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ESC/DAYTON (Department/Section)

ANALYTICAL (Type of Report)

REPORT NO.: MDA-567

JOB/PROJECT NO.: 303 (484.2430)

DATE: January 11, 1985

PERIOD COVERED:

TITLE: ANALYSIS OF SCRAPING SAMPLES FOR AROCHLOR FORMULATIONS

AUTHORS: B. N. Hughes

ABSTRACT: Scraping samples from the Queeny plant were analyzed for possible concentrations of Aroclor formulations. The results of these analyses are shown in this report.

TECHNICAL APPROVAL: P. L. Sherman
Sr. Research Group Leader

APPROVED BY: R. F. Ivory
Environmental Sciences Center
Service Project Manager

APPROVAL DATE: 1-11-85

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REPORT NO.: MDA-567
COPY NO.: 15
AUTHORS: B. N. Hughes
TITLE: ANALYSIS OF SCRAPING SAMPLES FOR AROCLOR FORMULATIONS

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H. A. Woltermann

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ACKNOWLEDGEMENT

Contributions of the following Dayton Laboratory personnel to this project are gratefully acknowledged:

Analyses: B. M. Hughes
D. McKenzie
C. Trang
B. Williams

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ANALYSIS REPORT

DATE: January 9, 1984

LOG NUMBER: 1-84-12-12-03

PROJECT NO: 760.18-30318
(484.24318 - previous No.)

TO: Queeny Plant/Ivory

Report sent Federal Express to:
Mr. Thomas R. Alvarez
Monsanto Co./Queeny Plant
1700 S. Second St.
St. Louis, Mo. 63177

FROM:

Environmental Sciences Center
Monsanto Company/Dayton Laboratory
1515 Nicholas Road
Dayton, Ohio 45418

Phone No: (513) 268-3411

Title: Analysis of Aroclor Formulations in Scraping Samples

Standard Sampling Method No(s): Samples supplied by customer.

Standard Extraction Method No(s): PCEX21

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A30318

Table File No(s): B30318, C30318

Enclosed are results and documentation for the analysis of 46 scraping samples for Aroclor formulations. Table 1 shows the results of Aroclor analyses and Table 2 shows quality control data for low and high native spikes of a clean soil sample. As described in Standard Extraction Method PCEX21 (shown in a later section of this report), each sample was spiked with two surrogate compounds. Recoveries of these two compounds (Cl5-benzene and 2,4,6-Cl3-biphenyl) are also shown in Tables 1 and 2.

Also enclosed in this report are brief descriptions of the extraction and analysis methods, sample login and custody information, and a copy of the ESC/MAC Good Laboratory Practices Manual. If you have questions concerning this report, please feel free to call.

ANALYST(S): B. Mason-Hughes, C. Trang, D. Mckenzie and Barbara Williams

ANALYST APPROVAL: B. Mason Hughes

TABLE [illegible] Summary of PCB Concentrations in Queeny Scrubber Samples.

RSC Project No: 760.18 - 30318 Date: January 9, 1985

Sample I.D.	Surrogate Recoveries(%Recovery)		PCB Concentration (ug/g)				
	CL5-benzene	2,4,6-CL3-biphenyl	A1248	A1254	A1260	A1262	Total
PCB101	83	93	15.0	-	-	160.7	175.7
PCB102	89	88	17.6	-	-	53.0	70.6
PCB102(DUP)	91	94	17.3	-	-	43.3	60.6
PCB103	87	89	69.0	172.3	-	87.3	328.6
PCB104	93	95	10.0	-	-	11.2	21.2
PCB105	103	98	10.0	-	-	16.6	26.6
PCB106	87	90	31.2	-	-	76.7	107.9
PCB107	94	99	70.3	-	-	65.0	135.3
PCB108	86	90	10.0	-	-	11.9	21.9
PCB109	87	95	19.6	-	-	109.2	128.8
PCB110	89	92	15.2	-	-	32.4	47.6
PCB111	70	70	125.4	172.0	-	101.9	399.3
Method Blank	103	90	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
PCB112	92	98	12.3	-	-	126.5	138.8
PCB113	84	87	57.8	-	-	11.0	68.8
PCB114	90	89	222.4	-	-	30.1	252.5
PCB115	86	93	812.6	-	-	-	812.6
PCB115(DUP)	84	93	790.0	-	-	-	790.0
PCB116	81	69	16.7	-	-	59.8	76.5
PCB117	111	104	696.6	-	-	-	696.6
PCB118	86	88	15.7	-	-	45.0	60.7
PCB119	80	89	20.7	-	-	38.3	59.0
PCB120	89	84	149.1	-	186.0	-	335.1
PCB121	92	89	146.0	-	93.5	-	239.5
PCB122	79	55	104.3	202.0	-	127.0	433.3
PCB123	87	93	22.7	51.5	-	480.0	554.2
PCB124	73	89	26.4	43.2	-	209.5	279.1
Method Blank	92	82	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
PCB125	85	95	13.1	-	-	171.4	184.5
PCB126	87	89	9.9	-	-	141.2	151.1
PCB127	81	89	10.0	-	-	11.9	21.9
PCB127(DUP)	82	87	10.0	-	-	8.5	18.5
PCB128	89	92	32.6	-	-	30.2	62.8
PCB129	94	101	25.0	-	-	426.0	451.0
PCB130	91	95	265.5	-	-	100.1	365.6
PCB131	86	89	18.7	-	-	266.0	284.7
PCB132	88	89	17.0	-	-	162.1	179.1
Method Blank	97	83	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
PCB133	85	93	-	-	-	21.0	21.0
PCB134	69	80	10.0	23.0	-	9.3	42.3
PCB135	85	86	11.7	-	-	117.0	128.7
PCB136	85	92	72.1	-	-	141.0	213.1
PCB136(DUP)	104	110	96.[illegible]	-	-	147.8	244.3
PCB137	90	80	30.3	-	-	33.2	63.5
PCB138	81	91	89.6	61.4	-	35.0	186.0
Method Blank	98	98	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
PCB139	94	94	198.0	251.8	-	624.8	1075.6

