl Acetate	2	5 - 20	S	2	300 - 700	S	
Chloro form	2	5 - 20	S	1	300 - 700	S	
Carbonyl Sulfide	1	5 - 20	S	3	100 - 300	S	
Methyl Mercaptan	3	3 - 10	Al	-		-	
Hexane	2	20 - 80	Al	1	1000 - 3000	LS	
1,2-Dichloroethane	4	5 - 20	AI	4	100 - 600	AI	
Cyclohexane	-			1	80 - 200	S	
Methyl Ethyl Ketone	1	30 - 80	S	-		-	
Methanol	2	30 - 80	Al	-		-	
1,2-Dichtoropropane	2	5 - 20	Al	2	300 - 700	Al	
Trichloroethylene	2	5 - 20	Al	2	100 - 600	Al	
1,1-Dichloro- ethylene	2	5 - 20	AI	2	100 - 600	Al	
*1,2-Dibromo- ethylene	-		**	-	a at ab ab ab ab ab ab ab	***	
Perchloro- ethylene	2	5 - 20	S	2	300 - 700	LS	
Vinyl Chloride	9	5 - 30	S	-			
1,3-Butadiene	1	5 - 30	S	-			
Acrylonitrile	1	5 - 20	Al	1	300 - 500 .	Al	
**Aniline	-			-	4 4 TO 10 TO		
Methyl Isobutyl Ket	rone 1	5 - 20	Al	-		••	

^{*}Cylinder constructions: Al = Aluminum, S = Steel, LS = Low-Pressure Steel

^{**}Cylinders are no longer available; the compounds were found to be unstable in the cylinders.

TABLE 1. AUDIT MATERIALS CURRENTLY IN THE REPOSITORY (Continued)

-	Low	Concentration	Range	Н	igh Concentration	Range
Compound (No. of Cylinders	Concentration Range (ppm)	Cylinder Construction*	No. of Cylinders	Concentration Range (ppm)	Cylinder Construction*
**Para-dichlorbenzene	e -		S	_		
Ethylamine	-	*	ΑI	•		
**Formal dehyde	-		-	-		
Methylene Chloride	3	1 - 20	AI	-		
Carbon Tetrachlorio	de 1	5 - 20	Al	•		
Freon 113	1	5 - 20	AI	•		
Methyl Chloroform	1	5 ~ 20	Al	-		
Ethylene Oxide	5	5 ~ 20	Al	-		
Propylene Oxide	1	. 5 ~ 20	AI	1	75 - 200	ΑI
Allyl Chloride	1	5 - 20	S	1	75 - 200	S
Acrolein	1	5 - 20	Al	-		
Chlorobenzene	3	5 ~ 20	ΑI	, •		
Carbon Disulfide	-	***	AI	1	75 - 200	AI
**Cyclohexanone	-		AI	•		
**EPA Method 25 Gas	6	100 - 200	Al	3	750 - 2000	AI
Ethylene Dibromide	2	5 ~ 20	S	2	100 - 300	S
Tetrachloroethane	1	5 - 20	S	-		

^{*}Cylinder construction: Al = Aluminum, S = Steel, LS = Low Pressure Steel

 $^{^{**}}$ Cylinders are no longer available; the compounds were found to be unstable in the cylinders.

^{***}The gas mixture contains an aliphatic hydrocarbon, an aromatic hydrocarbon, and carbon dioxide in nitrogen. Concentrations shown are reported in ppmC.

SECTION 2 EXPERIMENTAL PROCEDURES

Analysis of the cylinder gases is required to corroborate the concentrations reported by the company which prepared the gas mixtures and also to measure concentration changes with time, that is, estimate stability of the compounds.

INSTRUMENTATION

Analyses are presently performed with (1) a Perkin-Elmer Sigma 4 Gas Chromatograph with a flame ionization detector, and (2) a Tracor 560 Gas Chromatograph with a flame photometric detector. The flame photometric detector has been used principally for measurement of the sulfur-containing species. The gaseous samples are injected onto the columns by means of gas sampling valves constructed of Hastalloy C (high nickel content and low adsorptive properties). These valves are equipped with interchangeable sample loops to allow the injection of variable volumes of gas. A thermal oxidation system consisting of a 3/8 inch 0.D. stainless steel tube heated to $1350^{\circ}\mathrm{F}$ in a tube furnace is used to oxidize calibration mixtures to CO_2 for verification of concentration. The CO_2 concentration is measured with a Byron Model 401 equipped with a nickel based reduction catalyst and flame ionization detector.

The gas chromatographic parameters used in the measurement of individual compounds and any problems with the analysis are listed in Attachment 1.

CALIBRATION

Calibration of the gas chromatographs has involved measurement of known concentrations of gases in air or nitrogen. The source or method of preparation of calibration standards varies depending on the gas involved.

National Bureau of Standards, Standard Reference Materials (NBS-SRMs) of methane and propane were used for the calibration of the GC for the measurement of methane, ethane, propane, ethylene, and propylene audit materials. NBS-SRM of benzene was used for the calibration

of the GC for the measurement of low concentration benzene audit cylinders.

A second method for preparation of calibration standards involves the use of permeation tubes. For example, the calibration gases for vinyl chloride and ethylene oxide have been generated in this manner. The permeation tube is placed in a temperature-controlled chamber and nitrogen is passed over the permeation tube at a known flow rate. The resultant gaseous mixture is further diluted, if necessary, using additional nitrogen in a glass dilution bulb. The final mixture is collected in a gas sampling syringe and analyzed by GC-FID. The permeation rates of the tubes are determined periodically by weight loss.

A third method for developing a calibration standard is the pressure-dilution technique. A known volume of the compound, either gas or liquid, is injected into an evacuated glass bulb or stainless steel sphere of known volume. The volume of the bulb or sphere is determined gravimetrically. The bulb or sphere is then pressurized with a balance gas of choice. If a pure liquid is injected, total vaporization is assumed and the concentration is calculated by using the ideal gas law. Additional dilutions are also made, if necessary, by partially evacuating to a known pressure and pressurizing with a balance gas to a known pressure.

With each of these approaches, multipoint calibration curves are prepared each time a sample is analyzed.

OUALITY CONTROL

Replicate injections of both audit cylinder gases and calibration standards are performed until no trends in the detector response are observed and the relative standard deviation of replicate injections is less than 1 percent.

Instead of depending totally upon the pressure-dilution technique for the determination of calibration mixture concentrations, an internal quality control has been implemented to allow verification of the calibration mixture concentration. This involves passing a portion of the lowest concentration calibration mixture of a particular compound prepared in a bulb or sphere through the thermal oxidizer described in

Section 2.1 and analysis of the CO_2 produced with a Byron 401 analyzer. The CO_2 response is calibrated with a primary standard $(\pm\ 1\%)$ mixture of CO_2 in air. The concentration determined by this technique was then assigned to the lowest concentration calibration mixture and the concentrations of higher calibration mixtures were calculated by assuming quantitative dilution. This procedure was used for those organics listed in Table 2. The lowest standard calibration mixtures were only verified with this technique in order to limit the production of corrosive by-products of halogenated organics by the oxidizer.

TABLE 2. ORGANIC CALIBRATION MIXTURES VERIFIED BY BYRON 401 ANALYZER

Toluene M-xylene Methyl acetate Chloroform 1.1-dichloroethane Methanol 1,2-dichloropropane 1,1-dichloroethylene Perchloroet hylene Methyl isobutyl ketone Methylene chloride Carbon tetrachloride Freon 113 Allyl chloride Acrolein Chlorobenzene EPA Method 25 gas mixture

SECTION 3 PERFORMANCE AUDITS

RTI supplies repository cylinders for audits upon request from the EPA, state or local agencies or contractors. A contractor must be performing source emission tests at the request of EPA or a state or local agency in order to qualify for the performance audit. When a request is received, the contents of the cylinders are analyzed, the tank pressures are measured and the cylinders are shipped by overland carrier. Tank regulators are also provided when requested. A letter is included with the cylinders which provides general instructions for performance of the audit. The audit material concentration and cylinder pressure are provided to the requesting agency audit coordinator.

To date, 149 individual audits have been initiated, and 140 are complete. The audit results currently available are presented in Table 2. Generally, the results of the audits show agreement of \pm 15 percent with the audit material concentrations measured by RTI.

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Aud i †				RTI audit	Client audit	Status of
No.	Client***	Industry	Audit material	conc. (ppm)	% blas (Avg.)*	audi t**
1	Α	Ethylene oxide	Ethylene in N ₂	3,240	-22.5	E
•	.,	production	Ethylene in N ₂	21,200	-20.0	- .
2	A	Ethylene oxide	Methane/ethane in N ₂	1,710Me/220Et	+9/-20	E
		production	Methane/ethane in N ₂	8,130Me/597Et	+9/-1.00	
3	Α	Ethylene oxide	Methane/ethane in N ₂	1,021Me/315Et	+21.5/-4.50	E
		production	Methane/ethane in N2	6,207Me/773Et		
4	Α	Acetone	Benzene in N ₂	79.0	-19.0	E
		producti on	Benzene in N ₂	374.0	-11.0	
5	Α	Maleic anhydride	Benzene in N ₂	138	-9.40	E
		producti on	Benzene in N ₂	300	+4.70	
6	Α	Ethylene oxide	Ethylene in N ₂	5,440	-27.0	E
		producti on	Ethylene in N ₂	18,900	-33.0	
7	В	Maleic anhydride	Benzene in N ₂	80.0	+2.30	. Е
		production	Benzene in N ₂	355	+27.5	
8	С	Maleic anhydride	Benzene in N ₂	101	+12.9	E
		production	Benzene in N ₂	387	+14.5	
9	. D	Ethyl benzene	Benzene in N ₂	71.0	-2.80	E
		styrene manufacturer	Benzene in N ₂	229	-3.90	
10	E	Gasoline bulk	Benzene in N ₂	62.0	+3.80	E
		termi nal	Benzene in N ₂	80.0	+3.40	
11	F.	Gasoline transfer	Benzene in N ₂	142	-3.50	E
		termi na l	Benzene in N ₂	294	+3.20	
12	F	Gasoline transfer	Benzene in N ₂	268	-11.8	E
		termi na l	Benzene in N ₂	343	-1.00	

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued) RTI Audit Client Audit Status of						
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* audit**						
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Audit No.	Clien†***	Industry	Audit material	RTI audit conc. (ppm)	Client audit % bias (Avg.)*	Status of audit**
IVO.	CITOIT	industry	Add I material	conc. (ppm)	& blas (Avg.)	audi i
24	н	industri al	Toluene in N ₂	14.8	-1.90	E
		surface coating	Propylene in N ₂	474	+0.20	
		process	Propane In N ₂	20.3	-2.30	
			Methane/ethane in N ₂	1,640Me/195et	-13.5(as met)	nane)
25	С	Acrylic acid and	Propane in N ₂	10.1	+8.60	E
		ester Production	Propane in N ₂	710	+5.60	
26	С	Acrylic acid and	Propane in N ₂	5.1	+17.6	E
		ester Production	Propane In N ₂	607	-3.60	
27	E	Maleic anhydride	Benzene in N ₂	10.2	NA	F
			Benzene in N ₂	218	NA	
28A	Α	Carbon adsorber	Toluene in N ₂	8.55	-6.40	E
			Toluene in N ₂	405	-1.00	
28B	Α	Carbon adsorber	Toluene in N ₂	8,55	+4.10	E
			Toluene in N ₂	405	NA	
28C	Α	Carbon adsorber	Toluene in N ₂	8.55	-8.80	Ε
•			Toluene in N ₂	405	NA	
29	EPA, QAD	Instrument	Ethylene in N ₂	4.75	+4.00	E
		check-out	Ethylene in N ₂	19.6	+3.10	
			Ethylene in N ₂	312	-0.80	
			Ethylene in N ₂	3020	+5.30	
			Ethylene in N ₂	20400	-8.60	

Audi t	•			RTI Audit	Client Audit	Status of
No.	Clien+**	Industry	Audit material	conc. (ppm)	% blas (Avg.)*	audi t**
30	EPA, QAD	Instrument	Benzene in N ₂	8.20	+0.30	E
		check-out	Benzene in N ₂	78.0	-0.90	
			Benzene in N ₂	133	-4.00	
			Benzene in N ₂	348	-0.90	
31	EPA, QAD	Instrument	Toluene in N ₂	405	+3.20	E
		check-out	Toluene in N ₂	579	+1.00	
32	EPA, QAD	Instrument	Methyl acetate in N ₂	6.80	-2.60	E
		check-out	Methyl acetate in N ₂	17.2	+1.70	
			Methyl acetate in N2	326	-1.50	
			Methyl acetate in N ₂	455	-1.30	
33	EPA, QAD	Instrument	Propylene in N ₂	4.90	-22.4	E
		check-out	Propylene in N_2	19.7	-7. 80	
			Propylene in N ₂	300	+1.00	
			Propylene in N_2	685	-1.80	
34.	EPA, QAD	Instrument	Propane in N ₂	14.6	-0.70	E
		check-out	Propane in N_2	303	+7.60	
			Propane in N ₂	439	+6.20	
35a	1	Vegetable oii	Hexane in N ₂	82.2	+8.10	E
		plant	Hexane in N ₂	1980	+3.00	
35b	1 .	Vegetable oll	Hexane in N ₂	82.2	-1.20	Ε
טעע	• ,	plant	Hexane in N ₂	1980	-1.30	E
		Pram	110,0110 111 112	1700	-1.50	
36	A	Carbon adsorber	Toluene in N ₂	8.20	-2.40	E

