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QUALITY ASSURANCE PROJECT PLAN FOR THE NATIONAL PESTICIDE SURVEY OF DRINKING WATER WELLS

Prepared for:

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APPROVAL PAGE	
,	
Director, NPS	Date
QA Manager-OPP	Date
QA Manager-ODW	Date

NATIONAL PESTICIDE SURVEY QUALITY ASSURANCE PROJECT PLAN

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3. INTRODUCTION

This document was revised at the conclusion of the National Pesticide Survey (NPS) in order to have an accurate record of all the quality assurance/quality control features that were a part of the Survey quality effort. Although a draft QA program plan was available at the beginning of the Survey, a number of issues were raised during the course of the Survey that have been incorporated only into this, the final version of the program plan. The evolutionary nature of certain aspects of the NPS quality program will be evident to the reader since the document relies very heavily on original memorandum and unpublished narratives to document the requirements and execution of the QA program. The use of these memos and narratives resulted in the plan being organized into two rather distinct sections, the narrative, dealing with broad aspects of the QA program, and the appendices, which contain many of the more detailed QC requirements, particularly with respect to analytical methods requirements. If the reader wishes even more detailed QA information about a specific aspect of the Survey, they are referred to the individual project plans which will be available through the National Technical Information Service (NTIS), Springfield, Virginia.

The reader is also advised that the Environmental Protection Agency reorganized the Office of Water during the Spring of 1991. As a result, the Office of Drinking Water (ODW), a major sponsor of the NPS, no longer exists but has been incorporated into the Office of Ground Water and Drinking Water (OGWDW).

4. PROJECT DESCRIPTION

In 1981, the United States Environmental Protection Agency (USEPA) issued a policy requiring all environmental measurement data be collected under the auspices of a centrally managed quality assurance (QA) program. In response to this policy, EPA formed the Quality Assurance Management Staff (QAMS) and tasked them with developing QA guidance for all Agency environmental data collection efforts. Guidance developed by QAMS suggests that Quality Assurance Program Plans (QAPPs) be written to describe "the overall policies, organization, objectives, and functional responsibilities designed to achieve data quality goals...". The National Pesticide Survey (NPS), as a collector of environmental data, has developed the following QAPP to address these and other quality issues specific to the Survey.

4.1 Introduction

The National Pesticide Survey (NPS) is a jointly sponsored effort of the USEPA Offices of Drinking Water (ODW) and Pesticide Programs (OPP). The Survey has two primary objectives:

- 1. To determine the frequency and concentration of pesticide contamination in the Nation's drinking water supplies obtained from groundwater sources.
- 2. To examine the relationships of pesticide contamination to patterns of pesticide use and groundwater vulnerability.

To meet these objectives, substantial resources have already been committed to planning the Survey. In particular, a pilot study was conducted of sixteen wells during 1987 to field test essential components of the Survey design, logistics, and QA/QC procedures. Based on results of the pilot, a number of modifications were recommended by Mason, et al, 1988. Because the pilot was covered by a separate QA project plan (Kulkarni, et al, 1987), only activities conducted in support of the full Survey, from 1988 to 1991, will be covered under the QA program plan described in this document.

4.2 Purpose of Program Plan

The NPS will be conducted with assistance from several organizational groups within EPA and over 10 contract organizations, all operating from locations dispersed across the country. Given this level of complexity, the purpose of the QA program plan (QAPP) will be to communicate minimum standards for assuring quality to the primary organizations responsible for conducting the Survey. Each primary organization, whether EPA or contract, will be required to write a quality assurance project plan (QAPjP) describing to NPS management the procedures by which their organization plans to meet the requirements of the program plan. It will be left to the discretion of each organization, based on their own internal operations, to describe the exact procedures which will be used to meet Survey quality standards. Through the project plan review process, NPS management will examine the plans for completeness and conformity to the program plan, and if necessary, provide assistance in understanding the standards and implementing procedures to achieve them.

4.3 Data Quality Objectives (DQOs)

To ensure the usefulness of NPS data, management led an intensive planning effort prior to starting the full Survey, with the result that precision requirements for each of the Survey's domains of interest have been clearly defined and will be used to design and implement the Survey. To generate the requirements, Survey management, in discussions with their technical staff, considered the resources necessary for achieving different levels of confidence in the national estimates for pesticide occurrence. Separate sets of objectives were developed for the rural domestic wells and the community water systems and are presented in Exhibit 4-1. Following the NPS planning effort, QAMS institutionalized the planning process coincidently used by NPS as the Data Quality Objective (DQO) process. A report describing the Survey's planning process as it relates to the DQO process was then written (Nees, 1988).

Other measures of data quality which the Survey addressed were representitiveness and completeness. For representitiveness, the goal of the Survey will be to select and sample relatively few wells, which can then be used to characterize the status of pesticide occurrence in drinking water wells across the nation. This goal will be addressed through the Survey's statistical design, which will promote the selection of wells from all types of geohydrologic and pesticide use areas using stratification. For completeness, a minimum number of wells must be successfully sampled and analyzed in order to meet the precision requirements as stated for each of the domains of interest. This goal will be addressed in two ways; first, additional wells will be selected for sampling to account for anticipated losses of data in the range of 5-10% and second, data losses will be tracked as the Survey progresses, in order to select additional wells if losses exceed the estimated 5-10%.

4.4 Measurement Quality Objectives (MQOs)

MQOs for the full Survey have been established for detection limits, accuracy, and precision. Each laboratory will be required to demonstrate a limit of detection within a factor of two of that achieved during methods development, as reported in the method description. Detection limits that exceed this will be evaluated individually taking into consideration any known health effects levels.

Other MQOs established for the Survey include accuracy and precision. Accuracy will be evaluated using quarterly performance evaluation samples while precision will be evaluated using standard control chart plots of laboratory control samples. In addition to these overall MQOs, each analytical method has a number of quality control criteria that must be addressed in individual QAPjPs. All QC criteria are discussed in detail in Section 6.4 and Appendix A.

4.5 Key Features

The NPS QA program can only be understood within the context of the Survey's design, implementation, and analysis plans, therefore the key features of the Survey and their relationship to each other, as shown in Exhibit 4-2 will be described in this section.

Exhibit 4-1

NPS Data Quality Objectives

Rural, Domestic Wells

Domain Description	Domain Size, %	Probability of Detection, %
Wells nationally	1.0	63
Wells in counties with highest average pesticide use	0.14	75
Wells in counties with highest average ground-water vulnerability	0.25	75
Wells in cropped and culnerable parts of counties	0.25	97
Wells in counties with highest average pesticide use and ground- water vulnerability	0.3	47

Community Water Systems

Domain Description	Domain Size, %	Probability of Detection, %
Systems nationally	0.5	90
Systems in counties having the highest average ground-water vulnerbility	0.1	60

Exhibit 4-2

Survey Key Features

Step 1	Stratify
	UsageVulnerability
Step 2	Select Sites
	Rural, Domestic WellsCommunity Water Systems
Step 3	
Task A	Sample
	ICFState Personnel
Task B	Interview
	WestatState Personnel
Step 4	Analyze
	- Contract Labs (5) - Referee Labs (2)
Step 5	Report
	- EPA - Public

Statistical Design - By 1987, (and therefore not subject to this QA program plan), Research Triangle Institute (Research Triangle Park, North Carolina) had placed all counties in the US into one of 12 strata based on pesticide usage data (obtained from 1982 Census of Agriculture and Doanes Marketing Research, Inc.) and groundwater vulnerability (as defined by a modified DRASTIC method (Alexander et al., 1985). For the current effort, essentially two surveys will be conducted: one for community water systems (CWS) and one for rural domestic wells.

For the CWS, a complete listing of all CWSs can be found in the Federal Reporting Data System (FRDS). Using this listing, CWSs will be randomly selected from within county level strata. With the assistance of the well system operator, all system wells will be listed at the time of sampling and a specific well will be selected using a random selection process.

For rural domestic wells, the design calls for a second stage stratification, meaning counties that are selected for sampling will be further stratified into two sub-county strata: cropped/vulnerable and non-cropped/non-vulnerable. Cropping data will be obtained from field interviews of county agents about local pesticide use and cropping practices; vulnerability assessments will be based on DRASTIC scores developed using local hydrogeologic data, whenever possible. Strata will then be identified from maps on which information about cropping and vulnerability has been combined and specific wells will be randomly selected from within strata using telephone survey techniques. ICF, Incorporated, as the prime contractor for conducting all non-analytical phases of the Survey, and Westat, one of ICF's principal subcontractors, will be responsible for implementing the design begun by RTI.

<u>Training</u> - Implementation of the Survey design will require a significant commitment to training because field activities will be conducted by a large group of individuals from a number of diverse organizations including the States, EPA regional offices, ICF, and Westat. Therefore, to achieve consistency in field operations, ICF and Westat will share responsibility for developing and conducting a hands-on training course on NPS field protocols.

<u>Sampling</u> - The CWS samples will be collected by State sampling crews who will also administer questionnaires to the well operator and a local expert on pesticide usage. The rural, domestic well samples will be collected by ICF staff and questionnaires will be administered by professional interviewers employed by Westat. All sampling logistics including preparation of the sampling schedule, sample bottles, sample kits, and other supplies will be the responsibility of ICF.

<u>Chemical Analyses</u> - Samples will be analyzed using eight different analytical methods, several of which were developed specifically for NPS use. Exhibit 4-3 is a listing of the analytes associated with each of the methods. As discussed in Section 6, five contract laboratories and three EPA referee laboratories will perform the analyses.

Questionnaire Processing - All questionnaires completed at the time wells are sampled will be returned to Westat, where they will be coded and entered into a database.

Exhibit 4-3 Survey Analytes and Analytic Methods

NPS Method 1: Gas Chromatog (46 Analytes)	raphy with a Nitrogen-Pho	sphorus Detector	
Alachlor Ametryn Atraton Atrazine Bromacil Butachlor Butylate Carboxin Chlorpropham Cycloate Diazinon* Dichlorvos	Diphenamid Disulfoton* Difulfoton sulfone* Disulfoton sulfoxide* EPTC Ethoprop Fenamiphos Fenarimol Fluridone Hexazinone MGK 264 Merphos*	Methyl paraoxon Metolachlor Metribuzin Mevinphos Molinate Napropamide Norflurazon Pebulate Prometon Pronamide* Propazine	Simazine Simetryn Stirofos Tebuthiuron Terbacil Terbufos* Terbutryn Triademefon Tricyclazole Vernolate
NPS Method 2: Gas Chromatog (29 Analytes)	raphy with an Electron Ca	oture Detector	
4,4-DDD 4,4-DDE 4,4-DDT Aldrin Chlorobenzilate* Chloroneb Chlorothalonil DCPA	Dieldrin Endosulfan I Endosulfan II Endosulfan sulftate Endrin Endrin aldehyde Etridiazole Heptachlor	Heptachlor epoxide Hexachlorobenzene Methoxychlor Propachlor Trifluralin a-HCH b-HCH d-HCH*	g-HCH a-Chlordane g-Chlordane c-Permethrin t-Permethrin
NPS Method 3: Gas Chromatography with an Electron Capture Detector (17 Analytes)			
2,4-D 2,4-DB 2,4,5-TP 2,4,5-T 3,5-Dichlorobenzoic acid	4-Nitrophenol* Acifluorfen* Bentazon Chloramben* DCPA acid metabolites	Dalapon* Dicamba Dicamba, 5-hydroxy- Dichlorprop Dinoseb	PCP Picloram
NPS Method 4: High Performan (18 Analytes)	ce Liquid Chromatography	with an Ultraviolet Dete	ctor
Atrazine, ¹ Barban Carbofuran, phenol-3-keto Carofuran, phenol Cyanazine	Diuron Fenamiphos sulfone Fenamiphos sulfoxide Fluometuron Linuron	Metribuzin, DA Metribuzin, DADK* Metribuzin, DK* Neburon Pronamide metabolite	Propanil Propham Swep 1 deethylated

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Oxamyl

Exhibit 4-3 (continued)

NPS Method 5: Direct Aqueous Injection HPLC with Post-Column Derivatization (10 Analytes)

Aldicarb Aldicarb sulfone Aldicarb sulfoxide Baygon Carbaryl Carbofuran Carbofuran, 3hydroxy

Methiocarb Methomyl

NPS Method 6: Gas Chromatograhy with a Nitrogen-Phosphorous Detector (1 Analyte)

Ethylene thirourea (ETU)

NPS Method 7: Microextraction and Gas Chromatography

Ethylene dibromide (EDB) Dibromochloropropane (DBCP) c-1,3-dichloropropene** t-1,3-dichloropropene**

1,2-dichloropropane**

NPS Method 9: Automated Cadmium Reduction and Colorimetric Detector

Nitrate and nitrite measured as nitrogen (N)

* Qualitative only.

** Analytes previously detected by Method 8, which was dropped.

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<u>Data Synthesis</u> - Given the complexity of the NPS, data synthesis will occur in several steps. Initially, all data will be entered into databases at the location of the individual responsible for technical oversight of a specific area of the Survey, as follows:

Analytical data EPA-Cincinnati (Dave Munch, Chris Frebis)

Questionnaire data Westat (David Marker)
Field measurement data ICF (Cindy Jengleski)
Drastic data ICF (Bruce Rappaport)

The next step will be to forward the databases to ICF, who will be responsible, along with Westat for developing strategies to impute values for any missing data points. The last step will be to present the data to the public both in interpretive reports prepared by ICF under the direction of the NPS director and OPP and ODW management and in a public use database available on magnetic media.

4.6 Summary

Well water samples and related information will be collected from approximately 750 rural domestic wells and 600 community wells. These wells will be used to represent the approximately 10 million rural domestic wells and 96,000 community wells in the nation which depend on groundwater for drinking water purposes. Analysis of the well water samples will be for 126 pesticides and associated products. The presence and concentrations of nitrites/nitrates will also be determined. An alphabetical listing of all Survey analytes is provided in Exhibit 4-4. In addition to collecting well water samples, the Survey will administer questionnaires about pesticide usage, spillage, cropping patterns, well construction information, etc. for use in interpreting the results of the chemical analyses.

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Exhibit 4-4

NPS Analytes

Aciflurofen Alachlor Aldicarb Aldicarb sulfone Aldicarb sulfoxide

Aldrin Ametryn Atraton Atrazine

Atrazine, deethylated

Barban Baygon Bentazon Bromacil Butachlor **Butylate** Carbaryl Carbofuran

Carbofuran-3-hydroxy Carbofuran phenol Cabofuran pheno-3-keto

Carboxin Chloramben a-Chlordane g-Chlordane Chloroneb Chlorobenzilate Chlorothalonil Chlorpropham Cyanazine Cylcoate Dalapon **DCPA**

DCPA diacid* Diazinon

Dibromochloropropane

Dicamba Dichlorprop **Dichlorvos** Dieldrin Dinoseb Diphenamid

Disulfoton

Disulfoton sulfone Disulfoton sulfoxide Diuron

Endosulfan I Endosulfna II Endosulfan sulfate Endrin

Endrin aldehyde **EPTC**

Ethoprop Ethylene dibromide Ethylene thiourea Etridiazole

Fenamiphos Fenamiphos sulfone Fenamiphos sulfoxide

Fenarimol Fluometuron Fluridone a-HCH b-HCH d-HCH g-HCH Heptachlor

Heptachlor epoxide Heptachlorobenzene

Hexazinon Linuron Merphos Methiocarb Methomyl Methoxychlor Methyl paraoxon Metolachior

Metribuzin Metribuzin DA Metribuzin DADK

Metribuzin DK Mevinphos

MGK-264 Molinate Napropamide

Neburon Nitrate/nitrite

Norflurazon Oxamyl PCP Pebulate c-Permethrin t-Permethrin **Picloram**

Prometon Prometryn Pronamide Pronamide* Propachlor Propanil Propazine Propham Simazine Simetryn **Stirofos**

Swep Tebuthiuron Terbacil **Terbufos** Terbutryn Triademeton Tricyclazole Trifluralin Vernolate 1,2-DCP c-1,3-DCP t-1,3-DCP 2,4-DB 2.4-D 2,4,5-TP

3.5-Dichlorobenzoic

acid

2,4,5-T

4-Nitrophenol 4,4-DDD 4,4-DDE 4.4-DDT

5-Hydroxy Dicamba

Metabolite

5. QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

NPS management made a strong commitment to quality during the planning of the Survey, a commitment which will continue throughout the data collection and interpretation phases of the Survey. This chapter describes the formal QA organization which will be created to support management's commitment to quality and the responsibilities which will be delegated to individuals within the QA structure.