(continued)

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TABLE [illegible] (Continued)

PCB140	86	93	20.6	-	-	88.0	109.4
PCB141	86	91	24.0	14.4	-	18.5	56.9
PCB141(DUP)	93	89	22.3	-	-	73.6	95.9
PCB142	93	92	-	-	2.0	-	2.0
PCB143	94	95	19.0	-	-	141.0	160.0
PCB144	86	89	15.2	-	-	87.8	103.0
PCB145	95	95	-	-	-	30.3	30.3
PCB146	89	92	10.1	-	-	13.5	31.6
Method Blank	89	76	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
Solvent Blank	99	91	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)

Footnotes:

a. ND= None Detected (<1.0 ug/g)

TABLE FILE NO: B30318:MM

TABLE 2. Summary of Aroclor 1254 Spiking Concentrations and Recoveries in Quality Control Experiments for Ouseny Scraping Samples.

BSC Project No: 760.10 - 30310 Date: January 9, 1985

Sample I.D.	Surrogate Recoveries (%Recovery)		Aroclor 1254	
	CL5-benzene	2,4,6-CL3-Biphenyl	Amount Added (ug/g)	Recovery (%)
Blank-LS(PCB1105)	90	81	0.84	107
Blank-HS(PCB1106)	97	85	9.90	91
Blank-LS(PCB1122)	106	92	0.62	62
Blank-HS(PCB1123)	93	87	8.20	121
Blank-LS(PCB1133)	92	83	0.84	118
Blank-HS(PCB1134)	96	84	9.90	105
Blank-LS(PCB1143)	91	78	0.92	129
Blank-HS(PCB1144)	90	77	9.69	102
Blank-LS(PCB1154)	93	81	0.71	100
Blank-HS(PCB1155)	99	86	9.40	106

TABLE FILE NO: C30310:MH

The following pages describe Standard Extraction Methods and Standard Analysis Methods used for this project.

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Non-EC Active Matrices

ESC Standard Extraction Method No: PCEX21

MATRIX(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Soils and other non-EC active solids and solvents	0.5 - 5.0	10

TYPICAL ANALYTES: Aroclors 1232, 1242, 1248, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers

ESC Standard Analysis Method Nos: ECAM01, ECAM02

INTERFERENCES:	TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers that is non acid hydrolyzable	pentachlorobenzene	50 - 120
	2,4,6-trichlorobiphenyl	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson benzene or hexane for sample extraction or dilution.
Pierce vials.
Sulfuric acid at varying concentrations.
Ultrasonic bath.
Disposable glassware.
(See flow sheet for this extraction method in PCFS21.)

LIMITATIONS: Method applies only to matrices which do not contain interfering electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND UPDATES: 12/17/84

FLOW SHEET PCF821

FOR ESC EXTRACTION METHOD PCEX21

SAMPLE ID: ---------- MRC EXTRACT # ----------

---------- LOG NO: ----------

EXTRACTION DATE and TIME: ----------

SAMPLE COLOR AND DESCRIPTION ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[ ]	SAMPLE WEIGHT----------
SURROGATE SPIKE OF SAMPLE	[ ]	----------

ADD 10.0 mL BENZENE TO SAMPLE.	[ ]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 2 mL OF THE EXTRACT INTO A 7 mL VIAL	[ ]	
ADD 2 mL ------ SULFURIC ACID AND SHAKE	[ ]	
ALLOW PHASES TO SEPARATE REMOVE 1 mL FOR ANALYSIS.	[ ]	10.0 mL FINAL VOLUME ----------

EXTRACT NO: ----------

DATE/TIME COMPLETED ----------

ANALYST: ---------- --

ESC JOB NO: ----------

MONS 023180

ESC Standard Analysis Method (SAM) | ESC Standard Analysis Method No:
TITLE: Aroclor Formulations in Solvents | ESCA002

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5860 Capillary GC/EC interfaced to an HP-3357-LAS Laboratory Data System

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Aroclor formulations 1232, 1242, 1248, 1254, 1260, and 1262	0.1	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl through octachlorobiphenyl isomers are potential interferences. Exact retention times of most likely PCB isomers can be obtained from the analysis of Aroclor 1248, Aroclor 1254 or Aroclor 1260 formulations.

