1.0 SCOPE AND APPLICATION

1.1 This SOP is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). Tables 1a and 1b list the compounds that are routinely determined by this method, and the laboratory Reporting Limit for each analyte. Analytes with [ ] are only reported when requested by the client. These are provided for guidance and may not always be achievable.

1.2 Modifications to the analyte list or procedural changes to reach lower Reporting Limits are allowed if required by client, project or program. Any changes in the analytical procedures must be approved by the Section Chief and the Quality Services Manager before samples can be analyzed. Sample reporting limits are highly matrix-dependent.

TABLE 1a. METHOD 8081 ANALYTE LIST

ANALYTE:	CAS NUMBER	REPORTING LIMIT (ug/L)	REPORTING LIMIT (ug/kg)
Aldrin	309-00-2	0.05	1.7
alpha-BHC	319-84-6	0.05	1.7
beta-BHC	319-85-7	0.05	1.7
delta-BHC	58-89-9	0.05	1.7
gamma-BHC (Lindane)	319-86-8	0.05	1.7
[Chlordane (technical)]	57-74-9*	1.0	33
"-Chlordane	5103-71-9	0.05	1.7
(-Chlordane	5103-74-2	0.05	1.7
[Chlorobenzilate]	510-15-6	0.10	3.3
[Diallate]	2303-16-4	0.10	3.3
[DBCP (1,2-Dibromo-3-chlropropane)]	96-12-8	0.10	3.3
4,4'-DDD	72-54-8	0.10	3.3
4,4'-DDE	72-55-9	0.10	3.3
4,4'-DDT	50-29-3	0.10	3.3
Dieldrin	60-57-1	0.10	3.3
Endosulfan I	959-98-8	0.05	1.7
Endosulfan II	33213-65-9	0.10	3.3
Endosulfan sulfate	1031-07-8	0.10	3.3
Endrin	72-20-8	0.10	3.3
Endrin aldehyde	7421-93-4	0.10	3.3
Endrin ketone	53494-70-5	0.10	3.3
Heptachlor	76-44-8	0.05	1.7
Heptachlor epoxide	1024-57-3	0.05	1.7
[Hexachlorobenzene]	118-74-1	0.10	3.3
[Hexachlorocyclopentadiene]	96-12-8	0.10	3.3
[Isodrin]	465-73-6	0.10	3.3
Methoxychlor	72-43-5	0.50	17
Toxaphene	8001-35-2	5.0	170

TABLE 1b. METHOD 8082 ANALYTE LIST

ANALYTE	BZ #	CAS NUMBER	REPORTING LIMIT (ug/L)	REPORTING LIMIT (ug/kg)
PCB-1016	NA	12674-11-2	1.0	33
PCB-1221	NA	11104-28-2	2.0	67
PCB-1232	NA	11141-16-5	1.0	33
PCB-1242	NA	53469-21-9	1.0	33
PCB-1248	NA	12672-29-6	1.0	33
PCB-1254	NA	11097-69-1	1.0	33
PCB-1260	NA	11096-82-5	1.0	33
2,4'-Dichlorobiphenyl	8		0.1	3.3
2,2',5-Trichlorobiphenyl	18	37680-65-2	0.1	3.3
2,4,4'-Trichlorobiphenyl	28		0.1	3.3
2,2',3,5'-Tetrachlorobiphenyl	44	41464-39-5	0.1	3.3
2,2',4,5'-Tetrachlorobiphenyl	49		0.1	3.3
2,2',5,5'-Tetrachlorobiphenyl	52	35693-99-3	0.1	3.3
2,3',4,4'-Tetrachlorobiphenyl	66	32598-10-0	0.1	3.3
3,3',4,4'-Tetrachlorobiphenyl	69		0.1	3.3
2,2',3,4,5'-Pentachlorobiphenyl	87	38380-02-8	0.1	3.3
2,2',4,5,5'-Pentachlorobiphenyl	101	37680-73-2	0.1	3.3
2,3,3',4,4'-Pentachlorobiphenyl	105		0.1	3.3
2,3',4,4,5'-Pentachlorobiphenyl	118		0.1	3.3
3,3',4,4,5'-Pentachlorobiphenyl	126		0.1	3.3
2,2',3,3',4,4'-Hexachlorobiphenyl	128		0.1	3.3
2,2',3,4,4',5'-Hexachlorobiphenyl	138	35065-28-2	0.1	3.3
2,2',4,4',5,5'-Hexachlorobiphenyl	153	35065-27-1	0.1	3.3
2,3,3',4,4',5-Hexachlorobiphenyl	156		0.1	3.3
3,3',4,4',5,5'-Hexachlorobiphenyl	169		0.1	3.3
2,2',3,3',4,4',5-Heptachlorobiphenyl	170	35065-30-6	0.1	3.3
2,2',3,4,4',5,5'-Heptachlorobiphenyl	180	35065-29-3	0.1	3.3
2,2',3,4,4',5',6-Heptachlorobiphenyl	183	52663-69-1	0.1	3.3
2,2',3,4,4',6,6'-Heptachlorobiphenyl	184		0.1	3.3
2,2',3,4',5,5',6-Heptachlorobiphenyl	187	52663-68-0	0.1	3.3
2,2'3,3',4,4',5,6-Octachlorobiphenyl	195		0.1	3.3
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	206	40186-72-9	0.1	3.3
2,2',3,3',4,4',5,5',6'6'-Decachlorobiphenyl	209		0.1	3.3

2.0 SUMMARY OF METHOD

2.1 This SOP provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides and PCBs.

2.1.1 Prior to the use of this method, appropriate sample extraction techniques must be used.

2.1.1.1 A single extraction is done if no clean-up are required.

2.1.1.2 Should a clean-up be required, the sample extract is split prior to any clean up steps. One portion is processed for organochlorine pesticides and the other for PCB analysis.

