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Application of gas chromatography with photoionization and electron-capture detectors for field screening of semivolatiles in soil and water

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ABSTRACT

The Resource, Conservation and Recovery Act and the Comprehensive Environmental Response, Compensation and Liability Act expanded the list of hazardous chemicals under US Environmental Protection Agency (EPA) regulation during the 1970s and 1980s. This expansion aggravated the backlog of analyses in the EPA Contract Laboratory Program (CLP), and led to the development of field screening methods for volatile organics to augment CLP. We will review field methodology, compare laboratory to field methods, and discuss the applicability of on-site analysis for semivolatiles. Portable gas chromatographs developed for the analysis of volatiles can also be used for field screening of semivolatile organics such as polychlorinated biphenyls, polycyclic aromatic hydrocarbons, pesticides and phenols. The photoionization detector, a common detector for volatiles, would have to be supplemented with an electron-capture detector to analyze the wide range of analytes described in the EPA 8000 series methods. We will describe methods using these detectors for the analysis of soil and water samples.

INTRODUCTION

The passage of the Resource, Conservation and Recovery Act (RCRA) in 1976 and the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA or Superfund) in 1980 led to an expansion of the list of hazardous chemical compounds under US Environmental Protection Agency (EPA) regulation. A variety of new analytical methods were developed for their analysis. Those analyses conducted under the Superfund program during the nineteen eighties resulted in a tripling of the contract laboratory business yet remediation at the sites still progressed slowly as a result of problems with the turnaround time at the laboratories.

Under EPA's Contract Laboratory Program (CLP), turnaround time from laboratories in the program was typically greater than 30 days. Even a 30-day turnaround was not acceptable for the many critical and timely decisions that had to be made. It soon became apparent that there was a significant need for faster, lower cost field screening methods to supplement, and in some cases replace, the laboratory analytical methods [1]. This led to the evolution of field screening and analysis methods in the 1980s by the EPA and the Field Investigation and Technical Assistance Teams. Techniques such as soil gas monitoring [2], headspace [3], and static headspace [4] were adapted for field use instead of the time-consuming purge-and-trap [5] technique used in the laboratory for the analysis of volatiles.

With good technique, field methods, though quite different from laboratory methods, can provide similar results. In Table I, we compare results from the field static headspace and purge-and-trap laboratory methods. A portable gas chromatograph with a photoionization detector was used for static head-space while the laboratory results employed a purge-and-trap with a laboratory gas chromatograph and a mass-selective detector for a

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TABLE I

COMPARISON OF FIELD STATIC HEADSPACE METHOD WITH LABORATORY PURGE-AND-TRAP FOR VOLATILES

Field gas chromatograph: HNU Model 311 with PID; laboratory gas chromatograph: HP5890 with mass-selective detection.

	Static headspace (ppb)	Purge-and-trap (ppb)
Benzene	250	500
Toluene	4300	3200
Xylenes	7800	7200

detector. The correlation coefficient between the methods was 0.99 over the range from a few to several thousand ppb (v/v). Following proper quality control procedures in the field will improve the dependability of the data and its potential use in litigation.

Considerable effort was expended on the development of analytical capabilities to support new field methods. The principal performers among these methods were *portable* field instruments such as the total volatile organics analyzer based on photoionization or flame ionization detection, and portable gas chromatographs. These field methods focused primarily on the analysis of volatile organic compounds frequently lost by volatilization or biodegradation prior to analysis by the contract laboratories. Some of the field methods developed under the Superfund program were found to be very useful and could be modified for the analysis of semivolatile organic hydrocarbons.

We will describe some modifications to EPA methods to simplify for field use and discuss the substitution of the photoionization detection (PID) and electron-capture detection (ECD) methods for the detectors in the EPA 8000 series methods for the field analysis of semivolatile organic hydrocarbons.