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Aud i †				RTI audit	Client audit	Status of
No.	Client***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi t**
37	В	Coke oven	Benzene in N ₂	12.1	+0.80	εĨ
			Benzene in N ₂	105	+2,90	
38	D	Ethyl benzene/	Benzene in N ₂	9.90	+5.70	E
		styrene	Benzene in N ₂	77.9	+3.60	
			Benzene In N ₂	345	+1.50	
39	В	Coke oven	Benzene in N ₂	8.20	- 2.60	Ε
		Byproduct	Benzene in N ₂	85.4	-8.70	
40	D	Coke oven	Benzene in N ₂	10.9	+20.0	
		Byproduct	Benzene in N ₂	147	+6.80	
41 H	н	Paint spray	Benzene in N ₂	10.8	NA	F
			m-Xylene in N ₂	16.4	NA	
42	н	Ti re	Cyclohexane in N ₂	93.4	-11.1	£
		manufacturi ng				
43	В	Coke oven	Benzene in N ₂	7.54	+0.10	D
			Benzene in N ₂	225	+0.40	
44	D	Ethyl benzene/	Benzene in N ₂	8.20	-3.40	D
		styrene	Benzene in N ₂	74.5	-0.20	
			Propane in N ₂	10.6	-3. 00	
45	F	Industrial	Propane in Air	316	-3.20	E
		surface coating	Propane in Air	450	-2.00	
46	EPA, QAD	Ti re	Propane in Air	15.0	NA	F
		manufacturing	Propane in Air	316	NA	

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		TABLE J. JUNINA	Y OF PERFORMANCE AUDIT RE			
Audi t				RTI audit	Client audit	Status of
√o.	CII en t***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi t**
47	EPA, QAD	Tire	Propane in air	20.8	-18.4	Ε
••	2111) 4112	manufacturing	Propane in air	453	+13.4	_
		manuraerur riig	Tropuno III uli	1,25	.,5.	
48	. D	Dimethyl	Meta-xylene in N ₂	487	-2.10	Ε
		terephthalate	-			
		production				
49	EPA, QAD	Instrument	Toluene in N ₂	61.5	NA	F
	,	check-out	Methanol in N ₂	55.2	NA NA	•
		oneek out	1.01.1.01.01.11.11.2			
50	EPA, QAD	Tire oven	Propane in air	4.90	-48.8	E
		manufacturing	Propane in air	613	+16.9	
			Propane In air	718	+16.8	
51	EPA, QAD	Instrument	Propane in air	20.8	+20.0	E
		check-out	Propane in air	316	-9.20	
52	D	Styrene	Benzene in N ₂	106	-4.90	E
		manufacturing	Benzene in N ₂	358	-3.70	
		-	1,3-Butadiene in N ₂	20.9	+23.8	
53		Veg. oil	Cyclohexane in N ₂	99.0	-3.50	E
		manufacturi ng	2			
54	М	Research	Chloroform In N ₂	16.5	NA	F
- *	••		Chloroform in N ₂	531	NA NA	•
			3.1101010111111112	251	141	
55	J	Research	Ethylene in N ₂	300	+1.40	E

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

		TABLE 3. SUMMARY	OF PERFORMANCE AUDIT RE			
Aud i †				RTI audit	Client audit	Status of
No.	Cli ent***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi †**
56	К	Reactivity of vent activated charcoal	Chloroform in N ₂	8.11	NA	F
57	EPA, QAD	Instrument check-out	Hydrogen sulfide in N ₂	16.2	NA	F
58	С	Coll coating	Propane in Air Propane in Air	5•20 472	NA -8.40	E
59	L	Maleic anhydride	Benzene in N ₂ Benzene in N ₂	9•45 341	NA NA	F
60	М	Research	Audit not initiated			
61	EPA (State of Conn.)	Maleic anhydride	Benzene In N ₂	133	NA	F
62	0	,	Meta-xylene in N ₂ Hexane in N ₂	760 1990	NA NA	F
63	. м	Paper and pulp	Methyl mercaptan In N ₂	4.44	NA	F
64	Р	Research	Benzene in N ₂ Methyl ethyl ketone in N ₂	13.4 44.5	NA NA	F .
65	E	Coke oven Byproduct Recovery	Benzene in N ₂ Benzene in N ₂	7 . 93 132	-2.90 +1.39	E

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Audi t				RTI audit	Client audit	Status of
No.	Clien+***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi t**
66	D	Rubber	Benzene in N ₂	12.0	+14.2	Ε
		manufacturing	Benzene in N ₂	10.2	0	
		_	Benzene in N ₂	100	+6.40	
			Benzene in N ₂	335	+6.00	
			Hexane in N ₂	79.8	+1.80	
			Hexane in N ₂	3080	-7.50	
			Propane in Air	9.97	-3.20	
			Propane in Air	314	-10.8	
67	E	Coke oven	Benzene in N ₂	8,29	-2.20	E
		Byproduct	Benzene in N ₂	75.7	-2.50	
		Recovery	2			
68	EPA, Region II	Vinyl chloride	Vinyl chloride in N ₂	5.74	NA	F
		manufacturing	Vinyl chloride in N ₂	28.3	NA	
69	EPA, QAD	Instrument	Propylene in N ₂	328	-7. 00	E
		Check	Propylene in N ₂	725	-8.30	
70	EPA, Region I	Vinyl chloride manufacturing	Vinyl chloride in N ₂	7.50	NA	F
71	E	Degreasi ng	Trichloroethylene in N ₂	14.9	-0.40	E
		vent	Trichloroethylene in N_2	566	-8.70	
72	EPA, QAD	Instrument check-out	Hexane in N ₂	3080	NA	F
73	EPA, QAD	Combustion	Hydrogen sulfide in N ₂	16.2	-7. 50	E
		efficiency test	Methyl mercaptan in N ₂	8.22	-8.90	

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

Audi †				RTI audit	Client audit	Status of
No.	Cli ent***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi †**
74	Ε	Vinyl chloride	1,2-Dichloroethane in N ₂	9,30	+6.00	E
		manufacturing	1,2-Dichloroethane in N ₂	462	+3.70	
75	N	Coll coating	Propane in air	10.0	NA	F
			Propane in air	309	NA	
76	F	Coil coating	Propane in air	10.0	NA	F
			Propane in air	309	NA	
77	D.	Maleic	Benzene in N ₂	9.46	-6.60	E
		anhydr i de	Benzene in N ₂	66.9	-11.7	
78	EPA, Region VII	Instrument	Benzene in N ₂	120	NA	F
		checkout	Hexane in N ₂	30.2	NA ·	
79	D	Maleic	Benzene in N ₂	9.46	-4.60	E
		anhydr i de	Benzene in N ₂	128	+12.5	
80	f	Plywood/veneer	Propylene in N ₂	14.8	-4.70	E
		dryi ng	Propylene in N ₂	328	+4.40	
			Toluene in N ₂	430	-0.80	
81	P	Plywood/veneer	Propylene in N ₂	20.3	+18.2	E
		dryl ng	Propylene in N ₂	479	-22.5	
			Toluene in N ₂	487	+32.5	
82	J	Polypropylene	Propylene in N ₂	9,63	-0.35	E
		manufacturing	Propane in N ₂	19.7	+0.84	
		-	Propane in N ₂	296	+0.45	
83	ı	Coke oven	Hydrogen sulfide in N ₂	437	+4.90	E
			Hydrogen sulfide in N2	647	-16.5	
			Carbonyl sulfide	101	+1.98	

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Aud i †			•	RTI audit	Cilent audit	Status of
No.	Client***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi †**
84	J	Compliance	Benzene in N ₂	7.45	23.0	E
		testi ng	Hexane in N ₂	72.6	0.6	
		_	Toluene in N ₂	15.0	-8.7	
			Methyl mercaptan in N ₂	5.40	NA	
85	1	Steel	. Hydrogen sulfide in N ₂	647	.5.0	E
		manufacturing	Carbonyl sulfide in N ₂	9.08	1.0	
86	1	Oil shale	Hydrogen sulfide in N ₂	437	-3. 0	Ε
			Carbonyl sulfide in N ₂	117	-4.6	
			Methyl mercaptan In N ₂	8.42	-13.3	
87	Q	Maleic Anhy-	Benzene in N ₂	55.7	+528.4	E
		dride Produc- tion	Hexame in N ₂	324	+20.5	
88	R	Refining	Hydrogen sulfide in N ₂	17.5	21.1	E
00	.,	norring	Hydrogen sulfide in N ₂	437	22.0	L
89	Air Quality Bureau, New Mexico	Refining	Hydrogen sulfide in ${ m N}_2$	647	NA	F
90	S	Oil shale	Carbonyl sulfide in N ₂	· 117	-29.1	E
			Methyl mercaptan in N ₂	8.42	-14.8	
			Hydrogen sulfide in N ₂	437	-3.65	
91	F	Compliance	Trichlorethylene in N ₂	94.6	NA	E
		testing &	Propane in N ₂	10.0	NA	
		demonstration	Propane in N ₂	309	-54.0	
			Propane in N ₂	73.8	8.7	

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

Aud I †				RTI audit	Client audit	Status of
No.	Cilen†***	Industry	Audit material	conc. (ppm)	% blas (Avg.)*	audit**
92	EPA, Region 1	Research Method Development	Toluene in N ₂	347	NA	F
93	D	Method Validation	Hydrogen sulfide in N ₂	8.32	NA	F
94	USEPA, Region I	Research Method Development	Vinyl chloride in N ₂ 1,1-dichloroethylene in N ₂ Trichloroethylene in N ₂ Perchloroethylene in N ₂	8.39 14.2 13.5 7.94	-20.2 +10.6 +55.6 +48.1	E
95	E	Acrylonitrile Production	Acrylonitrile in N ₂ Acrylonitrile in N ₂	413 10 _• 8	NA 6 _• 94	E
96	USEPA, Region i	Resource Re- covery Garbage Burning Emis- sions	Propane in N ₂ Propane in N ₂	10 . 0 296	-35.0 -17.2	E
97	Tewksbury State Hospital, MA	Research- Method Develop- ment	Vinyl chloride in N ₂ 1,1-dichloroethylene in N ₂ Trichloroethylene in N ₂ Tetrachloroethylene in N ₂	8.39 14.2 13.5 7.94	+57 -9.9 -4.4 +48.6	E
98	Т	Plywood Veneer	Method 25 gas in N_2 Method 25 gas in N_2	102 as C 1940 as C	NA NA	F
99	U	Hazardous Materials Incineration	Trichloroethylene in N_2 Perchloroethylene in N_2 Chloroform in N_2	8.91 7.94 16.5	NA NA NA	F

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Audi †				RTI audit	Client audit	Status of
No.	Cli en†***	Industry	Audit material	conc. (ppm)	% blas (Avg.)*	audi t**
100	USEPA, Region i	Research Method	Chlorobenzene in N ₂	9.20	NA	F
		Development	Benzene in N ₂	128	NA	
		•	Hexane in N ₂	30.2	NA	
			Meta-xylene in N ₂		ld bulb) NA	
	•		, 2		rm bulb)	
101	U	Hazardous	Trichloroethylene in N ₂	13.5	NA	F
		Materials Incineration	Perchloroethylene in N ₂	14.5	NA	
102	Al legheny	Source Testing	Toluene in N ₂	8.51	NA	F
	County	-	Methyl ethyl ketone in N ₂	38.7	NA	
	·		Acrylonitrile in N ₂	11.6	NA	
			Methyl isobutyl ketone in		NA	
103	1	Hazardous Waste Incin- eration	Vinylidiene chloride in N ₂	14.2	12.3	E
104	ı	Hazardous Waste Incin- eration	Vinylidiene chloride in N ₂	9.00	10.0	E
105	USEPA, Region VI	Plastics	Vinyl chloride in N ₂	8.41	NA	F
106	USEPA, Region VI	Vinyl Chloride Manufacturing	Vinyl chloride in N ₂	8.44	NA	F
107	٧	Instrument	Methyl chloroform in N ₂	10.2	+7.8	Ε
	·	Check	Perchloroethylene in N ₂	7.94	+15.9	-
108	Q	Gasoline Termi- nal	Propane in air	1.18%	-4.2	Ε
109	Р	Chemi cals	Toluene in N ₂	16.4	17.3	Ε
		Manufacturing	Benzene in N ₂	7.3	NA	
			1,2-dichloroethane in N ₂	8.1	NA	
110	MD Dept. of Health	Instrument	Benzene in N ₂	9.64	-6.6	E
		Check	Perchloroethylene in N ₂	14.5	+60.1	**