2.1.2 The sample extract is injected into a gas chromatograph (GC) using an autosampler, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations.

2.2.1 If interferences prevent detection of the pesticide target analytes, this method may also be performed on samples that have undergone cleanups such as EAL-M-3620 (Florisil Column Cleanup) and for EAL-M-3660 (Sulfur Cleanup).

2.2.2 Extracts for PCB analysis may be subjected to a modified sulfuric acid cleanup designed specifically for these analyses. This cleanup technique will remove (destroy) many of the single component organochlorine or organophosphorous pesticides.

2.3 Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment.

2.3.1 Due to these difficulties, provisions are outlined for the determination of selected individual PCB congeners. The PCB congener approach potentially affords greater quantitative accuracy when PCBs are know to be present, especially in determining weathered Aroclors.

2.3.2 The laboratory must exercise caution using the congener method when regulatory requirements are based on Aroclor concentrations.

3.0 DEFINITIONS

3.1 **Organic-free reagent water** refers to water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water.

3.2 **Initial Calibration Verification (ICV)** is a second source calibration standard used to verify the initial calibration and evaluate method performance. It contains all the analytes listed in Table 1. The stock used to prepare the ICV must be from a source that is different from the stocks used to prepare the calibration standards.

3.3 **Continuing Calibration Verification(CCV)** is a mid-level calibration standard used to verify the initial calibration throughout the analytical sequence.

3.4 **Method Blank** is a reagent water or standard solid matrix spiked with all surrogates of interest and taken through the entire analytical procedure.

3.5 **Surrogate** is a non-target compound spiked into all samples and QC samples and taken through the entire analytical procedure to determine purging efficiency, and any possible matrix bias.

3.6 **Laboratory Control Sample (LCS)** is an aliquot of reagent water or a standard solid matrix, e.g. Na_2SO_4 or sand, spiked with a representative subset of the analytes of interest

and taken through the entire analytical procedure. It is used to monitor the analytical process and recoveries of target analytes are compared to laboratory or project specified control limits for precision and accuracy.

3.7 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are two sample duplicates spiked with a representative subset of the analytes of interest and taken through the entire analytical procedure. Results are used to evaluate measurement bias due to the sample matrix. Recoveries of target analytes are compared to LCS control limits.

3.8 **Reference** the terminology used to identify the native sample used for matrix spiking purposes.

4.0 SAFETY AND CHEMICAL HYGIENE

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available from the Laboratory Safety Officer.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4.4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds

4.3 Good laboratory technique dictates the use of appropriate dermal protection. A laboratory coat, eye protection, and gloves are the minimum requirements.

4.4 All wastes must be disposed of following the procedure outlined in EAL-SOP-018.

5.0 SAMPLE HANDLING AND PRESERVATION

5.1 Sample container, preservation and holding time requirements are given in Table 3.

5.2 While sample extracts are in the custody of the laboratory, they are stored in the semivolatiles laboratory at 4EC ± 2EC prior to and during analysis.

5.3 After analysis is completed, samples are stored by the Sample Management Office in laboratory walk-ins until disposal.

Table 3. RECOMMENDED SAMPLE CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples:	8-oz. wide mouth glass with Teflon liner	None	Extract samples within 14 days; analyze extracts within 40 days of extraction.
Liquid Samples: No Residual Chlorine Present	Amber glass , Teflon liner	Cool to 4EC	Extract samples within 7 days; analyze extracts within 40 days of extraction.

Liquid Samples: Residual Chlorine Present	Amber glass , Teflon liner	Add 3 mL of 10% $Na_2S_20_3$/gal. Cool to 4EC	Extract samples within 7 days; analyze extracts within 40 days of extraction.
Soil/Sediments and Sludges:	8 oz wide mouth glass with Teflon liner	Cool to 4EC	Extract samples within 14 days; analyze extracts within 40 days of extraction.

6.0 INTERFERENCES

6.1 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the injection syringe must be rinsed between samples with solvent. Whenever an unusually concentrated sample is encountered, the following sample(s) may need to be reanalyzed to determine if carryover contamination had occurred.

6.2 Analytical interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. All of these materials must be routinely demonstrated to be free of interferences, under the conditions of the analysis, by running laboratory reagent blanks.

6.3 Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

6.4 Interferences by phthalate esters introduced during in sample preparation can pose majors problems in pesticide determinations. These materials may be removed prior to analysis using EAL-M-3640 (Gel Permeation Cleanup).

6.5 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides.

6.5.1 Sulfur contamination should be expected with sediment samples. EAL-M-3660 is suggested for the removal of sulfur.

6.5.2 Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

6.6 Waxes, lipids, and other high molecular weight materials can be removed by EAL-M-3640 (Gel Permeation Cleanup).

6.7 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphourus pesticides are eliminated by EAL-M-3640. Co-eluting chlorophenols may be eliminated by using EAL-M-3620 (Florisil cleanup).

6.8 PCBs may also interfere with the analysis of organochlorine pesticides. This problem may be most severe for the analysis of multi-component analysis such as Chlordane, Toxaphene, and Strobane.