5.1 QA Policy Statement

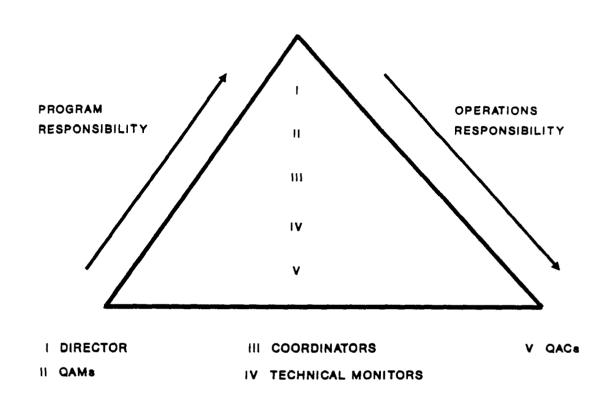
The policy of the National Pesticide Survey will be to participate in the Agency-wide quality assurance program, with the goal of collecting data of known and documented quality. All applicable guidance developed by the Quality Assurance Management staff will be followed and any additional requirements of the Office of Drinking Water and Office of Pesticide Programs will be addressed. The QA program will be operated with the philosophy that responsibility for quality belongs to everyone. To institutionalize this philosophy will require an active QA program within each participating organization, with visible support provided by the Survey both through frequent informal communication between the laboratory project managers and the technical monitors and through biannual on-site technical system audits.

5.2 QA Management Structure

The NPS QA management structure will be a refinement of the QA organization that was used during the pilot study and will be modified to account for those changes recommended by QAMS during their management system review (MSR) of the pilot. Recommendations by QAMS which were incorporated into the QA program included the hiring of a full-time QA specialist, development of a QA Management Plan, development of QA Project Plans for field and lab activities, development of procedures for data review and analysis, and development of clear, concise DQOs.

Using Exhibit 5-1 to illustrate the structure of the QA organization, five distinct levels of responsibility encompassing 26 individuals are identifiable. At Level I, leadership for quality rests with the Director of the Survey, who has assigned certain responsibilities for quality to the NPS Quality Assurance Manager (QA Manager), at Level II. The QA Managers for ODW and OPP, who are also included in Level II, will guide and assist the NPS QA Manager. At Level III are three coordinators for the analytical and implementation activities, while level IV is composed of Technical Monitors who have responsibility for oversight of day-to-day data gathering operations. The Quality Assurance Coordinators, at Level V, are staff employed by each organization participating in the NPS. As depicted, responsibility for QA policy and leadership will be the greatest at Level I and responsibility for QA of day-to-day operations will be the greatest at Level V.

Exhibit 5-1 NPS QA Organization



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Roles and responsibility for QA have been assigned as described below.

NPS Director -The Director has overall responsibility for Survey quality including:

Allocating resources for QA.

Prioritizing tasks, including QA/QC activities.

Requesting adjustments to work processes to improve quality.

Serving as the liaison with all parties interested in the quality of the Survey, such as the States, public interest groups, and groups within EPA.

OPP/ODW QA Manager - The OPP and ODW program QA Manager will be responsible for:

Directing the QA/QC program.

Formulation of NPS QA policy.

Providing guidance to the NPS Director, NPS QA Manager, and others about program office QA requirements.

Oversight of the activities of the NPS QA Manager.

Resolving QA/QC issues requiring programmatic input.

Approving QA plans and amendments.

Auditing, as needed.

NPS QA Manager - The QA Manager will be responsible for:

Advising the director on quality issues.

Leading audits of the implementation contractor and analytical laboratories, (both contract and referee) to assure that facilities, equipment, personnel, methods, records and quality control are in compliance with the QA Project Plans.

Reporting audit findings in written reports to the Survey Director.

Developing QA guidance documents and protocols, as needed.

Consulting and advising the QA Managers for ODW and OPP on QA issues.

Facilitating resolution of problems identified by Technical Monitors or Analytical Coordinators.

Building consensus on compliance and corrective action issues.

Forwarding QA documentation to ICF for archival.

Reviewing planning documents, draft reports and communications materials.

Presenting information on the QA Program at professional meetings.

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Analytical/Implementation Coordinator - The coordinators will be responsible for:

Elevating issues of concern to the NPS Director.

Informing the Technical Monitors about the progress of the Survey and quality issues.

Approving payment for work that meets NPS quality criteria.

Coordinating the data review activities of the Technical Monitors.

Technical Monitors - The Technical Monitors will be responsible for:

Oversight of day-to-day analytical method performance at the laboratories.

Informing the analytical/implementation coordinators about problems.

Facilitating resolution of problems in the work process.

Final review of data for acceptability.

Participation in on-site technical system and data audits.

Resolving discrepancies identified in performance evaluation studies.

Forwarding monthly progress reports to the NPS QA Manager.

Quality Assurance Coordinator Role - The QACs will report independently of project management and will be responsible for:

Performing internal technical system and data audits.

Overseeing corrective action.

Although the QA organization in Exhibit 5-1 is presented as a hierarchal structure, no individual in any one level is responsible administratively for an individual in the next level. Rather, because of the importance and visibility of the Survey, management has consented to allow individuals to participate in the QA program according to their expertise and on an as-needed basis.

6. QUALITY ASSURANCE PROJECT PLANS

Operational details of the NPS QA program will be described in individual quality assurance project plans (QAPjPs). Project plans will be written for each major element of the Survey, using QAMS guidance 005/80 for the analytical elements of the Survey. For non-analytical elements of the Survey, each task associated with a given element will be viewed as part of a process. The overall process will then be described in a logical order along with the QA/QC used to monitor and adjust it.

6.1 Scope

The criteria for QAPjPs contained in this chapter will apply to each of the elements listed below and will be written by the organization indicated.

Contract Analyses:

Methods 1 and 3	Montgomery Labs, Pasadena, California
Method 2	Clean Harbors, Boston, Massachusetts
Method 4	Radian Corporation, Austin, Texas
Method 5	ES&E, Gainesville, Florida
Method 6	Battelle, Columbus, Ohio
Method 7	ES&E, Gainesville, Florida

Referee Analyses:

Method 1, 3, and 6	OPP-Environmental Chemistry Section, Bay St. Louis,
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Mississippi

Method 2, 4, 5, 7, and 9 ODW-Technical Support Division, Cincinnati, Ohio and

Office of Research and Development (ORD) Risk Reduction

Engineering Laboratory, Cincinnati, Ohio

Non-Analytical Task Areas:

Sampling ICF Incorporated, Fairfax, Virginia Second Stage Stratification ICF Incorporated, Fairfax, Virginia

Statistics Westat, Rockville, Maryland

6.2 Philosophical Approach

The QAPjP will be viewed as each organization's agreement to implement specific procedures for assuring the quality of their data. As long as NPS issues are addressed, the manner in which they are addressed will be left to the discretion of the individual organization. The project plan will serve as the reference for discussions between the contractors and the analytical coordinators and technical monitors. During on-site technical system audits, the QAPjP will serve as the standard against which the organization's procedures can be judged. At this time the organizations implementation of the project plan can be verified and possible areas for improvement can be identified.

6.3 Minimum Criteria

Minimum criteria for approval of the project plans are:

- The organization's commitment to QA must be expressed.
- An individual, unattached to the project, must be identified who can serve in a QA
 oversight capacity with clearly identified responsibilities which will include the audit
 function.
- A system for records management must be described.
- 4. A system for supervisory or peer review of data must be described.

6.4 Requirements for Laboratory Plans

In addition to describing general aspects of the laboratories' operations, the more specific information found in Appendix A "Information Packet for NPS Laboratories" must be incorporated into the laboratory QA project plans, using the format recommended by QAMS (005-80), briefly described below.

Section 1: Title and Approval Page

Labs should list the Technical Monitor, Analytical Coordinator, and Project Officer as the EPA personnel responsible for approving the plan.

Also, a distribution list should be developed to include everyone working on the NPS. The list should be initialed and dated to indicate that all personnel have received and read a copy of the plan. The list will be made available to the Technical Monitor.

Section 2: Table of Contents

A table of contents will be included with the plan.

Section 3: Project Description

A brief description of the Survey and the role of the lab in relation to it must be described.

Section 4: Project Organization and Responsibilities

All individuals and their responsibilities with respect to NPS analyses must be identified. At a minimum, the program manager, quality assurance coordinator, sample receipt clerk, sample preparation personnel, analysts, and data clerks must be identified.

Section 5: QA Objectives for Measurement Data

Include the method for determining estimated detection limits (EDL's), and method reporting limits (MRL's) based on the guidance in Appendix A. Note that results must be forwarded to the Technical Monitor for approval.

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Describe the procedure that will be used for constructing control charts, including the use of Dixon's outlier test as described in Appendix A.

Note that EPA will provide field samples for spiking to be used for a study of recoveries from different matrices and to study analyte stability using NPS preservation and analysis schemes. Spiking levels and holding times for these studies can be found in Appendix A.

Section 6: Sampling Procedure

The implementation contractor, ICF, has provided information in Appendix A that the laboratories require on sampling procedures, such as sample containers and preservation schemes, sample labels and IDs and sample tracking procedures. This information will be included by the lab in its' plan.

Section 7: Sample Custody

ICF has provided information that needs to be included in this section, such as the communications system for notifying the labs about the sampling schedule and the labs notifying ICF about sample receipt (see Appendix A). ICF will also supply the labs with the procedure for returning sample kits, which should be included in this chapter.

The laboratory will describe its' procedures for sample receipt, sample storage for NPS samples and extracts, holding times and the manner in which they are tracked, and how it monitors environmental conditions of sample and extract storage areas. Policies for disposal of samples and extracts should also be provided. All procedures must meet the criteria presented in Appendix A.

Section 8: Calibration Procedures and Frequency

This section should acknowledge that EPA will supply all calibration standards. Based on the requirements of the methods, the lab should then describe their calibration procedures, frequency, and QC checks. Associated procedures should also be described, such as for standards preparation and retention of chromatograms. A statement should be included that any deviations from the validated method must be discussed with the Technical Monitor.

Section 9: Analytical Procedures

A brief summary of the method should be described in this section, with the full method included in an appendix. Major pieces of instrumentation should be described. Batch sizes including all required QC samples and their run order should also be given.

Section 10: Data Reduction, Validation, Reporting

The laboratories system for data reduction must be described. If an automated system is used, its' use and algorithms must be described. Peer review/supervisory review of the data must be described here. Data reporting must conform to the standard that has been provided by the NPS in Appendix A. All batch data, including QC and confirmation data, must be reported within 60 days of sample collection.

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Based on guidance in Appendix A, a fast track reporting system must be described in this section for confirmed positives with a known health effects level and for situations where results from confirmation columns do not agree with results from primary columns within 25%.

A system for retaining records in a retrievable manner must also be described in this section.

Section 11: Internal Quality Control Checks

A summary of all QC checks for analyses of NPS samples must be provided including the frequency of use, acceptable criteria, and corrective action. Confirmation procedures for positives identified on the primary column should be included. All these procedures should be based on guidance found in Appendix A.

Section 12: Performance and Systems Audits

The laboratory will be expected to participate in NPS sponsored quarterly performance evaluation samples and biannual technical system audits. Participation in both should be acknowledged in this section. Also, audits conducted by the laboratory of its own activities should be described, including the content, frequency, and reporting of the audits.

Section 13: Preventive Maintenance

A schedule of routine maintenance and replacement of parts should be described.

Section 14: Specific Procedures for Assessing Measurement System Data

Calculations should be described for assessing the results of QC samples such as the instrument control standard, for which resolution, peak symmetry factor, and peak geometry factor must be calculated and such as the laboratory control standard, for which standard deviation and relative standard deviation must be calculated.

Section 15: Corrective Action

Describe the organizations' procedures for taking corrective action, clearly identifying those individuals responsible for ensuring that problems have been resolved.

Section 16: QA Reports to Management

Describe both the internal reports generated for management and the monthly reports generated for the Technical Monitor. Reference the format as found in Exhibit 9.1.

6.5 Requirements for Other Plans

QAMS guidance 005/80 was written with a focus on the laboratory analysis steps of environmental data generating processes. Because the NPS wants to cover all steps in the data generating process, QA plans will be written for nonlaboratory aspects of the Survey as well. To implement this goal will require a broad and creative interpretation of the current guidance on QAPjPs,

especially in regards to discipline areas such as statistics, hydrology, and mapping. For these plans, the QAPjP format will be modified to account for the different orientation of the plans with the requirement that the minimum criteria listed in Section 6.3 be addressed and that the first four sections will be as described in 005/80 (i.e. Title - Approval Page, Table of Contents, Project Description, and Project Organization and Responsibilities.) The format for the rest of the plan will be flexible and allow for separate chapters on significant components of the work and the accompanying QC. All standard operating procedures are to be included as appendices. Approval of the plans will be the responsibility of the appropriate Technical Monitor and the QA Managers for OPP and ODW and the NPS Director.

6.6 Amendments to Plans

Changes to the plans are anticipated and NPS management has developed a procedure for approving amendments to the QAPjPs, which can be found in Appendix A-6.

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7. PROCESS CONTROL

During the Survey, to achieve the levels of precision expressed in the NPS DQOs, all aspects of the data generation process must be operated in a state of control. This will be accomplished through monitoring of intermediate and final NPS data, using statistical process control whenever possible. Each unique work element in the Survey, and its associated QA/QC will be described in individual QA project plans, as discussed in Section 6. The project plan will represent an agreement for a given level of quality between the organization performing the work and Survey management. This chapter will briefly summarize the controls that are explained in more detail in the project plans.

7.1 NPS Pilot

Prior to collecting data for inclusion in NPS final summary reports, a pilot Survey was conducted to field test major elements of Survey design, logistics, and QA/QC. The pilot proved to be an extremely effective tool for improving Survey quality because it led to overall process evaluation and improvement. Following a review by the Federal Insecticide, Fungicide, and Rodenticide Act, Scientific Advisory Panel Subpanel (1987), changes were made in several areas including the well selection process, the questionnaires, sampling schedule, and analytical methods.

7.2 Well Selection

The guiding philosophy behind all steps in the well selection process will be to meet requirements necessary to allow for subsequent statistical analysis. Given this philosophy, the capability will be present to associate error bounds with estimates derived during the data interpretation phase of the Survey. At the present stage of development of the NPS, no further process control can be applied to first stage stratification, since these activities were completed prior to the pilot. All remaining activities performed in the sample selection process will differ between the rural domestic survey and the CWS survey.

Controls for rural domestic well selection will begin with second stage stratification activities, which first involve the collection of hydrogeologic and pesticide information, using interviews and other means, and then involve mapping the collected information as DRASTIC vulnerability and cropping categories. Interviewers will be trained in proper interviewing techniques and questionnaires will be processed using standard controls for data coding, data entry, and range and logic checking. To provide consistency during mapping of DRASTIC scores, personnel will first be certified using reference counties.

During the Random Digit Dialing (RDD) effort, which will be used to locate qualified wells and well owners willing to participate in the Survey, several controls will be used. To control the actual telephone interviews, interviewers will be trained and then silently monitored by supervisory personnel for the accuracy and appropriateness of their conversations. To track progress within each county, control charts will be used to monitor the number of calls required for meeting the target number of

wells in each county. Out-of-control conditions will be investigated. RDD constitutes the third and last stage of the well selection process for rural domestic wells.

Controls for CWS well selection will be focused in two areas. The first will be to verify the information in the second stage sampling frame, i.e. the FRDS list. Attempts will be made to contact CWS owners for a large number of the entries and check the accuracy of the information in the FRDS database, particularly for the types of errors identified during the pilot, i.e. systems that are listed individually plus being included with parent companies and for the correct number of wells in the system. The second major control used in the CWS selection process will be to have a well defined standard operating procedure for systems with more than one well which can be used on-site by the sampling team leader to randomly select an individual well for sampling.

7.3 Sampling Controls

The following process control features will be instituted to both correct and improve on systems used during the pilot and will apply to samples from both rural domestic wells and CWSs. Two distinct phases occur in the sampling process: those activities performed prior to sample collection and those activities performed during or after sample collection. Process controls prior to sampling will include:

A thorough hands-on training program conducted jointly by ICF and Westat covering the NPS protocol for collecting samples and conducting interviews. All State and contract personnel will be required to attend a training session prior to collecting samples for the Survey and will be issued training manuals for later reference.

Centralized computerized management of all sampling activities at ICF, including final scheduling of sample collection dates. ICF can therefore control the rate of sampling to be both compatible with laboratory capacity and with the deadline for sampling completion.

ICF will contract for sample containers constructed of appropriate materials and precleaned and preserved to NPS specifications so that breakage and losses due to contamination or lack of preservative will be minimized or eliminated.

ICF will prepare and ship to the sample teams all sample kits (sample bottles and coolers) and supplies (including questionnaires and field manuals) so that the correct bottles, necessary equipment, and calibrated probes will always be used.

ICF and Westat will maintain a computerized inventory control system for both sampling supplies and questionnaires.

Process controls that will be used during and after sampling include:

Use of standardized and documented procedures by trained sampling teams.

Purging of wells prior to sampling.

Checking for any treatment upstream of sample collection.

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Checking well water for chlorine.

