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Analytical Protocol (GC/ECNIMS) for OSWER's Response to OIG Report (2005-P-00022) on Toxaphene Analysis

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Abbreviations

ASE: accelerated solvent extraction, Dionex instrument; NIMS: electron capture negative ion mass spectrometry; IS: internal standard; MDLs: method detection limits; MQLs: method quantitation limits; OCPs: organochlorine pesticides; OIG: Office of Inspector General; OSW: Office of Solid Waste; ppb: parts per billion (µg/kg); ppt: parts per trillion (ng/kg); SPE-Si: solid phase extraction using silica; TIC: total ion chromatogram

Background

This document offers a GC/MS protocol (attached as a text file and with supplementary data) for updating Method 8081a with (electron capture and dissociative electron capture) negative ion mass spectrometry (NIMS) for the determination/confirmation of selected analytes. The scope of the protocol covers most of the existing analytes in 8081a (first listing in section 1.1). This approach was developed in response to the OIG Report on Toxaphene [1] and to a specific request from Barry Lesnick, OSW [2]. The approach discussed here divides the analytes into four classes for either monitoring or method performance evaluation purposes: chlordane, toxaphene, organochlorine pesticides, and toxaphene congeners. The two complex mixtures of chlordane (chlordanes and related compounds heptachlor and nonachlor) and toxaphene (polychlorinated camphenes and bornanes) involve monitoring a series of ions representing various congener groups found in the mixtures and integrating all of these signals for a total toxaphene or chlordane response. In the case of the organochlorine pesticides (OCPs) and the toxaphene congeners, individual compounds are quantitated separately and reported separately.

Although the protocol does not address all of the 800-plus congeners that comprise toxaphene, additional toxaphene congeners can be added to the method by simply identifying retention time windows and associated responses as the additional congeners with the caveat that the responses are resolved from other congeners (arguably, a difficult procedure). The PCB congener #204 (2,2',3,4,4',5,6,6'-octachlorobiphenyl, not found in the Aroclors) originally used by Swackhamer et al. [3] was retained as internal standard although a number of different compounds could be used as internal standard (e.g., d10-chlorpyrifos). The methodology for toxaphene and chlordane was also published as a peer-reviewed article in our earlier work performed at NERL-Las Vegas

[4, 5].

The compounds 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT, among the analytes of Method 8081a, do not respond sensitively under NIMS conditions and should continue to be confirmed/quantitated by GC/MS using EI mass spectrometry and GC/ECD. The data we have obtained indicate that the oxygen reaction observed with PCBs that gives rise to ions potentially interfering with toxaphene determination (i.e., ions at the same nominal mass but not the same elemental composition) is completely eliminated in modern instruments under appropriate conditions, and the chosen internal standard serves to monitor this situation by the absence of $(M - Cl + O)^-$ ions (e.g., m/z 411) at its retention time (less than 0.5% possible attributable response). The protocol depends necessarily on the proper extraction, cleanup, concentration, and separations to achieve the quantitations and confirm the target analytes.

This report includes detailed data concerned with calibrations and separation conditions as well as ions monitored for each analyte class. Attached compressed files include the complete protocol from the method print out in text format and full size screen captures of the calibration plots with some data pairs visible. This method can be implemented directly by the user and enables complete reproduction of the experiments provided the instrumentation is available. Some performance data is included here for spiked soil for each of the analyte classes to assess the performance of the protocol in a realistic setting. The protocol exhibits acceptable stability normally spanning several weeks for a given calibration and excellent precision for the determinative step (replicate injections of a given sample extract) of about 2%. Variations in the replicates for spiked soil are chiefly the result of variability in the two concentration steps performed (one following ASE extraction, and one following SPE-Si cleanup) since the determinative step is tightly reproducible. The emphasis with the performance data is on assessing overall reproducibility, ruggedness, and applicability because recoveries are not the focus of this GC/MS protocol. Recoveries would be an appropriate emphasis for a complete new method development for SW-846. Thus, the GC/MS protocol here is an additional confirmation/quantitation option for the existing Method.

Experimental approach

Note regarding nomenclature: For the background regarding the term "parlars", see: [6] . The Hx-Sed and Hp-Sed congeners specifically mentioned in the OIG Report refer to the two environmentally significant toxaphene congeners, hexa- and hepta-chlorobornane, which had been isolated from sediment; see [7, 8, 9].