6.9 Glassware contamination

6.9.1 Clean all glassware as soon as possible after use by detergent washing with hot water,

and rinses with tap water and organic-free reagent water. The glassware is rinsed with isopropanol, drained and dried in an oven at 400°C for several hours.

6.9.2 Glassware contamination resulting in analyte degradation includes soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

NOTE: Oven-drying of glassware used for PCB analyses can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. It is important that glassware from samples containing high concentration of PCBs are not dried with glassware that may be used for trace analyses.

7.0 APPARATUS/INSTRUMENTATION

7.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, data system, gases, and syringes.

7.2 Columns: Wide bore capillary column (30m x 0.53 mm ID). Examples of phases include RTX-5 and RTX-35.

7.3 Detectors: Electron capture (ECD).

7.4 Microsyringe: various sizes

7.5 Vials: Glass, 2-, 10-, and 20-mL capacity with Teflon-lined screw cap.

8.0 STANDARDS AND REAGENTS

8.1 Reagents

8.1.1 Hexane (pesticide quality or equivalent).

8.1.2 Isooctane (2,2,4-trimethylpentane) (pesticide quality or equivalent).

8.2 Standards

8.2.1 The Standards Log Book must be filled out completely following EA-SOP-299.

8.2.2 Stock standard solutions are purchased as certified solutions. These solutions must be replaced after six months or sooner if routine checks indicate a problem.

8.2.3 All standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

8.2.4 Certificates of analysis for all purchased standards will kept on file.

8.2.5 Pesticide Calibration Standards:

8.2.5.1 Pesticide calibration standards are prepared at a minimum of five concentration levels and are prepared through dilution of the stock standards with hexane. One of the concentration levels should be at a concentration at the laboratory Reporting Limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC.

8.2.5.2 Stock Standards: Commercially prepared stock standards are purchased pre-mixed at various levels (8-80 µg/mL - Restek Pest Mix A and Pest Mix B or equivalent).

8.2.5.3 Intermediate standards are prepared by diluting the stock standards 500 µL to 100 mL (resulting in concentrations between 0.04 - 0.08 µg/mL).

8.2.5.4 Instrument calibration standards are prepared by diluting the working standards to the concentrations identified in Table 4.

8.2.5.5 Surrogates are added to the standards at the levels indicated in Table 4.

8.2.6 Multi-component Pesticide Standards.

8.2.6.1 Some Toxaphene components, particularly the more heavily chlorinated, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results.

8.2.6.2 Stock Standards: Commercially prepared stock standards are purchased pre-mixed.

8.2.6.3 Toxaphene and (technical) Chlordane working standards are prepared individually at 0.100 µg/mL for chlordane and at 0.500 µg/mL for Toxaphene All multi-component standards must contain the surrogates at 20.0 ng/mL.

8.2.6.4 Instrument calibration standards are prepared by diluting the working standards to the concentrations identified in Table 4 (50 µL **ö** 100 µL to a final volume of 100 mL).

8.2.7 Aroclor Calibration Standards

8.2.7.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Therefore, a five point initial calibration using a mixture of Aroclors 1016 and 1260 are sufficient to demonstrate the linearity of the detector response without having to perform initial calibration for each of the remaining Aroclors. This mixture is also used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors.

8.2.7.2 Single standards of each of the other Aroclors are required to aid in pattern recognition. Once linearity is demonstrated by 8.2.7.1, a single standard corresponding to the point of the linear range of the detector is prepared for the other Aroclors.

8.2.7.3 Stock Standards: Commercially prepared stock standards are purchased pre-mixed at a concentration of 1000 µg/mL..

8.2.7.4 Instrument calibration standards are prepared by diluting the stock standards to the concentrations identified in Table 4 (10 µL ö 200 µL to 100 mL final volume)..

8.2.8 PCB Congener Calibration Standards

8.2.8.1 If the samples are to be quantitated as individual PCB congeners, standards for the pure congeners are prepared.

8.2.8.2 Stock standards are purchased as commercially-prepared solutions.

8.2.8.3 Calibration standards are prepared at a minimum of five concentrations by dilution the stock standard with Isooctane or hexane at the concentrations specified in Table 4.

Table 4. Concentration of Calibration Standards (ug/mL)

Compound	CON1	CON2	CON3	CON4	CON5
tetrachloro-m-xylene (TCX) - surrogate	0.005	0.020	0.040	0.060	0.080
decachlorobiphenyl (DCB) - surrogate	0.010	0.040	0.080	0.120	0.160
alpha-BHC	0.005	0.020	0.040	0.060	0.080
beta-BHC	0.005	0.020	0.040	0.060	0.080
delta-BHC	0.005	0.020	0.040	0.060	0.080
gamma-BHC (lindane)	0.005	0.020	0.040	0.060	0.080
aldrin	0.005	0.020	0.040	0.060	0.080
heptachlor	0.005	0.020	0.040	0.060	0.080
heptachlor epoxide	0.005	0.020	0.040	0.060	0.080
alpha-chlordane	0.005	0.020	0.040	0.060	0.080
gamma-chlordane	0.005	0.020	0.400	0.060	0.080
dieldrin	0.010	0.040	0.080	0.120	0.160
endosulfan I	0.005	0.020	0.040	0.060	0.080
endosulfan II	0.010	0.040	0.080	0.120	0.160
endosulfan sulfate	0.010	0.040	0.080	0.120	0.160
endrin	0.010	0.040	0.080	0.120	0.160
endrin aldehyde	0.010	0.040	0.080	0.120	0.160
endrin ketone	0.010	0.040	0.080	0.120	0.160
DDT	0.010	0.040	0.080	0.120	0.160
DDE	0.010	0.040	0.080	0.120	0.160
DDD	0.010	0.040	0.080	0.120	0.160
methoxychlor	0.050	0.200	0.400	0.600	0.800
technical chlordane (TCHLOR)	0.100	0.200	0.400	0.800	1.600
toxaphene (TOXAPH)	0.500	1.000	2.000	4.000	8.000

