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Project Summary

Thermal Degradation Characteristics of Environmentally Sensitive Pesticide Products

Debra A. Tirey, Barry Dellinger, Wayne A. Rubey, and Philip H. Taylor

The thermal decomposition properties of the active ingredient of 16 pesticides have been theoretically examined. The parameter used to rank their stability was the temperature required for 99% destruction at a gas phase residence time of 2.0 sec under oxygen-starved conditions (T_{99}(2)). Experimental studies on five pesticide-related materials were also conducted under controlled laboratory testing.

Experimental studies of the high-temperature oxidation and pyrolysis of four key pesticide materials including the identification and quantification of products of incomplete combustion (PICs) were conducted. The four pesticides were Aldicarb* and Phorate (both insecticides) and Atrazine and Alachlor (both herbicides). These compounds are the active ingredients of Thimet, Temik, Aatrex-Nine-O, and Lasso II, respectively. A fifth material, a polyethylene blend bag which is used as an Atrazine container, was also examined.

The examination of the incineration ranking among the 16 subject pesticides indicated that they should be considered thermally fragile. However, each pesticide in the controlled laboratory testing decomposed to yield a large number of reaction intermediates. More intermediates were consistently produced under pyrolytic conditions. Although most of the intermediates were decomposed by 700°C, some persisted at the maximum testing temperature, 1000°C. It appears that these materials tested may be amenable to properly controlled, high-temperature incineration.

We also concluded that open burning of spent pesticide bags may not significantly reduce their effect on the environment and that the analytical protocols associated with the monitoring of decomposition products from pesticide materials should be further developed.

This Project Summary was developed by EPA's Risk Reduction Engineering Laboratory, Cincinnati, OH, to announce key findings of the SITE program demonstration that is fully documented in a separate report (see ordering information at back).

Introduction

Pesticides, insecticides, herbicides, and fungicides are applied worldwide to control rodents, insects, weeds, and fungi thought to be a direct threat to human health or to livestock and crops raised for human consumption. A renewed concern over the effect of applying these chemicals at an ever expanding rate, both to the environment and to people, has, however, recently been raised.

Many studies conducted to address these concerns have evaluated the persistence and toxicology of pesticide materials in plant and animal tissues and in soils. These studies indicate that most pesticides themselves are fragile compounds that are readily transformed in the environment to other metabolites that may or may not be more toxic or more persistent than the parent material.

What happens, however, when pesticides are thermally decomposed, as in the case of open burning of spent bag

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

materials containing trace quantities of pesticides—a common practice for many farmers? Or, what occurs when a pesticide has been determined through "persistence and toxicological" studies to no longer be suitable for widespread use and is suddenly banned? The method of choice to dispose of these materials is, in many instances, incineration. How will these materials react upon thermal decomposition? A review of the open literature suggests that only limited information is available.

In this study, the thermal decomposition properties of the active ingredient of 16 pesticides have been theoretically examined, and experimental studies on 5 pesticide-related materials were conducted. The theoretical stability evaluations were prepared with the use of available laboratory data or with data on structurally similar compounds in conjunction with chemical reaction kinetic theory (Table 1). The parameter used to rank their stability was the temperature required for 99% destruction at a gas phase residence time of 2.0 sec under oxygen-starved conditions, $T_{99}(2)$. Table 1 also indicates the thermal stability ranking and the thermal stability class ranking of each of the pesticides within the hierarchy of the 330 individual compounds and 7 classes currently classified (a ranking of 1 being most stable). Data sheets on each pesticide are presented in the full report.

Experimental Procedures

An experimental study of the thermal degradation characteristics of Aldicarb, Phorate, Atrazine, and Alachlor was undertaken. This included a successful atom balance for carbon, nitrogen, sulfur, phosphorus, and chlorine.

Instrumentation

All experiments were performed on the Thermal Decomposition Analytical System (TDAS). The TDAS is a closed, in-line, quartz flow reactor system capable of accepting a solid, liquid, or gas phase sample, of exposing the volatilized sample to a highly controlled thermal environment, and then of analyzing the effluents resulting from this exposure.