METHODOLOGY FOR LEVEL I AND II FIELD SCREENING

The framework of the EPA methodology involves five levels of investigative screening or analyses. The first level (level I) involves field screening with hand held analyzers (EPA protocol specifies a photoionization detector such as an HNU Model PI or HW101) and other site characterization equipment such as an oxygen meter, explosimeter, radiation survey equipment and chemical testing tubes [6]. Level I effort is designed to determine the real-time total level of contaminants present (*i.e.*, *total volatile organics*) that allows determination of the appropriate level of on-site respiratory protection and evaluation of air quality for existing or potential threats to surrounding populations [3]. It is possible to accomplish the following during level I screening: (i) identification of contamination source, (ii) monitoring soil vapor wells to determine the extent of the pollutant plume (*headspace*), (iii) measuring the total concentration profile in a borehole to determine contaminant migration in the ground water or leaching down through the soil and thus contaminating the underlying ground water and (iv) protecting the health of workers involved in the investigation and remedial work.

The two most serious threats from the volatiles involve evaporation into the air and migration away from the original source of contamination through the soil and into a source of ground water. Remediation of the ground water to EPA levels can take years. The plumes from contaminated sources can migrate long distances unless contained. The semivolatile hydrocarbons do not migrate but may have to be removed as a result of their proximity to a source of drinking water, toxicity, or other environmental concerns.

Once a level I screening identifies a contaminated area and delineates its extent, a level II screening can establish the identity of the compound(s) and relative concentrations. Previously, this was accomplished by sending samples to a laboratory for detailed analysis. EPA introduced the intermediate level II analysis in order to reduce both the times required to start remedial actions and the high costs associated with laboratory analysis and keeping trained personnel in the field waiting for results [6]. Level II involves field analysis with more sophisticated instrumentation (*i.e.*, portable gas chromatograph) to provide identification (as far as possible) of specific components present. The final three levels (levels III–V) use laboratories located "off-site" and frequently involve CLP analysis [6]. As such, we will not be concerned with these latter techniques. We are interested in rapid analysis which can be conducted in the field.

The new field methods for volatiles developed under the Superfund program were very successful and could be extended to include the semivolatiles, if some changes in detector technology and methodology are made in the process. Two potentially interesting GC detection systems for field use include PID and ECD since only carrier gas is needed for operation. The need for such field methodology for semivolatiles was obvious in a recent study at a hazardous waste site in Puerto Rico contaminated by DDT [7]. Samples were collected and sent to the laboratory on the two previous visits. We observed additional problems (areas of contamination), not previously encountered on the third visit with the "on-site" analysis by portable GC. During the three days sampling and analysis period, we collected and analyzed forty-four samples. We were finally able to clean up the site during the 1-week time period with field analysis of semivolatiles. This type of success in the field indicates both the advantage and necessity of developing field methodology for semivolatiles.

LABORATORY PROTOCOLS

When samples (containing semivolatiles) from the field site are returned to the laboratory for analysis under EPA protocol [8], an appropriate extraction method is selected for the samples. Manual or liquid–liquid extraction is used for water samples and Soxhlet extraction or ultrasonic extraction is employed for solid samples (soil or sludge). Manual extraction of water samples requires approximately 1 l of sample extracted with three 60-ml portions of methylene chloride in a 2-l separatory funnel. Formation of an unbreakable emulsion during the manual extraction forces the use of liquid–liquid extraction over a 16–24 h period. The organic layers are first dried then carefully concentrated in a Kuderna–Danish apparatus to 1 ml. If the analytical method requires a different solvent, now the solvent can be changed from methylene chloride.

Similarly, the ultrasonic extraction of solid samples (a faster process than Soxhlet extraction) requires that a large sample (on the order of 100 g) be extracted three times with solvent and the combined extracts be concentrated using a Kuderna–Danish apparatus with solvent change occurring during the concentration process.

The sample is analyzed to determine whether any further operations are needed prior to data reduction. If the initial chromatographic analysis shows that there are interfering peaks in the chromatogram, cleanup of the sample is necessary. Column chromatography on silica gel or Florasil or gel permeation chromatography will suffice. This entails a further concentration step with solvent change from the solvent used in the fractionation to the solvent desired for chromatography.

The preparation of the final analytical sample can take from 1 to 3 days for each sample submitted for analysis.

DISCUSSION OF FIELD METHODS

Field extraction of semivolatile hydrocarbons is a much simpler method than the laboratory methods described above. EPA has published a field screening methods catalog as a reference [9]. The development of field screening and analytical methods continued through the 1980s and into the 1990s thanks in large part to the Superfund Program. A procedure from that method's guide for semi-volatiles is as follows: a small sample, 10 ml of water or 800 mg of soil are mixed with 1 ml of a 1:4 mixture of water and methanol and 1 ml of hexane in a vial. The vial is shaken for 30 s and let stand for an additional 30 s to allow the layers to separate. Any emulsion that forms is easily broken by centrifuging the sample. The upper organic layer containing the extracted semivolatile hydrocarbons is injected directly into the gas chromatograph.