Table 4. Concentration of Calibration Standards (ug/mL)

Compound	CON1	CON2	CON3	CON4	CON5
PCB-1016 (AR1016)*	0.100	0.400	0.800	1.200	1.600
PCB-1221 (AR1221)	0.200	0.800	1.600	2.400	3.200
PCB-1232 (AR1232)	0.100	0.400	0.800	1.200	1.600
PCB-1242 (AR1242)	0.100	0.400	0.800	1.200	1.600
PCB-1248 (AR1248)	0.100	0.400	0.800	1.200	1.600
PCB-1254 (AR1254)	0.100	0.400	0.800	1.200	1.600
PCB-1260 (AR1260)*	0.100	0.400	0.800	1.200	1.600

*Multi-point initial calibration includes Aroclors 1016 and 1260 only. For all other multi-component analytes, a single point representing the reporting limit is used.

8.2.9 ICV/CCV Standards: Prepared from a separate stock as the calibration standards and at the mid-point of the calibration curve. (See Table 4.)

8.2.10 Matrix Spike Standards:

8.2.10.1 Pesticide stock standards are purchased as commercially-prepared solutions.

8.2.10.1.1 Working standards are prepared from the stock standard. The standard contains the entire target analyte list (not including Toxaphene and technical chlordane) at 0.25, 0.5, and 1,25 µg/mL.

8.2.10.1.2 2.0 mL of matrix spike working stock solution are added to every sample, resulting in 0.05, 0.1, and 0.25 µg/mL in the extract.

8.2.11 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with the pesticide surrogates tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB).

8.2.11.1 Pesticide and Aroclor surrogate stock standards are purchased as commercially-prepared solutions at 200 µg/mL.

8.2.11.1.1 The intermediate surrogate standard is prepared from a stock standard and contains both tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) by diluting 0.0750 µL if the stock solution to a final volume of 250 mL, resulting in a concentration of 0.6 µg/mL.

8.2.11.1.2 1.0 mL of surrogate stock are added to every sample, resulting in 0.06 µg/mL in the extract.

8.2.12 Column Degradation Check Standard:

8.2.12.1 Stock standards are purchased as commercially-prepared solutions.

8.2.12.2 The standard contains the following compounds at the specified concentrations:

Compound	Conc (µg/mL)
Endrin	0.050
4,4'-DDT	0.10

9.0 PROCEDURES

9.1 Sample Extraction

9.1.1 Samples may be extracted by several different methods for soil, water, and waste matrices:

Matrix	SW846 Method	EA SOP Number
aqueous	Method 3510 (Sep Funnel)	EAL-M3510
aqueous	Method 3520 (Cont Liq-Liq)	EAL-M-3520
soil	Method 3540 (Soxhlet)	EAL-M-3540
soil	Method 3550 (Ultrasonic)	EAL-M-3550
waste	Method 3580 (Waste Dilution)	EAL-M-3580

9.1.2 Hexane-acetone (1:1) may be more effective as an extraction solvent for OCP in some environmental and waste matrices than Methylene Chloride:Actone. The use of Hexane:Acetone generally reduces that amount of interferences that are extracted and improve signal-to-noise.

9.2 Extract Cleanup

9.2.1 Extracts may be “cleaned” using several different techniques depending upon the analytes of interest.

9.2.2 Option for extract cleanup include:

SW846 Cleanup Method	EA SOP Number
Method 3620 (Florisil Cleanup)	EAL-M-3602
Method 3640 (Gel Permeation Cleanup)	EAL-M-3640
Method 3660 (Sulfur Cleanup)	EAL-M-3660
Method 3665 (Sulfuric Acid [mod] Cleanup)	EAL-M-3665

9.3 INSTRUMENTATION

9.3.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injection. Hewlett Packard 5890.

9.3.2 Gas chromatography conditions. The chromatographic columns and operating condition for the instrument are:

9.3.2.1 Temperature program for columns 1 and 2:

Injector temperature:	240EC
Carrier gas (He) flow rate:	4-5 mL/min
Initial temperature:	140EC, hold for 5 minutes
Temperature program:	15EC/min to 210EC, then 6EC/min to 240EC, then 15EC/min to 300EC
Final temperature:	300EC, hold until all expected compounds have eluted.
Detector temperature:	325°C

9.3.3 Automated Data Acquisition System:

9.3.3.1 HP3365/Enviroquant. The system allows for the continuous acquisition and storage of all mass spectra obtained throughout the duration of the chromatographic program.

9.3.3.2 The data system has the capability to search any GC/MS data file for ions of a specific mass and plot such ion abundances versus time or scan number (Extracted Ion Current Profile (EICP). The most recent version of the EPA/NIH Mass Spectral Library is used.

9.4 Column Conditioning

9.4.1 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day.

