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REPORT NO.: MDA-639

JOB/PROJECT NO.: 322

DATE: 17 July 1985

PERIOD COVERED:

TITLE: ANALYSIS OF SOILS FOR PCB's

AUTHORS: B. M. Hughes

ABSTRACT: Soil samples from the Krummrich plant were analyzed for possible concentrations of PCB's. The results of these analyses are shown in this report.

TECHNICAL APPROVAL: P. L. Sherman

P. L. Sherman
Sr. Research Group Leader

APPROVED BY: R. P. Ivory

R. P. Ivory
Environmental Sciences Center
Service Project Manager

APPROVAL DATE: 7-17-85

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ABSTRACT ONLY

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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[redacted]

ANALYSIS REPORT

DATE: 6/16/85

LOG NUMBER: 05-24-07 PROJECT NO: 760 - 322

TO: Krumrich/Ivory

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

Phone No: (513) 268-3411

Title: Analysis of PCBs in Two Soil Samples

Standard Sampling Method No(s): Sample supplied by customer

Standard Extraction Method No(s): PCEX21

Standard Analysis Method No(s): ECAN02 and MSAN02

Final Report File No(s): A12216

Table File No(s): B12216 and C12216

Two soil samples were recieved from the Krumrich Plant for PCB analysis. GC characterization was also requested. Samples were subjected to Standard Extraction Method PCEX21.

PCEX21 requires an acid wash on part of the extract generated (see enclosed method). The non-acid washed portion of the extracts were analyzed by GC/MS, using Standard Analysis Method MSAN02 in an effort to characterize any oils present. No chromatographable components were detected in these analyses.

PCB analyses were completed on the acid washed portion of the extracts utilizing Standard Analysis Method ECAN02. PCBs were detected in both samples near the Limit of Detection (LOD = 1 ug/g) for these analyses. These values along with quality control data are reported in Tables 1 and 2.

Also included in this report are:

1) A copy of Log-in information generated.
2) A copy of the Standard Analysis Methods, and the Standard Extraction Method used in analyses.
3) An explanation of the tables generated for this project.
4) A copy of the ESC/UAG GLP Manual which explains quality control procedures utilized by ESC.

ANALYST(S): B. Mason Hughes, Chi Trang, David McKenzie and Barbara Williams

ANALYST APPROVAL: B. Mason Hughes

MONS 023490

[redacted]

[illegible]

ESC Project No: F60.18-322

Date: June 3, 1985

				SAMPLE IDENTIFICATION	
			Frame	Krumorich Log#02952-01 South Sewer Southwest Soil	Krumorich Log#02952-01 South Sewer Southwest Soil Duplicate
ANALYTE	ESC ANALYTICAL METHOD	LOD		CONCENTRATION (ug/g)	
Aroclor A1254	ECAN02	1.0	ND(a)	1.25	1.30
Rng of Values	-	-	-	8.29	6.69
No of Values	-	-	-	11	9
Std Deviation	-	-	-	.59	.31
% Rel Std Dev	-	-	-	47	24

SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)		RECOVERY (%)	
CL5-benzene	ECAN02	0.1	64	76	67
CL3-biphenyl	-	-	56	54	52

DOCUMENTATION			
ESC Extraction Method	PCEX21	PCEX21	PCEX21
Sample Amt extracted (g)	1.1000	1.1200	1.3500
Extract Final Volume (ml)	10	10	10
Extract Dilution	None	None	None
ESC Extract No.	PCE1350	PCE1351	PCE1352
Process File Name	BP505	BP506	BP507

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g

ND = none detected with a detection limit given by the LOD value

NA = not analyzed

Rng of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values

No of Values = Number of PCB isomers used to measure Aroclor concentration

% Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.