The field methods do not have a cleanup step; however, any observed interferences can be noted and the appropriate cleanup procedure applied to the sample undergoing laboratory analysis to provide confirmation of the field results.

One of the most widely used detectors in the laboratory is the flame ionization detector but the use of this detector in the field requires support gases (hydrogen and air) and the detector may take as long as 30 min to stabilize. Clearly, not ideal characteristics for field usage! Nitrogen–phosphorus (NPD) and flame photometric detection (FPD) have similar limitations to the flame ionization detection (FID), namely, the requirement for support gases and long warm-up times. *PID and ECD would appear to be ideal candidates for field detection for the GC analysis of semivolatiles.*

PID is the most frequently used laboratory detection method in a variety of EPA methods [5,8] for volatiles in water or soil. PID is again the most popular detection system for the field analysis of volatiles [7] as a result of its superior sensitivity to flame ionization detection (see Fig. 1 with the 50-fold improvement in sensitivity of PID; note the attenuation difference) and the lack of a support gases (hydrogen and air). Although PID has found considerable application for volatiles (a computerized literature search identified nearly 150 publications using PID for volatiles), there were few applications for the analysis of semivolatile hydrocarbons by PID. We believe that this is more indicative of the popularity of the volatiles methods than the applicability of PID for semivolatiles. Methods are described for the detection of polycyclic aromatic hydrocarbons (PAHs), nitrosamines, chlorinated pesticides, nitrogen containing pesticides, as well as nitro aromatics by PID. These compounds are the semivolatiles described in the EPA methods in Table II. The detection methods used for these meth-