9.4.2 Therefore, the GC column(s) must be primed or deactivated by injecting a PCB or pesticide standard mixture approximately 20 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

9.4.3 Several analytes, including Aldrin, may be observed in the injection just following this system priming. An acceptable blank must be achieved prior to analysis of any subsequent standards and samples.

9.5 Retention Time Windows : Default values of ±0.05 and ±0.07 minutes are used.

9.6 Initial Calibration

9.6.1 Pesticides

9.6.2 Introduce 2-5 FL of each calibration standard into the gas chromatograph using the same technique that will be used to introduce the actual samples.

9.6.3 Using peak responses and mass injected, the software calculates a response factor (RF) for each compound for each concentration in the standard curve, and calculate the percent relative standard deviation (%RSD)of the five responses for each compound.

9.6.4 If the percent relative standard deviation (%RSD) of the response factors is less than 20%

over the working range, linearity through the origin can be assumed, and the average response factor can be used in place of a calibration curve.

9.6.4.1 Should the %RSD fail to meet the 20% criteria, the following may apply:

9.6.4.1.1 Review the results (area counts, response factors) for those analytes which failed the %RSD acceptance criteria to determine if the problem is with just one of the standards. Should this be the case, the analyst has the option to reanalyze and replace the standard in question.

9.6.4.1.2 The analyst also has the option of narrowing the calibration range by replacing one or more of the standards with standards of different concentrations.

9.6.4.1.3 It is important to remember that this may cause samples to be diluted at the high end of the curve.

9.6.4.1.4 In addition, the analyst must verify that changing the standard concentration would not effect any client DQO's (that the new quantitation level is at least as low as any required regulatory limits or action levels).

9.6.4.2 In instances where the RSD for one or more analytes exceed 20%, the initial calibration may still be acceptable if the following conditions are met:

9.6.4.2.1 The mean of the RSD values for <u>all</u> analytes in the calibration is less than or equal to 20%.

9.6.4.2.2 The mean RSD is calculated by summing RSD values for each analyte and dividing by the total number of analytes.

9.6.4.2.3 the mean RSD criteria applies to **all analytes in the standards**, regardless of whether or not they are of interest for a specific project. (If the target analyte is part of the cal standard, its RSD value is included in the evaluation.)

9.6.4.2.4 The lab must provide either a summary of the ICAL data or a specific list of those compounds for which the RSD is exceeded and the results of the mean RSD calculation.

9.6.4.3 If the %RSD criteria for any compound in the initial calibration is exceeded, the lab may opt to use a regression analysis to establish the curve for that compound and used for quantitation.

9.6.4.3.1 The correlation coefficient must be r$0.990 ($r^2$$0.980).

9.6.4.3.2 If the r^2 value does not meet the acceptance criteria, remake the curve (or outlier points) and reanalyze.

9.6.4.3.3 The line must not be forced through the origin. Do not include the origin (0,0) as the sixth point.

9.6.4.3.4 If the y-intercept is higher than the reporting limit.

9.6.4.3.4.1 The best approach is to raise the reporting limit to above the y-intercept (or at least meet the y-intercept) if it would not change required client reporting limits. **This must be approved by the QC Chemist, QSM, and the LPM prior to reporting of the data package.**

9.6.4.3.4.2 *If the y-intercept is above the reporting limits and using average or mean RSD criteria is not an option (due to client DQOs), the initial calibration is not acceptable for that analyte and must be repeated.*

9.6.5 Aroclor Calibrations

9.6.5.1 When PCBs are to be determined as Aroclors, the following calibration scheme is used:

9.6.5.1.1 A 5 level calibration curve will be performed for PCB 1016 and 1260 (see Table 4).

9.6.5.1.2 A single standard corresponding to the reporting level is prepared for the other Aroclors.

9.6.5.1.3 In situations (i.e., client DQOs) where only a few Aroclors are of interest for a specific project, a five-points curve for individual Aroclors may be analyzed along with the 1016/1260 standard described above. (See Table 4.)

9.6.5.1.4 A minimum of 3 peaks must be chosen for quantitation of each Aroclor. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 24% of the height of the largest Aroclor peak. For each Aroclor, the chosen peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mix, none of which should be found in both of these Aroclors.

9.6.5.1.5 Aroclor 1016/1260 is used to demonstrate the detector response; therefore this mixture must be applied to the other five Aroclors for which only single standards are analyzed.

9.6.5.1.6 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration.

9.6.5.1.7 Alternately, a 3-5 point multi-point calibration may be performed for each individual Aroclor. When the multi point calibration approach is chosen,

use the calibration factors from these standards to evaluate linearity.

9.6.5.2 When PCBs are to be quantitatively determined as congeners, and initial five-point curve is performed that in includes standards for all of the target congeners.

9.6.5.2.1 This involves the calculation of the mean response or calibration factor, the standard deviation, and the relative standard deviation for each congener or Aroclor peak.

9.7 Initial Calibration Verification

9.7.1 The initial calibration curve must be verified immediately after the calibration is performed using a second source stock.

9.7.2 The % R calculated from the initial calibration curve must be ±15% of the true value.

$$\%R = \frac{CF - \overline{CF}}{CF} \times 100$$

where:

CF	=	Calibration factor from the analysis of the verification standard
$\overline{CF}$	=	Mean calibration factor from the initial calibration

9.7.3 If this standard fails, the standard should be remade immediately and the curve verified.

9.7.4 Should it fail a second time, the analysis is stopped and a new initial calibration curve is prepared.

9.8 Endrin/DDT Breakdown Check

9.8.1 Because Endrin and DDT are easily degraded if the injection port is contaminated with high boiling residue from sample injections or when the injector contains metal fittings, check for degradation by injecting a standard containing on 4,4'-DDT and Endrin. The presence of DDE, DDD, Endrin ketone, or Endrin aldehyde indicates breakdown is occurring.