TABLE FILE NO: B32218.MS

MONS 023491

[redacted]

[redacted] of Capillary GC/EC Analyses of Aroclor 1254 [redacted]
Krumrich Samples

ESC Project No. 760.18-322 Date: June 3, 1985

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Krumrich Log#02952-02 South Sewer Northwest Soil	Krumrich Log#02952-02 South Sewer Northwest Soil Low Spike	Krumrich Log#02952-01 South Sewer Southwest Soil High Spike
			CONCENTRATION (ug/g)		
Aroclor A1254	ECAN02	1.0	1.0	1.65	11.63
Rng of Values	"	"	7.76	5.28	14.74
No of Values	"	"	11	12	32
Std Deviation	"	"	.46	.59	3.21
% Rel Std Dev	"	"	48	36	28

SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)	RECOVERY (%)		
CL5-benzene	ECAN02	0.1	64	76	67
CL3-biphenyl	"	"	56	54	52
Native Spikine Study A1254		1.06,10.8	-	61	98

DOCUMENTATION

ESC Extraction Method	PCEX21	PCEX21	PCEX21
Sample Amt extracted (g)	1.2000	1.0930	1.0700
Extract Final Volume (ml)	10	10	10
Extract Dilution	None	None	None
ESC Extract No.	PCB1353	PCB1354	PCB1355
Process File Name	BP508	BP509	BP510

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g
ND = none detected with a detection limit given by the LOD value
NA = not analyzed
Rng of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values
No of Values = Number of PCB isomers used to measure Aroclor concentration
% Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.

TABLE/FILE NO: AC32218:MH

MONS 023492

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

[redacted]

[redacted] COMPANY/DAYTON LABORATORY

ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 5-25-85 NO: 1-85-05-24-02

REQUESTER: Krumrich PROJECT NO: 760.18 - 322

REPORT RESULTS TO: FILES/MEES, Ivory, Hughes, and Sherman

ANALYSIS REQUESTS SENT TO: FILES , Hughes, Sherman, Ivory, and McKenzie

SAMPLE PICKUP BY: D. McKenzie

SAMPLE LOCATION: Lab 28 building 2

SAMPLE DESCRIPTION FOR 2 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
Log # 02952-1 South Sewer	1 X 1L	PCB-1351, PCB-1352-dup
Log # 02952-2 South Sewer	1 X 1L	PCB-1353
Log # 02952-2 South Sewer + low native spike		PCB-1354
Log # 02952-2 South Sewer + high native spike		PCB-1355
Blank (Dayton soil)		PCB-1350

RECORDED BY: D.McKenzie

REQUIRED ANALYSES: PCBs and Wide Scan analysis to characterize all in Samples

ANALYTICAL TECHNIQUES:

TECHNIQUE (EM)	DATE COMPLETED	TECHNIQUE (EM)	DATE COMPLETED
x --- GC/EC ( )		x --- GC/MS ( )	

QUANTITATIVE: x

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

- HOLD FOR 60 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA322:L1

MONS 023494

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 Nor PCEX2]

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 1253, 1242, 1240, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers	ESC Standard Analysis Method Num "CAM01, ECAM02

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

APPARATUS AND MATERIALS:
Burrell and Jarrell 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 PCFS2].)

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

[illegible] SHEET PCEX21

FOR ESC EXTRACTION METHOD PCEX21

SAMPLE ID: ---------- ----- ESC EXTRACT # ----------

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

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

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

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[ ]	SAMPLE WEIGHT----------
SPIKE SAMPLE WITH SURROGATE SPIKE OF SAMPLE	[ ]	---------- ----------
ADD [illegible] mL BENZENE TO SAMPLE.	[ ]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SONIC 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.	[ ]	FINAL VOLUME 10.0 mL ----------

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

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

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

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

MONS 023497

ESC Standard Analysis Method (SAM) **ESC Standard Analysis Method No:**
TITLE: Organic Compounds in Solvents **HSAM02**

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5995B Capillary GC/MS-Scanning with RTE-6/5987 upgrade.

Typical Run Time:	3 - 40 min.
Typical Multiplier Voltage:	1900 - 2400 eV
Typical Threshold:	100 counts
Typical Mass Range:	10 - 500 amu
Typical Scan Rate:	2/second

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/mL)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Organic Compounds eluting between toluene and n-C34-ane	1	30	30

INTERFERENCES AND LIMITATIONS: Analyte must be chromatographically resolved from other organic compounds.

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

COLUMN TEMPERATURE PROGRAM: 50 - 1F 300 **INJECTION TYPE:** Splitless

CALIBRATION AND STANDARDIZATION:

Quantitative: Use internal standard quantitation technique and calibrate instrument response factors from internal standard.

Qualitative: Mass spectrum of unknown compound are compared to standard mass spectra in the NBS/EPA Mass Spectral Library which contains over 31,000 compounds. Positive identification is made only after a trained mass spectroscopist has visually compared the unknown compound mass spectrum with those identified as most probable compounds based upon their Match Factor.