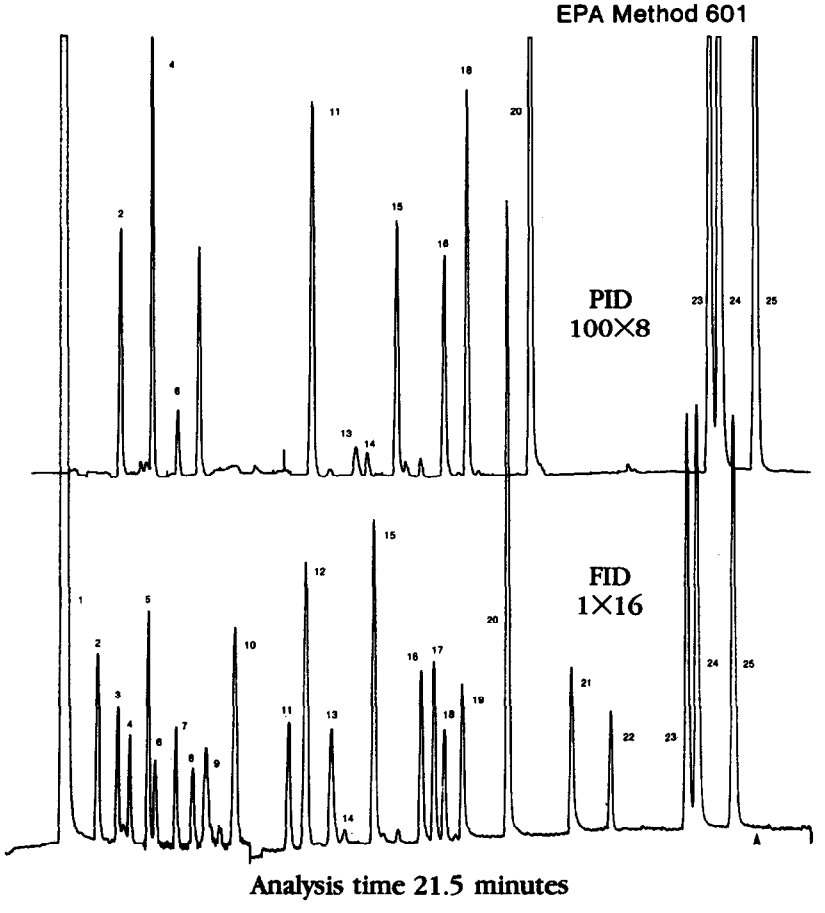

Fig. 1. Comparison of PID and FID sensitivity and selectivity for 601 priority pollutants. Conditions: HNU Model 421 GC; Quadrex Halomatics, 30 m × 0.53 mm I.D.; 3 min at 35°C then to 120°C at 8°C/min, hold at 120°C; 15 ml/min nitrogen. EPA 601 peak identification: 1 = solvent (methanol); 2 = 1,1-dichloroethene; 3 = methylene chloride; 4 = *trans*-1,2-dichloroethene; 5 = 1,1-dichloroethane; 6 = *cis*-1,2-dichloroethene; 7 = chloroform; 8 = 1,1,1-trichloroethane; 9 = carbon tetrachloride; 10 = 1,2-dichloroethane; 11 = trichloroethene; 12 = 1,2-dichloropropane; 13 = bromodichloromethane; 14 = 2-chloroethylvinylether; 15 = *trans*-1,3-dichloropropene; 16 = *cis*-1,3-dichloropropene; 17 = 1,1,2-trichloroethane; 18 = tetrachloroethene; 19 = dibromochloromethane; 20 = chlorobenzene; 21 = bromoform; 22 = 1,1,2,2-tetrachloroethane; 23 = 1,3-dichlorobenzene; 24 = 1,4-dichlorobenzene; 25 = 1,2-dichlorobenzene.

TABLE II

EPA GC METHODS (NON-GC–MS) FOR SOIL AND WATER ANALYSIS

These methods are analysis only (8000 series) and are applicable to ground water and solid waste samples. Sample preparation (extraction and cleanup) are covered by the 3500 series methods (extraction) and the 3600 series methods (cleanup). ELCD = electrolytic conductivity detector.

Method No.	Compounds	Detection
Volatiles		
8020	Volatile aromatics	PID, packed column
8021	Volatile aromatics	PID, capillary
8031	Acrylonitrile	NPD
8032	Acrylamide	ECD
8110	Haloethers	ELCD (ECD alternative)
Semivolatiles		
8040A	Phenols	FID and ECD
8060	Phthalate esters	ECD, packed column
8061	Phthalate esters	ECD, capillary
8070	Nitrosamines	NPD
8080A	Chlorinated pesticides	ECD, packed column
8080B	and polychlorinated biphenyls	
8081	Chlorinated pesticides and chlorinated biphenyls	ECD, capillary
8090	Nitro aromatics	ECD or FID
8100	Polycyclic aromatic hydrocarbons	FID
8120A	Chlorinated hydrocarbons	ECD, packed column
8121	Chlorinated hydrocarbons	ECD, capillary
8140	Organophosphorus pesticides	NPD or FPD, P Mode
8141	Organophosphorus pesticides	Capillary NPD or FPD
8150	Chlorinated herbicides	ECD, packed column
8151	Chlorinated herbicides	ECD, capillary
8410	Semivolatile organics	Capillary GC–Fourier transform IR

ods include ECD, FID, NPD, FPD, Fourier transform infrared and mass-selective detection.

What requirements are necessary to replace FID? The requirements would include a detector with a similar response (namely a carbon counter), a high sensitivity and a wide dynamic range. PID with a 10.2-eV lamp will not respond to small molecules such as methane, ethane–butane that have high ionization potentials (12.98–10.63 eV). For alkanes C_5 and above, PID will respond in a similar manner to FID. Langhorst [10] determined the sensitivities for nearly two hundred compounds for PID with a 10.2-eV lamp. She found that the photoionization detector was a carbon counter (on a molar basis), that the sensitivity for alkanes < alkenes < aromatics; that sensitivities for cyclic > non-cyclic and branched > non-branched; and that for substituted benzenes, ring activators increased sensitivity while ring deactivators decreased sensitivity. Driscoll [11] discussed the improved range and sensitivity for PID compared to FID. Thus, PID is a suitable replacement for FID for many environmental applications [5,11].