9.8.2 If the degradation of either DDT or Endrin exceed 15%, corrective action must be taken before proceeding with the calibration.

9.8.3 The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12 hour shift or every 20 samples, whichever is sooner.

9.8.4 Injector maintenance and recalibraton should be completed if the breakdown is greater than 15% of either compound.

$$\%Breakdown\ of\ DDT = \frac{(\text{area of DDT peak}) + (\text{area of DDE peak} + \text{area of DDD peak})}{(\text{area of Endrin peak} + \text{area of DDT peak} + \text{area of DDE peak} + \text{area of DDD peak})} \times 100$$

9.9 Continuing Calibration Verification

$$\%\ \text{Difference} = \left(\frac{\text{Initial Calibration Factor} - \text{Continuing Calibration Factor}}{\text{Initial Calibration Factor}}\right) \times 100$$

9.9.1 The calibration is verified every 12 hours by injecting the calibration verification standards prior to conducting any sample analyses.

9.9.1.1 A calibration standard must also be injected at intervals of not less than once every 20 samples [every 10 samples may cut down on repeat analysis] and at the end of the analysis sequence.

9.9.1.2 The high and low concentration mixtures of single component analyses and multi-component analyses are alternated from calibration verification.

9.9.2 The calibration factor for each analyte must not exceed a ±15% difference from the nominal (or true) value from the initial calibration.

$$\%\ \text{Difference} = \frac{CF - \overline{CF}}{CF} \times 100$$

where:

9.9.3 Alternatively, if the average of the responses for <u>all</u> analytes is within ±15% (absolute value), then the calibration has been verified. However, the average must include all analytes in the calibration, whether or not they are targets of interest for a specific project. Information regarding the compounds which exceed the 15% criteria must be provided to the client.

9.9.4 If the CCV fails to meet acceptance criteria, the standard should be reanalyzed immediately.

9.9.5 Should the standard fail a second time, the analysis is stopped and a new initial curve is analyzed.

9.9.6 All samples since the previous acceptable verification standard must be rerun. <u>This includes the ending verification standard.</u>

9.10 Matrix Spike (Matrix Spike Duplicate)

9.10.1 With each analytical batch of 20 or fewer client samples an MS/MSD pair must be

analyzed.

9.10.2 Spike each aliquot and analyze in the same manner as the reference sample.

9.10.3 Report the results of the both the %R in the MS and MSD samples and the %RPD between the MS and MSD. Note results in the case narrative.

9.10.3.1 Spike Recovery

$$\%\ R = (\%R) = \frac{SSR - SR}{SA} \times 100$$

where:
SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

9.10.3.2 %RPD

$$\%\ RPD = \frac{|MSR - MSRD|}{(MSR + MSRD)/2} \times 100$$

where:
MSR = Matrix spike recovery
MSRD = Matrix spike duplicate recovery

(The vertical bars in the formula above indicate the absolute value of the difference, therefore, RSD is always expressed as a positive value.)

10.0 IDENTIFICATION/QUANTITATION/CALCULATIONS

10.1 Quantitation of Pesticides

10.1.1 Analytes are quantitated by comparing the measured response in the sample against the initial standard curve.

10.1.2 The concentration in the extract for each analyte is taken directly from the standard curve.

10.2 Quantitation of Multi-component Analytes

10.2.1 Multi-component analyses present problems in measurement. Suggestions are offered in the following sections for handing Toxaphene and Chlordane.

10.2.1.1 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using either internal or external calibration procedures. If

compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

10.2.1.2 Toxaphene - Toxaphene is manufactured by the chlorination of camphenes. Quantitation of Toxaphene is difficult, but reasonable accuracy can be obtained.

10.2.1.2.1 Quantitate Toxaphene using the total area of the Toxaphene pattern or using 3 to 5 major peaks.

10.2.1.2.2 While Toxaphene contains a large number of compounds that will produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. This unresolved complex mixture results in the "hump" in the chromatogram that is characteristic of this mixture. Although the resolved peaks are important for the identification of the mixture, the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

10.2.1.2.3 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

10.2.1.2.4 When Toxaphene is determined using the 3 to 5 peaks approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionally larger or smaller in the sample compared to the standard.

10.2.1.2.5 The heights or areas of the 3 to 5 peaks should be summed together and used to determine the Toxaphene concentration. Alternatively, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for the sample.

10.2.1.3 Chlordane - Technical Chlordane is a mixture of at least 11 major components

and 30 or more minor components that is used to prepare specific pesticide formulations. The CAS Registry number for Technical Chlordane is properly given as 12789-03-6. Trans-Chlordane (or a-Chlordane, CAS RN 5103-71-9) and cares-Chlordane (y-Chlordane, CAS RN 5103-74-2), are the two most prevalent major components of Technical Chlordane.

10.2.1.3.1 The exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch. Moreover, changes may occur when the technical material is used to prepare specific pesticide formulations. The approach used for evaluating and reporting Chlordane results will often depend on the end use of the results and the analyst's skill in interpreting this multi-component pesticide residue.