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

Audit				RT1 audit	Client audit	Status of
No.	Client***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audit**
111	٧	Instrument	Chloroform in N ₂	16.5	+3	E
	·	Check	Carbon tetrachioride in N		+33.0	_
			Trichloroethylene in N ₂	13.5	+4.0	
			Freon 113 In N ₂	9.76	0	
112	J	Research, Method	Propane in N ₂	628	+0.6	Ε
		Development	Toluene in N2	347	+2.0	
113	GA State EPA	Plastics	Vinyl chloride in N ₂	8.44	+10.2	E
114	Sacremento County,	Instrument	Ethylene oxide in N ₂	10. 1	NA	F
	California	Check				
115	W	Instrument	Benzene in N ₂	389	-35.7	E
		Check	Chlorobenzene in N2	9.20	-43.1	
116	. V	Instrument	Methanol in N ₂	55, 2	NA	F
		Check				
117	X	Carbon Adsorp-	Toluene in N ₂	16. 1	NA	F
		tion	Methyl ethyl ketone in N_2	38.7	NA	
			Methylene chloride in N ₂	9, 67	NA	
118	F	Surface Coating	Method 25 gas in N ₂	96.8 as C	+127.3	E
119	К	Source Testing	Freon 113 in N ₂	9. 76	NA	F
120	Z	Source Testing	Toluene in N ₂	8, 51	+38.8	E
			Toluene in N ₂	558	-3, 1	
121	κ	Source Testing	Perchloroethylene in N ₂	7. 94	NA	F
122	LA State EPA	Source Testing	Benzene in N ₂	9. 64	-30, 5	E
			Vinyl chloride in N ₂	8.44	191.5	
			1,2-dichloroethane in N ₂	13.8	-37.0	
	•		Carbon tetrachloride in N2	10,5	-40.0	
123	С	Paper Manufac- turing	Vinyl chloride in N ₂	6,60	NA	F

TABLE 3. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

Aud I †		TABLE 3. SUMMARY	OF PERFORMANCE AUDIT RESUL	RTI audit	Client audit	Status of
No.	Clien†***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audi+**
124	LA State EPA	Instrument	Toluene in N ₂	8.51	-34.2	E
	2.7 37373 277	Check	Methylene chloride in N ₂	9.67	96.8	_
125	Y	Surface Coating	Method 25 gas in N ₂	107 as C	80.4	Ε
			Method 25 gas in N ₂	775 as C	39.5	_
126	1	Oil Shale	Carbonyl sulfide in N ₂	10.7	NA	F
			Carbonyl sulfide in N_2	116	NA	
			Hydrogen sulfide	627	NA	
			Methyl mercaptan	8.42	NA	
127	F ;	Surface Coating	Method 25 gas in N ₂	775 as C	-26.5, -18.7	E
128	Y	Surface Coating	Method 25 gas in N ₂	205 as C	21.5	E
			Method 25 gas in N_2	1040 as C	18.9	
129	J	Research, Method Development	Methyl ethyl ketone in N_2	38.7	NA	F
130	Region VII	Source Testing	Acrylonitrile in N ₂	11.6	-29.3	E
131A	South Coast Air	Hazardous Waste	Benzene in N ₂	134	-28	Ε
	Quality Manage- ment District	Landfill	Methane in N_2	6460	+0.6	
131B	South Coast Air Quality Manage- ment District	Hazardous Waste Landfill	Methane in N ₂	6460	-2,5	E
132	Maryland Dept.	Instrument	Benzene in N ₂	7.9	-11.1	E
	of Health	Check	Trichloroethylene in N ₂	9.4	- 31 . 6	
			Hexane in N ₂	32.8	-18.5	
			Methyl isobutyl ketone in	N ₂ 8.4	+15.4	
			1,2-Dichloroethane in N ₂	13.9	-2.1	
133	State of Call-	Quality Assur-	Methylene chloride in N_2	9. 2	+7.6	E
	fornia Air	ance Audit of	Chloroform in N ₂	4.6	+2.2	
	Resources Board	Standards	Perchloroethylene in N ₂	10,5	+14.3	
			Carbon tetrachloride in N ₂	9.6	+1.0	
			Trichloroethylene in N ₂	14.0	+7.1	
			Freon-113 in N ₂	11.0	-9.1	

TABLE 2. SUMMARY OF PERFORMANCE AUDIT RESULTS (Continued)

Audit	0.1	1-1-1-	A. 41A	RTI audit	Client audit	Status of
No.	Client***	Industry	Audit material	conc. (ppm)	% bias (Avg.)*	audit**
134	AA	Source Testing	Benzene in N ₂	310	+5.2	Ε
135	FF	Source Testing	Method 25 gas in N_2	103.8 as C	+28.1	E
136	J	Instrument Check	Benzene in N ₂ Benzene in N ₂	10.3 121	+12.2 +6.2	E
137	1	Source Testing	Method 25 gas in N ₂	195 as C		A
138	F	Compliance Testing	Methanol in N ₂	48.8	+10.7	E
139	ВВ	Source Testing	Methyl ethyl ketone in N_2 Method 25 gas in N_2	40.4 1060		Α
140	EPA Region II	Metal Refining	Benzene in N ₂	376	-11.2	Ε
141	Commonwealth of Massachusetts	Instrument Check	Benzene in N ₂ Trichloroethylene in N ₂ Methyl ethyl ketone in N ₂	7.9 14.0 40.4	+5.1 -4.3 +31.2	E
142	EPA Region II	Source Testing	Benzene in N ₂	7.8	-5 . 1	D
143	State of Dela- ware	Plastic Manufacturing	Vinyl chloride in N_2 Vinyl chloride in N_2	7.75 20.3	-11.0 -10.3	E
144	EE	Plastic Manufacturing	Method 25 gas in N ₂ Propane in air	96.1 as C 10.9	-24.5 -4.6	E
145	DD	Paper Coating	Toluene in N ₂	546		Α
146	State of Dela- ware	Instrument Check	Benzene in N ₂	7.9	-20.6	E
147	cc	Gasoline Terminal	Propane in N ₂	2052		A
148	, F	Viny1 Coating	Propane in N ₂	308		Α
149	ВВ	Plastic Manufacturing	Methyl ethyl ketone in N_2 Method 25 gas in N_2	40.4 1017 as C		A

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NA = Not analyzed
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**Status Codes:

- A = Cylinder shipped; audit results not yet received;
- B = Audit results received;
- C = Audit report submitted to EPA;
- D = Audit results received, audit report submitted to EPA, cylinder not yet returned by client;
- E = Audit complete;
- F = Audit completed without analysis of audit materials by client.
- ***Whenever the auditee is known, an alphabetical letter is shown. Whenever the auditee is unknown, the name of the agency requesting the audit is shown.

1977 - Audits	1-8	1982	-	Audits	83-86
1978 - Audits	9-28	1983	-	Aud i ts	87-106
1979 - Audits	29-49	1984	-	Aud i ts	107-130
1980 - Audits	50-75	1985	-	Aud i ts	131-149
1981 - Audits	76-82				

SECTION 4 STABILITY STUDIES

An ideal calibration standard or audit material should be stable over its total time of usage. The stabilities of the compounds in the repository were studied through periodic analysis of the cylinder contents. In this project, the gas mixtures in the repository are initially analyzed upon receipt from the specialty gas vendor to corroborate the vendor's analysis. If the RTI analysis result differs from the vendor's value by more than 10 percent, the cylinder is given to a third party (EPA or NBS) for analysis. The gas mixtures are again analyzed at 1 month, at 2 months, and at one year following the initial analysis to determine the stability of the gas mixtures. In some cases, analyses are not performed on the dates specified above; however, every attempt is made to acquire the data on this schedule. Cylinder concentrations are also usually determined prior to each performance audit, providing additional data for use in stability studies.

As the number of analyses per cylinder increases, statistical stability analyses will be performed. The results will be presented in a future report. Statistical stability analyses for ten (10) halocarbons and eight (8) other organics were recently published in the open literature (1,2).

Absolute accuracies of the cylinder analyses have not been determined due to lack of NBS standards for most of the organic gas mixtures above one ppm. Recently NBS has issued SRMs for tetrachloroethylene and benzene and is in the process of certifying a 4-component SRM containing aromatic species (benzene, toluene, chlorobenzene, and bromobenzene) and a 4-component SRM containing halocarbons (chloroform, carbon tetrachloride, tetrachloroethylene and vinyl chloride). Once these NBS-SRMs are available, they will be used in the future to estimate the absolute accuracy. An examination of the data in Attachment 1 shows values for individual cylinder analyses usually vary by less than 10 percent for 4-8 analyses over 2-6 years. This variation indicates changes in cylinder contents (i.e., instability) and the imprecision of the measurement process. The possible sources of experimental error

that could result in apparent differences in concentrations include (1) the variability of the analytical technique used for analysis, (2) stability of and/or accuracy of calibration standards, and (3) the accuracy of reproducing standards for which NBS-SRMs do not exist. these sources of variability contributes to the net uncertainty of the resulting data presented in Attachment 1. Estimates of day-to-day measurement uncertainty (repeatability) for all compounds have not been However, the measurement uncertainties for ten halocarbons were recently published (2). The measurement uncertainty varied from <1 to 10 percent depending on the compound, and the major portion of the uncertainty was attributed to the method of preparation of the calibration standard. The uncertainty for the gas chromatographic analysis was determined to be less than 2 percent by multiple injections of the gas during same day analysis. For some recent analyses of those organics listed in Table 2, the uncertainty in the concentration has These estimates are based on consideration of the been estimated. uncertainties of several parameters involved in the measurement and calibration standard preparation procedures. For example, for those analyses involving the use of the thermal oxidizer and Byron 401, the estimated uncertainties (percent coefficient of variation) were determined to be as follows:

- o CO₂ standard response uncertainty 2%
- o CO_2 standard concentration uncertainty 1%
- o CO₂ analyzer response linearity uncertainty 1%
- o $\,$ oxidized organic calibration mixture response uncertainty 1%
- o organic calibration mixture GC response uncertainty 1%
- o repository mixture GC response uncertainty 1%.

The equation below was then used to estimate the total uncertainty based on the above individual uncertainties.

Total Uncertainty =
$$2 \binom{n}{\sum_{i=1}^{n} e_i^2}^{1/2}$$

Where:

2 = two standard deviations (95 percent confidence limit)

e_i = individual component error, (percent coefficient of variation)

n = total number of error components.

Thus, a total uncertainty of 7.0 percent was obtained for all the compounds listed in Table 2. For those analyses involving the use of NBS-SRM's for calibration, the total uncertainty was determined to be 3.5 percent.

SECTION 5 SUMMARY AND CONCLUSIONS

Cylinder gases of hydrocarbons, halocarbons, and sulfur containing organic species have been used successfully as audit materials to assess the relative accuracy of gas chromatographic systems used to measure source emissions. Absolute accuracy has not been determined due to the lack of NBS standards for most of the organic gas mixtures above 1 ppm; instead an estimated interlaboratory bias has been reported for the performance audits conducted during source testing. This interlaboratory bias has been generally within 15 percent for both low and high concentration gases (Table 3).

Of the 45 gaseous compounds studied or currently under study, 39 have demonstrated sufficient stability in cylinders to be used further as audit materials. Five compounds (ethylamine, paradichlorobenzene, cyclohexanone, 1,2-dibromoethylene, and aniline) are not recommended as audit materials for various reasons as discussed in Attachment 1. One gaseous compound (formaldehyde) was ordered but the speciality gas manufacturer indicated that cylinder gases of this compound could not be prepared. Detailed statistical analyses which would separate statistical deviations from true concentration changes with time for 18 gaseous compounds have been published in a journal publication, and statistical analyses for the remaining compounds will be presented in a future report.

REFERENCES

- 1. R. K. M. Jayanty, C. Parker, C. E. Decker, W. F. Gutknecht, J. E. Knoll and D. J. VonLehmden, "Quality Assurance for Emissions Analysis Systems," <u>Environmental Science and Technology</u>, <u>17</u> (6), 257-263A (1983).
- 2. G. B. Howe, R. K. M. Jayanty, A. V. Rao, W. F. Gutknecht, C. E. Decker and D. J. VonLehmden, "Evaluation of Selected Gaseous Halocarbons for Use in Source Test Performance Audits," <u>J. of Air Pollution Control Association</u>, <u>33</u> (9), 823-826 (1983).