Analytical standards: The OCPs, toxaphene, and chlordane standards were purchased from Supelco (Bellafonte, PA, USA). The toxaphene congener solutions were purchased from LGC PromoChem (United Kingdom). The sediment mix was DE-TOX 484 and contained 2-endo,3-exo,6-exo,8,9,10-hexachlorobornane, 2-endo,3-exo,5-endo,6-exo,8,9,10-heptachlorobornane (Hp-Sed), 2-exo,3-endo,6-exo,8,9,10-hexachlorobornane (Hx-Sed), 2-exo,3-endo,5-exo,6-exo,8,9,10-heptachlorobornane (designated P1 in this discussion) . The important isomer mix

(parlars) was DE-TOX 483 and contained 2-exo,3-endo,5-exo,6-exo,8,9,10,10-heptachlorobornane (designated P2 in this discussion), 2-endo,3-exo,5-endo,6-exo,8,8,10,10-octachlorobornane(T2, parlar 26), 2,2,5,5,9,9,10,10-octachlorobornane (parlar 38), 2-endo,3-exo,5-endo,6-exo,8,9,10,10-octachlorobornane (parlar 40), 2-exo,3-endo,5-exo,6-exo,8,9,9,10,10-octachlorobornane (parlar 41), 2-exo, 5,5,8,9,9,10,10-octachlorobornane (parlar 44), 2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-nonachlorobornane (T12, parlar 50), 2,2,5,5,8,9,9,10,10-nonachlorobornane (parlar 62). The first mix identities were deduced from literature and analysis. The second mix contained only 5 distinct peaks for 8 components listed and were partially deduced from literature and analysis with coelution assumed among the remaining observed peaks which appeared to be multicomponent (i.e., parlars 38, 40,41 and 50, 62).

GC/MS Protocol Features:

The protocols reported here were developed on an Agilent GC/MSD 7673 (analytes chlordane and OCPs) and an Agilent GC/MSD 7675 (analytes toxaphene and toxaphene congeners) both fitted with 6890 gas chromatographs. Thus, the protocol involves both the previous generation and the latest generation of Agilent instruments, and both performed adequately. The column parameters on the 7673 were injector 250°C, transfer 280°C, 0.5 mL/min flow rate, 40 m Agilent J&W DB5MS, 0.18 mm ID, 0.18 μm film thickness. The column parameters used on the 7675 were injector 250 °C, transfer 280°C, 1.2 mL/min flow rate, 30 m Agilent HP5MSI, 0.25 mm ID, 0.25 μm film thickness. The GC program was the same for the two instruments: 60°C for 1 min, 60°C to 150°C at 10°C/min, 150°C to 250°C at 4°C/min, 250°C to 300°C at 10°C/min and hold for 6 min.

Ions monitored for the chlordane analysis: m/z 429.8 (IS); 303.9, 305.9, 337.9, 339.9, 341.9, 371.8, 373.8, 375.8, 407.8, 409.8, 411.8, 441.8, 443.8, 445.8, and optional 321.9 (d10-chlorpyrifos) all for 25 msec dwell time. Quantitation was performed by using manual integration of the total ion chromatogram (TIC) within the retention time window of chlordane.

Ions monitored for the OCPs analysis: m/z 429.8 (IS); Group1: 252.9, 254.9, 256.9, 263.9, 265.9, 267.9, 297.9, 299.9, 301.9 and Group 2 (start 26.50 min): 234.9, 236.9, 238.9, 327.9, 329.9, 331.9, 377.9, 379.9, 381.9, 385.9, 387.9, 389.9, 403.7, 405.7, 407.7, 419.7, 421.7, 423.7; and optional 321.9 (d10- chlorpyrifos) all for 25 msec dwell time. Quantitation was based on m/z 254.9 for α-BHC, β-BHC, γ-BHC, and δ-BHC; 299.9 for heptachlor; 329.9 for aldrin; 389.9 for heptachlor epoxide; 405.8 for endosulfan I and II; 379.9 for dieldrin, endrin, and endrin aldehyde; 421.7 for endosulfan sulfate. Alternatively, these ions can all be done in one group, eliminating the need for defining a group break time.