10.2.1.3.2 EA Laboratories uses two options for reporting Chlordane: reporting Chlordane (not otherwise specified, 57-74-9), and reporting the individual "- and (- isomers that can be identified under their individual CAS numbers.

10.2.1.3.3 When the GC pattern of the residue resembles that of Technical Chlordane, EA quantitates Chlordane residues by comparing the total area of the Chlordane chromatogram using three to five major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation of the residue. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area.

NOTE: Octachloro epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.

10.2.1.3.4 The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the pesticide active ingredient Technical Chlordane. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane (n.o.s.)" under the CAS number 57-74-9.

10.2.1.3.5 The second option is to quantitate the peaks of "-Chlordane, (-Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers. To measure the total area of the Chlordane chromatogram, inject an amount of a Technical Chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms.

10.3 Polychlorinated biphenyls (PCBs)

10.3.1 Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of toxaphene, Strobane, and chlordane. In each case, the chemical is made up of numerous compounds. So the chromatograms are multi-peak. Also in each case, the chromatogram of the residue may not match that of the standard.

10.3.2 Mixtures of PCBs of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the trade name Aroclor (1200 series and 1016). Though these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish.

10.3.3 PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue.

10.3.3.1 A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue.

10.3.3.2 This may also involve judgment about what proportion of the different Aroclors to combine to produce the appropriate reference material.

10.3.4 Quantitate PCB residues by comparing total area or height of residue peaks to total area of height of peaks from appropriate Aroclor(s) reference materials.

10.3.4.1 Measure total area or height response from the common baseline under all peaks. Use only those peaks from the sample that can be attributed to chlorobiphenyls.

10.3.4.2 These peaks must also be present in the chromatogram of the reference materials. Mixtures of Aroclors may be required to provide the best match of the GC patterns of the sample and reference.

10.4 Calculations

10.4.1 Water

$$Conc_{sample} = \frac{A_x \times V_t \times DF}{CF \times V_i \times V_s}$$

where:

$Conc_{sample}$ = Sample concentration (ug/L)
A_x = Area or height of the peak for the compound to be measured.
CF = Calibration factor from the initial calibration (area per pg)
V_t = Volume of the concentrated extract in milliliters (mL)
V_i = Volume of extract injected in microliter (uL). If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.
V_s = Volume of sample extracted in milliliters (mL)
DF = Dilution Factor. If no dilution is performed, DF = 1.0. The dilution factor is defined as follows:

$$DF = \frac{V_e + V_s}{V_e}$$

where:

V_e = Volume of extract used to make dilution (uL)
V_s = Volume of clean solvent (uL)

10.4.2 Soil/Sediment

$$Conc_{sample} = \frac{A_x \times V_t \times DF \times F_{clean}}{CF \times V_i \times W_s}$$

where:

$Conc_{sample}$ = Sample concentration on wet weight basis (ug/kg)
A_x = Area or height of the peak for the compound to be measured.
CF = Calibration factor from the initial calibration (area per pg)
V_t = Volume of the concentrated extract in milliliters (uL)
V_i = Volume of extract injected in microliter (uL). If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.
W_s = Weight of sample extracted in grams (g)
F_{clean} = Volume correction factor for cleanup procedures. When GPC cleanup is used the factor is 2.

DF = Dilution Factor. If no dilution is performed, DF = 1.0. The dilution factor is defined as follows:

$$DF = \frac{V_e + V_s}{V_e}$$

where:

V_e = Volume of extract used to make dilution (uL)
V_s = Volume of clean solvent (uL)

10.4.3 If sample concentrations are to be reported on a dry weight basis, the following equation is used:

$$Conc_{dry} = \frac{Conc_{sample}}{\%Solid} \times 100$$

where:

$Conc_{dry}$ = Concentration of the sample on a dry weight basis (ug/kg)
$Conc_{sample}$ = Concentration of the sample on a wet weight, or "as received" basis (ug/kg)
%Solid = Percent solids determined gravimetrically.

11.0 QUALITY CONTROL

11.1 Quality Control Acceptance Criteria for this method, including the frequency and corrective actions are shown in Table 9.

11.2 Initial Calibration

11.2.1 In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.

11.2.2 The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not permitted.

11.3 The standard curve is verified using a second source standard (ICV).

11.4 Method Blank: A method blank is analyzed once per analytical batch of 20 or fewer samples to determine whether or not the analysis has introduced any contamination to the samples.

11.5 Laboratory Control Sample: Analyzed once per analytical batch of 20 or fewer samples.

11.6 Matrix Spike/Matrix Spike Duplicate: Analyze one MS/MSD pair once per analytical batch of every 20 or fewer samples.

11.7 Confirmation

11.7.1 Second column confirmation is required for all reported results above the RL. .

11.7.2 All QC criteria must me met in order to use a column for second column confirmation.

11.7.3 If confirmed by GC/MS, a minimum concentration of 10 ng/uL in the final extract for each single-component compound is required.

12.0 REFERENCES

12.1 United States Environmental Protection Agency. 1997. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition, including Update III. U.S. EPA, Washington, D.C.

12.2 United States Environmental Protection Agency. 1995. Contract Lab Program , Statement of Work OLM03.2. U.S. EPA, Washington, D.C.