CHROMATOGRAPHIC COLUMN: 30-meter fused silica J&W DB-5 (0.25 uM) with wide bore (0.32mm).

COLUMN TEMPERATURE PROGRAM: 130(1)/4/300 | INJECTION TYPE: Splitless

CALIBRATION AND STANDARDIZATION: Use internal standard quantitation technique and calibrate instrument response factors from authentic Aroclor formulation standards. Use a minimum of 10 PCB isomers for the determination of formulation identity and quantity.

CALCULATIONS: Formulation concentration =
(isomer RRF)(isomer area/internal standard area)

where: RRF is the relative response factor for the appropriate PCB isomer in a given formulation. Formulation concentration is obtained from the average of at least 10 such calculations.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND METHOD UPDATES: 10/25/83, 2/3/84

MONS 023181

The following pages describe Analytical Request Information generated for this project.

ANALYSIS REQUEST

DATE: 12-12-84 | REQ. NO.: 1-84-12-12-03

[illegible] RESEARCH CORPORATION
DAYTON LABORATORY

REQUESTER**: Queen

CHARGE NO.**: 184.27 [illegible]

REPORT RESULTS TO**: FILES/ANALYSIS: Meel, Ivory, Sherman

SAMPLE DESCRIPTION | NUMBER OF BOTTLES: 46 | NUMBER OF SAMPLES ** 46

QNY-PCB-101	QNY-PCB-115	QNY-PCB-130
102	116	131
103	117	132
104	118	133
105	119	134
106	120	135
107	121	136
108	122	137
109	123	138
110	124	139
111	125	140
112	126	141
113	127	142
114	128	143
	129	145
		146

RECORDED BY: R F Ivory

ANALYSIS REQUEST TO FILES: M Hughes, Ivory, Sherman

SAMPLES FOR: M. Hughes

SAMPLE LOCATION: Lab 21

REQUIRED ANALYSIS**

C. PCB

KNOWN HEALTH HAZARD PRESENTED BY SAMPLE: **

REFERENCE TO PREVIOUS WORK (REQUEST #, JOURNAL, ETC.) **

COMPLETION DATE _____

ANALYTICAL TECHNIQUE **

- ☐ GAS CHROMATOGRAPHY
- ☐ ULTRAVIOLET
- ☐ VISIBLE
- ☐ INFRARED
- ☐ EMISSION
- ☐ EDAX XRF
- ☐ MICROSCOPY
- ☐ OTHER ____
- ☐ NMR
- ☐ HPLC
- ☐ XRD
- ☐ ICP
- ☐ ATOMIC ABSORPTION
- ☐ MASS SPEC
- ☒ GC/MS
- ☐ AUTO ANALYZER

QUAL. ____ SEMIQUANT. ____ QUANT. ____

TIME ALLOTED FOR ANALYSES

ANALYST	HOURS	INST. TIME

REQUIRED QA/QC

**SAMPLE DISPOSAL: AFTER REPORT COMPLETE ☐ DISCARD ☐ RETURN HOLD FOR ☒ 30 DAYS ☐ 60 DAYS ____ OTHER

** THIS INFORMATION MUST BE SUPPLIED BY ANALYSIS REQUEST ORIGINATOR

MONS 023183

ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 12/12/84 LOG NO: 1-84-12-12-03

REQUESTER: Queeny PROJECT NO: 484.24318

REPORT RESULTS TO: FILES/Mees, Ivory, and Sherman.

ANALYSIS REQUESTS SENT TO: FILES

SAMPLE PICKUP BY: David McKenzie

SAMPLE LOCATION: lab 28 building 2.

SAMPLE DESCRIPTION FOR 46 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
QNY-PCB-101	1	PCB-1093
QNY-PCB-102	1	PCB-1094,PCB1095-DUP
QNY-PCB-103	1	PCB-1096
QNY-PCB-104	1	PCB-1097
QNY-PCB-105	1	PCB-1098
QNY-PCB-106	1	PCB-1099
QNY-PCB-107	1	PCB-1100
QNY-PCB-108	1	PCB-1101
QNY-PCB-109	1	PCB-1102
QNY-PCB-110	1	PCB-1103
QNY-PCB-111	1	PCB-1104
QNY-PCB-112	1	PCB-1108
QNY-PCB-113	1	PCB-1109
QNY-PCB-114	1	PCB-1110
QNY-PCB-115	1	PCB-1111,PCB-1112-DUP

RECORDED BY: David McKenzie

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES: GC/EC

QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED: 1/15/85

COMMENTS: SEE PAGE 4 FOR QA/QC. (PAGE 1 OF 4) - Report sent to Alvarez on 1/9/85.

HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA2430

ANALYSIS REQUEST

DATE: 12/12/84 LOG NO: 1-84-12-12-03

REQUESTER: Queeny PROJECT NO: 484.24318

REPORT RESULTS TO: FILES/Moss, Ivory, and Sherpen.

ANALYSIS REQUESTS SENT TO: FILES

SAMPLE PICKUP BY: David McKenzie

SAMPLE LOCATION: lab 20 building 2.

SAMPLE DESCRIPTION FOR 46 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
QNY-PCB-116	1	PCB-1113
QNY-PCB-117	1	PCB-1114
QNY-PCB-118	1	PCB-1115
QNY-PCB-119	1	PCB-1116
QNY-PCB-120	1	PCB-1117
QNY-PCB-121	1	PCB-1118
QNY-PCB-122	1	PCB-1119
QNY-PCB-123	1	PCB-1120
QNY-PCB-124	1	PCB-1121
QNY-PCB-125	1	PCB-1125
QNY-PCB-126	1	PCB-1126
QNY-PCB-127	1	PCB-1127,PCB-1128-DUP
QNY-PCB-128	1	PCB-1159
QNY-PCB-129	1	PCB-1129
QNY-PCB-130	1	PCB-1130

RECORDED BY: David McKenzie

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES: GC/EC

QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED: 1/15/84

COMMENTS: SEE PAGE 4 FOR QA/QC. (PAGE 2 OF 4) - Report sent to Alvarez on 1/9/85.

HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA2430

[redacted] ENVIRONMENTAL SCIENCES CENTER [redacted]

ANALYSIS REQUEST

DATE: 12/12/84 LOG NO: [redacted]-12-03

REQUESTER: Queeny PROJECT NO: 484.24318

REPORT RESULTS TO: FILES/Hees, Ivory, and Sherman.

ANALYSIS REQUESTS SENT TO: FILES

SAMPLE PICKUP BY: David McKenzie

SAMPLE LOCATION: lab 28 building 2.

SAMPLE DESCRIPTION FOR 46 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
QNY-PCB-131	1	PCB-1131
QNY-PCB-132	1	PCB-1132
QNY-PCB-133	1	PCB-1136
QNY-PCB-134	1	PCB-1137
QNY-PCB-135	1	PCB-1138
QNY-PCB-136	1	PCB-1139,PCB-1140-DUP
QNY-PCB-137	1	PCB-1141
QNY-PCB-138	1	PCB-1142
QNY-PCB-139	1	PCB-1146
QNY-PCB-140	1	PCB-1147
QNY-PCB-141	1	PCB-1148,PCB-1149-DUP
QNY-PCB-142	1	PCB-1150
QNY-PCB-143	1	PCB-1151
QNY-PCB-144	1	PCB-1158
QNY-PCB-145	1	PCB-1152

RECORDED BY: David McKenzie

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES: GC/EC

QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED: 1/15/84

COMMENTS: SEE PAGE 4 FOR QA/QC. (PAGE 3 OF 4) - Report sent to Alvarez on 1/9/85.

HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA2430

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[redacted] PAGE 4

[redacted]

DATE: 12/12/84 Log no: [redacted]

REQUESTER: Queeny PROJECT NO: 484.2431B

REPORT RESULTS TO: FILES/Mess. Ivory, and Sherman.

ANALYSIS REQUESTS SENT TO: FILES

SAMPLE PICKUP BY: David McKenzie

SAMPLE LOCATION: lab 20 building 2.

SAMPLE DESCRIPTION FOR 46 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
QHY-PCB-146	1	PCB-1153
BLANK SOIL METHOD BLANKS		PCB-1107,PCB-1124 PCB-1122,PCB-1135 PCB-1145
BLANK SOIL + LOW NATIVE SPIKES		PCB-1105,PCB-1122 PCB-1133,PCB-1143 PCB-1154
BLANK SOIL + HIGH NATIVE SPIKES		PCB-1106,PCB-1123 PCB-1134,PCB-1144 PCB-1155
SOLVENT BLANK		PCB-1157

RECORDED BY: David McKenzie

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES: GC/EC

QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED: 1/15/85

COMMENTS: SEE PAGE 4 FOR QA/QC, (PAGE 4 OF 4) - Report sent to Alvarez on 1/9/85.

HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA2430

The following is a copy of the ESC/UAC Good Laboratory Practices Manual.

Copy Number: _____

GOOD LABORATORY PRACTICES MANUAL

A Guide to Quality Control/Quality Assurance

Ultratrace Analysis Group

March 1984

Joseph J. Brooks
Group Leader, Ultratrace Analysis

H. A. Wolternann
Manager, Environmental Sciences Center

Issued to: _____

Date: _____

Environmental Sciences Center
Dayton Laboratory
Dayton, Ohio 45407

JR3/JOB A

MONS 023189

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I. Objectives

The main objectives of the Ultratrace Analysis Group quality assurance/quality control program are: 1) to assure that our laboratory generates high quality results; and 2) to maintain the necessary records that document laboratory performance.