Ions monitored for the toxaphene analysis: m/z 429.8 (IS); 306.9, 308.9, 310.9, 340.9, 342.9, 344.9, 376.9, 378.9, 380.9, 410.8, 412.8, 414.8, 444.8, 446.8, 448.8, and optional 321.9 (d10-chlorpyrifos) all for 25 msec dwell time. Quantitation was performed by using manual integration of the TIC within the retention time window of toxaphene.

Ions monitored for the toxaphene congener analysis: m/z 429.8 (IS);306.9, 308.9, 310.9, 340.9, 342.9, 344.9, 376.9, 378.9, 380.9, 410.8, 412.8, 414.8, 444.8, 446.8, 448.8, and optional 321.9 (d10- chlorpyrifos) all for 25 msec dwell time. Quantitation was based on m/z 308.9 for Hx-sed, 342.9 for Hp-sed, m/z 378.9 for parlar 26 and 38, 40, 41, m/z 412.8 for parlar 50,62.

The reagent gas, methane, was set at a flow setting of 40 which means 40% of 2 mL/min. A number of other parameters characterize the conditions, some of which reside within the tune file that contains the calibration data. The instrument temperatures for the GC/MSD 5975 were 150°C for both source and quadrupole region and for the GC/MSD 5973 were 150°C for the source and 106°C for the quadrupole (these are the customary settings for each respective instrument as recommended by the vendor). The emission current is set automatically during the tune. The electron multiplier voltage was set at 2000V absolute. The appropriate tune file also is created with the polarity negative as befits the technique. Attached to this document in a zip file are the four acquisition file parameters as text files as put out by the data system for the four classes of analytes.

Spiked Soil Extractions/Cleanup:

A large number of development runs were made to somewhat refine the sample handling part of the methodology and ensure greater reproducibility, but no exhaustive attempt was made to reduce variations further. Seven replicates of soil (25 ppb toxaphene, 50 ppb chlordane, 50 ppb each of the OCPs, and 500 ppt each of the toxaphene congeners) were analyzed treating each class of analytes independently (total of 28 data sets reported). In each case 5 g of soil was spiked and extracted using ASE. The soil was New England Horizan A, sifted and homogenized (obtained from Dr. Brian Schumacher, EPA). The solvent was 50/50 acetone/methylene chloride and the conditions were 5 min equilibration, 15 min static extraction at 80°C, and 2500 psi pressure. The extract was then concentrated just to dryness under a gentle nitrogen stream and redissolved in hexane using sonication (not all components redissolve, presumably more polar analytes do not) with hexane for SPE cleanup. The 3 mL SPE Si cartridges were rinsed with 6 mL of hexane. The sample was applied in 1 mL of hexane and eluted with 3 mL hexane. The OCPs and parlars were not fully recovered with hexane elution and SPE cartridges were further subjected to 2 mL hexane/methylene chloride followed by 2 mL of methylene chloride. All eluants were combined and concentrated to about 1 mL. The internal standard was added and the sample extract was placed in a GC vial for analysis with the internal standard concentration at 10 pg/μL.

Calibrations

Calibration plots are included within the document and attached separately as *.png files (in the attached zip file) for all of the compounds. Calibrations were generally linear with r^2 of 0.98 or 0.99. The plots contain a visible tabulation of 3 or 7 pairs of the data that are used as area ratios versus amount ratios where the internal standard amount or area is the divisor. Additional data pairs may not be visible in the screen captures. These are captured plots afforded directly by the data system (EnviroQuant ChemStation version B.01.00 and Enhanced MSD ChemStation

D.02.00.275) . As such they give an idea of the linearity and intercept of the plots, and, additionally, show the equation and some of the raw data.

By using the lowest calibration point as the method detection limit, estimates of achievable detections in samples can be obtained. Detection limits are strongly dependent on sample and matrix interferences. Additional cleanup may be needed when multiple analyte classes and additional contaminants are present in real samples.

Results from Spiked Soils

In general the spiked soil results were reproducible within the 15-20% relative standard deviation range. There were some notable exceptions (certain OCPs) among the analytes that exhibited higher standard deviations and some obviously low recoveries. Method variability less than 10% is a desired goal but since the reproducibility of the determinative step is 2%, attempts at further refinement in the overall methodology is not germane to this report.