CALCULATIONS: Analyte concentration =
[(internal standard concentration)][(analyte area)/(internal standard area)]

where: Areas are the total ion areas of the analyte and internal standard.

Note: This calculation is made when unknown compounds are present or when the authentic standards are available.

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

DATE OF METHOD CREATION: 10/31/83 **Time Stamp -** (850309.1545).

ESC Standard Analysis Method (SAM) **ESC Standard Analysis Method No:**
TITLE: Aroclor Formulations in Solvents **ECAM02**

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5880 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 1221, 1242, 1248, 1254, 1260, and 1262	0.1	10	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 1242, 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)/e/150 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.

DATE OF METHOD CREATION: 10/25/83, 2/3/84 Time Stamp = (850201:1)23)

MONS 023499

The Following Page is a Detailed Explanation of Tables Generated for this Project

DETAILED EXPLANATION OF TABLES

In an effort to streamline reports generated by ESC on PCBs present in a wide variety of materials, using a wide variety of techniques, Standard Extraction Methods (SEMs) and Standard Analysis Methods (SAMs) have been developed which can be easily interfaced with each other. An abbreviated form of these SEMs and SAMs have been stored on the Hewlett-Packard 3357-LAS laboratory automation system used by ESC. In a effort to communicate these details with customers of ESC, this information is supplied with each PCB analysis. However, many projects involve a very small number of samples which are not necessarily the same matrix. Therefore, major information on the technical details of an analysis are presented in tabular form within the same table which reports the data.

Each table includes a general title, ESC Project Number and Date on which the table was generated. The sample identification is the customer identification which was on the sample bottle. The next section of the table gives results for the particular analyte listed. For Aroclor formulations, the formulation is identified either visually or from ESC developed data analysis programs. The concentration of the formulation is calculated as the average value of the Aroclor formulation, based upon the analysis of a number of individual PCB isomers in the formulation. The number of isomers used in this calculation is given as "No. of Values" and the range of Aroclor concentrations for this number of PCB isomers is given by "Rng of Values". The standard deviation and % relative standard deviation for this number of determinations of the Aroclor formulation are given in the following two lines of the tables.

The next section of the table deals with quality control used in each sample analysis. The surrogate spiking compound which is added to the sample before extraction and analysis is listed, along with its amount and % Recovery measured for each sample. In cases where PCB concentrations are very high and thus require extract dilution, no effort is made to quantify this compound. This could be done if ESC knows that these data are important, but typically 50 ug/g is the important level to quantify accurately so that surrogate data for concentrations larger than this value, from a regulatory standpoint, are not important.

The next section documents how each sample was extracted and analysed. The ESC Extraction Method, amount of sample extracted, extract final volume, extract dilution factor, ESC extract number and the computer data file in which the processed data is stored are given in this section. The last section gives a brief description of abbreviations or gives any footnotes that give additional information about specific samples.

This table format is both compact and precise. It was developed in an attempt to quickly and accurately generate data which the customer can quickly and accurately evaluate. If there are any suggestions concerning this format, please contact me at extension 396.

DESCRIPTION STORED IN TBEXOIMS

MONS 023501

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

Copy Number: _____

GOOD LABORATORY PRACTICES MANUAL

A Guide to Quality Control/Quality Assurance

Ultratrace Analysis Group

March 1984

Joseph J. Brooke
Group Leader, Ultratrace Analysis

H. A. Wolterzann
Manager, Environmental Sciences Center

Issued to: _____

Date: _____

Environmental Sciences Center
Dayton Laboratory
Dayton, Ohio 45407

JR3/JOB #

MONS 023503

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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

MONS 023505

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

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

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/EC 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.