The EPA semivolatiles methods that employ FID (Table II) include phenols, nitroaromatics and PAHs. Langhorst [10] detects these compounds with excellent sensitivity. PAHs in asphalt and diesel engine emissions by are analyzed by Arnold [12] with capillary GC and PID (8.3 eV). Sixteen EPA priority pollutants were analyzed in less than 14 min with better detection limits than FID. PAHs in water were separated by HPLC first, then injected into a gas chromatograph with PID to obtain detection limits of 50–100 pg or 10–40 times more sensitivity than FID [13], PAHs in sediment [14], nitroaromatics [15] and phenols [16] for methods 8100,

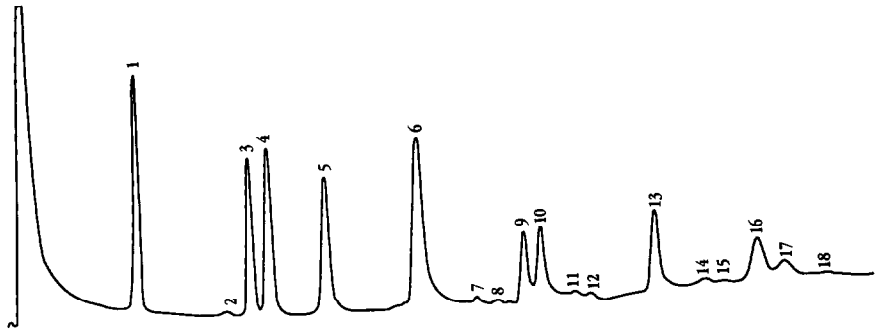

Fig. 2. Chromatograms of PAHs on portable GC. Conditions: HNU Model 311 portable gas chromatograph; NB30, 5 m × 0.53 mm capillary column (HNU); 1 min at 80°C then to 140°C at 16°C/min; hold 1 min, then to 270°C at 14°C/min for 8 min, flow 15 ml/min nitrogen through photoionization detector; 1.5 μl sample containing 20 ng/μl of each PAH; 25 min analysis time; Attennuation × 10. Peaks: 1 = naphthalene; 3 = acenaphthylene; 4 = acenaphthene; 5 = fluorene; 6 = phenathrene/anthracene; 9 = fluoranthene; 10 = pyrene; 13 = chrysene/benz[*a*]anthracene; 16 = benzo[*b*,*k*]fluoranthene; 17 = benzo[*a*]pyrene. Peaks not identified are impurities.

8090, and 8040A, respectively. Some PAHs analyzed on a field portable gas chromatograph with PID are shown in Fig. 2. The HNU GC311 was modified through the addition of temperature programming to enable more rapid elution of higher-molecular-mass PAHs. Nitrosamines are detected with NPD for the EPA method 8070. Meili *et al.* [17] described a GC–PID method for nitrosamines in meat with detection limits of 50–100 pg.

EPA methods 8060, 8061, 8080A and B, 8120A, 8121, 8150 and 8151 are all ECD methods. ECD is quite useful for the analysis of chlorinated compounds and has excellent selectivity. Some of these methods can be performed in the field on an isothermal GC with ECD. Temperature programming as shown in Fig. 2 would also be very useful for the analysis of pesticides to reduce analysis times and improve detection limits for the longer-eluting species. An example of a group of pesticides analyzed by field GC at a level of 0.1 μg/ml is shown in Fig. 3. Chlorinated hydrocarbons such as chlorophenols, chlorobenzenes, DDT, polychlorinated biphenyls and chlorinated pesticides [10,11,18,19] are also detected by PID. Thus, ECD and PID can be used as a complementary detection combination to detect or confirm isomers or less toxic compounds as described by Krull *et al.* [15]. Here, one would have to use a solvent that is optimized for both detectors.

The only other semivolatile methods are 8140 and 8141 for the detection of organophosphorus pesticides. PID will detect nitrogen-containing pesticides [20], nitrogen-containing hazardous pollutants [21] and phosphorus-based pesticides [19].