Table 1 lists the values obtained for determining toxaphene in spiked soils. The average recovery and percent relative standard deviation were 38.7 ppb for a 25 ppb spike and 17.8% relative standard deviation. There was a toxaphene-like background (appropriate ions in the retention time window) in the soil although the pattern was different from the standard. All soils available to us exhibited a background response for toxaphene ions. The reagent blank for toxaphene was 10 ppb while the unspiked soil contained 13 ppb level of toxaphene-like response that may indicate as much as a 25 ppb level of background when corrected for recovery. The background level and reagent blank thus explains the high recovery from the 25 ppb spike. In the face of a lack of a clean laboratory facility and the lack of a true blank media, it was not deemed essential to try to further improve the results reported here for spiked soils involving toxaphene.

Table 1 also lists the values obtained for determining chlordane. The average recovery and percent relative standard deviation were 31.5 ppb for a 50 ppb spike and 16.7% relative standard deviation. The chlordane blank soil response was about 10 ppb.

Table 2 tabulates values for the toxaphene congeners. The congeners were essentially not detected in blanks. The results are fairly consistent with the overall work reproducibility here with precision between 10% to 20% relative standard deviation.

Table 3 summarizes data for the OCPs. The OCPs were absent in soil and reagent blanks. The levels were consistent with the reproducibility found in this work with the exception of the endosulfan I and II and endrin aldehyde. These compounds are obviously subject to greater recovery variability than the other OCPs. It was first supposed that the β-BHC compound was coalescing with the γ-BHC peak at lower levels and the two compounds would be combined. Later it was realized that the β-BHC was indeed distinct but exhibited a response between 10 to 15 times less than that of γ-BHC and disappeared below the 250 pg/μL level. Distinct observations of β-BHC in spiked soils allowed requantitation from a limited but applicable

calibration response. A calibration plot derived from a new calibration involving higher values for β-BHC has been included. All of the BHC isomers exhibited poor chromatographic peak shape with pronounced tailing.

Method Quantitation Limits (MQLs)

Based on the lowest calibration point in the calibration data, the MQL (simple estimate of lowest reliable quantitation) for toxaphene is 50 pg/μL or 10 ppb for a 5 g soil sample. A similar approach for chlordane yields an MQL of 125 pg/μL or 25 ppb for a 5 g soil sample. The toxaphene congeners were responsive to at least 625 fg/μL which would be 125 ppt for a 5 g soil sample. The OCPs were calibrated to 12.5 pg/μL (except for β-BHC) resulting in a 2.5 ppb level per component in a 5 g soil sample. A more conservative estimate of MQLs can be based on the spiked soil data provided in this report. Method detection limits (MDLs) have not been determined in accordance with SW-846 guidance because the request for a GC/MS protocol was not linked to specific matrices, and performance data as would be required for a full method development were not obtained.

Concluding Remarks

The determination of the complex analytes chlordane and toxaphene was approached by using a number of ion groups spanning the congeners present. This ensures that transformation products will be included in the integrated signal in the context of a real-world analysis. The total ion current integration across the entire response envelop for the composite analyte is the most simple approach but tends to overemphasize the amount present in a real sample in some cases where there may be matrix contributions to monitored ions. In other cases where weathering or transformations have occurred, the comparison of sample signal to the response of a laboratory standard is inaccurate and likely to be inconsistent between NIMS and GC/ECD.

Because of environmental transformations of the multicomponent analytes, congener specific analysis is the more defensible form of determination. Weathered toxaphene determinations suffer from a lack of an appropriate standard to derive meaningful results. Some effort was made to develop macros to use more sophisticated integration but the results were not different enough to justify the increased complexity. The ChemStation software does not handle multiresponse/multicomponent analytes directly as it is optimized for a quantitation ion or a total ion current in the form of a peak at a given retention time. The approach described here thus requires manual intervention (or macro construction) and review, but review is essential even in trivial analyses which must be checked for proper integration and assignment in any serious quality control framework. The current application of the official Method by GC/ECD already integrates the signal over a window so that this approach is consistent with current practice. It should be further emphasized that the oxygen reaction with PCBs has been eliminated as a concern in toxaphene analysis by GC/NIMS, and the presence of internal standard PCB#204 ensures that this is properly demonstrated by monitoring m/z 411 at the retention time of the internal standard when it is run separately.