ATTACHMENT 1

Stability Data as of July 1985

- 1.0 BENZENE STABILITY STUDY
- 2.0 ETHYLENE STABILITY STUDY
- 3.0 PROPYLENE STABILITY STUDY
- 4.0 METHANE/ETHANE STABILITY STUDY
- 5.0 PROPANE STABILITY STUDY
- 6.0 TOLUENE STABILITY STUDY
- 7.0 HYDROGEN SULFIDE STABILITY STUDY
- 8.0 META-XYLENE STABILITY STUDY
- 9.0 METHYL ACETATE STABILITY STUDY
- 10.0 CHLOROFORM STABILITY STUDY
- 11.0 CARBONYL SULFIDE STABILITY STUDY
- 12.0 METHYL MERCAPTAN STABILITY STUDY
- 13.0 HEXANE STABILITY STUDY
- 14.0 1,2-DICHLOROETHANE STABILITY STUDY
- 15.0 CYCLOHEXANE STABILITY STUDY
- 16.0 METHYL ETHYL KETONE STABILITY STUDY
- 17.0 METHANOL STABILITY STUDY
- 18.0 1,2-DICHLOROPROPANE STABILITY STUDY
- 19.0 TRICHLOROETHYLENE STABILITY STUDY
- 20.0 1,1-DICHLOROETHYLENE STABILITY STUDY
- 21.0 1,2-DIBROMOETHYLENE STABILITY STUDY
- 22.0 PERCHLOROETHYLENE STABILITY STUDY

- 23.0 VINYL CHLORIDE STABILITY STUDY
- 24.0 1.3-BUTADIENE STABILITY STUDY
- 25.0 ACRYLONITRILE STABILITY STUDY
- 26.0 ANILINE STABILITY STUDY
- 27.0 METHYL ISOBUTYL KETONE STABILITY STUDY
- 28.0 CYCLOHEXANONE STABILITY STUDY
- 29.0 PARADICHLOROBENZENE STABILITY STUDY
- 30.0 ETHYLAMINE STABILITY STUDY
- 31.0 FORMALDEHYDE STABILITY STUDY
- 32.0 METHYLENE CHLORIDE STABILITY STUDY
- 33.0 CARBON TETRACHLORIDE STABILITY STUDY
- 34.0 FREON 113 STABILITY STUDY
- 35.0 METHYL CHLOROFORM STABILITY STUDY
- 36.0 ETHYLENE OXIDE STABILITY STUDY
- 37.0 PROPYLENE OXIDE STABILITY STUDY
- 38.0 ALLYL CHLORIDE STABILITY STUDY
- 39.0 ACROLEIN STABILITY STUDY
- 40.0 CHLOROBENZENE STABILITY STUDY
- 41.0 CARBON DISULFIDE STABILITY STUDY
- 42.0 METHOD 25 GAS MIXTURE
- 43.0 ETHYLENE DIBROMIDE
- 44.0 1,1,2,2-TETRACHLOROETHANE

NOTE: PPM concentrations shown in Attachment 1 are expressed on a mole/mole basis, except for EPA Method 25 mixture which is on a mole carbon/mole basis.

1.0 BENZENE STABILITY STUDY

Cylinder No Cylinder Constr		1A A1	1B Al	1C Al	1D Al	IE S	IF S	IG S
Manufacturer Concentration	ppm	65.4	324	200	117	61.0	71.0	80.0
	Date ppm	7/27/77 (79 . 0)	7/27/77 (374)	7/27/77 (241)	7/27/77 (138)	2/10/78 (62 . 0)	2/10/78 (71 . 0)	2/10/78 (80.0)
	Day ppm	136 (74.0)	136 (337)	247 (216)	29 (144)	78 (62 _• 0)	232 (73 _• 0)	78 (81 . 0)
	Day ppm	156 (78.0)	156 (350)	252 (215)	157 (1 3 4)	216 (61.0)	385 (75 . 0)	216 (81 . 0)
	Day ppm	167 (80 . 0)	167 (355)	381 (218)	252 (129)	385 (65 _• 0)	586 (74.5)	385 (84.0)
	Day ppm	630 (77 . 9)	402 (331)	**	290 (127)	722 (66•9)	882 (75 _• 7)	504 (85 . 4)
RTI Concentration	Day ppm	**	433 (343)		414 (127)	1337 (55•7)	1292 (65 _• 7)	1292 (74.0)
	Day ppm		969 (358)		1247 (132)	1858 (58•7)	2246 (70 _• 0)	2246 (78.3)
	Day ppm		1274 (348)		2438 (121)	2246 (60•4)		
	Day ppm		1491 (324)					
	Day ppm		2056 (305)					
	Day ppm		2438 (319)					

^{*}Al = Aluminum; S = Steel; LS = Low-Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 60°C.

CALIBRATION: Reagent-grade "Benzene" liquid is used as a standard. Pressure-dilution technique is used for making the series of standards for calibration.

^{**}Cylinder empty.

1.0 BENZENE STABILITY STUDY (Continued)

Cylinder No Cylinder Const		1H S	11 S	1J S	IK S	IL S	1M S	IN S
Manufacturer Concentration	рþm	100	139	232	265	296	326	344
	Date	2/8/78	2/9/78	2/9/78	2/9/78	2/9/78	2/9/78	2/9/78
	ppm	(101)	(139)	(229)	(264)	(295)	(319)	(332)
	Day	65	49	233	49	49	49	49
	ppm	(102)	(139)	(237)	(261)	(292)	(316)	(327)
	Day	206	50	386	50	51	51	54
	p pm	(98.0)	(142)	(243)	(268)	(294)	(318)	(342)
	Day	237	96	557	69	93	96	69
RTI Concentration	ppm	(101)	(139)	(225)	(254)	(298)	(323)	(335)
	Day	434	127	**	84	205	433	809
	b bw	(105)	(140)		(269)	(294)	(345)	(342)
	Day	773	205		**	237	830	**
	ppm	(106)	(138)			(302)	(335)	
	Day	831	505			809	1294	
	ppm	(100)	(147)			(295)	(320)	
	Day	1294	1293			1294	2379	
	ppm	(92.0)	(128)			(290)	(310)	
	Day	2380	1338			2379		
	ppm	(96.0)	(128)			(285)		
	Day		2380					
	p pm		(134)					

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

^{**}Cylinder empty.

1.0 BENZENE STABILITY STUDY (Continued)

Cylinder No Cylinder Const		10 S	1P S	1Q S	IR S	1 S S	IT S	IU S
Manufacturer Concentration	ррm	389	8.04	9.85	9.89	9,93	10.0	10.9
	Date	2/9/78	4/21/78	4/21/78	4/21/78	4/21/78	4/21/78	4/21/78
	ррm	(387)	(8.37)	(9,99)	(10.0)	(10.0)	(10.7)	(11.5)
	Day	64	4	5	4	4	25	4
	ppm	(369)	(8,33)	(9.88)	(10.1)	(10,1)	(10.2)	(10.7)
	Day	205	25	25	13	26	146	25
	ppm	(396)	(8,20)	(10.1)	(9.73)	(9.80)	(9.20)	(10.8)
	Day	809	26	332	332	56	362	332
	ppm	(396)	(8.34)	(9.71)	(9.77)	(9.50)	(9.90)	(10.7)
	Day	1294	56	**	1018	146	1222	434
RTI Concentration	ррm	(389)	(8.19)		(9.46)	(8.90)	(9.56)	(10.9)
	Day	2247	134		1270	628	**	759
	ppm	(376)	(7.81)		(9,64)	(9.57)		(10.2)
	Day	•	434			738		1222
	ppm		(8.21)			(9.45)		(9.69)
	Day		766			**		2175
	ppm		(7.93)					(9.90)
	Day		1222					
	ppm		(7.68)					
	Day		2175					
	p pm		(7.90)					

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

^{**}Cylinder empty.

1.0 BENZENE STABILITY STUDY (Continued)

Cylinder N	o .	17	1W	1X	1Y	IZ	1 AA	1 AB
Cylinder Const	ruction*	S	S	S	S	S	\$	\$
Manufacturer Concentration	ppm	12.2	8.09	11.0	11.2	8.09	9.14	270
	Date ppm	4/25/78 (12 , 7)	5/19/78 (8,10)	5/4/78 (11 . 2)	5/4/78 (10•9)	5/4/78 (8,20)	5/4/78 (9.10)	7/27/77
	Day ppm	1 (12,5)	105 (7.70)	132 (10 . 2)	132 (9.90)	132 (7 _• 04)	132 (7.80)	29 (319)
	Day ppm	21 (12 . 3)	287 (8,10)	**	302 (10•7)	302 (7.70)	302 (8,50)	157 (312)
RTI Concentration	Day ppm	109 (12.0)	488 (8 _• 20)		393 (10.8)	473 (7.54)	1005	2056 (305)
	Day ppm	358 (12.1)	784 (8.30)		2162 (10.3)	**	1209 (8 . 42)	**
	Day ppm	755 (12•0)	1194 (7 . 45)				2162 (8.40)	
	Day ppm	1218	2147 (7.80)					
	Day ppm	2171 (11•9)						

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

^{**}Cylinder empty.

2.0 ETHYLENE STABILITY STUDY

Cylinder No		2A	2B	2C	·20	2E	2F	2G
Cylinder Constr		A I	A1	A1	A1	. AI	Al	A1
Manufacturer Concentration	ppm	2920	3000	4960	4970	19900	19900	4.95
	Date	2/23/78	2/23/78	2/23/78	2/23/78	2/24/78	2/24/78	4/27/78
	ppm	(3070)	(3130)	(5210)	(5200)	(20400)	(20600)	(4 _• 70)
	Day	49	49	48	48	48	48	29
	ppm	(3120)	(3180)	(5340)	(5280)	(20800)	(20800)	(4,70)
RTI	Day ppm	198 (2880)	198 (2940)	201 (4660)	201 (4910)	200 (20200)	200 (20300)	106 (4.85)
Concentration	Day	809	809	809	809	808	808	741
	ppm	(3200)	(3270)	(5380)	(5340)	(18900)	(19000)	(4.62)
	Day	2291	2291	2291	2291	2290	2290	1180
	ppm	(3280)	(3350)	(5520)	(5480)	(20600)	(20700 ₎	(5.12)
	Day ppm							2224 (4 _• 50)

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Durapak n-octane on Porasil C column at 30 degrees Celsius.

CALIBRATION: NBS-SRM Propane is used for standard calibration.

2.0 ETHYLENE STABILITY STUDY (Continued)

Cylinder No Cylinder Constr		2H A1	21 A1	2J Al	2K A1	2L A1	2M A1	2N Al
Manufacturer Concentration	рpm	10.0	15.0	19.9	300	448	603	701
	Date	4/27/78	4/28/78	4/28/78	4/28/78	4/28/78	4/28/78	4/28/78
	p pm	(9.70)	(14.4)	(19.2)	(306)	(468)	(629)	(740)
	Day	29	28	28	33	33	34	34
	ppm	(9.60)	(14.4)	(19.3)	(319)	(493)	(646)	(749)
	Day	106	104	104	105	104	104	104
RTI	ppm	(9.90)	(14.9)	(20.3)	(312)	(473)	(636)	(737)
Concentration	Day	740	739	739	728	740	740	740
	ppm	(8.40)	(18.0)	(21.5)	(300)	(457)	(606)	(703)
	Day	1180	1179	1179	2225	2225	2225	2225
	ÞÞm	(10.0)	(14.4)	(18.9)	(291)	(435)	(583)	(678)
	Day	2224	2223	2223				
	ppm	(9.50)	(14.2)	(18.9)				
	Day	2587 (9.54)						

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

3.0 PROPYLENE STABILITY STUDY

Cylinder No Cylinder Constr		3A A1	3B A1	3C Al	3D A I	3E A I	3F Al	3G Al	3H A I
Manufacturer Concentration	рpm	4.94	9.91	14.8	20.0	298	446	585	683
	- Date	4/27/78	4/27/78	4/27/78	4/27/78	4/27/78	4/27/78	4/27/78	4/27/78
	p pm	(4.86)	(9.83)	(14.6)	(19.8)	(296)	(442)	(577)	(672)
	Day	26	26	26	27	27	27	27	27
	ppm	(4.94)	(9.85)	(14.5)	(19.0)	(286)	(428)	(560)	(655)
	Day	27	104	104	104	104	105	104	105
RTI	ppm	(4.78)	(10.3)	(14.8)	(20.0)	(317)	(474)	(629)	(729)
Concentration	Day	104	749	749	749	750	750	750	750
	ppm	(4.98)	(9.76)	(14.8)	(20.3)	(324)	(479)	(620)	(721)
	Day	749	1250	**	2229	820	2229	2229	820
	ppm	(4.93)	(9.63)		(19.7)	(328)	(444)	(579)	(725)
	Day	2229	2229			**			2229
	p pm	(4.80)	(9.80)						(676)
	Day	2601							
	b bw	(4.75)							

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Durapak n-octane on Porasil C column at 30 degrees Celsius

CALIBRATION: NBS-SRM Propane is used for standard calibration.