MONS 023508

HP 5985A

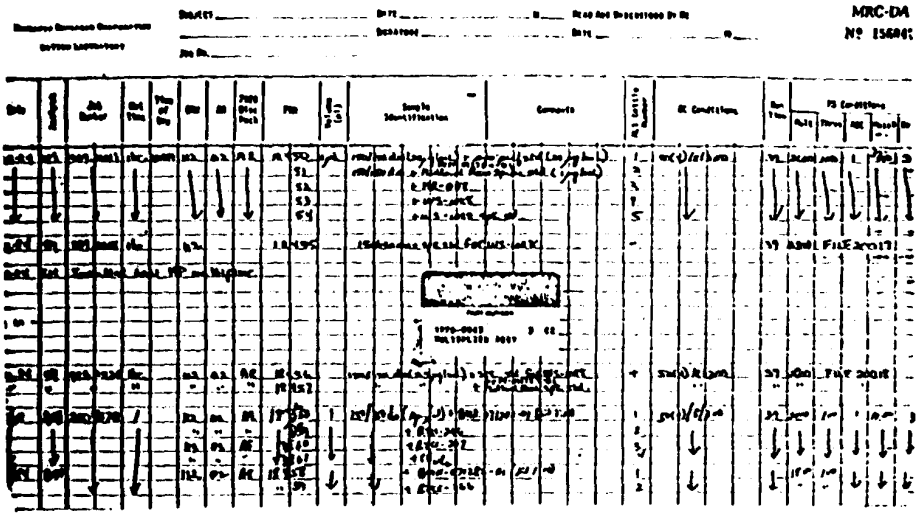

FIGURE 1. Example of GC/MS Logbook page.

HP 5880 GC/EC

MRC-DA
№ 15636

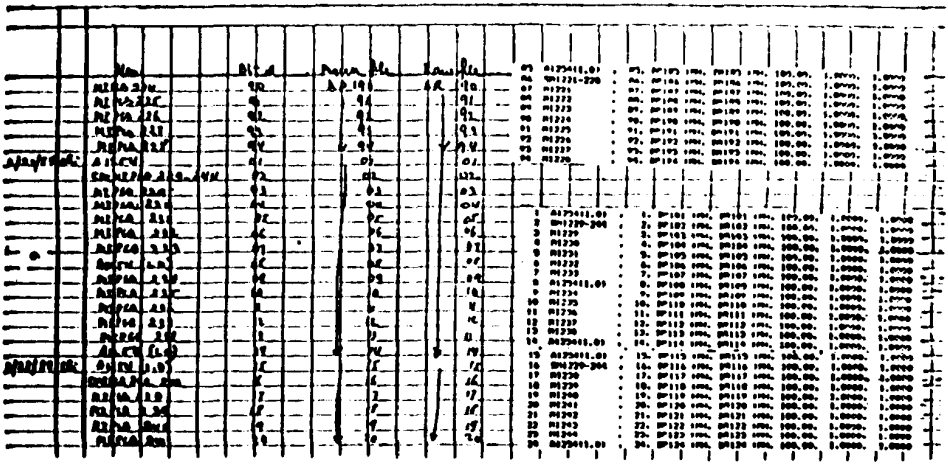

FIGURE 2. Example of GC/EC Logbook page.

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.

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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 supervision 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.

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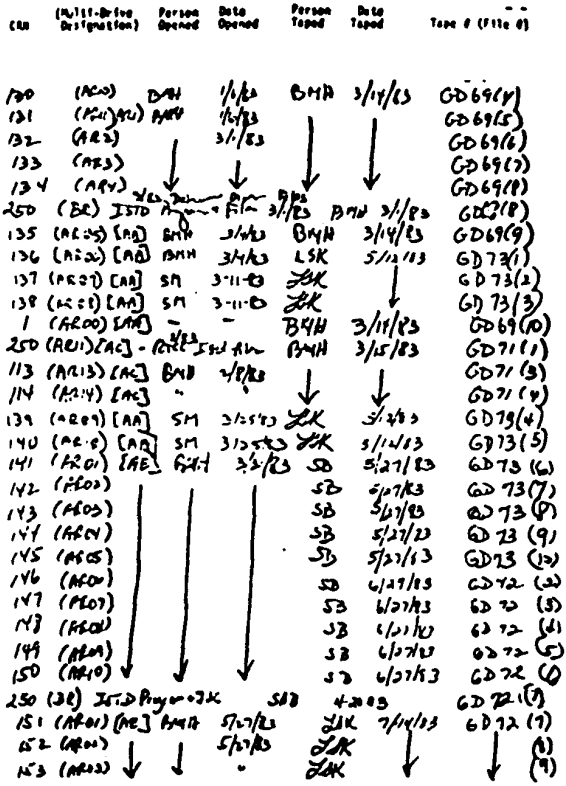

FIGURE 3. Example of DISC/Tape Logbook page.