II. Scope

The scope of this manual includes areas of GLP not specifically addressed in the ESC section manual and expands upon certain areas where QA/QC practices differ from those described in the section manual. It is intended to be used along with the section manual to provide a complete picture of the GLP within the Ultratrace Analysis Group.

III. Analysis

The generation of analytical results within the Ultratrace Analysis Group routinely involves two processes: sample preparation and instrumental analysis. Sample preparation involves one or more of a variety of techniques including extraction, dilution, acid/base washes, column chromatography, concentration and numerous other techniques to render the sample in a suitable condition for instrumental analysis. Instrumental analysis takes the suitably prepared sample and introduces it into an analytical instrument to measure the desired parameter(s). Both of these processes can be described by clearly defined and separable methods called sample preparation methods (SPMs) and instrumental analysis methods (IAMs). SPMs and IAMs may be combined in numerous ways (depending upon the nature of the samples and the needs of the customer) to produce a successful analytical protocol. For example, several different SPMs may be used to produce samples that are suitable for analysis by a single IAM. The measures incorporated into each of these experimental processes to ensure that an assessment of the quality of the results can be made constitute the QA/QC program for a project. The following subsections describe the elements of the analysis process:

A. Methods

Methods are the written documentation that describe the analytical process. They contain sufficient detail such that a competent scientist or technician can readily perform the required analyses. The usual practice is to have methods that describe the sample preparation process (SPMs) and methods that describe the instrumental analysis process (IAMs). Each method developed within the Ultratrace Analysis Groups is assigned a method number as described in the ESC Section Manual.

Methods are obtained from various sources including in-house development, modification/adaptation of literature methods, or specific protocol methods dictated by government agencies. Methods may be formally validated by a series of experiments that describe a classical validation protocol or may be less vigorously proven owing to factors such as frequency or use, customer needs and

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cost/timing restrictions. In any case a QA/QC program is applied to the analysis process to provide a measure of the quality of the results. Unless a QA/QC program is prescribed by a required protocol (e.g., EPA Dioxin in Soil protocol), certain minimum QA/QC measures (depending upon the analysis type) are incorporated into all Ultratrace Analysis projects. The following subsection describes the Ultratrace QA/QC program:

b. QA/QC Program

An effective QA/QC program incorporates experiments that provide information relative to each of the following questions:

(1) Are there any impurities, background or contamination that will influence the results?

(2) How reproducible are the results?

(3) How well can the analyte be recovered from the sample matrix?

(4) What is the limit of detection for the analysis?

(5) What is the dynamic range of the analysis?

(6) What is the accuracy of the analysis?

All of these questions are addressed by properly selecting/applying the use of blanks, replicates, spikes and standards in a QA/QC program. The discussion of the use of these QA/QC techniques as they apply to Ultratrace Analysis Group activities is best done by considering the sample preparation and instrumental analysis processes separately.

1. Sample Preparation QA/QC

   a. Method blanks - Representative glassware, solvents and reagents are used with sample matrices which are similar to those for which results are being reported, yet do not contain the analytes of interest. Method blanks are analyzed at a frequency equal to 10% of the analyzed samples for sample sets which are multiples of 10. At least one method blank is analyzed for sample sets containing between 1 and 9 samples.

   b. Replicates - If sufficient sample amounts have been submitted for analysis, replicate frequency is the same as the method blank frequency. If at all possible, replicates should be obtained from the same sample bottle in order to minimize sample inhomogeneity problems due to samples being taken at slightly different times.

   c. Spikes - If sufficient sample is available for each sample type, the sample is split and spiked at two levels with

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the native (unlabeled) analyte(s). The levels are typically 2x (low) and 20x (high) the anticipated detection limit and serve as a technique for estimating the limit of detection for each matrix. These low and high spikes are conducted at a frequency equal to the Method Blank frequency. Obviously spiking with the native analyte cannot be done for widescan analysis since this presupposes knowledge of the identity of the analyte(s) in the sample.

Other spiking techniques are used to evaluate the extraction/recovery efficiencies for the analyte(s) of interest. Ideally extraction/recovery efficiencies are determined for each sample, since, in principle, each sample may represent a different matrix. This can be done very easily when the detection technique is mass spectrometry if an isotopically labeled version of the analyte(s) is available. Documentation of extraction/recovery efficiencies is not required for each sample if the sample matrix for a set of samples is constant. However, for a sample set for which the similarity of samples is not known, and for which adequate sample is available, each sample must be split and spiked with a known amount of the analyte(s) of interest to determine extraction efficiency. If adequate sample size is not available or the identity of the analyte(s) is not known before the fact (i.e., widescan analysis), surrogate spiking compounds may be used to assess extraction efficiencies in each sample without actually spiking each sample with the analyte(s) of interest. The choice of the surrogate spiking compound is tailored for each analyte. This surrogate must be chosen so that it is either chemically identical to the analyte of interest (for example using isotopically labeled analogues of the analytes), or is closely similar in terms of liquid/liquid partition properties, chemical reaction properties, column elution properties or other matrix isolation properties which are used to isolate the analyte(s) from the sample matrix. Another property of the surrogate spiking compound is that it not be present in the sample being analyzed.