^{**}Cylinder empty.

4.0 METHANE/ETHANE STABILITY STUDY

Cylinder No. Cylinder Construction*		4A A1		4B A1		4C A I		4D A1	
Audit Material**		М	Ε	М	Ε	М	Ε	М	E
Manu facturer Concentration	ppm	6000	714	8130	597	1000	295	1670	202
	Date ppm	7/21/78 (6210)	7/21/78 (773)	7/21/78 (8130)	7/21/78 (654)	7/21/77 (1020)	7/21/77 (315)	7/21/77 (1710)	7/21/77 (220)
	Day	264	163	35	35	264	163	35	29
	ppm	(5980)	(715)	(7550)	(663)	(983)	(292)	(1560)	(218)
RTI	Day ppm	662 (6580)	264 (684)	264 (7820)	163 (606)	1027 (1290)	264 (283)	264 (1640)	157 (202)
Concentration	P P	(0,00)	(00.7	((000)	(,,_,,,	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	,,,,	,
	Day ppm	2145 (6460)	662 (703)	662 (8590)	264 (577)	2510 (1068)	1027 (284)	1027 (1950)	258 (195)
	Day		2145	2145	662		2510	2510	1027
	ÞÞm		(730)	(8430)	(598)		(300)	(1770)	(206)
	Day				2145				2510
	рpm				(619)			•	(207)

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Durapak n-octane on Porasil C column at 30 degrees celsius.

CALIBRATION: NBS-SRM methane is used for standard calibration.

^{**}M = Methane; E = Ethane.

5.0 PROPANE STABILITY STUDY

Cylinder No Cylinder Const		5A A I	58 A1	5C Al	50 A I	SE Al	5F Al	5G Al	5H Al
Manufacturer Concentration	ppm	5.01	10.0	14.6	20.0	303	439	604	708
	Date ppm	4/25/78 (4 . 90)	4/25/78 (9,70)	4/25/78 (14 . 3)	4/25/78 (19 _• 5)	4/26/78 (304)	4/26/78 (441)	4/26/78 (615)	4/27/78 (730)
	Day	24	24	25	25	24	24	27	26
	ppm	(4,90)	(9.80)	(14.5)	(19.8)	(301)	(436)	(615)	(723)
	Day	108	108	108	108	107	107	107	106
	p pm	(5.10)	(10.1)	(14.9)	(20.3)	(305)	(440)	(607)	(710)
	Day	605	513	582	582	530	530	604	603
	p pm	(4.89)	(10.6)	(15.0)	(20.8)	(316)	(450)	(613)	(718)
	Day	729	752	736	736	581	581	735	734
RTI Concentration	p pm	(5.20)	(10.0)	(14.7)	(20.1)	(316)	(453)	(628)	(734)
CONCENT ATTOM	Day ppm	**	914 (10.0)	2220 (14.8)	1252 (19•7)	735 (313)	728 (472)	2218 (607)	2218 (715)
	Day		2220	2589	2220	752	**		
	p pm		(10.9)	(14.8)	(20.0)	(314)			
	Day					913			
	ppm					(309)			
	Day					1251			
	ppm					(296)			
	Day		•			2219			
	рpm					(308)			

^{*}Al = Aluminum; S = Steel, LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Durapak n-octane on Porasil C column at 30 degrees Celsius.

CALIBRATION: NBS-SRM Propane is used for standard calibration.

UNCERTAINTY OF REPORTED CONCENTRATIONS: + 3.5%

^{**}Cylinder empty.

5.0 PROPANE STABILITY STUDY (Continued)

.	51	5J	5K	5L
ruction*	AI	Al	Al	AI
ppm	1000	2000	10,000	20,000
Date ppm	3/3/83 (1027)	3/3/83 (2100)	3/3/83 (11800)	3/3/83 (20700)
Day ppm	452 (1070)	452 (2180)	452 (13000)	452 (21000)
Day ppm	734 (1006)	734 (2052)	734 (13021)	734 (21302)
	Date ppm Day ppm	ppm 1000 Date 3/3/83 ppm (1027) Day 452 ppm (1070) Day 734	ppm 1000 2000 Date 3/3/83 3/3/83 ppm (1027) (2100) Day 452 452 ppm (1070) (2180) Day 734 734	Date 3/3/83 ppm 3/3/83 3/3/83 3/3/83 ppm 3/3/83 3/3/83 3/3/83 ppm 3/3/83 2/100 11800 Day 452 452 452 452 ppm 452 452 (13000) 13000 Day 734 734 734

^{*}Al = Aluminum; S = Steel, LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Durapak n-octane on Porasil C column at 30 degrees Celsius.

CALIBRATION: NBS-SRM Propane is used for standard calibration.

UNCERTAINTY OF REPORTED CONCENTRATIONS: + 3.5%

6.0 TOLUENE STABILITY STUDY

Cylinder No Cylinder Constr		6A LS	6B LS	6C S	6D S	6E S	6F S
Manu facturer Concentration	ppm	408	606	16.2	9,11	9.00	430
	Date ppm	12/6/78 (405)	12/6/78 (585)	10/3/78 (17.3)	10/3/78 (9,62)	3/29/83 (8•51)	7/1/80 (430)
	Day ppm	3 (405)	3 (579)	48 (14•9)	64 (8 _• 50)	744**** (8 ₊ 04)	861 (347)
RTI	Day ppm	86 (394)	86 (577)	365 (15 _• 0)	66 (8,60)		1115 (338)
Concentration	Day ppm	100 (393)	358 (615)	1373 (14 . 8)	160 (8,20)		1505 (427)***
	Day ppm	**	2079 (663)***	**	**		1765 **** (351)
	Day ppm		2338**** (603)				

^{*}Al = Aluminum, S = Stee!, LS = Low Pressure Steel.

Questionable value.

Concentration uncertainty: + 7%

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 60 degrees Celsius.

CALIBRATION: Reagent grade "Toluene" liquid is used as a standard. Pressure-dilution technique is utilized for generation of series of standards for calibration.

^{**}Cylinder empty.

6.0 TOLUENE STABILITY STUDY (Continued)

Cylinder No Cylinder Constr	•	6G A I	6H A I	61 A1	6J Al	6K LS	6L LS
Manufacturer Concentration	ppm	18.2	9.0	10,3	21.7	196	310
	Date ppm	7/27/83 (16•1)	7/1/80 (8,50)	12/11/84 (9 . 27)	12/11/84 (20,3)	12/11/84 (183)	12/11/84 (290)
RTI Concentration	Day ppm	383 (19 _• 1)***	1505 (9 . 40)	192**** (8.70)	121**** (18 . 9)	141 **** (184)	141**** (281)
		**	**				

^{*}AI = Aluminun, S = Steel, LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 60 degrees Celsius.

CALIBRATION: Reagent grade "Toluene" liquid is used as a standard. Pressure-dilution technique is utilized for generation of series of standards for calibration.

^{**}Cylinder empty.

^{***}Questionable value.

^{****}Concentration uncertainty: \pm 7%

7.0 HYDROGEN SULFIDE STABILITY STUDY

Cylinder No Cylinder Const		7A A I	7B A I	7C Al	7D A I	7E A1	7F A I	7G A I
Manufacturer Concentration	ppm	399	9,15	16.7	649	6.95	6,45	671
	Date	10/1/78	7/7/78	10/1/78	10/1/78	10/1/78	10/1/78	3/2/83
	ppm	(371)	(9,73)	(16.1)	(641)	(7,05)	(4,94)	(628)
	Day	38	87	38	38	87	38	687
	p pm	(424)	(6,72)	(16.5)	(655)	(5, 75)	(5, 14)	(683)
	Day	111	124	111	111	124	111	833
	o pm	(414)	(7,11)	(15,7)	(690)	(5,62)	(4.81)	(654)
RTı	Day	1030	197	580	1030	197	580	
Concentration	p pm	(437)	(6,36)	(16,2)	(647)	(5, 23)	(4,35)	
	Day	2270	696	1030		696	1030	
	p pm	(444)	(6, 23)	(17.5)	**	(5, 14)	(3,71)	
	Day	2446	1116	2270		1116	2325	
	p pm	(401)	(8, 32)	(14,5)		(5,38)	(4.3)	
	Day		2399	2300		2325	2446	
	рpm		(8.0)	(15.3)		(4.6)	(4.1)	
	Day		2424	2446		2446		
	p pm		(6,6)	(15.6)		(4,4)		
	Day		2545					
	ppm		(6.0)					

^{*}Al = Aluminum, S = Stee!, LS = Low Pressure Stee!.

ANALYTICAL CONDITIONS: Flame photometric detector, Chromosil 330 column at 60 degrees Celsius.

CALIBRATION: Reagent grade pure "Hydrogen sulfide" gas is used as a standard. Dilutions are made in a Tedlar bag for generation of series of standards for calibration. Permeation tube is used as a standard for calibration for the last two analyses of low concentration cylinders.

ANALYTICAL PROBLEMS: Only a Teflon® column and Teflon® lines should be used. The air-to-hydrogen ratio is critical to the sensitivity of the FPD.

^{**}Cylinder empty.

7.0 HYDROGEN SULFIDE STABILITY STUDY (Continued)

									
Cylinder N	٥.	7H	71	7J	7K	7L	7M	7N	70
Cylinder Const	ruction*	Al	AI ·	Al	AI	AI	Al	Al	AI
Manufacturer Concentration	ppm	20.77	29,27	39.14	97.31	206.3	323,2	417	503.2
RTI	Date	1/17/85	1/17/85	1/17/85	1/17/85	,1/16/85	1/16/85	1/16/85	1/16/85
Concentration	ppm	(17.7)	(22.6)	(31.6)	(83,7)	(200)	(291)	(398)	(489)
	Day	25	25	25	146	147	147	147	147
	p pm	(20.6)	(30.4)	(42.4)	(92.1)	(210)	(320)	(415)	(514)
	Day	146	146	146					
	ppm	(21.0)	(30,5)	(40.5)					

^{*}AI = Aluminum, S = Steel, LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame photometric detector, Chromosil 330 column at 60 degrees Celcius.

CALIBRATION: Reagent grade pure "Hydrogen sulfide" gas is used as a standard. Dilutions are made in a Tedlar bag for generation of series of standards for calibration. Permeation tube is used as a standard for calibration for the last two analyses of low concentration cylinders.

ANALYTICAL FROBLEMS: Only a Teflon® column and Teflon® lines should be used. The air-to-hydrogen ratio is critical to the sensitivity of the FPD.

8.0 M-XYLENE STABILITY STUDY

Cylinder No Cylinder Consti		8A LS	88 LS	8C S	80 S	8E LS	8F LS	8G A1
Manufacturer Concentration	ррm	405	613	17.3	7,33	······································		· · · · · · · · · · · · · · · · · · ·
	Date	10/5/78	10/5/78	10/5/78	10/5/78	6/7/85***	6/7/85***	6/7/85***
	o pm	(480)	(720)	(16.6)	(6,20)	(596)	(362)	(11.5)
	Day	63	63	63	63			
	p pm	(445)	(676)	(17.2)	(6,81)			
	Day	158	1 58	166	166		•	
RTI Concentration	ppm	(425)	(656)	(20.8)	(6,82)			
	Day	412	606	302	1036			
	p pm	(487)	(760)	(16.4)	(5,66)			
	Day	606	2140	1036	•			
	p pm	(507)	(598)	(19.0)				
		**	**	**	**			

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

CALIBRATION: Reagent grade "M-Xylene" liquid is used. Pressure-dilution technique is used for generation of series of standards for calibration.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 60, 120 or 140 degrees Celsius.

^{**}Cylinder empty.

^{***}Concentration uncertainty: \pm 7%

9.0 METHYL ACETATE STABILITY STUDY

Cylinder No Cylinder Consti		9A S	9B S	9C S	9D S
Manu facturer	ppm	326	455	6.84	17.2
Concentration					
	Date	10/13/78	10/13/78	10/13/78	10/13/78
	p pm	(271)	(428)	(5,29)	(12.9)
	Day	230	230	230 [°]	230
	рpm	(340)	(437)	(4,86)	(12,5)
RTI	Day	286	286	286	286
Concentration	p pm	(324)	(442)	(5,02)	(11.8)
	Day	629	629	630	630
	ppm	(348)	(479)	(5.88)	(12,5)
	Day	2442**	2442**	2442**	2442**
	p pm	(336)	(470)	(5,32)	(17 _* 2)* ¹

^{*} Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Fiame ionization detector, 10% OV-275 on Chromosorb WHP column at 50 degrees Ceisius.

CALIBRATION: Reagent grade "Methyl acetate" liquid is used as a standard. Pressure-dilution technique is utilized for generation of series of standards for calibration.

^{**}Concentration uncertainty: \pm 7%

 $^{^{***}}$ Questionable value.