Use of the NPS Hotline (1-800-451-7896) for assistance with problem sites.

Use of a computerized sample tracking system, to assure that scheduled sampling events occurred as planned and that samples reached the laboratories in satisfactory condition.

ICF will also maintain a problem file to document any problems affecting quality that occurred during the sampling process. Information contained in the problem file will include the identity of the individual who discovered the problem, the nature of the problem, who was contacted to discuss the problem, and how the problem was resolved.

7.4 Questionnaire Administration and Processing Controls

A large part of the data collection effort for the NPS will be through the use of questionnaires. Process control features associated with this effort include:

Expert evaluation of questionnaire wording and construction.

Use of trained, State personnel to administer questionnaires for the CWS survey and use of trained, professional, Westat interviewers for the rural, domestic well survey.

Processing questionnaires in a controlled environment where data retrieval is performed as necessary, where coding decisions are checked for consistency between coders, and where data entry is performed twice and checked for accuracy.

Development of range and logic checks to evaluate data for reasonableness.

Develop imputation strategies after reviewing Survey results, so that the best possible estimates for missing data points can be made.

Choose imputation classes carefully, so that donor records will be reasonable approximations of the missing values.

Check the frequency of use for each donor record, so that no single record contributes an inordinate number of values.

Flag all imputed values in the database.

7.5 Laboratory Controls

The analytical portion of the Survey will be tightly controlled in order to guarantee both the correct identity and the correct concentration for any analytes that are reported. Process control features associated with this effort include:

Use of standardized methods.

Use of a centralized source of standards

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An initial demonstration of capabilities.

Demonstrated control of the analytical measurement system through calibration requirements, method blanks, surrogate recoveries, internal standard responses, use of laboratory control standards with control charts, and instrument control standards.

A study to examine potential interferences from sample matrices.

A study to examine false negative and false positive rates through the use of a referee lab.

A study to determine analyte stability for NPS sample preservation and analysis.

Use of Method 7 for checking trihalomethane concentrations, which might indicate the sample was chlorinated and therefore not a valid NPS sample.

Participation in quarterly performance evaluation studies.

7.6 Database Management Controls

Data will initially be stored in three databases before being consolidated into the complete NPS database, which will then be prepared for use by the public. Process control features associated with this effort can be placed into three categories: analytical, questionnaire, and merged database, as described below.

Analytical

All laboratory data will be subjected to an automated data audit before being entered into the database. Any deviations to requirements in the QAPjPs will be reviewed by the appropriate Technical Monitor, who will have final authority for accepting or rejecting the data that will be included in the database.

The problem file maintained by ICF concerning problems encountered in the field during sample analysis will be consulted at the end of the Survey. Any samples with problems which would invalidate the analytical analyses will be removed from the database. A typical problem of this type might be that the sample was not collected according to protocol, such as being collected following treatment by chlorination.

Questionnaire

Data retrieval will be performed for all "key" data items identified by EPA as critical and for which additional resources should be committed in order to retrieve missing data points.

The nonresponse rate for each data item will be calculated in order to identify any problem questions and to screen for questions which might not be amenable to imputation.

The professional interviewers hired by Westat will participate in a debriefing session during which they will subjectively evaluate the effectiveness of the questionnaire instruments.

Merged Database

The merged database will clearly identify any imputed values with flags.

Stringent security measures will be implemented so that the merged databases cannot be altered after a final QC check has been performed.

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8. AUDITS

A schedule of frequent audits will be a major component of the QA program administered by the NPS QA Manager. The audits will encompass analytical activities, as well as "implementation" activities, such as sample selection, sampling, questionnaire administration, etc. Audits will be conducted to independently verify that processes are in place that are capable of achieving agreed upon levels of quality. The different types of audits which will be employed include technical system audits (TSAs), audits of data quality (ADQs), and performance evaluation studies (PEs). These are described in detail below. A fourth type of audit, a close-out audit, is described in Section 9.

8.1 Philosophical Approach

All audits will be conducted with the philosophy that the results will be used for continuous improvement, rather than to assign blame for deficiencies. Audits will focus on the activities of each individual, giving equal importance to work accomplished at every level of the organizational hierarchy. The emphasis will therefore be to hold individuals accountable for the quality of their own work.

8.2 Technical Systems Audits

Technical system audits (TSAs) will be used to make an on-site assessment of both the resources and processes used to accomplish NPS work. The QAPjP will serve as the standard against which the organization will be evaluated. As a general rule, these audits will be conducted by a team led by the NPS QA Manager with assistance from the Technical Monitor(s) and, at times, the QA Managers for ODW and OPP. Depending on the organization being audited, one of three checklists will be used, as follows:

- Laboratory Audit Checklist (Appendix B) will be used for organizations supplying analytical services.
- Field Sampling Audit Checklist (Appendix C) will be used at both CWS and rural, domestic well sites during the collection of water samples.
- General NPS Audit Checklist (Appendix D) will be used for all office activities, such as sample kit preparation, DRASTIC mapping, and questionnaire processing.

Typically, the audit will be tentatively scheduled by the Technical Monitor at a date and time mutually agreeable to both the organization being audited and the audit team. The NPS QA Manager will then provide formal notification of the audit in a letter to the project manager at the organization being audited.

The audit will commence with an introduction by the audit team leader describing the scope and agenda for the audit, in addition to any relevant updates on the progress of the Survey. At that time, project management will be requested to provide an update on personnel and their responsibilities. A tour of the facilities and/or observation of the work will occur next. The responsible party for each item on the checklist will be interviewed, at their work station, if possible, to verify that operations conform

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to the QA project plan. In conjunction with the interviews, all supporting documentation will be reviewed.

During audits of the analytical laboratories, a data audit will be conducted, during which a member of the audit team will manually verify the analytical result and all accompanying QC. Any questions arising during the data audit will be directed to the analyst who performed the work.

During audits of field operations, the auditor(s) will not interrupt the field crew, especially in the presence of the occupants of the home where sampling occurs. Rather the auditor(s) will act as impartial observers and discuss any problems with the field crew in private.

At the conclusion of the audit, a meeting will be held with the project manager during which the audit findings will be discussed and an agreement will be reached on corrective action measures. A report describing the audit will be written by the team leader and circulated for review to all participants, including the project manager, before being forwarded to the Survey Director.

TSAs will be held semiannually for laboratory and office activities. The goal for auditing field activities will be 1% of all wells sampled.

8.3 Audits of Data Quality

Audits of data quality (ADQs) will be a major part of the laboratory technical system audit (see Section VIII of the laboratory audit checklist) and will be conducted in order to verify the accuracy of analytical identifications and quantifications. The procedure will be to track a sample from the time it was taken, through analysis, and finally to the reporting of the analytical result to the Survey database manager. In addition to sample results, the preparation and analysis of all QC samples and standard solutions will be checked and calculations will be verified.

8.4 Performance Evaluation Studies

Performance evaluation (PE) studies will be used to challenge the capabilities of the analytical laboratories and will be conducted quarterly. The contents of the standards will be determined each quarter in discussions between the NPS QA Manager and the Technical Monitors. The Technical Monitors will consider several factors when recommending analytes for inclusion in the PE samples such as whether an analyte has been detected in well water samples, if an analyte has been difficult for the laboratories to analyze, and has the analyte been included in a previous PE sample. The Technical Monitors will be counseled to include the analytes at three to five times the limit of quantification, unless conditions specifically warrant a different level.

Bionetics, a contractor with EMSL-Cincinnati, will be responsible for preparing standard solutions and packaging them in sealed glass ampules. The referee laboratories will be responsible for verifying their contents. The QA Manager will forward the PE samples to the labs with instructions for preparation, as shown in Exhibit 8-1. The labs will be given approximately four weeks to analyze the

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Exhibit 8-1

Example of Instructions for PE Samples

INSTRUCTIONS FOR NATIONAL PESTICIDE SURVEY PERFORMANCE EVALUATION SAMPLES February 7, 1990

Methods 1, 2, 3, 4, 5, 6, and 7

As part of the Survey quality assurance program, performance evaluation samples will be analyzed by every laboratory once each calendar quarter. The samples will be prepared from concentrates contained in sealed glass ampules. Each lab will receive two identical vials per analytical method, so that a back-up vial will be available. To prepare the samples, follow the directions given below. The analyte concentrations will be within the normal working ranges of the method. Analyze once and report the value as you would any other NPS samples, using the sample ID from the concentrate vial. In addition please forward your result(s) in memo form to your technical monitor no later than March 16, 1990.

DIRECTIONS: Partially fill a 1000 mL Class A volumetric flask with laboratory pure water. Add 1 mL of concentrate. Fill the volumetric to the mark with water and invert several times to mix. Use only that portion of the sample that is required by the analytical method.

Please contact your technical monitor if you have any questions.

DUE DATE IS MARCH 16, 1990

CC:

Battelle Clean Harbors ECS Hunter/ESE Montgomery Labs Radian TSD

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samples and report back to their Technical Monitor. The results will also be reported via floppy disc along with sample batch data.

Criteria for passing the PE samples will be developed on an analyte-by-analyte basis. A 99% confidence interval will be computed for the known "true" value of each analyte in the PE sample based on results of seven separate analyses by the referee lab and compared to a 99% confidence interval for the reported result. (The latter will be computed from precision estimates from past laboratory performance for the given analyte). If the two confidence intervals overlap, the criteria will have been met. If the intervals do not overlap, the criteria will not have been met and corrective action must be instigated in consultation with the Technical Monitor. Each laboratory and Technical Monitor will receive the results for their methods. A summary report of all analyses will be prepared and forwarded to the Survey Director and the QA Managers for OPP and ODW.

8.5 Corrective Action Verification

Any deficiencies identified during the course of an audit will require that the audited organization develop a plan immediately for correcting the problem. The plan must be discussed with the Technical Monitor, and, as appropriate, with the NPS QA Manager. At the minimum, verification of problem resolution will occur at the next on-site audit.

9. QA COMMUNICATION STRATEGIES

Since its inception, NPS has committed significant resources towards developing effective communication strategies which will greatly facilitate the information exchange processes necessary for the QA program to operate successfully.

9.1 Status Reports

NPS management has developed a routine schedule of teleconference calls which will include QA as an agenda item. Weekly calls will be hosted by NPS staff for key EPA and contract personnel to discuss progress, problems, and QA issues that have developed over the past week. The calls will serve an important function in coordinating real-time activities that impact more than one organization, such as the sampling schedule on the analytical labs. The calls will be used to keep NPS management informed of the audit schedule and to report informally on audit findings. Monthly teleconference calls will be hosted by NPS management for EPA staff located in the ten regional offices who have been designated as official points of contact on Survey issues. The overall progress of the Survey will be described during the call, as will any associated QA issues.

Quality assurance issues will also be communicated in several other formats. Specific QA accomplishments will be described in monthly and quarterly reports by the QA Manager to the EPA project officer for quality assurance support, who resides at the Environmental Monitoring Systems Laboratory (EMSL) in Cincinnati. An annual report on QA accomplishments and plans for the following fiscal year will be prepared by the NPS QA Manager for the OPP QA Manager for inclusion in the OPP annual quality assurance report and workplan to QAMS. Lastly, as described in Section 8, all audit results will be documented in free-standing reports and forwarded to the Survey Director, the QAMs for the Offices of Pesticides Programs and Drinking Water, and the NPS archives operated by ICF.

9.2 Operations Communications

Separate communications networks will be developed for tracking the progress of operations level activities on a day-to-day basis. ICF will develop a computerized tracking system accessible by both field crews and lab personnel that will contain information on the planned dates of sample collection and actual progress in the field. A system of frequent phone contacts will also be encouraged between the project managers at the laboratories and the Technical Monitors, especially in cases where problems arise with an analytical method or the laboratory experiences any type of difficulty which would preclude its ability to analyze samples. The laboratories will also provide written monthly progress reports to the Technical Monitors according to the format presented in Exhibit 9-1. This level of communication should benefit the quality of Survey data because managers will have access to information that will allow them to make the necessary adjustments for meeting Survey goals on completeness.

Exhibit 9-1

Format for Monthly Progress - QA Report

EPA Contract Laboratories Progress - QA Report

Meth	nod #
Rep	ort Period
Anal	lyst
Date	·
1.	Progress:
	# of samples received
	# of samples analyzed
	# of samples invalidated
	Set ID numbers forwarded to data manager
2.	Bench Level Corrective Action(s):
	Date
	Problem
	Action Taken
	Verification of Correction
	Sample set analyzesd prior to problem
	(Use back of page if additional room is required.)
3.	Problems (Project Related):
4.	Information requested by Technical Monitor.
5	Changes in Personnel

6.

Comments:

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9.3 Final Report

Information on the QA program and its impact on the Survey will be included in the Survey's summary interpretive reports, following all data collection and processing activities. In addition to providing specific information about the QA program in the final reports, each report will be reviewed by the NPS QA Manager from a QA perspective and comments will be forwarded to the Director.

10. CLOSE-OUT ACTIVITIES

As each organization completes its work for the Survey, a final update to each of the project plans will be required, as will participation in a close-out audit, as described below.

10.1 Final Update to QA Project Plans

All QA/QC conducted in support of the Survey will be described in one of the QA project plans previously listed in Section 6. The QA project plans are designed to be "living" documents, therefore if planned procedures are not effective or implementable, the organization performing them will be free to develop alternative approaches, provided the revision procedure in Appendix A-6 is followed. These types of changes are anticipated to occur with relative frequency and it will be the responsibility of the organization performing the work to accurately record changes in the QA project plan.

A final review of the project plans will be performed by the Technical Monitors and the QA Manager, who will request that missing updates be included before final payment is made for the project plans at the end of the Survey. An electronic version of the plan will also be requested after final changes are made. The final updates to the project plans will form an appendix in the final report and will be available individually from the National Technical Information Service (NTIS), Springfield, Virginia.

10.2 Close-Out Activities

As requested in the Survey policy on data archival, shown in Exhibit 10-1, every organization participating in the Survey will be responsible for archiving data they have generated in support of Survey results. Basically the policy states that the data must be stored at a secure location in an organized and retrievable fashion for a minimum of one year after release of Survey results. A close-out audit will be conducted by the NPS QA Manager and Technical Monitors to evaluate the archival procedures adopted by each organization and a written report will be made to the Survey Director on their suitability. During the audit, the files will be checked for completeness and the storage space will be inspected. The close-out audit will be the last on-site review performed under the auspices of the NPS QA program, therefore each organizations QAC will be responsible for providing written verification that any necessary corrective action was implemented.

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Exhibit 10-1

Example of NPS Archival Policy

ATTACHMENT 1

NATIONAL PESTICIDE SURVEY (NPS) POLICY FOR ARCHIVAL OF RAW DATA JANUARY, 1990

POLICY STATEMENT

It is the policy of the National Pesticide Survey that all raw data that have been collected in support of Survey activities will be stored and managed in a systematic manner such that data may be retrieved in a timely fashion for reference purposes.

BACKGROUND

The NPS has conducted sampling, interviewing, and chemical analyses for approximately 1,350 drinking water well sites across the U.S. As a result, a considerable amount of "raw" data has been generated. To assure the continued availability of these data for reference purposes, each organization contributing to the Survey is requested to abide by the following guidelines.

GUIDELINES

1) Scope:

This policy applies to all data and other materials that have been generated either through the analytical effort or the implementation/data synthesis effort; both hard copy and/or electronic media are covered. Also covered are any formal documents used for the Survey, such as training manuals.

2) Accessibility:

Materials must be managed so that they can be accessed within 5-10 working days. Access to materials should be limited to internal staff and representatives of the EPA.

3) Length of Storage:

At the minimum, materials must be stored until October of 1992. Even after this date, the Survey Director (or designee) must be contacted for permission to purge Survey data/documents. Survey management also reserves the right to have all files surrendered upon request at any time.

4) Privacy Act (5 U.S. Code 552a):

Organizations that have recorded information on specific individuals must manage their records so as to guarantee confidentiality in accordance with the requirements of the Privacy Act.

APPROVAL

This policy has been reviewed and approved by the Director, National Pesticide Survey, 401 M Street, S.W., WH550, Washington, D.C. 20460.

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11. REFERENCES CITED

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- Mason, R.E., L.L. Piper, W.J. Alexander, R.W. Pratt, S.K. Liddle, J.T. Lessler, and M.C. Ganley, 1988.