Notice

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Table 1. Quantitations of spiked soil for toxaphene (25 ppb, 5 g sample) and chlordane (50 ppb, 5 g sample).

Replicate no.	Toxaphene in ppb	Chlordane in ppb
1	34.2	31.4
2	46.4	35.2
3	42.7	29.4
4	35.0	41.6
5	27.0	27.0
6	42.0	27.8
7	43.9	28.0
avg	38.7	31.5
% rel std dev	17.8	16.7

Table 2. Quantitations of spiked soil for toxaphene congeners (500 ppt, 5 g sample).

Replicate no.	Hx-Sed	Hp-Sed	P1	Parlar 26	P2
1	468	532	454	552	542
2	690	722	698	788	894
3	768	746	720	778	836
4	688	724	742	808	910
5	780	752	758	804	942
6	778	786	756	822	928
7	672	784	782	706	1032
avg	692	720	702	752	870
rel std dev %	15.8	12.1	16.0	12.7	17.9

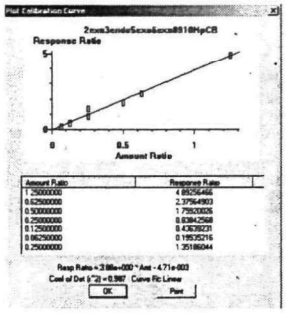
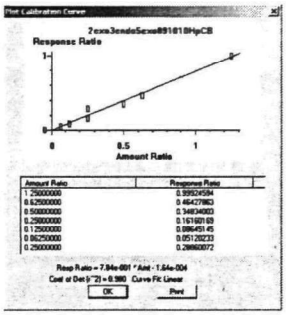
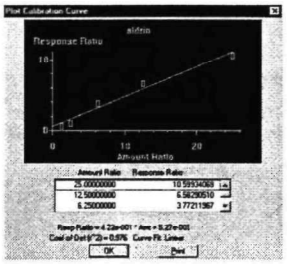
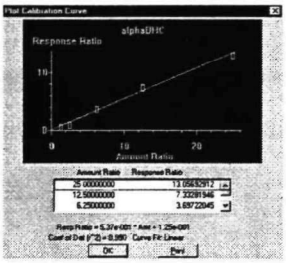
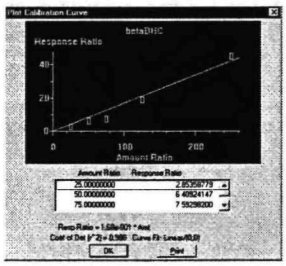
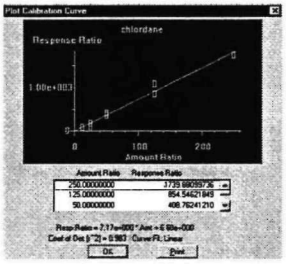
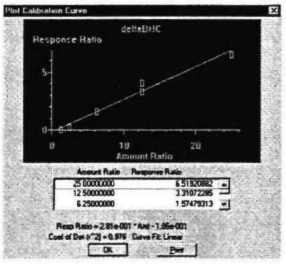
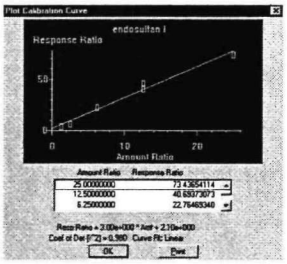
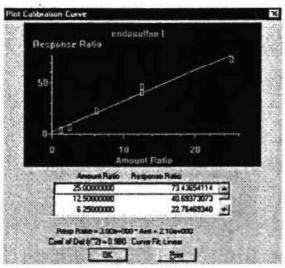
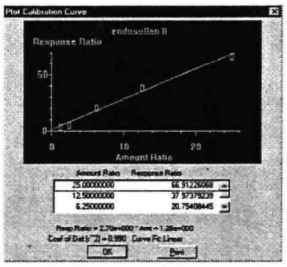
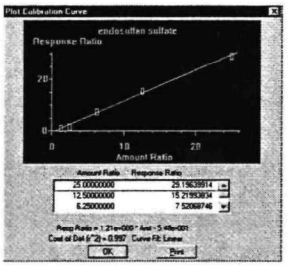
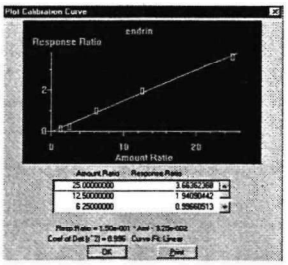
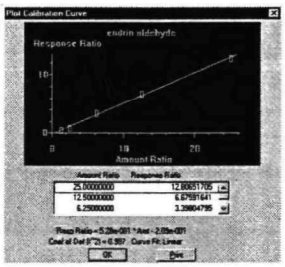
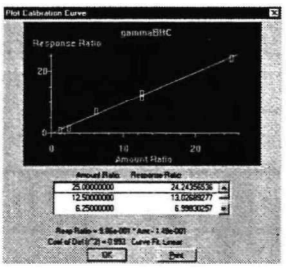
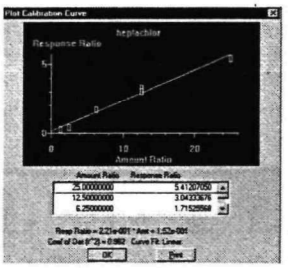
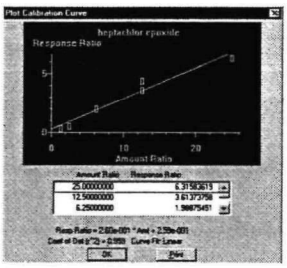
Replicate no.	Parlar 38,40,41	Parlar 44	Parlar 50,62		
1	508	696	544		
2	792	930	812		
3	808	876	702		
4	856	1016	844		
5	860	986	790		
6	826	942	750		
7	866	1266	772		
avg	788	958	744		
rel std dev %	16.1	17.8	13.4		