In summary, we have shown that PID and ECD

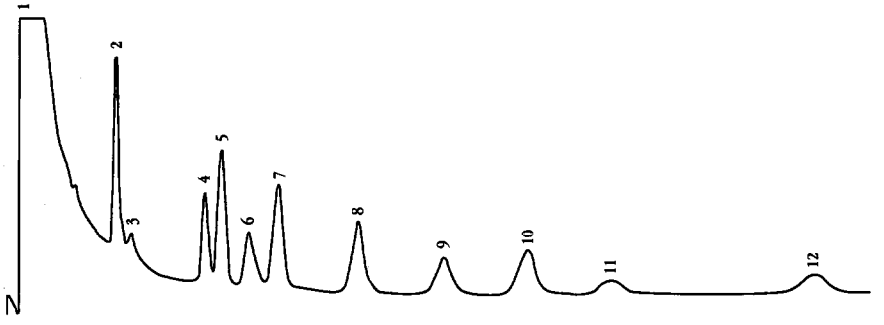

Fig. 3. Analysis of pesticides on portable GC with ECD. HNU Model 311 portable gas chromatograph; conditions: 1 μl containing 0.1 μg/ml of each pesticide; NB30 25 m × 0.32 mm (HNU), isothermal 180°C, column flow 3 ml/min argon–methane; analysis time 14 min; Attenuation × 1. Peaks: 2 = dichlorane; 4 = vinclozolin; 5 = heptachlor; 6 = dichlorfluanid; 7 = aldrin; 8 = heptachlor epoxide; 9 = α-endosulphan; 10 = dieldrin; 11 = β-endosulphan; 12 = endosulphan sulphate. Peaks not identified are impurities.

TABLE III

QUALITY CONTROL PROTOCOL FOR FIELD SAMPLING FOR DDT IN SOIL

An are sample numbers; 1, 2, 3 represent replicates.

Chronological order of samples and standards					
10 ng/μl	Calibration		H1		
1 ng/μl	Standard	91.44% Yield	I1		
A1				Calibration	
A2			J1		
B1			K1		
B2			L1		
C1			L2		
C1	Duplicate	3.48% RPD	L3		
C2				Calibration	
D1			M1		
	Standard	86.53% Yield	N1		
D2			O1		
D3			M2		
	Calibration		N2		
New septa			N2	Duplicate	16.4% RPD
	Calibration			Standard	110.8% Yield
E1			P1		
E2			Z1		
E3			P2		
F1			1 ppm	Standard	93.5% Yield
F2			10 ppm	Standard	121.7% Yield
F3			Z2		
G1			Z3		
G2			Z4		
G3			NH1 (N Horizontal)		
	Standard	84.4% Yield	NH2		
			DDD 10 ppm		
Day 2			10 ppm	Standard	93.5% Yield
	Calibration				
	Calibration				

are useful alternatives for detection methods such as FID, FPD and NPD for the semivolatile compounds. One might trade off some selectivity in the process but the advantages of timely on-site analyses surely outweigh any other considerations.

IMPROVEMENT OF FIELD METHODS

Field analytical methods were determined to be most useful when the contaminants of concern were already identified, so that appropriate methods, dilutions, calibration ranges, etc. could be employed [22]. It was also found that credibility could be lent to field data by using quality control techniques similar to laboratory methods (*i.e.*, duplicates, standards run at regular intervals, etc.). Note that the quality control protocol described in Table III is very similar to laboratory protocol. A quality control protocol was maintained for the analytical results obtained in the field [6] which consisted of analysis of a standard to determine percent recovery and analyzing duplicates on sample extracts to verify analyst reproducibility. The instrument was recalibrated at the beginning of each morning and afternoon shift and at any change in condition (new septa), or shift in peak retention time. Fifteen percent of the samples were returned to the laboratory for verification. In this case, excellent agreement was obtained [6] although different methodology was used in the field and laboratory. The data are shown in Table IV.

The agreement found between the two methods

TABLE IV

COMPARISON OF FIELD *VS.* LABORATORY

Field GC: HNU Model 311 with PID; laboratory GC: HP5890 with ECD.

DDT (ppm)	
Field GC–PID	Lab GC–ECD
0.314	0.170
0.500	0.400
0.774	0.410
5.26	5.560
	6.000
10.235	12.150

was excellent with a correlation coefficient (r^2) of 0.997. Thus with good technique, equipment and quality control, level III type results can be obtained in the field while maintaining flexibility of remediation activities at the site. An example of the quality control protocol used for the field data above is given in Table III.

Field analytical equipment is currently used for on-site detection and identification of volatile organic contaminants in air, water and soil. Portable gas chromatographs such as the HNU Model 311 which are frequently used for characterization of volatile organic compounds can also be used for semivolatile organics, such as pesticides, polychlorinated biphenyls and PAHs. Temperatures of 200°C are required along with dual detector capability to be able to analyze the semivolatiles.