Gas-phase samples are swept with carrier gas of helium through heated transfer lines into a quartz flow reactor where controlled high-temperature exposure occurs. Mean residence times of 0.5 to 6.0 sec may be achieved. Thermal decomposition data may be taken over the temperature range 200° to 1050°C.

The effluent resulting from thermal exposure is swept by carrier gas to a Hewlett Packard 5890 gas chromatograph (GC) where it is cryogenically focused on the head of a capillary column located inside the GC oven. Later, the oven temperature is raised at a specified rate and the separated compounds eluting from the column can then be sent to either the ion source of an HP 5970B mass selective detector (MSD), or to a flame ionization detector (FID) located within the GC assembly.

Data acquisition and analysis for the TDAS was done with the aid of an HP 59970 ChemStation and the accompanying system software that includes an online National Institute of Health/U.S. Environmental Protection Agency (NIH-EPA) mass spectral library. It was also necessary to develop an interface between the GC and the MSD. This interface, the insertion-split, was designed as a compromise between the typically used "direct-to-source interface" and the "open-split interface" and incorporated the meritorious aspects of each. The direct-to-source interface promotes heightened sensitivity for the mass spectrometer because effluents from the column are deposited directly into the source. However, having the GC column outlet placed directly in the source of the mass spectrometer (typically held at 10-6 or 10-7 torr) creates a huge pressure drop across the column that literally pulls volatile compounds through the latter portion of the column without allowing for any separation that may be attained by interaction with the liquid stationary phase. For these experiments, it was paramount that such compounds as CO_2, CH_4, and the light C_2 gases be separated, since these were predicted to be major PICs.

The open-split interface provides for the use of much larger column bores and larger sample sizes but protects the source of the mass spectrometer from undue wear since much of the column effluent is diverted before it enters the source. One of the chief drawbacks of this type of interface is the loss of sensitivity relative to the direct-to-source interface. Since one of the goals of this study was to perform a mass balance of the data, isolating and analyzing as many of the products as possible was imperative. The insertion-split interface provided the best answer to these two dilemmas.

Essentially the insertion-split interface is a small-bore transfer tube placed within a capillary column; the tube is surrounded by carrier gas that is constantly being swept away. A drawing of this interface as it is installed in the TDAS is presented in Figure 1. A piece of narrow-bore, fused, silica tubing (uncoated but deactivated) is positioned in the source of the mass spectrometer in much the same way that the capillary column would be positioned if it were direct-to-source, with a fixed length left on the oven side of the connection nut (can be a variable length, 10 to 20 cm). The end of this tubing remaining in the oven is then placed inside the outlet end of the GC capillary column. Obviously,

Table 1. *Pesticide Thermal Stability Data*

Pesticide Compound	*Oxygen-Starved Condition, $T_{99}(2)$(°C)*	*Stability Ranking*	*Stability Class Division**
DCPA	*750*	*105-114*	*3*
Alachlor†	*620*	*185-189*	*4*
Acephate	*595*	*207*	*5*
Pronamide	*570*	*220*	*5*
Carbonfuran	*560*	*226-228*	*5*
Triallate	*550*	*231-234*	*5*
Fonofos	*530*	*237-241*	*5*
Ethoprop	*530*	*237-241*	*5*
Chlorprifos	*510*	*249-251*	*5*
Atrazine†	*510*	*249-251*	*5*
Terbufos	*510*	*249-251*	*5*
Cyanazine	*500*	*253-258*	*5*
Azinphos methyl	*460*	*266-269*	*5*
Phorate†	*400*	*276-277*	*6*
Methomyl	*200*	*318-320*	*7*
Aldicarb†	*200*	*318-320*	*7*