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- Nees, M. and C. Salmons, 1987. <u>National Survey of Pesticides in Drinking Water Wells, A Review of the Planning Process and the Data Quality Objectives</u>, RTI/7801/08/01F.
- United States Environmental Protection Agency, <u>A Set of Scientific Issues Being Considered by the Agency in Connection with the National Pesticide Survey Pilot Study</u>, October 9, 1987, Federal Insecticide, Fungicide, and Rodenticide Act Scientific Advisory Panel Subpanel
- United States Environmental Protection Agency, <u>Interim Guidelines and Specifications for Preparing</u>

 <u>Quality Assurance Project Plans</u>, December 29, 1980, QAMS-500/80

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APPENDIX A INFORMATION PACKET FOR NPS LABORATORIES

Appendix A-1

Guidance for Quality Assurance Project Plans:

Section 5

- Initial Demonstration of Capabilities: Determining Reporting Limits
- Initial Demonstration of Capabilities: Use of Control Charts
- Additional Guidance on Establishing Control Charts for NPS Methods
- Dixon's Test

INITIAL DEMONSTRATION OF CAPABILITIES DETERMINING REPORTING LIMITS 2/3/88

- 1. Determine concentration of standard necessary to produce an instrument detector response with a 5/1 signal to noise ratio.
- 2. Spike eight reagent water samples at the concentration determined above, and analyze in a single day.
- 3. Compute Minimum Detectable Level (MDL) by multiplying the standard deviation by the student's t value, appropriate for a 99% confidence level, and a standard deviation estimate with n-l degrees of freedom. (Note that for n=8, t=2.998 at the 99% confidence level and 7 degrees of freedom).
- 4. The Estimated Detection Level (EDL) equals either the concentration of analyte yielding a detector response with a 5/1 signal to noise ratio, or the calculated MDL, whichever is greater.
- 5. Determined EDLs must be no greater than twice those determined during methods development, with the following exceptions:
 - a. Method 5 target values will be supplied by EPA, since the EDLs included in the method were determined using a less sensitive detector than currently available.
 - b. Methods 7 and 9 will be evaluated by the technical monitors, since target EDLs are not included in the methods.
- 6. The acceptability of EDLs exceeding the above limits will be determined by the technical monitor, based on health effects values.
- 7. Reanalyze the standards using the confirmation column. the EDLs determined on the confirmation column must equal those determined on the primary column. Again, EDLs exceeding this requirement will be approved on a case by case basis, by the technical monitors.
- 8. The laboratories will be required to perform up to six analyses per analyte mix by GC/MS, for the appropriate methods. These analyses will be performed by Multiple Ion Detection (MID), using the three ions specified by EPA. The purpose of these analyses are to determine the concentration at which a 5/1 signal to noise ratio, for the least intense of the three ions, is obtained.

9. The Minimum Reporting Levels (MRLs) are defined as the following mutiple of the EDL.

Method #	<u>Multiple</u>
1	4 x EDL
2	5 x EDL
3	5 x EDL
4	5 x EDL
5	3 x EDL
6	3 x EDL
7	3 x EDL
9	3 x EDL

- 10. Any chromatographic peak occurring at the proper retention time of an NPS analyte, at a concentration level between .5 x MRL and MRL will be confirmed and reported as an occurrence of that analyte. Exact quantification will not be required. Any frequent occurrence of a peak which is not an NPS analyte, or any occurrence of a non-NPS analyte at what appears to be a high concentration, should be noted.
- 11. The lower concentration calibration standard must be prepared at a concentration equal to the MRL.

INITIAL DEMONSTRATION OF CAPABILITIES USE OF CONTROL CHARTS 2/3/88

- A. Contractors for Methods 1-4 and 6 will be required to demonstrate control of the measurement system via use of control charts. Control must be demonstrated for each analyte for which quantitation is required and for the surrogate at a concentration equal to that spiked into samples.
- B. To establish the control charts, following initial demonstration of capability, 5 reagent water samples will be spiked at 10 times the Minimum Reporting Limit (MRL) for the method and carried through extraction and analysis. An additional 15 samples will be spiked and analyzed, 5 on each of 3 days. The data from these 20 spiked samples will be used to construct control charts.
- C. Criteria for Accuracy and Precision
 - 1. The RSDs for any analyte must be less than or equal to 20%, except where data, generated by Battelle at the corresponding level, indicated poorer precision. The RSDs exceeding 20% will be evaluated on a case-by-case basis by technical moniotrs for each method.
 - 2. The mean recovery (x) of each analyte must lie between Battelles' mean recovery for each analyte (at the corresponding level) +/- 3 times the relative standard deviation (RSD) for that analyte as determined by Battelle during methods development, but no greater than Battelle's mean recovery +/-30%.

Example:

For an analyte "A"

- i. Battelle demonstrated recovery (x) of 80% for Analyte "A" with RSD of 5%. Acceptable recoveries will be 80% +/- 3(5%) = 80% +/- 15% = 65% 95%.
- ii. Or, Battelle demonstrated recovery (x) of 80% with RSD of 15% for analyte "A". The acceptable recovery would then be limited to 80% +/- 30% 50% 110%.

3. Surrogate

In establishing the control chart for the surrogate, criteria C(1) and C(2) above, apply; it follows that one of the spike mixes must contain the surrogate at the concentration as spiked into actual samples.

Surrogate recoveries from samples (Methods 1-4, 6-7) will be required to be within +/- 30% of the mean recovery determined for that surrogate during the initial demonstration of capabilities.

4. Warning Limits/Control Charts

The control charts will be drown up so as to depict both warning limits (+/-2) standard deviations (s) and control limits (+/-3).

D. Outliers

Dixon's test will be used to determine outliers. There can be no more than 3 outliers per analyte from the 20 spiked controls.

E. Out-of-Control Situations

- 1. In the following instances, analytical work must be stopped until an "in-control" situation is established.
 - a. More than 15% of the analytes of a particular method are outside \pm 3s.
 - b. The same analyte is outside +/- 3s twice in a row, even though >85% of the total analytes are in control.
- 2. An "alert" situation arises when one of the following occurs:
 - Three or more consecutive points for an analyte are outside
 +/- 2s but inside the +/- 3s.
 - b. A run of 7 consecutive points above or below the mean.
 - c. A run of 7 points for an analyte in increasing or decreasing order.

The "alert" situation implies a trend toward an "out-of-control" situation. The contractor is required to evaluate his analytical system before proceeding. If "alert" or "out-of-control" situations occur frequently, reestablishing control charts may be required by the technical monitor before analytical work can proceed.

3. Other Factors

a. Method blank

If the "method blank" exhibits a peak within the retention window of any analyte and is greater than or equal to one-half the MRL for that analyte, an "out-of-control" situation has developed.

b. Performance Evaluation Samples

If the contractor fails on one of these samples, an "out-of-control" situation is present.

4. Up-Dating Control Charts

Following establishment of the control chart, a spiked control is part of each analytical or "sample set". When 5 such controls have been run, the recoveries of these analytes will be incorporated into the control charts by adding these 5 most recent recoveries to the 20 original points and then deleting the first 5 of the original points. Accuracy and precision are recalculated and the chart is redrawn. The newly drawn chart will then apply to all data in sample sets subsequent to the last one used to update the chart.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

ENVIRONMENTAL CHEMISTRY LABORATORY NASARISTL BUILDING 1105. NSTL STATION. MISSISSIPP 38529

April 25, 1988

MEMORANDUM

SUBJECT: Additional Guidance on Establishing Control Charts for NPS Methods

Bob marker

FROM:

Bob Maxey, NPS Analytical Coordinator

Environmental Chemistry Laboratory

NSTL, MS

TO:

Addressees

In reviewing the following guidance, please refer to the attached instructions, "INITIAL DEMONSTRATION OF CAPABILITIES, CONTROL CHARTS", which were disseminated at the NPS meeting in Cincinnati in February.

When you or your your laboratory have completed analyses of the 4 sets of 5 samples (see Attachment, part B) and when these 20 values have been tested for and found to contain 3 or fewer outliers, the sample mean (x) of these 17-20 values is determined in ppb units along with the standard deviation also in ppb units. Refer to your formulas in Section 14 of your QAPP.

Divide the standard deviation (in ppb units) by the sample mean (\overline{x}) in ppb units and multiply the result by 100 to obtain the Relative Standard Deviation (RSD). RSD is the standard deviation expressed as a percentage of the sample mean, (\overline{x}) .

The guidance in the Attachment, Section C-2, calls for \overline{x} as a mean recovery, and I should have more clearly specified \overline{R} as the mean recovery. Divide the value \overline{x} of an analyte (in ppb from the second paragraph above) by the spiking level, in ppb, for that analyte and multiply by 100 to obtain \overline{R} , the mean recovery in percent of the 17-20 values. Repeat for each analyte in the Method.

 \overline{R} becomes the central line on the control chart about which upper (UWL, UCL) and lower (LWL, LCL) warning and control limits are depicted (see Attachment, Section C-4). The warning limits become $\overline{R}+2$ (RSD), and the control limits become $\overline{R}+3$ (RSD). There is a separate but similarly constructed control chart for each analyte, including the surrogate.

I apologize to those of you confused by the control chart instructions. If you have additional questions, call me at FTS 494-1225 or commercial (601) 688-1225.

Addressees:

Dave Munch

Dr. Robert Clark

Dr. A. Dupuy, Jr.

Dr. C. Byrne

W. Dreher

J. Watkins

cc:

Dr. A. Yonan

INITIAL DEMONSTRATION OF CAPABILITIES CONTROL CHARTS

- A. Contractors for Methods 1-4, and 6, will be required to demonstrate control of the measurement system via use of control charts. Control must be demonstrated for each analyte for which quantitation is required and for the surrogate at a concentration equal to that spiked into samples.
- B. To establish the control charts, following initial demonstration of capability, 5 reagent water samples will be spiked at 10 times the Minimal Reporting Level (MRL) for the method and carried through extraction and analysis. An additional 15 samples will be spiked and analyzed, 5 on each of 3 days. The data from these 20 spiked samples will be used to construct control charts.
- C. Criteria for Accuracy and Precision
 - The RSDs for any analyte must be ≤20%, except where data, generated by Battelle at the corresponding level, indicated poorer precision. The RSDs exceeding 20% will be evaluated on a case-by-case basis by Technical Monitors for each method.
 - The mean recovery (x̄) of each analyte must lie between Battelles' mean recovery for each analyte (at the corresponding level) ± 3 times the RSD for that analyte as determined by Battelle during methods development, but no greater than Battelle's mean recovery ± 30%.

Example:

For an analyte "A"

- o Battelle demonstrated recovery (X) of 80% for Analyte "A" with RSD of 5%. Acceptable recoveries will be 80% ± 3 (5%) = 80% ± 15% = 65% 95%;
- o or, Battelle demonstrated \overline{x} of 80% with RSD of 15% for analyte "A". The acceptable recovery would be limited to 80% ± 30% = 50% 110%.

3. Surrogate

In establishing the control chart for the surrogate, criteria in C(1) 2nd (2) above, apply; it follows that one of the spike mixes must contain the surrogate at the concentration as spiked into actual samples.

Surrogate recoveries from samples (Methods 1-4, 6-7) will be required to be within ± 30% of the mean recovery determined for that surrogate during the initial demonstration of capabilities.

4. Warning Limits/Control Limits

The control charts will be drawn up so as to depict both warning limits ($\pm 2\sigma$) and control limits ($\pm 3\sigma$) about the mean.

D. Outliers

Dixon's test will be used to determine outliers. There can be no more than 3 outliers per analyte from the 20 spiked controls.

E. Out-of-Control Situations

- 1. In the following instances, analytical work must be stopped until an "in-control" situation is established.
 - a. More than 15% of the analytes of a particular method are outside ± 3σ.
 - b. The same analyte is outside ± 3σ twice in a row, even though >85% of the total analytes are in control.
- 2. An "alert" situation arises when one of the following occurs:
 - a. Three or more consecutive points for an analyte are outside ± 2σ but inside the ± 3σ.
 - b. A run of 7 consecutive points above or below the mean.
 - c. A run of 7 points for an analyte in increasing or decreasing order.

The "alert" situation implies a trend toward an out-of-control" situation. The contractor is required to evaluate his analytical system before proceeding. If "alert" or "out-of-control" situations occur frequently, re-establishing control charts may be required by the Technical Monitor before analytical work can proceed.

3. Other Factors

a. Method blank

If the "method blank" exhibits a peak within the retention window of any analyte and is greater than or equal to one-half the MRL for that analyte, an "out-of-control" situation has developed.

b. Performance-Evaluation Samples

If the contractor fails on one of these samples, an "out-of-control" situation is present.

F. Up-dating Control Charts

Following establishment of the control chart, a spiked control(s) is part of each analytical or "sample set". When 5 such controlShave been run, the recoveries of these analytes will be incorporated into the control charts by adding these 5 most recent recoveries to the 20 original points and then deleting the first 5 of the original points. Accuracy and precision are recalculated and the chart re-drawn. The newly drawn chart will then apply to all data in sample sets subsequent to the last one used to update the chart.

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DIXON'S TEST

Dixon's test is used to confirm the suspicion of outliers of a set of data (for example, control chart data points). It is based on ranking the data points and testing the extreme values for credibility. Dixon's test is based on the ratios of differences between observations and does not involve the calculation of standard deviations.

The procedure for Dixon's test is as follows (from Taylor, 1987):

1) The data is ranked in order of increasing numerical value. For example:

$$X_1 < X_2 < X_3 < \dots < X_{n-1} < X_n$$

- 2) Decide whether the smallest, X_1 , or the largest, X_n , is suspected to be an outlier.
- 3) Select the risk you are willing to take for false rejection. For use in this QAPP we will be using a 5% risk of false rejection.
- 4) Compute one of the ratios in Table 1. For use in this QAPP we will be using ratio r_{22} , since we will be using between 20 and 17 points for the control charts.
- 5) Compare the ratio calculated in Step 4 with the appropriate values in Table 2. If the calculated ratio is greater than the tabulated value, rejection may be made with the tbulated risk. Fort his QAPP we will be using the 5% risk values (bolded).

Example (from Taylor)

Given the following set of ranked data:

The value 10.58 is suspected of being an outlier.

1) Calculate r₁₁

- 2) A 5% risk of false rejection (Table 2), $r_{11} = 0.477$
- 3) Therefore there is no reason to reject the value 10.58.
- 4) Note that at a 10% risk of false rejection $r_{11} = 0.409$, and the value 10.58 would be rejected.

TABLE 1
CALCULATION OF RATIOS

Ratio	For use if n is between	if X_n is suspect	if X_1 is suspect
r ₁₀	3 - 7	$(X_n - X_{n-1})$ $(X_n - X_1)$	$(X_2 - X_1)$ $(X_n - X_1)$
r ₁₁	8 - 10	$(X_n - X_{n-1})$ $(X_n - X_2)$	$(X_2 - X_1)$ $(X_{n-1} - X_1)$
r ₂₁	11 - 13	$(X_n - X_{n-2})$ $(X_n - X_2)$	$(X_3 - X_1)$ $(X_{n-1} - X_1)$
r ₂₂	14 - 25	$(X_n - X_{n-2})$ $(X_n - X_3)$	$(X_3 - X_1)$ $(X_{n-2} - X_1)$

Note that for use in this QAPjP ratio $\ensuremath{r_{22}}$ will be used.

TABLE 2

VALUES FOR USE WITH THE DIXON TEST FOR OUTLIERS

Risk of False Rejection <u>Ratio</u> n 0.5% <u>1%</u> <u>5%</u> 10% 3 0.994 0.988 0.941 0.806 0.926 4 0.889 0.765 0.679 5 0.821 0.780 r_{10} 0.642 0.557 6 0.740 0.698 0.560 0.482 7 0.080 0.637 0.507 0.434 8 0.725 0.683 0.554 0.479 9 0.677 0.635 r₁₁ 0.512 0.441 10 0.639 0.597 0.477 0.409 11 0.713 0.679 0.576 0.517 12 0.675 0.642 0.546 0.490 r₂₁ 13 0.649 0.615 0.521 0.467 14 0.674 0.641 0.546 0.492 15 0.647 0.616 0.525 0.472 16 0.624 0.595 0.507 0.454 17 0.605 0.577 0.490 0.438 18 0.589 0.561 0.475 0.424 19 0.575 0.547 0.462 0.412 r_{22} 20 0.562 0.535 0.450 0.401 21 0.524 0.440 0.391 22 0.514 0.430 0.382 23 0.505 0.374 0.421

Note that for this QAPjP the 5% risk level will be used for ratio r_{22} .

0.497

0.489

0.413

0.406

0.367

0.360

24

25

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Reference:

John K. Taylor, <u>Quality Assurance of Chemical Measurements</u>, Lewis Publishers, Chelsea, MI, 1987.