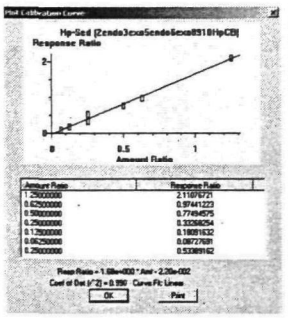
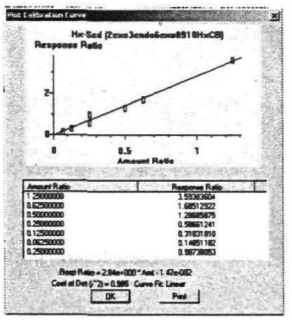
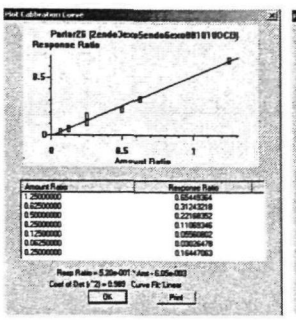
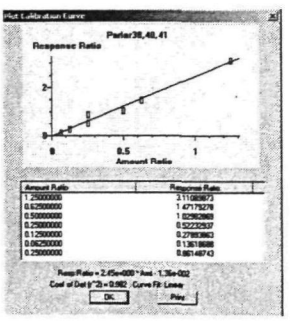
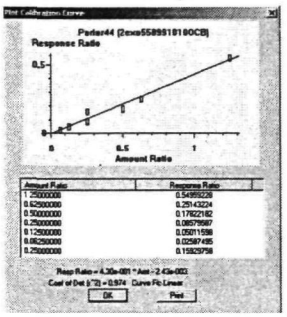
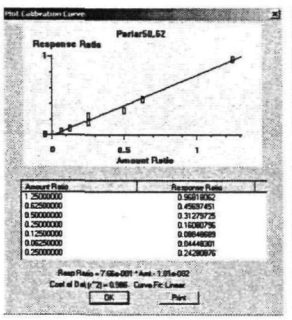
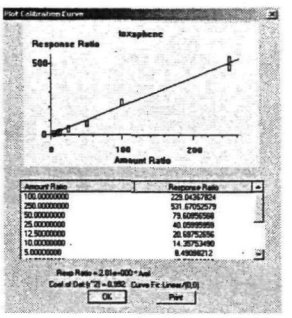
Table 3. Quantitations of spiked soil for OCPs (50 ppb, 5 g sample).