** Ranking of 1 indicates most stable.*
† Derived from experimental data obtained from this study.

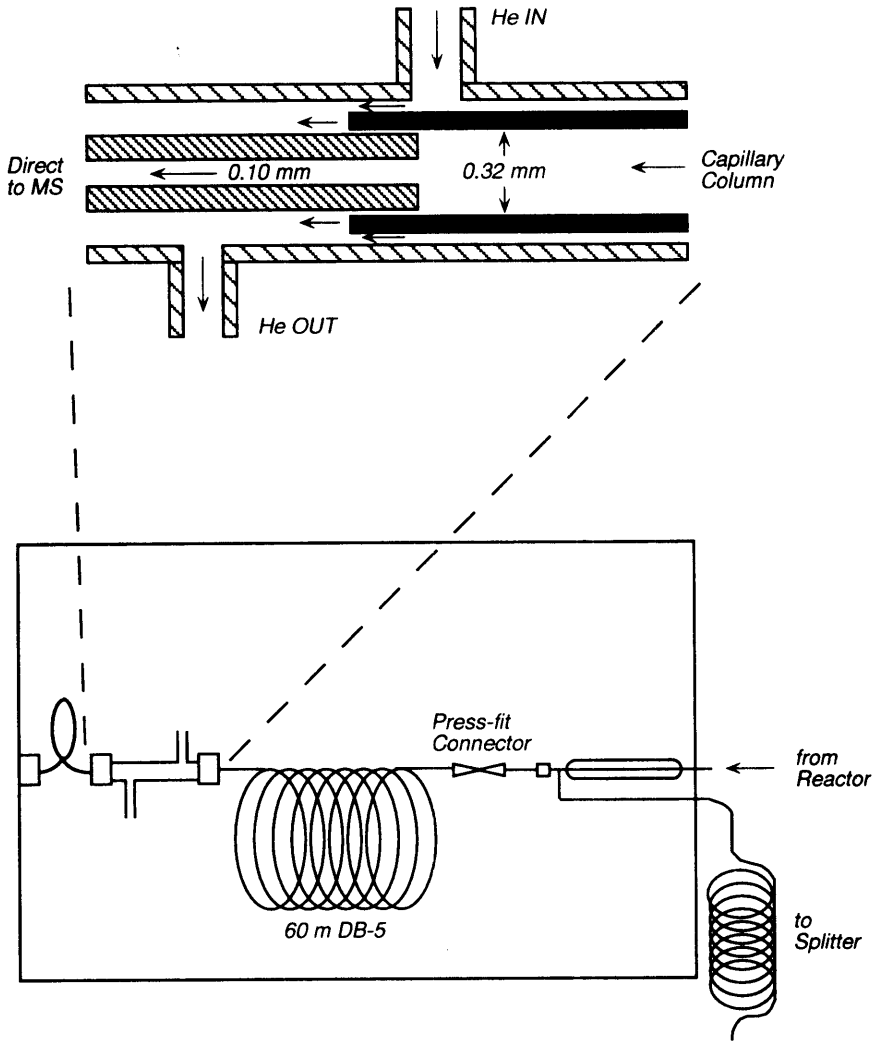

Figure 1. *Close-up schematic of the insertion-split interface as it is configured in the TDAS.*

attention must be paid to the inner and outer dimensions of the two tubes. The film thickness of the capillary column is also important relative to gas chromatograph-mass spectrometer (GC-MS) operation. The ends of the transfer tubing and the capillary column that overlap are housed within a stainless-steel piece of tubing fitted with inlet and outlet gas flows; this allows a gaseous carrier to be purging the area surrounding the junction at all times. Finally, the entire stainless-steel miniaturized housing is firmly mounted inside the GC oven so as to remain stationary even while the GC oven fan is running.

Because there is a finite annular gap between the outer wall of the transfer tubing and the inner wall of the capillary column and because this junction is kept pressurized by the addition of flowing helium carrier surrounding the two overlapping tubes, the outlet to the capillary column now experiences approximately atmospheric pressure. Thus, lightweight materials are not "pulled" through the column without being separated as with the direct-to-source interface. Also, because the transfer tubing is placed inside the capillary column, transfer of sample from column to mass spectrometer (MS) is almost continuous; there is no axial or open gap in the flowpath of the column effluent as would be experienced by the open-split interface. This fact helps to maintain relatively good sensitivity for this interface despite the fact that there is some splitting of the sample at the column overlap junction.

Before invoking the insertion-split interface for this program, considerable developmental work went into testing the linearity of splitting for lightweight as well as for heavy materials (i.e., whether heavy materials would be preferentially split relative to lightweight compounds because of the axial position they would tend to occupy while traveling through the capillary column). The insertion-split interface design used a bluff-body mixing principle at the annular split location as a contingency for this concern. Test results indicated that, indeed, the bluff-body design performed as expected; splitting of test samples containing a wide-molecular range of compounds was consistently the same for light, intermediate, and heavy compounds. (The compounds used were octane, octadecane, and octacosane in cyclohexane). We also found that for a given volumetric column flow, linear velocity, and head pressure, the insertion-split interface response was always a fixed fraction (approximately 40% to 50%) of that experienced with the direct-to-source interface. This number depends on the length and diameter of the transfer tubing positioned in the source of the mass spectrometer.