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Appendix A-2

Guidance for Quality Assurance Project Plans:

Section 6

- Sample Container Types, Sizes, and Preservatives
- Sample Label and ID Information
- Sample Tracking Form

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Sample Container Preservation Requirements

NPS METHOD	BOTTLE, CAP, AND PRESERVATIVE
NPS-1	Clear l-liter borosilicate glass bottles with teflon-lined caps with 10 ml of mercuric chloride.
NPS-2	Clear l-liter borosilicate glass bottles with teflon-lined caps with 10 ml of mercuric chloride.
NPS-3	Clear l-liter borosilicate glass bottles with teflon-lined caps with 10 mi of mercuric chloride.
NPS-4	Clear l-liter borosilicate glass bottles with teflon-lined caps with 10 ml of mercuric chloride.
NPS-5	250 ml amber screw-cap glass bottles with teflon faced septa with 7.5 ml of pH 3 buffer.
NPS-6	60 ml screw-cap glass bottles with teflon-faced septa with 0.6 ml of mercuric chloride.
NPS-7	60 ml screw-cap glass bottles with teflon-faced septa with 0.6 ml of mercuric chloride.
NPS-9	125 ml polyethylene bottles with 0.25 ml of sulfuric acid.
	rative should be \leq 1% of the final sample volume for all methods. the preservative should be 10 mg/L for all methods.

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Description of Sample Code Numbers for Sampling Scenario and Lab Spike Assignments

PD-0001-1-1-01 D Method Number Lab Name Number Sample Type Well Type PC = Community Well 0001 1 = JMM 01 = Field Sample 2 = ATI02 = Shipping Blank 2 PD = Domestic Well 3 = RAD 03 = Backup sample PR = Resampled Well 3 4 = ESE PB = PE Sample 04 = Lab spike (mix A, level 1)=A0 1500 5 = BCL 5 05 = Lab spike (mix A, level 2)=A1 6 = BSL 06 = Lab spike or time storage - Day 0 (mix A, level 3)=A2 7 = TSD 07 = Lab spike (mix B, level 1)=B008 = Lab spike (mbx B, level 2) = 8109 = Lab spike or time storage - Day 0 (mix 8, level 3) =82 10 = Lab spike (mix C, level 1)=C0 11 = Lab spike (mbx C, level 2)=C1 12 = Lab spike or time storage - Day 0 (mbr C, level 3) = C2 13 = Time storage (Day 0 duplicate) 14 = Time storage - Day fourteen 15 = Time storage - Day fourteen duplicate Lab performing the analyses for the NPS: FS = Field Sample 1 = JMM (Montgomery Laboratories) 2 = ATI (Alliance Technologies Inc./Clean Harbors Analytical Services) LS = Lab Spike 3 = RAD (Radien, Inc.) TS = Time Storage 4 = ESE (ESAE) 5 = BCL (Battelle, Columbus Division) 6 = BSL (Bay St. Louis (EPA/Environmental Chemistry Lab)) 7 = TSD (EPA/Technical Support Division Lab)

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NATIONAL PESTICIDE SURVEY

SAMPLE #:

METHOD# KIT:

PRESERVATIVE:

DATE : TIME : SAMPLER

Blank Sample Bottle Label

NATIONAL PESTICIDE SURVEY

SAMPLE #: PD-9999-7-7-01

TSD - METHOD# 7 KIT: 711

FIELD SAMPLE

PRESERVATIVE: HgC12

DATE : TIME : SAMPLER

Example Sample Bottle Label

TERRORETESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATESTETTTSTATES

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Appendix A-3

Guidance for Quality Assurance Project Plans:

Section 7

- NPS Sample Tracking Program
- Minimum Requirements for Sample Management and Spiking
- Spiking Update

THE MPSIS SAMPLE RECEIPT PROCEAN

NPSIS is designed to keep track of the day to day operations of the National Pesticide Survey. You play an important role in NPS and your timely notification of receiving a kit of samples is essential to the success of NPS. We have designed the Sample Receipt Program with your busy schedule in mind. NPSIS will obtain the minimum amount of information necessary while still maintaining a secure system. You will be entering data into the NPSIS personal computer via your own computer, modem, and Carbon Copy software.

1.1 Hardware and Software Requirements.

The NPSIS Sample Receipt Program has a minimum hardware and software requirement. Here is a list of items you will need:

Hardware:

- One (1) IBM PC, XT, AT, or Personal System model with at least 640K memory.
- One (1) 2400 or 1200 baud Hayes or Hayes compatible modem with cables. (See Carbon Copy guide for cabling requirements and a description of usable modems)
- One (1) data transmission phone line.

Software:

- NPSIS Sample Receipt Program access provided for you by ICF.
- One (1) copy Carbon Copy software which is provided to you by ICF for the duration of NPS.

1.2 Initial Installation Steps.

Before you can access and use NPSIS, you must first load the Carbon Copy software onto your PC. The directions are provided in the Carbon Copy manual. One item you will want to include is an entry into the "Call Table". This entry will include a name, telephone number, and password for the NPSIS computer. To enter these items into the Call Table, press "2" from the Carbon Copy Parameters' Screen. The information you must enter consists of the following:

• Name: NPS

• Telephone Number: 703-961-0629

Password: NPS

1.3 Parameters for Communications.

NPSIS will maintain a set configuration throughout operation. Any changes due to updates in equipment or the system which will affect your ability to communicate through Carbon Copy will be forwarded to you. The parameters which will be maintained at this time are:

- 2400 baud modem speed.
- Answer ring count equal to one.
- Re-boot on exit after 5 minutes. (If there is a power failure or some other type of interruption, you can log back on to NPSIS and resume your session.)
- Five minute inactivity time constraint.
- Two password attempts.

2 REPORTING A SAMPLE RECEIPT TO MPSIS.

2.1 Establishing a Communications Link.

Once you have installed Carbon Copy and have all of the necessary hardware, you are ready to "log on" to the NPSIS computer at ICF. To do this:

Type: C:> CCHELP HPS in your directory containing Carbon Copy.

This command will automatically dial the NPSIS computer, send your password for verification, and establish a data link between the two computers. You will be able to discern what is taking place by messages to your screen.

2.2 Entering A Sample Receipt Into NPSIS.

Once you have established a data link, (e.g., are "logged on"), you will see on the screen exactly what is on the screen of the NPSIS computer. This screen you are viewing is the main menu for the Sample Receipt Program. Remember that you are controlling the NPSIS computer via a 2400 baud phone line and your typing will appear on the screen at a much slower rate than you are accustomed to. A few tips on how to use the system are outlined in the next section.

2.2.1 Useful Tips on How to Use NPSIS.

Before you start, a few things to remember are:

- Pressing the "Esc" key will cancel all changes for the screen you are currently in and return you to the previous screen. Pressing "Esc" at the Searching Screen returns you to the main menu.
- Pressing "PgDn" or "PgUp" will save the items you have entered in the current screen and place you in the next or previous screen, respectively. This feature is handy to use when you only have a few items to enter in a screen which prompts for several items.
- Pressing "Enter", "arrow up", or "arrow down" will move the cursor from field to field in each screen. Remember that using the sideways arrows will not work.
- Pressing the "Alt" and "Right Shift" keys together will place the Carbon Copy Control Screen over the NPSIS Sample Receipt Program. You can then use the communications features in Carbon Copy. Pressing "Fl0" again when you are through will replace the NPSIS Sample Receipt Program screen you were currently in back on your screen, and
- Because you will are most likely to be entering information regarding a number of kits at one time, after you save or cancel your entries for one kit, you will be placed at the initial Sample Searching Screen for a new kit. If you are finished with your data entry, simply press "Esc" to exit the Sample Searching screen and be placed in the main menu.

2.3 A Basic Outline of the Sample Receipt Program.

The NPSIS Sample Receipt Program has three basic features:

- Initial reporting of a NPS sample kit of sample bottles.
- Ability to edit or re-edit an existing report of a kit receipt, and
- Access to ICFs computerized mail system which provides the ability to send memoranda to ICF staff.

The information obtained in an entry for a kit of bottles is:

- The kit identification number, the FedEx airbill number, and the last name of the person making the entry.
- Any damage to the kit as a whole such as melted ice or any breakage of the cooler.

- Verification of which bottles belong in a kit or cooler, notification of any missing bottles or any additional bottles, and
- Any damage to each sample bottle which renders it unusable for analysis and testing.

2.4 NPSIS Sample Receipt Program Screens.

When you have completed the logon procedure, you will see the following main menu on your computer screen:

NATIONAL PESTICIDE SURVEY INFORMATION SYSTEM

SELECTION MENU FOR REPORTING SAMPLE RECEIPTS 04/05/88

Report \ Edit a Sample Receipt Send a Memo

Press <Alt><Right-Shift> to Logoff

use † Land 🚽 to select option.

The screens provided in this memo will show all of the screens available and thus represent the maximum number of screens you will encounter with NPSIS. It is most likely that you will not have the need to enter information reporting damaged kits or samples. Therefore, not all of the screens depicted below will appear in your normal session.

If you choose the first item on the menu, "Report \ Edit a Sample Receipt", you will then be prompted for the kit identification number and the FedEx airbill number associated with the specified kit. The screen will appear like this:

NPS Sample Receipt Searching Screen

** Enter the following items to access kit information **

To find the Kit information in NPSIS in the most complete and accurate fashion, please enter the Kit number and the FedEx airbill number.

Enter kit number: ---> FD-0001-151

Enter FedEx airbill #:
---> 1111111111

Enter your last name: ----> CHIANG

* Press ESC to exit the searching *

If the kit number you have entered is incorrect, or if the kit number and FedEx airbill number combination is incorrect, NPSIS will prompt you to try to enter these number again, as illustrated on the next page. It is possible that the FedEx airbill number on the kit is not the same as the FedEx airbill number which was entered into the NPSIS system. This could happen if the field team loses or damages the airbill.

ERROR!! The kit you entered cannot be found. . .

Kit number: PD-0001-151

AND

FedEx airbill number: 1111111111

Please check these numbers and try again!

NPSIS is designed to track Kits and FedEx airbill numbers.
The Kit and FedEx airbill number combination you have entered does not match what is currently in the system. Please enter the correct combination. If you still have problems, try leaving the FedEx airbill # BLANK. Only enter the Kit number.

Press any key to continue...

Then, you will encounter this screen insuring that you have entered the FedEx airbill number:

Kit No.: PD-0001-151

Did you enter the correct Kit number and FedEx airbill number?

NPSIS is designed to store and track all FedEx airbill numbers. This Kit may have a different FedEx airbill number than the system, please enter the new FedEx airbill number:

---->

Note: if the correct airbill number was entered before, hit ENTER.

PgDn (Next page), PgUp (Previous page), Esc (Exit)

Once you have correctly identified the sample kit, NPSIS will ask you if there is any damage to the kit as a whole:

Kit No.: PD-0001-151

Was there any damage to the sample kit? (Y/N) Y

PgDn (Next page), PgUp (Previous page), Esc (Exit)

If you press "Y", NPSIS will then prompt you for the apparent cause of damage:

Was there any damage to the sample kit? (Y/N) Y

Please indicate the cause for damage:

Kit is broken (Y/N) Y

Ice is melted (Y/N)

Other Reason (Y/N)

Please enter any comments about the sample kit.

Comments: Broken upon arrival.

Comments:

PgDn (Next page), PgUp (Previous page), use # dor do select field.

There may already be comments regarding the kit in the comment field shown in the above screen. In this case, please enter your comments after any which already appear. This insures that no information is destroyed.

Next, NPSIS will ask you to survey the contents of the kit and check that which bottles are contained within the kit. You should then look at the bottle labels and determine if any are missing. Don't forget to check and determine if any bottles have been included in the kit which do not appear on the list provided by NPSIS on this screen:

Kit No.: PD-0001-151

Please compare the following bottle numbers with those in the sample kit.

 Bottle No:
 PD-0001-1-1-01

 Bottle No:
 PD-0001-1-3-01

 Bottle No:
 PD-0001-1-3-03

 Bottle No:
 PD-0001-1-9-01

 Bottle No:
 PD-0001-1-9-03

Did you receive exactly these bottles in the sample kit? (Y/N) H

PgDn (Next page), PgUp (Previous page), Esc (Exit)

If you have pressed "N", indicating that you did not receive exactly what NPSIS assumes you have received, you will be prompted to enter the appropriate information. This information includes pressing a "Y" or a "N" beside each bottle, and entering the bottle number found on the labels of any additional bottles you have received:

```
PD-0001-151
Kit No.:
     Please indicate which bottles you received:
        Bottle No:
                            Received (Y/N)
        .......
      PD-0001-1-1-01
                                PD-0001-1-1-03
                               PD-0001-1-3-01
                                T
      PD-0001-1-3-03
                                Y
      PD-0001-1-9-01
      PD-0001-1-9-03
       Please indicate any additional bottles you received:
 1. Bottle No.: PD-0002-1-1-05
                               2. Bottle No.: PD-0002-2-2-01
 3. Bottle No.: PD-0004-4-4-01
                               4. Bottle No.: - - - -
 5. Bottle No.:
                      . . .
                                6. Bottle No.:
 7. Bottle No.:
                                8. Bottle No.:
```

PgDn (Next page), PgUp (Previous page), use ♠ dor ← to select field.

Notice that the user has indicated that he did not receive the first two bottles on the list. Also note that the user has indicated additional bottles which have come in the sample kit, but which were not on the list.

Next, NPSIS prompts you to indicate if any of the individual bottles have been damaged and rendered unusable for analysis:

Was there any damage to the sample Bottles? (Y/N) Y

PgDn (Next page), PgUp (Previous page), Esc (Exit)

In order to complete the appropriate information on damaged samples, you must first press a "Y" or a "N" in the field labeled "Damaged Y/N". If you have entered a "Y" in this field, you must then identify what the cause of the damage is, to the best of your abilities. As noted on the computer screen below, the "Other" category should be used if the sample is unusable but is not broken. Please try to comment whenever possible.

Kit No.: PD-0001-151 Please indicate which bottles are damaged by entering Y or N, and for those which are damaged, indicate the cause of damage. --- C A U S E ---Bottle No: Damaged Broken Other Comment (Y/N)(Y/N)(Y/N). PD-0001-1-3-01 PD-0001-1-3-03 H PD-0001-1-9-01 Ħ PD-0001-1-9-03 H PD-0002-1-1-05 PD-0002-2-2-01 Y PD-0004-4-4-01 H

The 'Other' cause category is for reporting contamination of a sample, e.g. contamination noted on the Sample Tracking Form, air bubbles, or other reasons a sample is unusable.

PgDn (Next page), PgUp (Previous page), use ♣ d or ← to select field.

Now you have completed all of the necessary information needed to verify that the proper samples have reached their final destination in usable condition. You may save your kit entry by pressing "Enter". If you wish to cancel your kit entry and try again, press "N" and "Enter". If you wish to view or edit the current kit entry, press "R" and "Enter" and NPSIS will place you back at the beginning of your entry.

By pressing "Enter", you have saved all of the information necessary for a particular sample kit. NPSIS assumes that you will enter more than one kit entry per session. Therefore, you will be placed at the initial "Searching Screen". If you are finished, press "Esc" and you will be returned to the main menu. You can then log off of NPSIS by pressing "Alt" and "Right shift" at the same time. You may also send a memo through the ICF computerized mail system. To do this, cursor down to the second menu choice and press "Enter".

The next two pages of this memo describe how to use the ICF electronic mail system. Note that the password for you is NPS. The mail system software program will prompt you for this password before it will allow access to the system. Also, when you are selecting the recipients of your memo, please press the space bar beside the initials "NPS". This will send your memo to all ICF staff involved in the NPS project. If you wish to send memos to a particular ICF staff member, please call Beth Estrada for the identification number of the desired ICF employee.

ELECTRONIC MAIL

Function

Augment office communications with electronic transfer of notes and files.

Summary

Electronic Mail (E-Mail) allows you to send, receive, read, and subsequently save or discard notes and attached files.

When you power up your workstation you will automatically enter E-Mail if you have received any mail. Enter your password to check your mail, or press <ESC> twice to avoid E-Mail and continue to the Assist main menu.

Instructions

Operation of E-Mail is similar to Lotus 1-2-3. Press the F1 key to receive help at any time during operation. If any more help is needed contact workstation support to receive a manual.

For more information on any feature of electronic mail, use Network Courier's online help or refer to the User's Manual.

Passwords

Your password will be "password" until you change it yourself. Once you have given your password and entered E-Mail, you can change your password by selecting Options, then Password.

Reading Mail

- Select "Read" from your menu. Highlight read, then press <ENTER>.
- Select the note to read:
 - a. Highlight the note (using the arrow keys); and press <ENTER>.
 - B. To save the note, select "Storage", then "Save". Enter the name of the file to which the note should be saved.
- 3. Press <ESC> to select another note.

Writing Mail

- 1. Select "Compose", then "edit".
- 2. Press <ENTER> when the highlight moves to "TO".
- 3. Select the recipients(s):
 - Move the highlight to the first recipient's initials.
 - b. Press the space bar. A small mark will appear.
 - c. Repeat steps a and b for all recipients. Press the space bar twice to "de-select" recipients.