10.0 CHLOROFORM STABILITY STUDY

Cylinder N		IOA	108	100	100	
Cylinder Construction*		\$ 	S	S	S	
Manufacturer Concentration	ppm	520	348	8.70	16.9	
	Date	10/17/78	10/17/78	10/17/78	10/17/78	
	p pm	(529)	(345)	(8.08)	(17.6)	
	Day	161	161	161	161	
	b bw	(515)	(351)	(7.39)	(16.5)	
	Day	256	256	256	256	
RT1 Concentration	ppm	(514)	(340)	(7.50)	(16.2)	
	Day	553	975	553	553	
	ppm	(531)	(325)	(8.11)	(16.5)	
	Day	**	2422***	2422***	2422***	
	ppm		(333)	(4.26)	(14.9)	

^{*}AI = Aluminum; S = Steel; LS = Low Pressure.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 50 or 100 degrees Celsius.

CALIBRATION: Reagent grade "Chloroform" liquid is used as a standard. Pressure-dilution technique is utilized for generation of series of standards for calibration.

^{**}Cylinder empty.

^{****}Concentration uncertainty: \pm 7%.

11.0 CARBONYL SULFIDE STABILITY STUDY

Cylinder No Cylinder Constr		1 1A S	1 1B S	11C S	1 1D S	11E AL	11F AL
Manufacturer Concentration	ppm	251	100	9, 96	7.03	9.54	101
	Date	11/3/78	11/3/78	11/3/78	11/3/78	9/18/81	9/18/81
	p pm	(276)	(109)	(9,10)	(6.81)	(12.9)	(111)
RTI	Day	78	78	78	78	35	35
Concentration	рpm	(281)	(111)	(8.66)	(6,48)	(12.5)	(117)
	Day	185	185	185	185	222	
	p pm	(275)	(95.0)	(8,23)	(6.41)	(9.08)	
		**	**	**	**		**

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame photometric detector, Carbopak B column at 50 degrees Celsius or Chromosil 330 column at 60 degrees Celsius.

CALIBRATION: Reagent grade pure "Carbonyl Sulfide" gas is used as a standard. Dilutions are made in Teflon® bag for generation of series of standards for calibration.

ANALYTICAL PROBLEMS: Only a Teflon column and Teflon lines should be used. The air-to-hydrogen ratio is critical to the sensitivity of the FPD.

^{**}Cylinder empty.

11.0 CARBONYL SULFIDE STABILITY STUDY (Continued)

Cylinder No Cylinder Constr		1 1G A1	1 1H Al	111 A1
Manu facturer Concentration	рpm	99•2	225	414
RTI	Date	1/11/85	1/11/85	1/11/85
Concentration	ppm	(101)	(228)	(423)
	Day ppm	150 (96•5)	150 (199)	150 (404)

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame photometric detector, Carbopak B column at 50 degrees Celsius or Chromosil 330 column at 60 degrees Celsius.

CALIBRATION: Reagent grade pure "Carbonyl Sulfide" gas is used as a standard. Dilutions are made in Teflon® bag for generation of series of standards for calibration.

ANALYTICAL PROBLEMS: Only a Teflon column and Teflon lines should be used. The air-to-hydrogen ratio is critical to the sensitivity of the FPD.

12.0 METHYL MERCAPTAN STABILITY STUDY

Cylinder No		12A A1	128	120	12D A1	
Cylinder Construction*			AI	A1		
Manu facturer Concentration	ppm	8,03	10.0	3, 55	4.22	
	Date	1/24/79	1/24/79	1/24/79	1/24/79	
	ppm	(5,66)	(7, 94)	(3,65)	(4.23)	
	Day	104	104	104	104	
	рpm	(5,60)	(8, 10)	(3,50)	(4,76)	
RTI	Day	139	139	139	139	
Concentration	ppm	(5,65)	(7.90)	(3,56)	(4,54)	
	Day	985	985	985	**	
	ppm	(5,40)	(8,42)	(3,64)		
	Day	2194	2194	2194		
	ppm	(5,45)	(8.00)	(3.80)		
	Day	2331	2331	2331		
	p pm	(4.70)	(8.00)	(3,40)		

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame photometric detector, Carbopak B column at 50 degrees Celsius or Chromosil 330 column at 60 degrees Celsius.

CALIBRATION: Reagent grade pure "Methyl mercaptan" gas is used a standard. Dilutions are made in a Teflon® bag for generation of series of standards for calibration. Permeation tube was used as a standard for calibration for the last two analyses.

ANALYTICAL PROBLEMS: Only a Teflon column and Teflon lines should be used. The air-to-hydrogen ratio is a critical variable.

^{**}Cylinder empty.

13.0 HEXANE STABILITY STUDY

Cylinder No Cylinder Const		I 3A LS	13B LS	13C A1	130 A1	1 3E A I
Manufacturer Concentration	ppm	1975	2973	30.6	79.2	80.0
	Date	2/6/79	2/6/79	2/6/79	2/6/79	3/25/83
	ppm	(2170)	(3070)	(30.8)	(82.2)	(83,2)
	Day	6	6	296	296	376
	p pm	(1980)	(2860)	(30.1)	(81.0)	(88.2)
	Day	337	338	337	337	
	рpm	(2070)	(2950)	(30.6)	(81.3)	
RTI	Day	469	469	469	469	
Concentration	ppm	(1990)	(3080)	(32.0)	(79.8)	
	Day	1886	1886	523	835	
	ppm	(1990)	(2980)	(30.0)	(80.2)	
	Day		**	835	1247	
	ppm			(30.2)	(82.7)	
	Day			1886	**	
	ppm			(32.8)		

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 60 or 100 degrees Celsius.

CALIBRATION: Reagent grade "Hexane" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Cylinder empty.

14.0 1,2 DICHLOROETHANE STABILITY STUDY

Cylinder No Cylinder Constr		14A A1	14B Al	I 4C A I	14D Al	14E Al	14F Al	I 4G A I	14H Al
Manu facturer Concentration	ppm	14.4	9.64	100	526	6,92	12.5	97•9	439
	Date ppm	1/19/79	1/19/79 (9•20)	1/19/79 (96•2)	1/19/79 (498)	4/5/79 (10 . 0)	4/5/79 (15 , 2)	4/5/79 (102)	4/5/79 (463)
	Day	58	58	58	58	30	30	30	30
	ppm	(15.2)	(10.8)	(103)	(534)	(9,42)	(14.7)	(105)	(451)
	Day	155	155	155	155	69	69	69	69
	p pm	(14.9)	(10.0)	(98,2)	(524)	(9.30)	(14.3)	(99.0)	(462)
	Day	811	81 1	501	501	586	811	425	589
RTI	p pm	(14.2)	(9,56)	(87.3)	(592)**	(9.14)	(14.5)	(87.3)	(432
Concentration	Daγ	835	835	920	920	811	835	844	697
	p pm	(13.5)	(9.19)	(102)	(502)	(9,70)	(13.8)	(101)	(451
	Day	1964	1964	1964	1964	835	1888	1888	844
	p p m	(13.9)	(9.68)	(94.9)	(477)	(9, 16)	(13.9)	(92.4)	(453)
	Day	2333***	2333***	2333***	2333***	2247***	2247***	2247***	1888
	ppm	(14.1)	(9.30)	(96.7)	(496)	(9.32)	(14.3)	(96.0)	(416)
	Day								2247**
	ppm								(427)

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Fiame ionization detector, 10% OV-101 on Chromosorb WHP column at 100 degrees Celsius.

CALIBRATION: Reagent grade "1,2 Dichloroethane" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

 $^{^{**}}$ Questionable value.

^{***}Concentration uncertainty: \pm 7%

15.0 CYCLOHEXANE STABILITY STUDY

Cylinder No	^	15A	
Cylinder Consti		Al	
Manufacturer Concentration	ppm	99.1	
	Date	3/19/79	•
	ppm	(106)	
	Day	147	
	ppm	(93.4)	
RTI	Day	394	
Concentration	ppm	(99.0)	
	Day	926	
	рpm	(102)	
	Day	1966	
	p pm	(95.9)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 100 degrees Celsius.

CALIBRATION: Reagent grade "Cyclohexane" liquid is used as a standard. Pressure-dilution technique is used for making series of standards for calibration.

16.0 METHYL ETHYL KETONE STABILITY STUDY

Cylinder N		16A	
Cylinder Const	ruction*	S	
Manufacturer Concentration	ppm	43.7	
	Date	5/23/79	
	ppm	(42.3)	
	Day	28	
	ppm	(40.0)	
	Day	58	
RT1 Concentration	ppm	(39,9)	
	Day	380	
	bbw	(44.5)	
	Day	653	
	ppm	(38.7)	
	Day	1847	
	ppm	(40.4)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Chromosorb 101 column at 180 degrees Celsius.

CALIBRATION: Reagent grade "Methyl ethyl ketone" liquid is used as a standard. Pressure-diultion technique is utilized for making series of standards for calibration.

17.0 METHANOL STABILITY STUDY

Cylinder No. Cylinder Construction*			
Manufacturer Concentration	ррт	50.0	97.2
	Date	5/17/79	11/28/84
	ppm	(58.8)	(106)
	Day	21	202**
	p pm	(52,3)	(88.4)
	Day	51	
RTI Concentration	ppm	(51.1)	
	Day	196	
	ppm	(55,2)	
	Day	2020	
	ppm	(48.8)	
	Day	2224**	
	p pm	(45.8)	

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, Chromosorb 101 column at 50 degrees Celsius or 0.2% Carbowax 1500 plus 0.1% SP-2100 on Carbowax C at 60 degrees Celsius.

CALIBRATION: Reagent grade "Methanol" is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Concentration uncertainty: \pm 7%

18.0 1,2-DICHLOROPROPANE (PROPYLENE DICHLORIDE) STABILITY STUDY

Cylinder No Cylinder Consti		18A A1	18B A1	18C Al	18D Al
Manufacturer Concentration	p pm	7.07	14.6	476	664
	Date	7/10/79	7/10/79	7/10/79	7/10/79
	ppm	(6.06)	(15.6)	(496)	(685)
	Day	28	28	28	28
	ppm	(5,52)	(16.4)	(455)	(621)
	Day	48	48	48	48
	p pm	(5,94)	(15.0)	(480)	(675)
	Day	497	749	372	372
RTI Concentration	p pm	(6,03)	(16.3)	(497)	(685)
	Day	749	1793	1793	1793
	ppm	(5,59)	(12.1)	(402)	(557)
	Day	1793	1845	1845	1845
	ppm	(3.12)	(13,2)	(424)	(574)
	Day	1845	2155**	2155**	2155**
	ppm	(3.86)	(13.3)	(441)	(594)
•	Day	2155**			
	ppm	(3,49)			

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 100 degrees Celsius.

CALIBRATION: Reagent grade "1,2-Dichloropropane" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Concentration uncertainty: \pm 7%

19.0 TRICHLOROETHYLENE STABILITY STUDY

•	Cylinder No. Cylinder Construction*		19B A1	19C Al	19D A1	
Manufacturer Concentration	ppm	9,23	14.7	100	505	
	Date	5/24/79	5/24/79	5/24/79	5/24/79	
	ppm	(9.58)	(14.3)	(102)	(506)	
	Day	77	77	77	77	
	ppm	(10.2)	(15.1)	(103)	(503)	
	Day	92	92	92	92	
RTI Concentration	ppm	(9.78)	(14.9)	(100)	(499)	
	Day	683	683	810	810	
	ppm	(9.03)	(13.6)	(105)	(522)	
	Day	820	820	820	820	
	p pm	(8.91)	(13.5)	(94.6)	(490)	
	Day	1853	1853	1853	1853	
	ppm	(9,40)	(14.0)	(105)	(523)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 100 degrees Celsius.

CALIBRATION: Reagent grade "Trichloroethylene" liquid is used as a standard. Pressure-dilution technique is used for making series of standards for calibration.

20.0 1,1-DICHLOROETHYLENE (VINYLIDENE CHLORIDE) STABILITY STUDY

Cylinder No Cylinder Const		20A 20B AI AI		20C Al	20D A I	
Manu facturer Concentration	рpm	9,58	14.8	96.8	490	
	Date	6/1/79	6/1/79	6/1/79	6/1/79	
	p pm	(10,3)	(15.6)	(101)	(524)	
	Day	35	35	35	35	
	ppm	(9,90)	(15.1)	(99.0)	(510)	
	Day	62	62	62	62	
	ppm	(10.1)	(15.5)	(102)	(505)	
RTI	Day	404	404	817	404	
Concentration	рþm	(11.5)**	(17.1)**	(94.0)	(498)	
	Day	818	818	1831	1831	
	p pm	(9.00)	(14.2)	(98.4)	(488)	
	Day	1831	1831	2190***	2190**	
	o pm	(9.00)	(13.2)	(94.7)	(479)	
	Day	2190***	2190***			
	p.pm	(8.78)	(14.1)			

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Fiame ionization detector, 10% OV-101 on Chromosorb WHP column at 100 degrees Celsius or 10% SP-2100 on Supelcoport column at 100 degrees Celsius.