Sample Introduction

Standards of each of four pesticides were received from the National Repository located at Research Triangle Park, NC. The purity of each was certified as greater than 98% (i.e., Aldicarb 99.8%, Phorate 98.2%, Atrazine 99.4%, and Alachlor 99.6%). Aldicarb, Atrazine, and Alachlor were solids at room temperature, whereas Phorate was a viscous liquid. The Atrazine 90 DF bag was clean—no pesticide material had been placed in it.

Target test conditions for the four pesticide active ingredients were (1) 1% mol/mol of pesticide in carrier gas, (2) a gas phase residence time (tr) = 2.0 sec, and (3) two reaction atmospheres: pyrolysis at a fuel/oxygen equivalence ratio (phi) of 10 and oxidation at phi = 0.5 (100% excess oxygen). The Atrazine bag material was run only under oxidative conditions.

Sample delivery for the three solid pesticide active ingredients (Aldicarb, Atrazine, and Alachlor) in this set of experiments involved dissolving the solid material in a suitable solvent and depositing an aliquot of the solution into a quartz pyroprobe tube. Once the solvent had evaporated, the tube was placed in the platinum coil of a CDS Model 120 pyroprobe assembly (Chemical Data System, Inc.) that was then placed into the insertion region of the TDAS. With the use of temperature programming of both the insertion region itself and/or the pyroprobe heating coil, each pesticide was volatilized into flowing carrier gas at a

specific, reproducible rate. Separate thermal gravimetric analysis (TGA) experiments were performed in flowing air and nitrogen to aid in determining the first approximation of these temperature protocols.

Approximately 40 quartz tubes were loaded with sample from the same stock standard solution, and solvent was allowed to evaporate. The tubes were then kept covered at room temperature in a laboratory hood until just before their use. Loading the quartz tubes in this manner provided the best reproducibility with regard to sample size.

Sample delivery for the only liquid active ingredient, Phorate, was more straightforward than that for the three solids. The pure liquid (0.5 µL) was injected into an insertion region held isothermally at 100°C by using a 0.5 µL full-scale liquid syringe fitted with a 6-in. needle.

For the polyethylene bag, approximately 2 mg of bag material was placed in a quartz pyroprobe tube (loaded with a plug of quartz wool to keep the piece of polymeric material from falling out). The tube was then placed in the platinum coil of the CDS pyroprobe assembly and put into the insertion region of the TDAS. As with the solid pesticides, temperature programming of both the insertion region itself and the pyroprobe heating coil volatilized the sample into flowing carrier. A separate TGA experiment was performed in flowing air to aid in determining the first approximation of this temperature protocol.

The profiles generated using these sample introduction techniques delivered the maximum possible part per million (mol/mol) concentration of pesticide in the carrier gas while also delivering a suitable sample size to the analytical system downstream of the reactor for adequate conversion to products. The concentration of active ingredients ranged from 0.1% to 0.5% mol/mol in the carrier, with the sample sizes ranging from 77 to 500 µg. Table 2 presents a summary of the pesticide concentrations in carrier gas used in this study, the insertion region protocols that delivered these values, and the accompanying oxygen concentrations required for phi = 0.5.

Degradation of each of the three pesticides was conducted under both oxygen-deficient (fuel/oxygen equivalence ratio of 10) and oxygen-rich (fuel/oxygen equivalence ratio of 0.4 to 0.5) conditions. Approximately 0.5% oxygen in helium (mol/mol) was available for combustion in the oxygen-deficient conditions (as determined by actual measurement of the reaction gas), whereas a 10% mix of oxygen in a balance of 51% helium/39% nitrogen was used for the oxygen-rich experiments (these were the values obtained by mixing compressed air and helium at a one-to-one ratio vol/vol). Gas mixtures were prepared by using a gas mixing device developed inhouse. Residence time at temperatures for all exposures regardless of atmosphere was held constant at 2.0 sec; the reactor temperature varied over the range 200° to 1000°C. Experiments were conducted at 1.23 atm. Sample was introduced by using the protocols described in preceding paragraphs.