 The small mark will disappear.
 - d. Press <ESC> to cancel the entire list.
- 4. Select the initials of those who will receive copies:
 - a. Press the down arrow to move to "CC".
 - b. Select recipients as instructed above (step 3, a-d)

Writing Mail, continued

- 5. Enter a subject and priority. (optional)
- 6. Select attachments (optional):
 - a. Press <ENTER> and type the path for the document(s).
 - b. Press <ENTER> and select the document(s) to be attached.
 - c. Repeat steps a and b for documents in another directory.
- 7. Enter the text of your message.
- 8. Press <ESC> when finished.
- 9. Select "Transmit" to post the note and attachments.

Quitting the Mail Program

- 1. Press <ESC> from the menu.
- 2. Select "YES".

SAMPLE MANAGEMENT 2/3/88 (Sample preservation requirements and holding times)

- 1. Samples must arrive at the laboratory with ice still remaining in the shipping box. If a sample box arrives at the laboratory without any ice remaining, the laboratory should contact the technical monitor immediately.
- 2. The pH of all samples collected for analyses using either Method 5 or 9 must be tested prior to analyses. If the pH is found to be >4 for Method 5, or >3 for Method 9, the laboratory should contact the technical monitor prior to analysis.
- 3. Except for Method 9, strict adherence to sample and extract maximum holding times (14 days) is required for both primary and secondary column analyses. All analyses should be completed as soon as possible, but under extenuating circumstances, the maximum extract holding time may be extended to 28 days for GC/MS analyses only, if approved by the technical monitor. Method 9 samples must be analyzed within 28 days.
- 4. Water samples are to be disposed of after the 14 day sample holding time has been exceeded, except for Method 9, which is 28 days. Sample extracts must be maintained until disposal is approved by the TSD or OPP Laboratory Coordinator.
- 5. Additional samples will be collected at 10% of the sample sites for spiking at the laboratory to test matrix interferences. These samples are to be spiked at analyte concentrations equal to 2, 10, or 20 times the minimum reportable level for each analyte, except for Method 9. Method 9 spiked samples are to be spiked at 2 or 10 times the reporting limit. Samples collected for the analyte stability studies are to be spiked at 10 times the minimum reportable level for each analyte. ICF will have the spiking level (and mix for those methods with more than one mix) printed on the sample label.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

CINCINNATI OHIO 45268

MEMORANDUM

July 14, 1988 DATE:

SUBJECT: Changes in NPS Laboratory Procedures

David J. Munch, TSD Project Manager DD 702

National Pesticide Survey

TO: NFS Technical Monitors (See below)

The following minor changes in laboratory operations are being made.

1. Spiking Levels (Mathods 1-7)

Currently, selected MPS samples are being spiked at either Level 1 (5 times MRL), Level 2 (10 times MRL), or Level 3 (30 times MRL). In many cases, spiking at Level 3 has created analyte concentrations in samples which exceed the linear range of the instrumentation. Any Level 3 spiked samples currently on hand should be analyzed: however, no further requests will be made to spike samples at level 3.

In order to maintain three spiking levels, a Level 0 (2 times MRL) is being added. Laboratory Control Standards and Time Storage Samples are to continue to be spiked at Level 2 (10 times MRL)

2. Spiking Levels (Method 9)

Currently, sample spiking levels used for Method 9 are, Level 1 (2 times MRL), Level 2 (10 times MRL), and Level 3 (10,000 ug/L). The spiking levels are to remain the same; however, Level 0 will now be 2 times MRL, Level 1 10 times MRL, and Level 3 10,000 ug/L.

3. Data Reporting Format

In order for the data reporting format to match the requirements for reporting suspected NPS analytes observed on the primary column, at a concentration between 1/2 MRL and MRL (see memorandum entitled "Determining and Reporting the Presence of NPS Analytes below the Minimum Reporting Levels and Identifying Unknown Peaks," by Bob Maxey, 6/1/88), further clarification is required. In those cases where the presence of an NPS analyte at a concentration between 1/2 MRL and the MRL is successfully confirmed, the primary and confirmational column data for that analyte should be reported as "-111". In those cases where confirmational analyses are either not required, or the confirmational analyses did not confirm the presence of the analyte, the primary column data for that analyte should be reported as "-222". Please transmit this information to both your contract and referee laboratories, as soon as possible. If you have any questions concerning these items, please let me know.

Addressees ·

- A. Dupuy
- L. Kamphake (TSD)
- C. Madding (TSD)
- R. Maxey (OPP)
- K. Sorrell (TSD)
- R. Thomas (TSD)

cc:

- H. Brass (TSD)
- C. Freebis (CSC)
- A. Kroner (TSD)

Appendix A-4

Guidance for Quality Assurance Project Plans:

Section 10

- Data Reporting Format
- Data Reporting Format Changes
- Data Reporting Codes
- Rapid Reporting System

There are two ways the data is received at the EPA in Cincinnati. The first is on a floppy disk and cover the majority of the data received. The other way is on a hard copy. Data that is received on a hard copy includes method 9 referee data, methods 2, 4 and 5 referee data for the instrument control standards, methods 2, 4, 5 and 7 referee data for the confirmation column, all GCMS data run at the referee laboratories, and miscellaneous samples from all the laboratories.

Due to the different nature of the data transmissions, there are different methods of entering them into the data files. For the data received on a floppy disk, the disk is copied to a master diskette and a backup diskette for each laboratory/method combination as well as by analysis data and instrument control standard data. Its unique name comes from the disk number that is sequentially assigned as the data arrives. These diskettes are checked to guarantee the copy was complete. The data is then uploaded from the PC to the NCC mainframe in North Carolina. It is stored in a partitioned data file by the same scheme used for storing it on diskette (the membername being the sequential disk number). These data files are checked for completeness and accuracy of transfer. The data files are then transferred to the mainframe at Cincinnati, since this is where the processing of the data will be done. They are transferred to partitioned data sets for processing (the membername being the laboratory/method combination). For data received on a hard copy, the data is exitered directly onto the Cincinnati mainframe in the processing data sets. The hard copies are then stored in a file folder by laboratory-/method combination.

Now that the data is in a processing data set it is edited to comply, with the format set-up at the beginning of the survey. This includes deleting extra blank lines, deleting method number in the set number, deleting lab pH when not required, adding dots in blank fields, moving headers and data to the correct column, etc. This also includes changing the spelling of nine analytes (eight were misspelled in the original format given to the laboratories and one which was misspelled by a laboratory in creating the format). Also a line of asterisks was added between samples to help the technical monitors in perusing the data. Once the data was believed to be in an acceptable format, a program was run to determine if the computer would read the right data for the right piece of information. If the data reading check program proved the data to be in the acceptable format, the remaining QA/QC check programs were run on the data. If the data was not in the acceptable format, the data was edited to comply with the correct format.

A hard copy of the data is computer generated and this copy is used to highlight problem data and give to the technical monitor. The output from the QA/QC check programs is used to detect data that did not meet QA/QC criteria as well as check for positives, positives above the rapid reporting limit, and proper mix, sample id, and reporting codes. The hard copy is marked to show any, data discrepancies or special notes. The hard copy is dated and either hand delivered or mailed to the technical monitor.

When the technical monitor finishes reviewing the data, the hard copy is returned with their comments as to the disposition of the data. The data is edited to meet with the critique of the technical monitor, as well as to add any necessary comments and to eliminate the line of asterisks. The data is

then transferred to a finished data set on the Cincinnati mainframe. It resides there until a "batch" of data is defined.

A "batch" of data is an arbitrary group of sample sites broken down by the sampling date. The cut-off date for the end of a "batch" is chosen in such a way as to minimize the amount of work and to report data in complete sets. Once a "batch" is determined, data is transferred to its final resting place. When all of the data from all of the labs resides in this final data set, various check programs are run to ensure the data's quality. These programs include checks on the following: date sampled, date shipped, date received, time sampled, type codes, site numbers, positives, reporting codes, field *pH, stabilizing temperature, and conductivity. If there is any data of suspect quality, it is investigated and appropriately edited. A report is then generated and sent to the respective analytical coordinators for their review and approval.

Once the data is returned from the analytical coordinators, it is edited per their comments. It is then transferred to a data file which contains only finalized field sample data. (This copy is for backup purposes only, in case the ICF file gets lost.) This data file is then copied to the ICF account on the Cincinnati mainframe for their use.

FORMAT FOR NATIONAL PESTICIDE SURVEY (NPS) DATA

LINE	COLUMNS	DESCRIPTION
1	57-64	I_Temp S_Temp Date_Sam Date_Shp Date_Rec Time_Sam Time_Ice METHODS 5 AND 9 ONLY] pH
2	1-6 9-14 17-24 27-34 37-44 47-54 57-64 [FOR	
3	BLANK	
4	1-7	Receipt Condition
5	1-80	enter CONDITION OF SAMPLE UPON RECEIPT AT LABORATORY
6	BLANK	
7	1-6 16-18 21-25 28-35 38-45 48-55 58-63	_
8	48-55 58-63	enter LAB ABBREVIATION enter SET NUMBER

LINE	<u>COLUMNS</u>	DESCRIPTION
9	BLANK	
10	1-4 8-13 16-22 25-31 34-40 43-49 52-60 65-70	Type Spiker Extract Analyst Sam_Vol Ext_Vol Int. Std. % Surr
11	8-13 16-22 25-31 34-40 43-49	enter EXTRACTOR'S INITIALS enter ANALYST'S INITIALS enter VOLUME OF SAMPLE
12	BLANK	
13	1-8	Comments
14	1-80	enter ANY PERTINENT COMMENTS ON SAMPLE AND ANALYSIS
15	BLANK	
16	1-7 29-33 39-45 67-71	Analyte Conc. Analyte Conc.
17?	1-25 28-34 39-63 66-72	enter ANALYTE'S NAME enter CONCENTRATION OR PERCENT RECOVERY enter ANALYTE'S NAME enter CONCENTRATION OR PERCENT RECOVERY

FORMAT FOR NATIONAL PESTICIDE SURVEY (NPS) DATA

LINE	COLUMNS	DESCRIPTION
1	1-6 9-14 17-24 27-34 37-44 47-54 57-64 68-69	Date_Sam Date_Shp Date_Rec Time_Sam
2	37-44 47-54 57-64	enter STABILIZED TEMPERATURE OF WATER
3	BLANK	
4	1-17	Receipt Condition
5	1-80	enter CONDITION OF SAMPLE UPON RECEIPT AT LABORATORY
6	BLANK	
7	1-6 16-18 21-25 28-35 38-45 48-55 58-63	Set # Date_Spk
8	1-13 16-18 21-25 28-35 38-45 48-55 58-63	
9	BLANK	

DATE: September 9, 1988

SUBJECT: Data Reporting Codes

FROM: Christopher Frebis, CSC Statistician

TO: Distribution

The purpose of this memorandum is to discuss the reporting codes used in the National Pesticide Survey. There has been some confusion over these codes as to when and where to use them and their exact meaning.

Table 1 identifies the unique sample types (SAMP - field sample, MELK - method blank, SELK - shipping blank, LCS - lab control standard, and LSS, DTS, HTE, and HTS - spiked field samples — these last three are each a type of time storage sample). Under each unique sample type are the only possible codes that can appear for that sample type. (Note: -555 has been added for the situation where the contract lab sends the extract to the referee lab for GCMS analysis, and the code -222 has been deleted.) There is also a type of decision tree for field samples since they are a little more complicated with three analyses for confirmation and qualitative only analytes.

I hope this memorandum helps to put everyone on similar terms as well as clearing the muddy water. If there are any questions of different scenarios you wish to discuss, please call me at (513) 569-7498.

Distribution: Herb Brass, Technical Support Division

Aubry Dupuy, Environmental Chemistry Laboratory

Carol Madding, Technical Support Division
Bob Maxey, Environmental Chemistry Laboratory

Dave Munch, Technical Support Division Kent Sorrell, Technical Support Division Bob Thomas, Technical Support Division



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY CINCINNATI, OHIO 45268

MEMORANDUM

DATE: April 12, 1988

SUBJECT: NPS Rapid Reporting System

FROM:

David J. Munch, Chemist DJ 970 Drinking Wasser Drinking Water Quality Assessment Branch

TO: NPS Technical Monitors

Jerry Kotas has requested that any confirmed results of health significance be reported as quickly as possible. Therefore, if an analyte listed in the attached tables is observed in the primary analyses, at or above the rapid reporting limit, the following actions should be instituted. For any listed analyte where the rapid reporting level is less than or equal to 1/2 the minimum reporting level (MRL), any occurrence at or above 1/2 the MRL should also be processed as below. (Note: The procedures for determining the occurrence of NPS analytes that may occur below the MRL, and are not listed on the attached tables, have not yet been finalized.)

- The appropriate confirmational analyses (GC/MS for methods 1-3, 6-7, second column for Method 5) should be performed as soon as practical.
- The laboratory should telephone their Technical Monitor, the same 2. day the confirmation is completed.
- The laboratory should immediately document the observed result in 3. a letter to their Technical Monitor.
- As quickly as possible on the day the above telephone call is received from the laboratory, the Technical Monitor should inform their Laboratory Analytical Coordinator of the finding. The Technical Monitor should forward on to the Laboratory Analytical Coordinator the above documentation, with any comments he/she may have concerning the validity of the result.
- The Laboratory Analytical Coordinator should inform Jerry Rotas and the second Analytical Coordinator of the finding by telephone the same day if possible, and in writing after the documentation is received from the Technical Monitor.
- The Analytical Coordinators are to request, through the 6. appropriate Technical Monitors, that all analyses for this sample site be conducted, and reported in writing, as soon as practical.

If you have any questions concerning these procedures, please let Bob Maxey or me know. Also, please pass on this information to your contract and referee laboratories. They will need to have this information in hand prior to their conducting the dry run.

Attachment

Addressees:

- A. Dupuy
- L. Kamphake C. Hadding
- R. Maxey
- R. Sorrell
- R. Thomas

cc:

- J. Kotas
- H. Brass
- A. Kroner
- J. Orme

METHOD #1

ANALYTE	RAPID REPORTING LEVEL
Alachlor	44 ug/L
Ametryn	300 ug/L
Atrazine	35 ug/L
Bromacil	2,500 ug/L
Butylate	700 ug/L
Carboxin	1,000 ug/L
Diphenamid	300 ug/L
Fenaniphos	5.0 ug/L
Hexazinone	1,050 ug/L
Metolachlor	300 ug/L
Metribuzin	250 ug/L
Propazine	500 ug/L
Simazine	50 ug/L
Tebuthiuron	125 ug/L
Terbacil	250 ug/L

METHOD #2

ANALYTE	RAPID REPORTING LEVEL
alpha-Chlordane	0.5 ug/L
gamma-Chlordane	0.5 ug/L
Chlorothalonil	150 ug/L
Dacthal (DCPA)	5,000 ug/L
Dieldrin	0.5 ug/L
Propachlor	130 ug/L
Trifluralin	25 ug/L

HETHOD #3

ANALYTE	RAPID REPORTING LEVEL
Acifluorfen	130 ug/L
Bentazon	87.5 ug/L
2,4-D	100 ug/L
Dalapon	800 ug/L
Dicamba	13 ug/L
Dinoseb	3.5 ug/L
Pentachlorophenol	300 ug/L
Picloran	700 ug/L
2,4,5-T	105 ug/L
2,4,5-TP	70 ug/L

METHOD 44

ANALYTE	RAPID REPORTING LEVEL
Cyanazine	13 ug/L
Diuron	70 ug/L
Fluometuron	438 ug/L
Prophan	595 ug/L

METHOD #5

ANALYTE	RAPID REPORTING LEVEL
Aldicarb	10 ug/L
Baygon	40 ug/L
Carbaryl	1,000 ug/L
Carbofuran	50 ug/L
Methomyl	250 ug/L
Oxamyl	175 ug/L

METHOD #6

ANALYTE

RAPID REPORTING LEVEL

ethylene thiourea

1.05 ug/L

HETHOD #7

ANALYTE	RAPID REPORTING LEVEL
dibromochloropropane	2.5 ug/L
1,2-dichloropropane	56 ug/L
cis/trans 1,3-dichloropropene	11 ug/L
ethylene dibromide	0.04 ug/L

METHOD #9

ANALYTE RAPID REPORTING LEVEL

Nitrate/Nitrite 10,000 ug/L

Appendix A-5

Guidance for Quality Assurance Project Plans:

Section 11

Laboratory QC Requirements (including time-storage)

LABORATORY QC REQUIREMENTS FOR PRIMARY ANALYSES GENERAL INFORMATION 2/3/88

- 1. Laboratory control standard mixes, which together contain all method analytes, will be analyzed with each set of samples, (except Methods 5, 7, and 9).
- 2. A set of samples is defined as all samples, blanks, spiked samples, etc., which are extracted at the same time, or for methods 5, 7, and 9, which are analyzed by the same person within a 12 hour period.
- 3. The internal standard area checks detailed in Methods 1-6 will be used as stated. However, the control limits will be reassessed following completion of the initial demonstration of capabilities.
- 4. The measurement system is to be evaluated whenever any analyte is observed in a method blank, at a concentration greater than or equal to 1/2 the minimum reportable level (MRL). Method blanks are to be analyzed with each set of samples.
- 5. The criteria for monitoring instrument control standards will be utilized as stated in the methods, for Methods 1-6.
- 6. Surrogate recoveries (Methods 1-4, 6-7) will be required to be within +/- 30% of the mean recovery determined for that surrogate during the initial demonstration of capabilities.
- 7. Time storage samples must be extracted within +/- 4 days of the proper date, and analyzed within 4 days of extraction. For example, non-stored time storage samples must be spiked within the 14 day holding time for samples, and must be analyzed within 4 days of extraction. A stored time storage sample, must be analyzed within 4 days of extraction. A stored time storage sample must be extracted by no sooner than 10 days and no later than 16 days after being spiked, and must be analyzed within 4 days of being extracted. Samples will be spiked at 10x MRL.
- 8. The requirements for monitoring calibration standard responses will be followed as written in the methods.
- 9. Samples failing any QC criteria must be reanalyzed at the contractors expense.
- 10. Only qualitative analyses will be required for Chloramben, Diazinon, Disulfoton, Disulfoton sulfone, Disulfoton sulfoxide, Endosulfan I, Endosulfan II, Metribuzin DADK, Metribuzin DK, Prometon, Pronamide, and Terbufos. While these analytes are to be analyzed in at least one of the concentration levels of the calibration standards, they are not subject to any of the QC requirements.