CALIBRATION: Reagent grade "1,1-Dichloroethylene" pure liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

 $^{^{**}}$ Questionable value.

^{***}Concentration uncertainty: \pm 7%.

21.0 1,2-DIBROMOETHYLENE STABILITY STUDY

Cylinder No Cylinder Consti		21A LS	9 6/18/79 6/1/79) (12 _• 2) (110)		21D LS
Manufacturer Concentration	ppm	10.0	14.9	99.9	301
	Date	6/18/79	6/18/79	6/1/79	6/18/79
	ррm	(7.90)	(12.2)	(110)	(265)
	Day	61	61	61	61
RTI	ppm	(7.80)	(12.0)	(107)	(266)
RTI Concentration	Day	89	89	89	89
	ppm	(7.40)	(11.6)	(105)	(257)
	Day	722	772	787	643
	ppm	(7.72)	(8.02)	(99.2)	(309)
		**	**	**	**

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 100 degrees Celsius.

CALIBRATION: Reagent grade "1,2-Dibromoethylene" pure liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

ANALYTICAL PROBLEMS: The gas mixtures and the calibration standards contain substantial amounts of both the <u>cis</u> and the <u>trans</u> isomers of 1,2-Dibromoethylene. The first three sets of analyses are questionable because only one isomer was measured during the calibrations and cylinder analyses. During the GC analyses on Day 1864, it was found that dibromoethylene partially converted to an unknown compound. Hence, dibromoethylene is not practical as an audit material.

 $^{^{**}}$ Cylinders returned due to partial conversion to an unknown compound.

22.0 PERCHLOROETHYLENE STABILITY STUDY

Cylinder N	o.	22A	228	22C	220		
Cylinder Constr	inder Construction*		ler Construction* S S		\$	LS	LS
Manu facturer Concentration	ppm	7.98	13.0	487	629		
	Date	7/6/79	7/6/79	7/6/79	7/6/79		
	ррm	(8.40)	(15.0)	(419)	(624)		
	Day	35	35	35	35		
	p pm	(7.97)	(14.9)	(453)	(642)		
	Day	52	52	52	52		
	ppm	(7.92)	(14.7)	(440)	(619)		
RTI	Day	376	376	677	677		
Concentration	ppm	(7.94)	(14.5)	(361)	(542)		
	Day	1818	1818	713	713		
	p pm	(6.88)	(13.7)	(387)	(571)		
	Day	2162**	2162**	1818	1818		
	ppm	(6.88)	(13.3)	(349)	(557)		
	Day			2162**	2162**		
	ppm			(353)	(564)		

^{*} Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 50 or 100 degrees Ceisius.

CALIBRATION: Reagent grade "Perchloroethylene" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Concentration uncertainty: \pm 7%.

23.0 VINYL CHLORIDE STABILITY STUDY

Cylinder No Cylinder Const		23A S	23B S	23C S	230 S	23E S	23F S	23G \$	23H S	23I S
Manufacturer Concentration	ppm	5.94	8.00	8.03	8.52	20.0	20.1	30.0	30.3	7.98
	Date	10/1/79	10/1/79	10/1/79	10/1/79	10/1/79	10/1/79	10/1/79	10/1/79	10/1/79
	ppm	(5,87)	(7.71)	(7.82)	(7.85)	(19.7)	(20.1)	(29.6)	(29.8)	(7.31)
	Day	18	18	18	18	18	18	18	18	18
RTI Concentration	ÞÞW	(5,74)	(7.50)	(7.45)	(7.61)	(19.1)	(19.3)	(28.3)	(28.7)	(7.12)
	Day	700	**	700	700	700	700	700	700	700
	p pm	(6.60)		(8,44)	(8,41)	(20,7)	(20.9)	(29.4)	(29.4)	(8.39)
	Day	1812		1812	1812	1812	1812	1812	1812	1812
	ppm	(6.10)		(8.10)	(8, 15)	(20.3)	(20.6)	(30.3)	(30.6)	(7.75)

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 0.4% Carbowax 1500 on Carbopak C at 50 degrees Celsius.

CALIBRATION: Vinyl chloride permeation tube purchased from Metronics is used for calibration. Permeation tube is maintained at $30^{\circ}\text{C}_{\bullet}$

^{**}Cylinder empty.

24.0 1,3 BUTADIENE STABILITY STUDY

Cylinder No	3-	24A	
Cylinder Constr		S	
Manufacturer Concentration	ppm	22.6	
	Date	3/21/80	
	ppm	(20.9)	
	Date	95	
RT1 Concentration	ppm	(23.1)	
	Day	480	
	ppm	(24.0)	
	Day	1718	
	p pm	(22.9)	
			

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 0.1% SP-1000 on Carbopak C column at 90 degrees Celsius or 10% OV-101 on Chromosorb WHP column at 60 degrees Celsius.

CALIBRATION: Reagent grade "1,3 Butadiene" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

25.0 ACRYLONITRILE STABILITY STUDY

Cylinder No Cylinder Const		25A LS	25B LS	25C LS	250 LS	25E AL	25F AL
Manufacturer Concentration	p pm	20.1	348	11.7	638	400	10.0
	Date	7/24/79	7/24/79	7/24/79	7/24/79	11/8/82	11/18/82
	p pm	(14.6)	(411)	(6,38)	(678)	(413)	(10.8)
	Day	185	185	185	185	134	1 39
	ppm	(12.7)	(416)	(3,35)	(699)	(410)	(11.7)
RTI	Day	349	349	349	349	787	787
Concentration	p pm	(13.2)	(441)	(2.87)	(703)	(421)	(10.8)
	Day	841	841	84 1	84 1		
	p pm	(9,96)	(397)	(4.05)	(667)		
	Day	**	**	**	**		
	ppm						

^{*}AI = Aluminum; S = Steel; LS= Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 4% Carbowax 20M on Carbopak B at 50 or 150 degrees Celsius.

CALIBRATION: Acrylonitrile permeation tube or pressure-dilution technique is used for GC-FID calibration. Permeation tube is maintained at $30^{\circ} \pm 0.1^{\circ}\text{C}_{\bullet}$

ANALYTICAL PROBLEMS: The large changes noted at the low concentration levels are, at least in part, a result of difficulty in making precise measurements at these levels.

^{**}Cylinder empty

26.0 ANILINE STABILITY STUDY

Cylinder No. Cylinder Construction*	26A A1	26B
Manufacturer ppm Concentration	11.3	18.4
RTI Analysis	See Analytica	al Problems

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 250 degrees Celsius.

CALIBRATION: Reagent grade "Aniline" pure liquid is used as a standard. "Glass bulb" dilution technique is utilized for making series of standards for calibration.

ANALYTICAL PROBLEMS: Because aniline has an extremely high boiling point (186°C), special handling would be required to measure this compound. A completely heated system for sampling in the vapor phase and for preparing standards would be required. Temperature-dependent condensation in the cylinder and the regulator causes the amount of aniline which is delivered by the cylinder to vary. As a result, aniline is not considered to be practical as an audit material.

27.0 METHYL ISOBUTYL KETONE STABILITY STUDY

Cylinder No Cylinder Consti		27A Al	27C Al
Manufacturer Concentration	ррm	9, 51	72.9
	Date	12/18/80	7/8/81
	ррm	(10.2)	(75.4)
	Day	27	See Analytical
	bbw	(10.6)	Problems
	Day	83	
RTI Concentration	p pm	(9,53)	
	Day	202	
	ppm	(9.49)	
	Day	1275	
	p pm	(8.40)	
	Day	1643**	
	p pm	(10.3)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 0.1% SP-1000 on Carbopak C column at 180° degrees Celsius.

CALIBRATION: Reagent grade "Methyl isobutyl ketone" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

ANALYTICAL PROBLEMS: Methyl isobutyl ketone at high concentratrations is not practical as an audit material because pressurization of the cylinder above approximately 200 psi results in condensation of the analyte.

^{**}Concentration uncertainty: \pm 7%.

28.0 CYCLOHEXANONE STABILITY STUDY

Cylinder N Cylinder Const		28A A1	28B Al
lanufacturer Incentration	ppm	10.1	19.0
	Date	12/11/80	12/11/80
RTI	ppm	(8,19)	(25,5)
Analysis	Day	85	85
	ppm	(3,26)	(17.1)
		See Analytical	Problems.

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 10% SP-1000 on Supelcoport column at 200 degrees Celsius.

CALIBRATION: Reagent grade "Cyclohexanone" liquid is used as a standard. Pressure-dilution technique is used for making series of standards for calibration.

ANALYTICAL PROBLEMS: The analysis of cyclohexanone gas is dependent on the temperatures of the cylinder and the regulator and on the length of the sampling line between the regulator and the gas chromatograph. The concentration in the cylinder decreases with time. Therefore, cyclohexanone is not practical as an audit material.

29.0 PARADICHLOROBENZENE STABILITY STUDY

Cylinder No. Cylinder Construction*	29A S	29B \$
Manufacturer ppm Concentration	15.6	38.1
RTI Analysis	See Analyti	cal Problems

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 10\$ SP-1000 on Supelcoport column at 200 degrees Celsius.

CALIBRATION: Reagent grade "Paradichlorobenzene" is used as a standard. "Glass bulb" technique is used for making series of standards for calibration.

ANALYTICAL PROBLEMS: The stability study for this compound was terminated bacause of analytical difficulties and because the cylinder pressure was less than 200 psig. Paradichlorobenzene is a solid at room temperature with a melting point of 54°C. Condensation in the cylinder, regulator and sampling lines was extreme. Paradichlorobenzene is not practical as an audit material.

30.0 ETHYLAMINE STABILITY STUDY

Cylinder No. Cylinder Construction*	30A S	30B S			
Manufacturer ppm Concentration	10	20			
RTI Analysis	See Analyt	ical Problems			

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 10% OV-101 on Chromosorb WHP column at 250 degrees Celsius.

CALIBRATION: Reagent grade "Ethylamine" liquid is used as a standard. "Glass bulb" technique is utilized for making series of standards for calibration.

ANALYTICAL PROBLEMS: Because of vapor pressure considerations, the cylinders could not be fully pressurized. The pressure in the cylinder is less than 200 psi. A completely heated system for sampling in the vapor phase and for preparing standards would be required. Temperature-dependent condensation in the cylinder and the regulator causes the amount of ethylamine which is delivered by the cylinder to vary. As a result of these problems, ethylamine is not considered to be practical as an audit material.

31.0 FORMALDEHYDE STABILITY STUDY

RTI				
Requested	ppm	10	20	
Concentration				

The speciality gas supplier indicated that they could not make gas mixtures containing formaldehyde.

32.0 METHYLENE CHLORIDE STABILITY STUDY

Cylinder No. Cylinder Construction*		32A A1	32B Al	3 <i>2</i> C AI
Manufacturer Concentration	ррт	10.2	1.0**	5 . 0**
	Date	3/5/82		
	ppm	(10.8)		
	Day	31		
	ppm	(10.8)		
	Day	70		
	ppm	(10.6)		
	Day	96		
	ppm	(11.2)		
	Day	124		
RTI Concentration	ррm	(11.4)		
ooncentration	Day	160		
	ppm	(10.9)		
	Day	278		
	ppm	(10.2)		
	Day	381		
	ppm	(9, 70)		
	Day	843		
	ррm	(9.20)***		
	Day	1198***		
	ppm	(11.5)		

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 20 ft. \times 1/8" SS column packed with 10% SP-1000 on 80/100 Supelcoport. 30 cm³/minute He carrier gas. Column temp. = 100°C. Detector temp. = 175°C.

CALIBRATION: Reagent grade "Methylene chloride" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Nominal concentrations ordered from the manufacturer.

^{***}Questionable value.

^{****}Concentration uncertainty: \pm 7%.

33.0 CARBON TETRACHLORIDE STABILITY STUDY

Cylinder No. Cylinder Constru		33A AL	
Manufacturer Concentration	∕ ppm	11.3	
	Date	3/4/82	
	ррm	(12.7)	
	Day	74	
	ppm	(11.7)	
	Day	74	
	ppm	(10.2)	
	Day	98	
	ppm	(11.1)	
RTI	Day	124	
Concentration	ppm	(10.6)	
	Day	161	
	p pm	(10.2)	
	Day	382	
	p pm	(10, 5)	
	Day	832	
	p pm	(9 _• 60)**	
	Day	1199***	
	ppm	(12,2)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 20 ft. \times 1/8" SS column packed with 10% SP-1000 on 80/100 Supel-coport. 30 cm³/minute He carrier gas. Column temp. = 100°C. Detector temp. = 175°C.

CALIBRATION: Reagent grade "Carbon tetrachloride" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Questionable value.

^{***}Concentration uncertainty: \pm 7%.