The effluent resulting from a single reactor exposure (unreacted parent material and all PICs) was directed to a 60 m, DB-5, 0.32-mm i.d. column (J&W Scientific, Inc.) held at -60°C with the use of liquid nitrogen as coolant. Individual reaction products were separated by programming the GC oven from -60° to 290°C at 10°C/min with a 15-min hold at -60°C and a 25-min hold at 290°C. Detection was accomplished with the aid of an HP 5970B quadrupole mass spectrometer. The mass spectrometer was operated in full-scan mode with an electron energy of 70 eV and an electron multiplier setting of 1700. To optimize detection of products, during the first 20 minutes of the GC program, the mass range scanned was 10 to 200 amu; from

Table 2. *Volatilization Parameters and Concentration of O_2 in the Carrier Used to Achieve Target Test Conditions*

				Pyrolysis		*Oxidation*	
Compound	*Insertion Region Program*	*Pyroprobe Program*	*Conc. (mol/mol)*	*% O_2* in Carrier*	*phi**	*% O_2 in Carrier*	*phi*
Aldicarb/s‡	*Isothermal @200°C*	*No Program*	*4000*	*0.5(0.5)*	*10(10)*	*10(9)*	*0.5(0.5)*
Phorate/1	*Isothermal @100°C*	*Not used*	*5000*	*0.5(0.5)*	*10(10)*	*10(7)*	*0.5(0.5)*
Atrazine/s	*Isothermal @275°C*	*Ambient to 275°C@ 20°C/ms hold 20s*	*3000*	*0.5(0.5)*	*10(10)*	*10(10)*	*0.5(0.5)*
Alachlor/s	*Isothermal @ 300°C*	*Ambient to 300°C @ 20°C/ms hold 20s*	*4000*	*0.5(0.5)*	*10(10)*	*10(8)*	*0.5(0.5)*
Atrazine DF bags	*Isothermal @ 250°C*	*Ambient to 500°C @ 20°C/ms*	*Not applicable§*	*0.5*	*Not applicable*	*10*	*Not applicable*

* *Concenration of oxygen mol/mol in carrier required for complete combustion based upon the stoichiometric equation for the pesticide listed as: actual (theoretical).*

\+ *Phi value for the oxidative experiments listed as: actual (theoretical).*

‡ *"s" indicates solid phase sample. "1" indicates liquid phase sample.*

§ *No attempt was made to calculate a "concentration" or "phi" value for the polymer.*

20 to 80 min, the mass range scanned was 10 to 500 amu. This allowed for maximum detection of light gasses during the first part of the GC program. Quantitation and identification of products was determined with the aid of an HP ChemStation data system and an on-line NIH-NBS mass spectral library as well as through manual interpretation.

Analytical standards for observed products were run wherever possible to obtain quantitative response factors. Where obtaining a product was either impossible, extremely difficult, or untimely (i.e., a 6-wk or 2-mo waiting period), analytical standards were run for compounds in the same class or closely related in structure to the compound of interest. These response factors were then used to estimate the response factors for the actual products seen in thermal decomposition experiments. Response factors were typically obtained from 4 or 5 point calibration curves with some replicate points being performed where possible.

Standards were injected into the TDAS using the same timetable, valve switching, split ratio, and GC program as were the pesticides. Thus, response factors generated from the curves could be used directly to perform absolute quantification of the area responses reported in each data run by using the following equation:

Ng of compound detected =
Area counts of
compound/response factor

The "ng detected" values were then converted with the use of molecular formulas and molecular weights to yield mass and/or moles of carbon, nitrogen, sulfur, phosphorus, and chlorine. In this way, a balance of the atoms at each temperature could be evaluated. The compiled list of analytical standards run for this program and the response factors determined in both reaction atmospheres are given in Appendix 2 of the full report.

Results

The chromatograms generated in this study were very complex, especially at intermediate destruction temperatures. For example, the thermal decomposition of Alachlor under oxygen deficient conditions yielded greater than 80 different PICs over the temperature range of 275° to 1000°C. A typical example chromatogram obtained from the Alachlor experiments is presented in Figure 2.