GENERAL INFORMATION (CON'T)

- 11. If any analyte is observed at a concentration greater than the minimum reportable level, during sample analyses using Method 7, the corresponding shipping blank must also be analyzed, and if necessary, confirmed.
- 12. Each time that new calibration standard dilutions are prepared they must be compared to the existing calibration curve, and the observed concentration must agree within +/- 20% of the expected concentration.
- 13. Any deviation from the analytical procedures or QC requirements, must be approved by the appropriate technical monitor, and documented in writing.

LABORATORY QC REQUIREMENTS (CON'T)

SECOND COLUMN CONFIRMATION (Methods 1-7)

- Quantitate by comparison to a calibration standard, which is within +/- 20% of the concentration of the sample determined using the primary column.
- 2. The concentrations determined on the secondary column, must agree within +/- 25% of the result determined on the primary column.
- 3. If the concentration determined on the secondary column does not agree within the limits stated above, the contractor must confer with the technical monitor concerning resolution of the discrepancy.

GC/MS CONFIRMATION (Methods 1-3,6-7)

- 1. The sample is to be compared to a standard prepared at the concentration determined for the sample, on either the primary or secondary column, whichever concentration is the lower.
- 2. If additional sample treatment is performed for GC/MS analysis (blowdown, etc.), the standard and sample must both undergo the same treatment.
- 3. Results of the GC/MS analysis are simply reported as the presence or absence of the analyte.
- 4. The sample extract is to be shipped to one of the referee laboratories (Methods 1, 3, or 6 to BSL, Methods 2, or 7 to TSD) for high resolution GC/MS analysis if confirmation of the analyte is not possible using quadrupole GC/MS due to the concentration of the analyte or if the concentration is equal to or greater than 1/2 the lowest adverse health effect level for that analyte, or if requested by the technical monitor.

Appendix A Date: February 1992 Page 61 of 63

Appendix A-6

Guidance for Revisions to Quality Assurance Project Plans



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D.C. 20460

JUL 7 1987

OFFICE OF PESTICIDES AND TOXIC SUBSTANC

MEMORANDUM

SUBJECT: Addenda to Quality Assurance Plans

FROM:

James Boland, Acting Director James Boland, National Pesticide Survey of Orinking Water Wells

TO: NPS Technical Monitors and Quality Assurance Coordinators

This memorandum is in response to questions on the procedures for revision of Quality Assurance Project Plans. It is anticipated that QA Project Plans will be approved by the QAOs for the Office of Drinking Water and Office of Pesticide Programs and myself during July. The documents should not be revised, instead revisions should be in the form of addenda.

The addenda can be a memorandum from the analytical contractors, coordinators or technical monitors. The memorandum should be specific with regard to the page or section and sentences being revised and the wording of any changes. For instance,

Change Section 12, page 3, second paragraph, third sentence:

"The QAC will..... basis."

"The QAU will audit each analysis set."

Delete Section 12, page 3, second paragraph, third sentence: OT

"The QAC willbasis."

The memorandum should have an approval space at the end such as:

Approved _		
	QAO-ODW	
Approved _		
	QAO-OPP	
Approved _		
	NPS Director	

Addenda will be approved by the Quality Assurance Officers and myself and there should be a prior consensus from all involved on the contents and wording of any addendum. Copies of an addendum once approved will be distributed to the analytical coordinators who will distribute it to the technical monitors. Technical monitors will be responsible for distributing addenda to their contractors. Copies will be retained by the NPS Director and QAOs.

These addenda will be incorporated into the final edited document for publication. This final document incorporating the addenda and ICF editoral and typographical comments will be needed in September for publication by the National Technical Information Service. This procedure should reduce the number of complete documents which need to be generated and the amount of effort required to finalize the laboratory QAPjPs for publication. If further addenda are needed after publication, the form and procedure will be the same unless otherwise notified.

Further revision after September will also take the form of addenda as previously described. Once the Survey QAO is retained, he/she will coordinate the process for approving addenda. If you have any questions, contact one of the QAOs.

cc: M. Gomez-Taylor

E. Leovey

Appendix B Date: February 1992 Page 1 of 12

APPENDIX B LABORATORY AUDIT CHECKLIST

NATIONAL PESTICIDE SURVEY LABORATORY AUDIT CHECKLIST

AUDITOR(S)		LABORATORY				
		ANALYTICAL METHOD				
		PE	RSONS	CON	TACTE	O/TITLE
	CONTRACT NO.					
	REPORT NO.					
SEC	TION I: QA MANAGEMENT SYSTEMS FOR	NPS	ANALY	SES		
	OUESTION		Yes	No	N/A	Comments
ı.	Is the latest copy of the QA Plan available?					
2.	Does the QAPjP contain all the applicable signatures?					
3.	Are personnel familiar with the QAPjP?				-	•
4.	Is the QA function being implement as described in the QAPjP?	ed				
5.	Do internal organization charts sh QA function which operates outside of the technical unit which genera the measurement data?					
6.	Does the QA function located exter to the project review data?	nall	Ly_			
7.	Is a record maintained of internal laboratory audits?					
8.	Does the audit record show that the system and performance audits are conducted as described in the QAPj					

Page ___ of _

	OUESTION	Yes	No	N/A	Comments
9.	Is a system in place for determining that method QC criteria have been met?				
10.	Are failures in method QC documented?				
11.	If failures have occurred, has corrective action been documented?		_		
12.	Have long-term problems been encountered?		_		
13.	If yes, has the problem and subsequent corrective action been documented?		_		
14.	Are control charts being prepared according to the QAPjP?		_		
15.	Are personnel at all levels aware of the recourse within their organization for correcting problems?		_		
16.	does management show visible support for quality assurance?				
17.	Have questions been openly and honestly answered?				

SECTION II: PROJECT MANAGEMENT SYSTEM

	QUESTION	Yes	No	N/A	Comments
1.	Are the individuals currently performing the work the same individuals who were originally assigned to perform the work as described in the QAPjP?		_		
2.	If deviations have occurred, have they been documented?				
3.	Does the line manager allow for quick resolution of problems?				
4.	Is the phone number and address of the project manager current?	_	_		
5.	Have new analysts been adequately prepared for NPS work?				
6.	Does a supervisor review and initial daily logs for content and completeness?			-	
7.	Have monthly reports of laboratory activity been submitted to the technical monitor?				
8.	Are current staffing levels sufficient to meet the needs of the NPS in a timely and efficient manner?		_		
9.	Do laboratory personnel have a copy of the analytical method at their workstation?				
10.	Are SOPs listed in the QAPjP being followed?				
11.	Has sufficient communication occurred between the lab and ICF so that the goals of the project can be met?				

SECTION III. SAMPLE TRACKING SYSTEM

	OUESTION	Yes	No	N/A	Comments
1.	Have samples been assigned a unique control number?				
2.	Can samples be cross-referenced to NPS control numbers?				
3.	Are sample tracking forms properly completed?		_		
4.	Are sample tracking records filed?				
5.	Is the storage facility for samples adequate?				
6.	Are refrigerator/freezer logs for samples, extracts, and standards available and current?		_		
7.	Are samples, extracts, and standards stored in a manner that prevents contamination?		_		
8.	Are procedures developed to alert analysts to the sample receipt schedule?		_		
9.	Is the movement of samples and extracts within the lab documented?				
10.	Are procedures developed for tracking holding times for samples and extracts?	?			
11.	Are time storage samples being analyzed according to the QAPjP?	<u> </u>	_		
12.	Are procedures available for shipping extracts to the referee labs?		_		
13.	Are procedures available for sample disposal?				
14.	Have sample supplies (i.e. cooler, shipping box, and bottles) been				

SECTION IV. SYSTEMS FOR MANAGING AND DOCUMENTING ANALYTICAL OPERATION

	OUESTION	<u>Yes</u>	No	N/A	Comments
1.	Is the following information documented for all reagents used?				
	a. Manufacturer		_		
	b. Date of receipt				
	c. Date opened		_		
	d. Purity				
	e. Lot number		_		
2.	Does documentation exist for standards preparation that uniquely identifies the reagents/solvents used and the method of preparation?		_	-	
3.	Does documentation exist for identification of standard preparer and date of standard preparation?				
4.	Are new standards being prepared at the proper intervals?		_		•
5.	Are calibration standards validated prior to use?		_		·
6.	Are calibration procedures being followed according to the QAPjP?		_		
7.	Do balances have calibration stickers showing date of last certified calibration and date of next scheduled calibration?				
8.	Do balances have logs indicating calibration checks performed in-house?				
9.	Are maintenance logs kept for lab equipment?			_	
10.	Is the analytical method being performed as described in the QAPjP?				
11.	Are deviations to the method documented?		_	_	

Page ___ of _

SECTION V: DATA MANAGEMENT SYSTEMS

	OUESTION	Yes	No	N/A	Comments
1.	Are entries to logbooks signed, dated, and legible:				
2.	Are changes to logs dated and initialed by the person who made them?	_			
3.	Are the required calculations being performed as described in the QAPjP?		_		
4.	If not, have the calculations being used been documented?		_		
5.	Are hard copies of sample preparation records and chromatograms stored in the project files?		_		
6.	Have lab data management systems been validated prior to use?				
7.	Does the data validation staff periodically duplicate the calculations performed by the LIMS?				
8.	Can the instrument on which the analysis was performed by identified from the project files?				
9.	Is data stored in an accessible yet securable area?				
10.	Do the procedures for reporting data follow NPSIS guidelines?		_		
11.	Are the project files checked for completeness?			_	
12.	Does the lab have a document archival system in place?				

SECTION VI: LABORATORY MANAGEMENT SYSTEMS

	OUESTION	<u>Yes</u>	No	N/A	Comments
1.	Is service on instruments readily available?				
2.	Are replacement parts for instruments available?				
3.	Has a contamination free area been provided for trace level work?				
4.	Is the analytical balance located an area free from drafts and rapid temperature changes?	-		44	
5.	Are reagent grade or higher purity chemicals used to prepare standards?				
6.	Is the manufacturer's maintenance manual available?				
7.	Has sufficient laboratory space been allocated to perform all phases of the analytical method?				
8.	Are glassware cleaning procedures adequate?		_		

SECTION VII: FOLLOW-UP ON PREVIOUSLY-IDENTIFIED PROBLEMS

	OUESTION	<u>Yes</u>	No	<u>N/A</u>	Comments
1.	Has a person been designated to follow-up on previously-identified problems?		_		
2.	Has a timeframe been stipulated for resolving problems?				
3.	Does documentation of the resolution of problems exist?				

SECTION VIII: DATA AUDIT

2. Analyst:

3. Date of Analysis:

4. Calculated days from date of extraction:

5. Internal Standard ID:

The more	following information should be confirmed than one sample is tracked, make additionable to the same additionable to the s	ed using laboratory records if ional copies of Section VIII.
A.	SAMPLE RECEIPT INFORMATION	
1.	NPS ID Number:	
2.	Laboratory ID Number:	
3.	Date Sampled:	
4.	Date Received:	
5.	Other Comments:	
в.	EXTRACTION	
1.	Sample Set Number:	
2.	Analyst:	
3.	Date of Extraction:	
4.	Calculated days from date of sampling:	-
5.	Surrogate Solution ID:	
6.	Can surrogate solution preparation be validated?	
7.	Method Blank ID:	
c.	PRIMARY ANALYSIS	
1.	Sample Set Number:	

Page or _	Page		of	
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		Sample ID:
6.	Can internal standard preparation be validated?	
7.	Instrument ID:	
8.	Do records show the instrument calibration was validated per QAPjP, Section 8?	
9.	Do records show all required QC checks for the sample's set were evaluated per QAPjP, Section 11?	
10.	Do records show the course of action taken if any QC checks did not meet criteria per QAPjP, Section 11?	
11.	Were data reduced as described in the QAPjP, Section 10 and 14?	
12.	If an analyte hit was observed, could the qualitative and quantitative results reported for the sample be reproduced using the laboratory data?	
D.	SECONDARY ANALYSIS	
1.	Sample Set Number:	
2.	Analyst:	
3.	Date of Analysis:	
4.	Calculated days from date of extraction:	
5.	Internal Standard ID:	
6.	Can internal standard preparation be validated?	
7.	Instrument ID:	
8.	Do records show the instrument calibration was validated per QAPjP, Section 8?	
9.	Do records show all required QC checks for the sample's set were evaluated per QAPjP, Section 11?	

Page ___ of _

		Sample ID:
10.	Do records show the course of action taken if any QC checks did not meet criteria per QAPjP, Section 11?	
11.	Were data reduced as described in the QAPjP, Section 10 and 14?	
12.	If an analyte hit was observed, could the qualitative and quantitative results reported for the sample be reproduced using the laboratory data?	
E.	CONFIRMATION ANALYSIS BY GC/MS	
1.	Sample Set Number:	
2.	Analyst:	
3.	Date of Analysis:	
4.	Calculated days from date of extraction:	
5.	Internal Standard ID:	
6.	Can internal standard be validated?	
7.	Instrument ID:	
8.	Was the instrument tuned to manufacturers specifications?	
9.	Are instrument operating conditions recorded?	
10.	If an analyte hit was observed, could the qualitative results reported for the sample be reproduced using the laboratory data?	

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APPENDIX C FIELD SAMPLING AUDIT CHECKLIST

NATIONAL PESTICIDE SURVEY FIELD AUDIT CHECKLIST

Audi	tor:		{	Sampling Organi	zation:		
Date	:		F	Field Team Lead	er:		
Site	:		(Sampler:			
Well	ID No.:	:		Interviewer:	· · · · · · · · · · · · · · · · · · ·		
Othe	r Team M	dembers:					
			PRE-F	IELD PREPARATIO	ONS		
	Questio	on			Yes	No	Comments
1.	been re		DWS samp?	sampling locations and the Well ?		_	
2.	san Red	nple househo	old been i Team Lead	the head of the recorded in the der Introduction	Interview		
	ead			des been assign nead of the sam		_	
3.	sample	container k	(it numbe	ducted of the rs for each wel formation Sheet			
ADD I	TIONAL (COMMENTS					
					Report No.	:	
					D		. .