The lack of temperature programming can be offset in some cases by calculating the response factors of the peaks and comparing them with standards in the lab at a later time. This was used by Krull *et al.* [15] to determine nitro containing aromatics in mixtures. The PID response changed slightly with nitro substitution (factor of 3–5) while the ECD response changed by three to five orders of magnitude.

Another method for improving the identification is the retention index monitoring (RIM) system [23] which is a tool for the automatic interpretation (identification) of complex mixtures based on a unique pattern recognition algorithm for search of index peaks. Compound identification is based on two columns of different polarity. Compound libraries are available with space up to 1100 compounds. Separations are accomplished on a pair of fused-silica capillary columns; one using NB-1701 and the other an NB-54 bonded stationary phase. This can be performed in the field isothermally or with temperature programming. Both column inlets are installed into a single column injector. Identifications were made with the Micman identification software (available from HNU Systems) which compares the results on both columns to a pre-established library and then lists results only when the compound is found on both columns. Identifications were based on absolute retention time. The PID results are shown in Fig. 4a for a 10-ppm sample. The detection limit for this method was 0.1 ppm

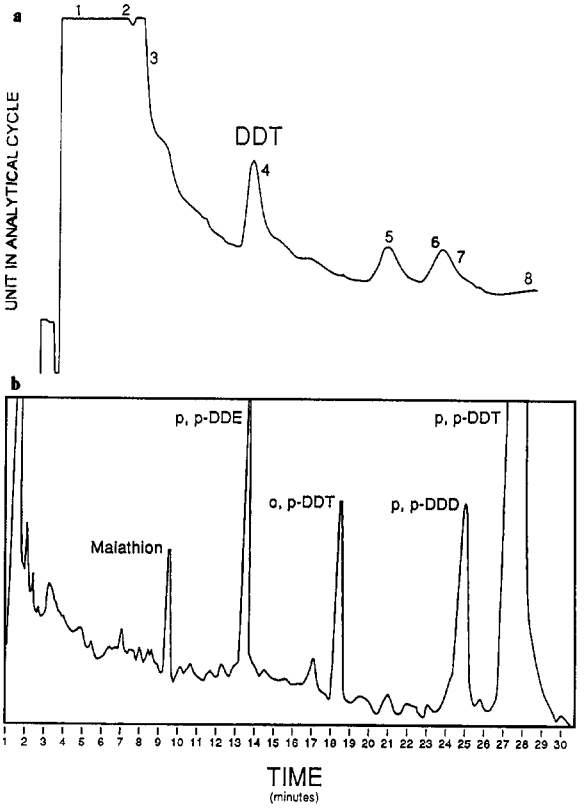

Fig. 4. PID and ECD chromatograms for DDT in soil. HNU Model 311 portable gas chromatograph; (a) PID, conditions (field): NB30 15 m × 0.32 mm capillary column (HNU), isothermal 180°C, column flow 15 ml/m in of prepurified nitrogen; attenuation × 1; analysis time 10 min. (b) ECD, conditions (laboratory): NB30 25 m × 0.32 mm capillary column (HNU), isothermal 150°C, column flow 15 ml/min of prepurified nitrogen; attenuation × 10. Peak: 4 = DDT (10.235 ppm). Peaks not identified are impurities.

and the action limit for DDT was 2 ppm so that the method had sufficient sensitivity to determine whether the soil was contaminated. ECD is a more selective detection method for this application with a detection limit two orders of magnitude lower, but for field applications, we can see that the additional sensitivity or specificity was not needed. In Fig. 4b, other chlorinated isomers of DDT were identified along with low levels of malathion which was not supposed to be present at this site. The peaks with a longer retention time than DDT do not appear to be pesticides since there was no response with ECD. They are probably hydrocarbon impurities in the solvent. The detection limit for DDT with ECD was < 1 ppb.

We have shown that the sample preparation for field methods can be simplified for semivolatiles. This will enable more rapid and "on-site" availability of tests needed for remediation. GC with a PID and ECD would appear to have sufficient sensitivity and selectivity to analyze all of the 8000 series semivolatiles for water and soil. Typical methods could be performed in the field with analysis times of between 20 and 60 min.

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