2. Instrumental Analysis QA/QC

The final step in obtaining analytical results involves the use of an appropriate instrumental technique to produce the analytical data. Several QA/QC practices must be incorporated in this process to ensure validity of the data.

a. Instrumentation

Careful documentation of instrument usage, calibration, and maintenance is essential for the generation of high quality results. Each major instrument within the Ultratrace Analysis Group will be the assigned responsibility of a professional employee designated as the Instrument Steward. The Instrument Steward is responsible for making sure that logbooks are available and properly completed by instrument users. It is the

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responsibility of each individual using the equipment to record all the pertinent data required in the logbook. An example of a page from a GC/MS logbook is given in Figure 1 and that of a GC/LC logbook is given in Figure 2.

Instruments will be calibrated according to the instrument manufacturers' specifications or according to the method being used with the instrument. A log of instrument calibration will be maintained with the instrument.

The Instrument Steward will be responsible for ensuring that preventative and corrective maintenance programs are carried out on all instruments under his/her responsibility. If a specific problem is noted by an instrument user, it should be noted in the logbook and the Instrument Steward notified so that appropriate corrective action can be taken.

b. Blanks

A solvent blank will be analyzed at a frequency equal to the method blank. The method blank can serve as the solvent blank provided it is clean in the area of the analyte(s) of interest. This practice eliminates the possibility of background instrumental contamination.

c. Quantitation Internal Standard

A quantitation internal standard will be added to each sample following sample preparation but prior to instrumental analysis. This compound will serve for determining relative response factors and for correcting for variations in sample injection volumes and evaporation of solvents.

d. Standards

Standards are solutions of the analyte(s) of interest used to determine the response of the detector over the range of analyte concentrations found in the samples. For Quantitative analyses, sufficient standards and replication of standards will be run to demonstrate linearity and precision of the detector response over the range of analyte concentrations. Should sample concentrations fall outside the demonstrated linear range, either additional standards are prepared to expand the range, or appropriate dilution/concentration of the sample is done to place it in the proper range.

C. Degrees of Quantitation

The information provided for any particular analysis is tailored to the needs of the customer. In actual practice these needs vary across the broad continuum from the grossly qualitative to the precisely quantitative. It is informative to examine what is involved in providing the varying degrees of quantitation required by our customers.

HP 5985A

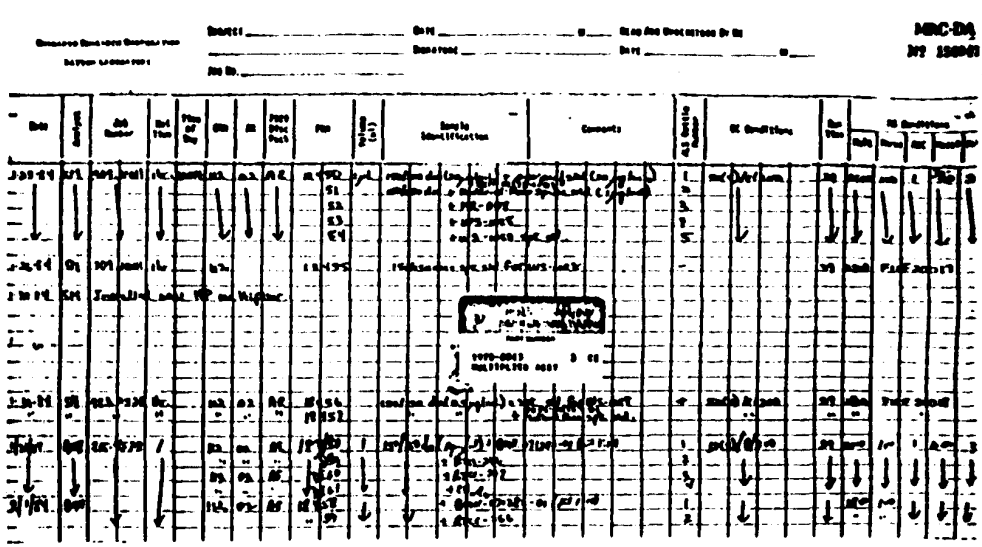

FIGURE 1. Example of GC/MS Logbook page.