PRE-FIELD PREPARATIONS (Continued)

		0	eport No.: ate: age:		
<u>, , , , , , , , , , , , , , , , , , , </u>					
ן טטא	1 1 ON	AL COMMENTS			
	• •	Dinted time?			
2.		the sampling team plan for and arrive at	the		
l .		sufficient ice (at least one bag per kit ained?	.)		_
	Que	stion	Yes	No	Comments
I.	MORN	ING			
		DWS SITE VISIT			
7.		e all the necessary sampling and inter- wing materials taken to the DWS site?	_	_	
	b.	Was the Federal Express three character code crossed out on the box?		_	
6.	a.	Were old Federal Express bar code labels and airbills removed from the sample con kit and supply coolers when first receiv	ntainer	_	
5.	to del	the local Federal Express Office contact ensure that samples could be picked up by ivered to the office after sampling was pleted?		_	
4 .		an equipment inventory conducted against Supply Kit Equipment Checklist?		_	
	Que	stion	Yes	No	Comments

Question 1. If the head of the sample household was not at the site were attempts made to contact the person or to solicit an applicable substitute? 2. Did the Field Team Leader consult the head of the sample household and introduce the sampling team? 3. Did the selected sampling location represent freshly pumped water, and not from a holding tank? 4. Did the Field Team Leader ask if there was a treatment system? 5. After selecting the well, did the Team Leader complete the well and area sketches using the list on Page 7? 6. Were all applicable portions of the Team
site were attempts made to contact the person or to solicit an applicable substitute? 2. Did the Field Team Leader consult the head of the sample household and introduce the sampling team? 3. Did the selected sampling location represent freshly pumped water, and not from a holding tank? 4. Did the Field Team Leader ask if there was a treatment system? 5. After selecting the well, did the Team Leader complete the well and area sketches using the list on Page 7? 6. Were all applicable portions of the Team
the sample household and introduce the sampling team? 3. Did the selected sampling location represent freshly pumped water, and not from a holding tank? 4. Did the Field Team Leader ask if there was a treatment system? 5. After selecting the well, did the Team Leader complete the well and area sketches using the list on Page 7? 6. Were all applicable portions of the Team
freshly pumped water, and not from a holding tank? Did the Field Team Leader ask if there was a treatment system? After selecting the well, did the Team Leader complete the well and area sketches using the list on Page 7? Were all applicable portions of the Team
treatment system? After selecting the well, did the Team Leader complete the well and area sketches using the list on Page 7? Were all applicable portions of the Team
Leader complete the well and area sketches using the list on Page 7? Were all applicable portions of the Team
· · · · · · · · · · · · · · · · · · ·
Leader Introduction and Well Observation Record completely filled out?
ADDITIONAL COMMENTS

Report	No.:		
Date	:		
Page:	:	 of	

III. DWS WELL SAMPLING PROCEDURES

A. WELL PURGING Question Yes Comments No 1. Was a port or tap available for sampling ahead of any pre-treatment and specific to the well selected? b. If not, was the ICF Hotline called before samples were collected? 2. a. Was the air temperature measured and recorded on the DWS Well Purging Parameters Record provided in the field logbook? b. Was a dry temperature probe used for the measurement? a. Were the measurements for pH, temperature, and conductivity of the $T_{\rm O}$ sample taken within 25 seconds after submersion into the sample? b. Was the water gently stirred, but readings taken with the water and meters at rest? Were the measurement recorded on the Well Purging Parameters Record as To? Was the reading obtained on the conductivity meter multiplied by 10 and reported as ppm? ____ 4. Was the water allowed to run an additional five minutes before extracting another sample for the measurements of pH, temperature and conductivity? b. Were the results recorded on the DWS Well Purging Parameters Record as T_5 ? ADDITIONAL COMMENTS

Report No.: ____

Page: _____ of

Date: ____

III.	DWS	WELL SAMPLING PROCEDURES (Continued)			
	Α.	WELL PURGING			
	Ques	stion	Yes	No	Comments
5.	a.	Was the water allowed to run an additional five minutes before extracting another same for the measurements of pH, temperature are conductivity and the results recorded on the DWS Well Purging Parameters Record as T_{10} ?	iple id :he	_	
	b.	Were the readings checked for stability?			
	с.	If readings were not stable, was the well checked at five minute intervals until two consecutive samples produced stable readings or 30 minutes had elapsed?			
	d.	Were the criteria for stability properly applied?	_	-	
	В.	SAMPLE COLLECTION			
	Ques	stion	Yes	No	Comments
1.	Was	only one sample kit worked with at a time?			
2.	bott	e the kits checked for broken bottles, tle labels, and were labels checked against sample numbers shown on the Sample Trackin n?		_	
3.	Were	e samples collected from plastic or rubber es?		_	
ADDI	TION	AL COMMENTS			
			· · · · · · · · · · · · · · · · · · ·		
		Dat			of

III.	DW:	S WELL SAMPLING PROCEDURES (Continued)				
	В.	SAMPLE COLLECTION				
	Que	stion	`	⁄es	No	Comments
4.	App ¹	licable to kits designated ESE.				
	a.	Was the shipping blank bottle label initialed, dated, and the time recorded with a waterproof pen?	_	 -		
	b.	Was the shipping blank placed back into the kit without opening it?	-			
5.	boti	collection of samples using the 1-liter tles, were the following conditions erved:				
	a.	Initialing, dating and recording of tim on the bottle label?	e -			
	b.	Keeping the bottle cap in hand to avoid contamination?	_		_	
	c.	Sampling close to the tap without touch ing the spout?	-		_	
	d.	Preservative not lost because of rinsin overfilling or spillage?	g, -		_	
	e.	Filling the bottle slightly above the 1000 mL mark?	_		_	
	f.	Replacing of cap to seal the bottle?	-			
	g.	Recording of the date and time of sampland the sampler's initials on the Samplaracking Form?			_	
ADD I 1	IONA	AL COMMENTS				
			····			
			Report Date: Page:	No.:	of	

III.	DW:	S WELL SAMPLING PROCEDURES (Continued)			
	В.	SAMPLE COLLECTION			
	Ques	stion	Yes	No	Comments
5.	h.	Placing each filled bottle back into the sample container kit before taking the nobottle out?	ext —	_	
6.		collection of samples using the 250 mL tles, were the following conditions obser	ved:		
	a.	Initialing, dating, and recording of time the bottle label?	e on		
	b.	Keeping the bottle cap in hand to avoid contamination?			
	с.	Adding water to the bottle until almost full?			
	d.	Gentle tapping of bottles with fingers to bring air bubbles to the surface?	o 	_	
	e.	Use of bottle cap to complete filling; creation of convex meniscus prior to sealing the bottle?			
	f.	Sealing bottle so that the teflon side of the septum is in contact with the sample		_	-
	g.	Inversion of the bottles and tapping fire to check for air bubbles?	mly		·
	h.	Refilling of bottle, if necessary, to eliminate air bubbles?	-	_	
ADD I 1	TION/	AL COMMENTS			
		D.	ate:		
		Y-	age:		

		•			
III.	DW:	S WELL SAMPLING PROCEDURES (Continued)			
	В.	SAMPLE COLLECTION			
	Que	stion	Yes	No	Comments
6.	i.	Recording of the date and time of sampling and the sampler's initials on the Sample Tracking Form?	, 	_	
	j.	Placing each filled bottle back into the sample container kit before taking out the next bottle?			
7.	a.	Initialing, dating and recording of time on the bottle label?			
	b.	Keeping the bottle cap in hand to avoid contamination?		_	
	c.	Filling the bottle to the start of the neck?		_	
	d.	Replacing the cap to seal the bottle?			
	e.	Recording of the date and time of sampling and the sampler's initials on the Sample Tracking Form?			
	f.	Placing each filled bottle back into the sample container kit before taking out the next bottle?		_	
8.	bott	collection of samples using the 60 mL tles, were the following conditions erved:			
	a.	Initialing, dating and recording of time on the bottle label?		_	***************************************
	b.	Keeping the bottle cap in hand to avoid contamination?			
ADDIT	[ION/	AL COMMENTS			
					
		Repo	rt No.:		
					F
		, age	• ——		

III. DWS WELL SAMPLING PROCEDURES (Continued) B. SAMPLE COLLECTION Question Yes No Comments c. Adding water to the bottle until almost 8. full? d. Gentle tapping of bottles with fingers to bring air bubbles to the surface? e. Use of bottle cap to complete filling; creation of convex meniscus prior to sealing of bottle? f. Sealing bottle so that the teflon side of the septum is in contact with the sample? Inversion of the bottles and tapping firmly to check for air bubbles? h. Refilling of bottle, if necessary, to eliminate air bubbles? i. Recording of the date and time of sampling, and the sampler's initials on the Sample Tracking Form? Placing each filled bottle back into the sample container kit before taking out the next bottle? 9. Were any bottles overfilled during sampling? If any bottles were overfilled, were they noted on the Sample Tracking Form? Were any bottle caps or septa dropped during the course of sampling? ADDITIONAL COMMENTS

Report No.: __

____of

Date: _

Page: _

III. DWS WELL SAMPLING PROCEDURES (Continued)

B. SAMPLE COLLECTION

	Question	Yes	No	Comments
12.	If item #11 was checked yes, were the caps and septa rinsed with well water prior to placemen on the sample bottle?	t	_	
13.	If item #11 was checked yes, was the event not on the Sample Tracking Form?	ed	_	
14.	After all bottles in the kit were filled, did sample team members check the Sample Tracking Form to ensure all samples had been collected?		_	
15.	Did the team member who completed the Sample Tracking Form sign their name in the space provided on the form?			***************************************
16.	Was the white copy of the Sample Tracking Form placed in its original ziplock bag, which is taped to the sample kit lid?			
17.	Was the pink copy of the Form placed in the pocket of the Field Logbook?		_	
18.	Was ice placed around the sample bottles for each kit by first placing the small plastic bag enclosed in each sample container into the open spaces of the kit, then placing the ice into the bag to fill the sample kit?			***************************************
19.	a. Was the sample kit sealed at this time?			
	b. If yes, will the kit be delivered to Federal Express the same day?	-	_	
20.	Was a final pH, temperature and conductivity reading taken after all sample container kits had been completed?		_	
ADDI	TIONAL COMMENTS			
		ort No. e:	·	
	Page		0	f

III.	DWS WELL SAMPLING PROCEDURES (Continued)			
	B. SAMPLE COLLECTION			
	Question	Yes	No	Comments
21.	Was the water run continuously during the time purging and sampling?			
22.	Was excess water collected and/or directed to a drain to prevent making a mess?		_	
	C. AFTER SAMPLE COLLECTION			
	Question	Yes	No	Comments
1.	a. Was additional ice added as needed to the kits after the sampling activities?b. Was the neck of the inner bag twisted and taped so that leaks would not occur?		_	
2.	Was the Federal Express airbill removed from inside the ziplock bag and kept separate to avoid confusion with other airbills?		_	
3.	Did the sampler properly replace the styrofoam inserts and the sample container kit lid?			
4.	Was there a plastic bag without holes or tears between the kit and the cardboard box?		_	
5.	Were the ends of the plastic bag twisted together to form a neck which was taped at the base?		_	•
6.	Was the plastic bag end folded and taped again, or taped down onto the kit?		_	
ADDIT	FIONAL COMMENTS			
			· <u>·····</u>	
	Date	:		
	Page	:	of	

III.	DWS WELL SAMPLING PROCEDURES (Continued)			
	C. AFTER SAMPLE COLLECTION			
	Question	Yes	No	Comments
7.	Was the box taped shut, being careful not to cover the labels located on the two opposite sides of the box?			
8.	Was the pre-addressed Federal Express airbill placed in the clear adhesive pouch on the appropriate box?		_	
9.	Was Part A of the Final Community Water System Checklist completed?		_	
10.	Was the Community Water System Well Observation Record completed and checked?			
11.	Was the site policed for trash prior to leaving?		_	
	D. SHIPMENT OF SAMPLES TO LABORATORIES			
	Question	Yes	No	Comments
1.	a. Did Federal Express pick up the samples?			
	b. Were the samples taken to a Federal Express office?		_	
2.	Were samples relinquished to a Federal Express agent?			
3.	Was a copy of each airbill collected from the Federal Express agent and placed in the Field Logbook?			
ADDIT	IONAL COMMENTS			
		t No.:		
			_ of	

III.	DW:	S WELL SAMPLING PROCEDURES (Continued)			
	D.	SHIPMENT OF SAMPLES TO LABORATORIES			
	Que	stion	Yes	No	Comments
4.	Fede	unused material shipped back to ICF by eral Express "Standard Air" two-day oment?			
5.	Was	the Final DWS Checklist, Part C, complete	ed?		
6.	info	the sampling team call the NPS Hotline to orm the Sample Tracking System that kits been sent to the laboratory?	· —	_	
7.	West	e logbooks/questionnaires either sent to tat or plans made to send them after the al Area Interview?	_	_	
		COMPLETION OF DWS QUESTIONNA	AIRE		
	Ques	stion	Yes	No	Comments
1.		Was the DWS Questionnaire administered to head of the sample household?	·		
	b.	If the answer to item #1 is no, then list to whom it was administered.	: 	_	
2.	a.	Was the DWS Questionnaire administered du the time that samples were collected?	uring ——		
	b.	If no, then list when the interview occurred.			
	c.	Did the interview take place in an appropriate setting?		_	
ADD I T	T I ON/	AL COMMENTS			
					
		Re Da Pa	eport No.: ate:	of	

COMPLETION OF DWS QUESTIONNAIRE (Continued)

	Question	Yes	No	Comments
3.	Are the name(s) of all respondents supplied on the Interview Record of Contacts?			
4.	Was the respondent's name correctly printed on Page 1 of the questionnaire?		_	
5.	Was the respondent's telephone number recorded in appropriate boxes?			
6.	Were all applicable questions answered in Section A of the questionnaire?			
7.	Were all applicable questions answered in Section B of the questionnaire?	_	_	
8.	Were all applicable questions answered in Section C of the questionnaire?	_		
9.	Were all applicable questions answered in Sect D of the questionnaire?	i on		
10.	Did the interviewer check the appropriate slot if drilling records or inspection documents were used to obtain information in Section D?	-	_	
11.	Were all written comments and responses legible?		_	
12.	a. Were all responses correctly coded?		_	
	b. Were prompted responses marked with an (X)?	? —	-	
	c. Were questions re-read or clarified so that bias was not introduced?		_	
ADDI	TIONAL COMMENTS			
		-		
	0	No		
	Date			
	Page	:	of	

COMPLETION OF DWS QUESTIONNAIRE (Continued) Ouestion Yes No Comments 12. d. Was the interviewer always in control of the situation? Did the interviewer act in a professional but courteous manner? f. Did the respondent become enraged, upset, or annoyed during the interview? q. Did the interviewer use exact NPS definitions and not introduce their own definitions or opinions? 13. Was evidence of erasures present in the completed questionnaire? 14. Did the interviewer use the following criteria for making corrections to responses: a. Draw a line in blue pencil through the original answer. b. Write the corrected answer in blue pencil above or beside the original response. Did the interviewer examine the completed forms and questionnaires prior to leaving the site to ensure no questions were accidentally skipped? Did the Field Team Leader review all completed questionnaires and forms prior to leaving the site? ADDITIONAL COMMENTS

Report No.: Date:		
Page:	of	

APPENDIX D
GENERAL NPS AUDIT CHECKLIST

NATIONAL PESTICIDE SURVEY GENERAL AUDIT CHECKLIST

DATEQAPjI			jP					
AUDITOR(S)ORGA			ION					
	PER	RSONS C	ONTA	CTED/	TITLE			
CONT	RACT NO.							
REPO	RT NO	 						
SECT	ION I: QA MANAGEMENT SYSTEMS FOR NPS A	CTIVIT	IES		•			
	Question	Yes	No	N/A	Comments			
1.	Is the latest copy of the QA Plan available?							
2.	Does the QAPjP contain all the applicable signatures?							
3.	Are personnel familiar with the QAPjP?							
4.	Is the QA function being implemented as described in the QAPJP?	·						
5.	Do internal organization charts show QA function which operates outside of the technical unit which generates the measurement data?							
6.	Does the QA function located externall to the project review data?	Ly		 .				
7.	Is a record maintained of internal audits?		_					
8.	Does the audit record show that the system and performance audits are conducted as described in the QAPjP?							

	Question	 _	Yes	ИO	N/A	
9.	Is a system in place for determining that protocol ahs been followed?	 				
10.	Are failures in protocol documented?	 				
11.	If failures have occurred, has corrective action been documented?	 				
12.	Have long-term problems been encountered?	 				
13.	If yes, has the problem and subsequent corrective action been documented?	 				
1 4 .	Are personnel at all levels aware of the recourse within their organization for correcting problems?	 				
15.	Does management show visible support for quality assurance?	 _				
16.	Have questions been openly and honestly answered?					

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-

SECTION II: PROJECT MANAGEMENT SYSTEM

	Question	<u>Yes</u>	No	N/A	Comments
1.	Are the individuals currently performing the work the same individuals who were originally assigned to perform the work as described in the QAPjP?		_		
2.	If deviations have occurred, have they been documented?				
3.	Does the line manager allow for quick resolution of problems?		_		
4.	Is the phone number and address of the project manager current?				
5.	Have new employees been adequately prepared for NPS work?				
6.	Does a supervisor review and initial daily logs for content and completeness?		_		
7.	Have monthly reports been submitted to the technical monitor?				
8.	Are current staffing levels sufficient to meet the needs of the NPS in a timely and efficient manner?				
9.	Do personnel have a copy of the appropriate SOP at their workstation?				
10.	Are SOPs listed in the QAPJP being followed?				

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SECTION III. DATA MANAGEMENT SYSTEMS

_	<u> </u>	Yes	No	N/A	Comments
1.	Has a document filing system been established?				
3.	Are written procedures available for storage and retrieval of files?				
4.	Are files stored in an accessable yet securable area?		_		
5.	Is the storage facility for files adequate?				
6.	Have standards been established for contents of the file?				
7.	Are the files checked for completeness against the standard?				
8.	Have data management systems been validated prior to use?		_		
9 .	Are computer system checks performed and documented?				
10.	Are software packages documented?				
11.	Is routine maintenance being performed on the computer systems?				
12.	Is service readily available for computer hardware and software?				