Table 9. Organochlorine Pesticides and PCBs by GC/ECD: SW846 Methods 8081A and 8082

EAL-M-8081A / 8082 Organochlorine Pesticides and Polychlorinated Biphenyls

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	LABORATORY CORRECTIVE ACTION
Holding time	Aqueous samples within 7 days; Solid and waste samples within 14 days; Analyze all extracts within 40 days	Extraction and analysis must be completed within holding time.	Notify client to determine if laboratory is to proceed with analysis or if client will resample
Initial Calibration	Initial 5 point calibration Low standard #PQL.	Initial calibration for all single peak target analytes, and Aroclors 1016 and 1260: <20 %RSD; or mean %RSD for all analytes in the standard #20%; or use calibration curve ($r \geq 0.990$).	Verify standard preparation and instrument operations, correct problem. Recalibrate instrument. Document actions taken.
Initial Calibration Verification (ICV)	Second source mid-level standard following initial calibration	%D from initial calibration no greater than ±15%.	Verify standard preparation, if incorrect reprepare ICV Verify preparation of calibration standards if incorrect reprepare standards, and recalibrate instrument. Document actions taken.
Continuing Calibration Verification (CCV)	Beginning of each 12 hour analytical shift and after every 20 samples (10 recommended)	%D from initial calibration no greater than ±15%.	If %D>15 and no target analytes are found, no further action is required. Verify standard preparation and instrument operations, correct problem. Repeat initial calibration if problem cannot be identified. Document actions taken.
Degradation Standard	Beginning of analytical sequence	Breakdown of Endrin : <15% Breakdown of DDT : <15%	Evaluate system perform system maintenance. Recalibrate as necessary.

Table 9. Organochlorine Pesticides and PCBs by GC/ECD: SW846 Methods 8081A and 8082

EAL-M-8081A / 8082 Organochlorine Pesticides and Polychlorinated Biphenyls

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	LABORATORY CORRECTIVE ACTION
Method Blank	1 per analytical batch Must be analyzed on each instrument used to analyze samples	All target compound concentrations must be <PQL.	Determine source of contamination, i.e. instrument, reagent water, reagents. Take appropriate corrective action and document. If preparation in error reanalyze or prepare analytical batch. If samples cannot be reanalyzed or reprepared, qualify data. document actions taken.
Laboratory Control Sample (LCS)	1 per analytical batch	Values are within project specified control limits (or lab determined) for precision and accuracy.	Reanalyze the LCS. Check instrument parameters, sensitivity and linearity. Correct any problems. Validate LCS preparation. If error is found, reprepare the LCS, and reanalyze the method blank, LCS and all field samples in the batch. If LCS is valid, evaluate against project specific DQOs and report data if there is not impact on data usability. If data is not usable, reprepare and reanalyze the method blank, LCS and all field samples in the batch. If repreparation of samples is not possible, qualify data, and note in the report narrative. Document all actions taken in a NCR and in the report narrative.

Table 9. Organochlorine Pesticides and PCBs by GC/ECD: SW846 Methods 8081A and 8082

EAL-M-8081A / 8082 Organochlorine Pesticides and Polychlorinated Biphenyls

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	LABORATORY CORRECTIVE ACTION
Surrogate spike	All field and QC samples	Evaluate %R for primary surrogate. If low, evaluate secondary surrogate compound. Secondary surrogate values must be within project specified control limits (or lba detremined) for precision and accuracy.	Examine all QC (including but not limited to LCS, MB). IF surrogate in LCS and/or MB is out-of-control, check quantitation. If quantitation is correct reanalyze. If similar results are obtained from reanalysis, obtain fresh, verified surrogate solution, and reprepare and reanalyze the analytical batch. If samples cannot be reprepared, qualify data. If surrogate recoveries in LCS and MB are acceptable but below the control limit for any sample, validate sample preparation. Correct any problem then restrict and reanalyze the sample If surrogate recoveries in LCS and MB are acceptable but above the upper control limit for any sample and no target analytes are detected, data is reported with discussion in the analytical narrative.
MS/MSD	1 set per analytical batch	Values are within project specified control limits (or lab determined) for precision and accuracy	I analyte recovery is outside control limits in LCS and data is judged unusable, reanalyze the analytical batch. If LCS acceptable but recovery is outside control limits in MS/MSD, validate preparation of samples. If no errors or problems are discovered for sample preparation, data is reported with discussion in analytical narrative.
2nd Column Confirmation	100% for all positive results	Same acceptance criteria as for primary column.	Same corrective actions as for primary column.

EA Engineering, Science, and Technology, Inc.

EA Laboratories

METHOD STANDARD OPERATING PROCEDURE

Number: EAL-M- 8081A/8082 **Rev. No.:** 0

Title: Organochlorine Pesticides and PCBs, and PCB Congeners by Gas Chromatography

Approved By: _____ _____
Glen Gregory, Section Chief Date

Approved By: _____ _____
M. M. Uhlfelder, Quality Services Manager Date

Approved By: _____ _____
A. R. Karimi, Laboratory Director Date

EA Engineering, Science, and Technology, Inc.

EA Laboratories

METHOD STANDARD OPERATING PROCEDURE

Number: EAL-8081A/8082 **Rev. No.:** 0

Revisions		
Rev. No.	**Date of Release**	**Description**
0		Initial distribution for updated/new methods

EA Engineering, Science, and Technology, Inc.

EA Laboratories

METHOD STANDARD OPERATING PROCEDURE

Number: EAL-8081A/8082 **Rev. No.:** 0

Controlled Distribution

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Natasha Sullivan	8
Craig Schenning	17
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Mohammed Haq	19