HP 5880 GC/EC

MRC-DA
N? 156363

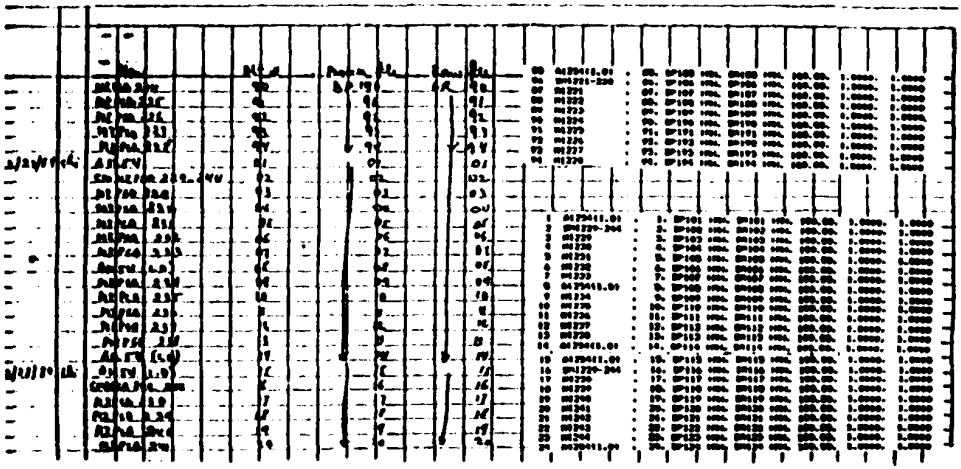

FIGURE 1. Example of GC/EC Logbook page.

1. Qualitative Analysis

Qualitative analysis implies no quantitation at all but rather seeks only the identity of the compound(s) present. GC/MS is particularly applicable to this type of analysis problem. Two types of identifications can be made:

a. Tentative

A tentative identification is made on the basis of the analyst's judgement of a match between the mass spectrum from the sample and a mass spectrum from a reference library. This is often done for the large number of compounds found in a widescan organic analysis.

b. Confirmed

A confirmed identification requires that the chromatographic retention time and the mass spectrum of the unknown compound match those of a known standard of the tentatively identified compound. This is often done in widescan analyses when standards can be obtained for tentatively identified compounds that are of concern.

The normal QA/QC requirements for qualitative analysis are (1) analysis of a blank, (2) analysis of duplicate samples and (3) use of a calibration compound (usually Decafluorotriphenylphosphine, DFTPP) to assure that the mass spectrometer will produce mass spectra that can be compared with library spectra.

2. Semi-Quantitative Analysis

Semi-Quantitative Analysis extends the information provided by a qualitative analysis to include some estimate of the amount of analyte(s) present. The method of semi-quantitation involves the comparison of detector response of the analyte(s) with that of an internal standard. The estimated concentration is obtained by assuming a unit response factor between the analyte(s) and the internal standard(s). An indication of recovery from the sample matrix is obtained from the recovery of the surrogate internal standard(s).

3. Quantitative Analysis

Quantitative Analysis provides for the accurate assessment of the amount of analyte(s) present in a sample. To be able to do quantitative analysis one must know the identity of the analyte so that standards can be prepared for spiking and for the generation of calibration curves. The method of quantitation is based on a three-point calibration curve using standards of the analyte(s) of interest over the range of concentrations in the samples. A one-point calibration method can be used (as with the laboratory data

system) provided that linearity has been demonstrated over the concentration range of the samples using standards of the analyte(s) at three concentrations in the range. This calibration curve is then used to provide concentrations of the analyte(s) in the samples via an external standards method or more preferably via an internal standards method. For the internal standards method, the quantitation internal standard is added to each of the standards (just as with the samples) prior to instrumental analysis and relative response factors are determined.

For quantitative analysis the mass spectrometer is operated as a gas chromatographic detector. The MS is therefore tuned and operated in a manner specified in the approved method rather than using a calibration compound as required for qualitative analyses.

D. Quality Criteria and Remedial Action

The inclusion of QA/QC measures into an analysis program has little meaning unless the results are interpreted and appropriate action is taken when necessary. The ability to do this assumes some set of criteria that will trigger a response or remedial action. The following subsections describe the criteria that are used in the Ultratrace Analysis Group to evaluate when quality control data indicate a need for corrective measures and what remedial action will be taken.

1. Quality Criteria

The overriding quality criterion above all others is: Do the results meet the needs of the customer. This determination will be made jointly between the customer and the analyst. The analyst must exercise caution in not allowing the customer to overstate, misinterpret or unintentionally misrepresent the data. In addition to this prime quality criterion, the following conditions must be met:

a. Blanks - In the event that a blank produces a positive value, it may not be used as a valid blank if it exceeds 10% of any reported value for a sample.

b. Recoveries - Recoveries for all surrogate and native spikes must lie in the range of 50%<Recovery<150%.

c. Precision - Precision will be calculated based on the recoveries of surrogate internal standards. Average recoveries will be calculated with ± standard deviation for each sample set. Replicate analyses and low and high native spike recoveries may also be used to provide additional information/insight into the precision of the analysis.

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d. Limit of Detection - The limit of detection (LOD) will normally be stated as ½ the value of the low level native spike if it is recovered and quantifiable. Should the low level native spike not be recovered, the LOD will be stated as ½ the value of the high level native spike if it is recovered and quantifiable. Should neither spike be recovered, a decision must be made regarding the advisability of reprocessing the sample. Should all samples contain such large quantities of the analyte(s) that the levels of the low and high spikes are insignificant (<10%) by comparison, the value of the LOD becomes less important and is simply stated as the LOD for the instrumental analysis process determined by the analysis of standards and the ability to recover the surrogate spikes.

e. Accuracy - The accuracy of the analyses is best determined by analyzing some sample which contains the analyte at some predetermined level (e.g., an NBS standardized sample). If this type of standard is not available then the low level and high level spikes can be used as a measure of the accuracy of the analyses. Results must fall within a range of 75% to 120% of the excepted value for the analyses to be considered valid.