34.0 FREON 113 STABILITY STUDY

Cylinder No. Cylinder Constru	ction*	34A A1	
Manufacturer Concentration	p pm	10.4	· · · · · · · · · · · · · · · · · · ·
	Date	3/3/82	
	bbw	(10.8)	
	Day	34	
•	ppm	(10.1)	
	_		
	Day	70	
	bbw	(10.0)	
	Day	70	
	ppm	(9,60)	
	Day	98	
RTI	ppm	(10.0)	
Concentration	pp	(1000)	
	Day	125	
	p pm	(10.0)	
	Day	162	
	ppm	(10.3)	
	Day	704	
	Day ppm	384 (9 . 80)	
	PP'''		
	Day	857	
	ppm	(11.0)	
	Day	1200**	
	ppm	(8.79)	

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 20 ft. \times 1/8" SS column packed with 10% SP-1000 on 80/100 Supelcoport. 30 cm³/minute He carrier gas. Column temp. = 100°C. Detector temp. = 175°C.

CALIBRATION: Reagent grade "Freon 113" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Concentration uncertainty: \pm 7%.

35.0 METHYL CHLOROFORM STABILITY STUDY

Cylinder No. Cylinder Construction*		35A . Al	
Manufacturer Concentration	ppm	10.2	
	Date	3/2/82	
	b bw	(10.3)	
	Day	70	
	ppm .	(11.8)	
	Day	99	
	ppm	(10.7)	
RTI	Day	136	
Concentration	ppm	(10.6)	
	Day	161	
	p pm	(10.0)	
	Day	381	
	p pm	(10.4)	
•	Day	858	
	p pm	(10.0)	

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 20 ft. \times 1/8" SS column packed with 10% SP-1000 on 80/100 Supel-coport. 30 cm³/minute He carrier gas. Column temp. = 100°C. Detector temp. = 175°C.

CALIBRATION: Reagent grade "Methyl chloroform" is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

36.0 ETHYLENE OXIDE STABILITY STUDY

Cylinder No. Cylinder Constru		36A A1	368 Al	36C A1	36D Al	36E Al
Manufacturer Concentration	ррт	10.0	1.0**	5.0**	15.0**	20.0**
	Date	3/12/82				
	ppm	(11.2)				
	Day	73				
	p pm	(9,60)				
	Day	88				
RT! Concentration	ppm	(9.80)				
	Day	122				
	ррm	(9,60)				
	Day	157				
	ppm	(9,80)				
	Day	1012				
	ppm	(9,70)				

^{*}AI = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 6 ft. x 1/8" SS column packed with 80/100 mesh Porapak QS. 30 cm 3 /minute Helium carrier gas. Column temp. = 150°C. Detector temp. = 175°C.

CALIBRATION: Ethylene oxide permeation tube purchased from Metronics is used for GC-FID calibration.
Permeation tube is maintained at 30°C.

ANALYTICAL PROBLEMS: There appeared to be some loss of ethylene oxide when a brass regulator was used on the cylinder.

 $^{^{**}}$ Nominal concentrations ordered from manufacturer.

37.0 PROPYLENE OXIDE STABILITY STUDY

Cylinder No.		37A	37B
Cylinder Constru	ction*	Al	Al
Manufacturer Concentration	ppm	9.48	96.0
	Day	8/4/82	8/4/82
	ppm	(12.3)	(89.5)
	Day	55	55
	ppm	(11.8)	(86.9)
	Day	76	76
RTI Concentration	ppm	(10.6)	(83.6)
	Day	743	121
	ppm	(8.10)**	(90,8)
	Day	844	743
	p pm	(9.24)	(75.7)*
	Day	1057	844
	ppm	(9.65)	(82.8)
	Day		1057
	ppm		(91.7)

^{*} Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 6 ft. x $1/8^{\rm m}$ SS column packed with 80/100 mesh Porapak QS. 30 cm³/min Helium carrier gas. Column temp. = $150^{\rm o}$ C. Detector temp. = $175^{\rm o}$ C.

CALIBRATION: Reagent grade "propylene oxide" is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

^{**}Questionable value.

38.0 ALLYL CHLORIDE STABILITY STUDY

Cylinder No.		38A	388	38C	38D
Cylinder Constr	uction*	\$ 	\$ 	S	\$
Manu facturer Concentration	ppm	10•2	99•5	8.7	92.4
	Date ppm	8/13/82** (11.6)	8/13/82** (124)	4/24/85**** (8 _e 99)	4/30/85**** (95 , 7)
	Day ppm	75 (5 . 25)	74 (87 . 2)		
RTI	Day ppm	110 (5.08)	110 (87•7)		
Concentration	Day ppm	167 (5.36)	167 (83 . 4)		
	Day ppm	727 (4.53)	727 (53.6)		
	,	***	***		

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 20 ft. x 1/8" SS column packed with 10 \$ SP-1000 on Supelcoport. 30 cm³/minute Helium carrier gas. Column temp. = 100°C. Detector temp. = 175°C,

CALIBRATION: Reagent grade "Allyl chloride" is used as a standard. Pressuredilution technique is utilized for making series of standards for calibration.

 $^{^{**}}$ Initial analysis was questionable

^{***} Returned due to impurities.

^{*****}Concentration uncertainty: \pm 7%.

39.0 ACROLEIN STABILITY STUDY

Cylinder No.		39A	39B	
Cylinder Constru	ction*	Al	Al	
Manu facturer Concentration	ppm	10.2	107	
	Date	8/18/82	8/18/82	
	ppm	(10.6)	(90.4)	
	Day	28	28	
	ppm	(11.0)	(103)	
	Day	69	69	
RTI	ppm	(9.74)	(106)	
Concentration	Day	AI 10.2 te 8/18/82 (10.6) y 28 (11.0) y 69 (11.0) y 728 (6.90)** y 833 (8.97) y 1031***	728	
	ppm		(80,8)**	
	Day	833	833	
	ppm	(8,97)	(97.3)	
	Day	1031***	1031***	
	ppm	(9.11)	(98.4)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detector, 20 ft. x 1/8" SS column packed with 10 % SP-1000 on 80/100 Supericoport. 30 cm³/min Helium carrier gas. Column temp = 100°C. Detector temp. = 175°C.

CALIBRATION: Reagent grade "acrolein" is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.

 $^{^{**}}$ Questionable value.

^{***}Concentration uncertainty: \pm 7%.

40.0 CHLOROBENZENE STABILITY STUDY

Cylinder No. Cylinder Construction*		40A S	40B A1	40C Al
Manu facturer Concentration	ppm	9, 66	14.84	4.89
	Da te	8/6/82	10/11/83	10/11/83
	ррm	(9,03)	(14.7)	(4.19)
	Day	39	612**	612**
	ppm	(9.15)	(13.4)	(4.74)
RTI	Day	75		
Concentration	ppm	(9.20)		
	Day	380		
	ppm	(9,62)		
	Day	1043**		
	ppm	(8.11)		

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame ionization detection, 20' X 1/8" stainless steel column packed with 10% SP-1000 on 80/100 mesh Supelcoport. 30 cc/min Helium carrier gas. Column temp. = 150°C. Detector temp = 175°C.

CALIBRATION: Reagent grade chlorobenzene was used as a standard. Pressure-dilution technique is utilized for making a series of standards.

^{**}Concentration uncertainty: \pm 7%.

41.0 CARBON DISULFIDE STABILITY STUDY

Cylinder No. Cylinder Construction*		41A Al	41B Al
	······································		· · · · · · · · · · · · · · · · · · ·
Manufacturer Concentration	ppm	108	108
	Date	7/14/82	2/21/85
	ppm	(100)	(101)
	Day	34	110
RT1 Concentration	ppm	(114)	(98•0)
	Day	72	
	ppm	(116)	
		**	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel.

ANALYTICAL CONDITIONS: Flame photometric detector, 4.6° X $1/4^{\circ}$ Teflon® column packed with Carbopak BHT 100. 90 cc/min Helium carrier gas. Column temp. = 75° C. Detector temp. = 175° C.

CALIBRATION: Reagent grade carbon disulfide is injected into a Teflon® bag being filled with N $_2$ at 5 L/min. through a mass flow controller. The injection fitting is heated slightly to ensure volatilization.

ANALYTICAL PROBLEMS: There is significant peak "tailing" unless a very high flow rate is used. "Tailing" is also caused by "bleed" from the sample loop. Sample valve should be in the inject position for exactly 5 seconds and then switched back to the sampling position to attenuate tailing. All sample lines and regulators must be conditioned extensively.

^{**}Cylinder empty.

42.0 EPA METHOD 25 GAS MIXTURE STABILITY STUDY*

Cylinder No.		42A	42B	42C	420	42E	42F
Cylinder Constru		A1	A1	Al	Al	Al	A1
Manufacturer Concentration	рртС	100	100	200	750	1000	2000
	Date	3/16/83	3/16/83	3/16/83	3/16/83	3/16/83	3/16/83
	ppmC	(102)	(107)	(205)	(775)	(1040)	(1940)
RTI	Day	483	483	**	483	483	483
Concentration	ppmC	(97 . 9)	(104)		(779)	(1060)	(1930)
Concentr at ton	Day ppmC	**	**		726**** (765)	726**** (1020)	726**** (1930)

 $^{^{*}}$ Gas Mixture contains an aliphatic hydrocarbon, an aromatic hydrocarbon, and carbon dioxide in nitrogen.

ANALYTICAL CONDITIONS: Flame ionization detector Durapak n-octane on Poracil C column at 30 °C for separation of aliphatic hydrocarbon and 10 % OV-101 on chromosorb WHP column at 60 °C for separation of aromatic hydrocarbon.

CALIBRATION: NBS-SRM was used as a standard for aliphatic hydrocarbon and Reagent grade liquid is used as a standard for aromatic hydrocarbon. Pressure-dilution technique is utilized for generation of series of standards for calibration.

^{**}Cylinder empty.

^{***}Al = Aluminum; S = Steel; LS = Low Pressure Steel

^{****}Concentration uncertainty: + 7%.

42.0 EPA METHOD 25 GAS MIXTURE STABILITY STUDY* (Continued)

Cylinder No. Cylinder Constru		42G A1	42H A1	42! Al	42J Al	42K A1	42L Al
Manufacturer Concentration	p pmC	96, 7	98.6	147.6	151	198	197.5
	Date	12/11/84	12/11/84	12/11/84	12/11/84	12/11/84	12/11/84
RTI Concentration	ppmC	(96.4)	(98.9)	(149)	(153)	(195)	(195)
	Day	90***	90***	90***	90***	192	90***
	ppmC	(95.8)	(93,3)	(144)	(145)	(183)*	*** (187)

 $^{^{*}\}mathsf{Gas}$ Mixture contains an aliphatic hydrocarbon, an aromatic hydrocarbon, and carbon dioxide in nitrogen.

ANALYTICAL CONDITIONS: Flame ionization detector Durapak n-octane on Poracil C column at 30°C for separation of aliphatic hydrocarbon and 10% OV-101 on chromosorb WHP column at 60°C for separation of aromatic hydrocarbon.

CALIBRATION: NBS-SRM was used as a standard for aliphatic hydrocarbon and Reagent grade liquid is used as a standard for aromatic hydrocarbon. Pressure-dilution technique is utilized for generation of series of standards for calibration.

^{**} Al = Aluminum; S = Steel; LS = Low Pressure Steel

^{***}Concentration uncertainty: \pm 7%.

^{****}Questionable value.

43.0 ETHYLENE DIBROMIDE STABILITY STUDY

Cylinder No. Cylinder Construction*		43A S	43B S	43C S	430 S
Manu facturer Concentration	ppm	10	20	100	300
RTI Concentration	Da te	10/24/84	10/24/84 (17•5)	10/24/85 (96 . 1)	10/24/84
	Day ppm	54 (9.3)	54 (17•5)	55 (107)	55 (344)**
	Day ppm	243 (8,66)	243 (15•4)	243 (84 _• 0)	

^{*}Al = Aluminum; S = Steel; LS = Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 5% OV-101 on Chromosorb WHP at $60\,^{\circ}\text{C}_{\bullet}$

CALIBRATION: Reagent grade "ethylene dibromide" liquid is used as a standard. Pressure—dilution technique is utilized for making series of standards for calibration.

^{**}Questionable value.

44.0 1,1,2,2-TETRACHLOROETHANE STABILITY STUDY

Cylinder No. Cylinder Constru	44A Al		
Manu facturer Concentration	ppm	12.2	
RTI Concentration	Date ppm	10/9/84 (11.6)	

^{*} Al = Aluminum; S = Steel; LS = Low Pressure Steel

ANALYTICAL CONDITIONS: Flame ionization detector, 5% OV-101 on Chromosorb WHP at 100°C.

CALIBRATION: Reagent grade "1,1,2,2-Tetra-chloroethane" liquid is used as a standard. Pressure-dilution technique is utilized for making series of standards for calibration.