Replicate no.	alphaBHC	betaBHC	deltaBHC	heptachlor	aldrin	heptachlor epoxide
1	28.0	52.0	39.8	34.8	40.8	51.2
2	40.6	57.6	50.0	44.6	53.4	53.0
3	33.0	46.4	37.8	35.4	42.0	46.2
4	27.0	71.6	40.4	28.2	34.4	51.8
5	42.4	71.4	53.4	49.2	56.6	59.6
6	43.2	64.4	51.2	53.0	59.8	62.4
7	37.6	43.2	32.2	49.0	53.0	54.2
avg	36.0	58.0	43.6	42.0	48.6	54.0
% rel std dev	18.7	19.9	18.3	22.0	19.5	10.0

Replicate no.	endosulfan I	dieldrin	endrin	endosulfan II	endrin aldehyde	endosulfan sulfate
1	16.8	73.8	73.6	17.7	26.0	36.6
2	21.4	70.4	72.6	29.4	44.6	47.4
3	37.8	61.2	63.6	38.4	19.7	37.6
4	51.0	69.6	68.6	43.0	25.6	42.8
5	25.2	74.4	72.8	37.8	44.2	51.0
6	20.4	74.2	70.6	29.8	42.8	52.4
7	13.0	62.8	66.6	11.1	37.8	46.2
avg	26.6	69.4	69.8	29.6	34.4	44.8
% rel std dev	50.1	7.9	5.3	39.2	30.1	13.8

Attachments imbedded:
Calibrations plots
Attachments as zip file
Calibrations plots (full size)
Method ouputs as text files

7.7 NIMS GC/MS confirmation/quantitation may be used in conjunction with either single-column or dual-column analysis.

7.7.1 Selected ion monitoring NIMS GC/MS is capable of reaching similar detection limits to those of GC/ECD. The ions monitored and quantitation ions are given in Table 1.

7.7.2 The NIMS GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

7.7.3 NIMS GC/MS may not be used for quantitation when concentrations are below MDLs.

7.7.4 NIMS GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis and the extract of the associated method blank.

7.7.5 The base/neutral/acid extract and the associated blank may be used for NIMS GC/MS confirmation if the surrogates and internal standard do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed..

7.7.6 A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by NIMS GC/MS or EIMS GC/MS.

Table 1. Selected Ion monitoring for analytes by GC/NIMS

Compound	Quantitation ion	Other ions	Comments
toxaphene	TIC	306.9,308.9,310.9,340.9,342.9,344.9,376.9,378.9,380.9,410.8,412.8,414.8,444.8 446.8,448.8	Manual integration
chlordane	TIC	303.9,305.9,337.9,339.9,341.9,371.8,373.8,375.8,407.8,409.8,411.8,441.8,443.8 445.8	Manual integration
alpha-BHC	254.9	252.9,254.9,256.9	Group 1
beta-BHC	254.9	252.9,254.9,256.9	Group 1
gamma-BHC	254.9	252.9,254.9,256.9	Group 1
delta-BHC	254.9	252.9,254.9,256.9	Group 1
heptachlor	299.9	297.9,299.9,301.9,263.9,265.9,267.9	Group 1
aldrin	329.9	327.9,329.9,331.9	Group2 (26.5 min)
heptachlor epoxide	389.8	387.9,389.9,391.9	Group2 (26.5 min)
endosulfan I, II	405.7	403.7,405.7,407.7	Group2 (26.5 min)
dieldrin	379.9	377.9,379.9,381.9	Group2 (26.5 min)
endrin	379.9	377.9,379.9,381.9	Group2 (26.5 min)
endrin aldehyde	379.9	377.9,379.9,381.9	Group2 (26.5 min)
endosulfan sulfate	421.7	419.7,421.7,423.7	Group2 (26.5 min)
parlar 26,38	378.9	toxaphene ions	
parlar 38,40,41	378.9	toxaphene ions	
parlar 50,62	412.8	toxaphene ions	
Hx-Sed	308.9	toxaphene ions	
Hp-Sed	342.9	toxaphene ions	
IS, PCB#204	429.8	410.8	Monitor oxygen reaction