If the above quality criteria are not met the analyst will immediately assess the need for and extent of remedial action that may be necessary.

2. Remedial Action

The first course of action will be for the analyst and/or group leader to contact the customer and discuss the results pointing out the limitations of the data indicated by the quality control results. At this point the overriding quality criterion of customer needs will be addressed first. It could be that the customer's needs are met without meeting one or more of the other criteria. In such a case no remedial action is necessary. It could also be that the customer has such a demand for tightness in the results that his needs are not met even though the other quality criteria are. This would trigger further analyses with tighter quality control designed around the results obtained (e.g., replications of positives to better establish precision or spikes of non-detected samples to better establish the limit of detection).

In consultation with the customer additional experiments will be designed including the appropriate quality control to ultimately provide the results of sufficient quality to meet the customer's needs.

IV. Data

This section deals with the subject of data and how they are obtained, recorded and reduced so as to be useable in a report and yet readily available for referral and inspection.

The samples will be labeled with a log number at sample login according to the procedure described in the ESC Section GLP Manual. When delivered to the responsible technical person, the samples may then receive additional labeling as deemed necessary. This additional labeling will be documented in a laboratory logbook and/or notebook. These logbooks and notebooks will be used by the analysts to record all pertinent work and results obtained from the samples. The laboratory notebooks will be filled out in accordance with Monsanto Uniform Notebook Procedures.

All raw data generated for a given project, which cannot be conveniently placed in the laboratory notebook (e.g., chromatograms, computer output, etc.), will be filed under some prescribed order (i.e., log number) in a central file under the supervison of the Project Leader and/or QA Specialist.

Most of the data generated by the Ultratrace Analysis Group are computer data stored on magnetic discs. In order to archive these data the specific file name and disc number associated with the analysis will be written down in the laboratory notebook and/or instrument logbook. When the disc is completely full of data an entire unedited disc image or copy of the files on the disc will be transferred to magnetic tape. A logbook will be kept for recording the disc number, name and prefix associated with it, along with the associated tape and file number to which the disc was transferred. An example of such a logbook is given in Figure 3. When the data are transferred a verification program is used to check all transferred records for correctness. When tapes are filled (they typically hold seven to ten disc images) they should be stored in a fireproof vault.

All calculations of the results of the data will be specified in the method used. These calculations will be entered in the analysts laboratory notebook unless they are computer generated. In this case, a copy of the computer output will be filed with the raw data. The Project Leader will be responsible for checking the correctness of the data.

V. Sample Disposal

Samples will be disposed of in accordance with the instructions on the Analysis Request form. The only exception to this would result when the analysis reveals a problem which requires sample disposal in a manner other than that originally specified. If the Project Leader notes from the results of the analysis that a potentially toxic product was present in the samples then the samples will be disposed of in a manner deemed safe by the Safety Department. The date of sample disposal and the manner should be noted on the request form stored with the raw data completing the chain of custody of the sample.

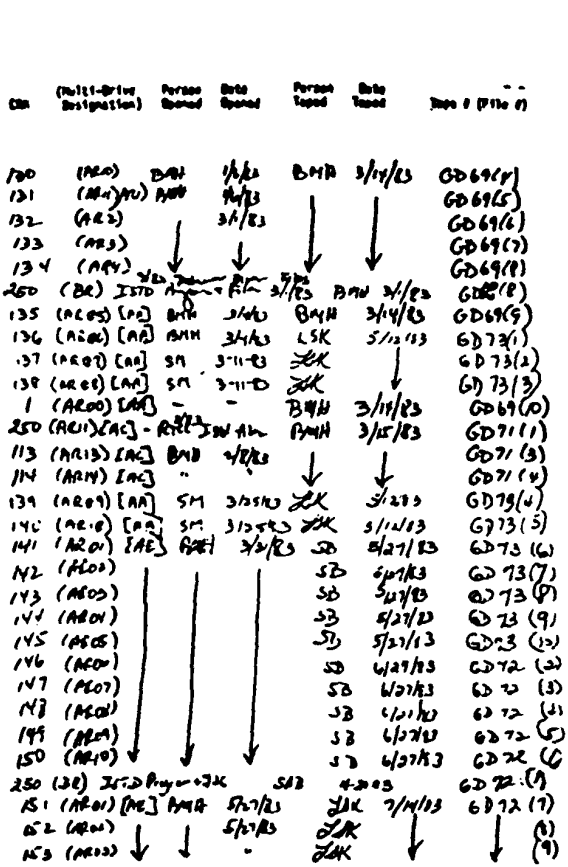

FIGURE 3. Example of DISC/Tape Logbook page.

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