Although the excess oxygen chromatograms were generally less complex than were the pyrolysis ones, a relatively large number of products were nevertheless detected in these experiments as well. The number of byproducts observed for each of the pesticides is summarized in Table 3.

The metabolite studies found in the literature reported that these four pesticides were not persistent in the environment and that they were readily transformed to other compounds. Their thermal stability as determined under the conditions of this study was analogous to this behavior. All four compounds themselves were labile, disappearing by 600°C under both pyrolytic and oxidative conditions. The relative stabilities can be conveniently ranked by the temperature required for 99% destruction for a 2.0 sec residence time ($T_{99}(2)$) (see Table 4). The relative stabilities under both sets of conditions in this study were: Alachlor > Atrazine > Phorate > Aldicarb. Aldicarb and Phorate exhibited degradation at the lowest reactor temperature possible on the TDAS, 200°C. Because no Aldicarb was detected in the quantitative transport run at 200°C, this temperature was assigned as its $T_{99}(2)$ value. In the case of Phorate, the $T_{99}(2)$ value is the temperature at which no Phorate was detected in replicate runs. A more in-depth explanation of the problems associated with running Phorate are discussed in the full report.

Interestingly, none of the pesticides displayed a large dependence upon reaction atmosphere. From this observation, one can infer that the decomposition mechanisms may be dominated by unimolecular pathways. A more pictorial representation of this can be seen in the thermal decomposition composite curves generated for the pesticides Alachlor and Atrazine in Figure 3.

The specific products detected in the decomposition studies of Aldicarb, Atrazine, and Alachlor in the form of weight % yield (relative to parent) for each atmosphere can be found in Tables 5 through 10 of the full report. Many of the products detected in these experiments may be environmentally significant.

Because of the viscosity of Phorate, the small volume available for sampling (i.e., only 50 µL in a 1.5-mL vial volume) and the short "shelf-life" of Phorate once exposed to the atmosphere, the reproducibility of injection was not good. Replicate and triplicate injections at each reaction condition resulted in relative standard deviations of as much as ± 36%. For this reason, only qualitative analysis of the Phorate decomposition products are listed in Tables 11 and 12 of the final report. There, all areas of peaks not identifiable by their mass spectra are summed under the heading "Unidentified."

In the full report, the results obtained from the Atrazine bag oxidation experiments are similarly presented (i.e., as qualitative analysis of the decomposition products). The act of volatilizing the polymer in the insertion region necessarily makes "weight % yield (relative to parent)" type data meaningless. As with Phorate, areas of peaks we were unable to identify are summed under the heading "Unidentified." The products seen from this series of experiments were the same ones observed in previous studies conducted in this laboratory in which polyethylene and polyethylene/polypropylene blends were thermally decomposed.

Mass balance for the pesticide experiments was achieved with a fairly good degree of success. Although some temperature data points were clearly outliers, most data points were within ± 30% of the 100% recovery mark. A listing of the atom balances for C, N, S, or Cl where appropriate are presented in Appendix 3 of the full report. Because of the high degree of uncertainty associated with the Phorate data, no atom balances were attempted.

Conclusions

The experimental results of this study are very complex. Some simple conclusions are, however, readily apparent.

1. Based on the stability of the parent pesticides and their thermal byproducts, these materials may be amenable to properly controlled, high-temperature incineration. The number yields and stability of the byproducts suggest, however, that open burning of spent bag materials containing pesticide residues may not significantly reduce their effect on the environment.
2. When compared with a previously generated ranking of hazardous waste incinerability, the 16 subject pesticides (with the possible exception of DCPA) should be considered thermally fragile (i.e., $T_{99}(2) < 600°C$).
3. With the exception of Alachlor, reaction atmosphere had almost no effect on pesticide stability. This suggests that the primary mechanisms of decomposition are unimolecular (e.g., simple bond rupture or more complex concerted intramolecular reactions).
4. Each pesticide decomposes to yield a large number of reaction intermediates. More intermediates were consistently produced under pyrolytic conditions. Most intermediates were decomposed by 700°C; however, some persisted at

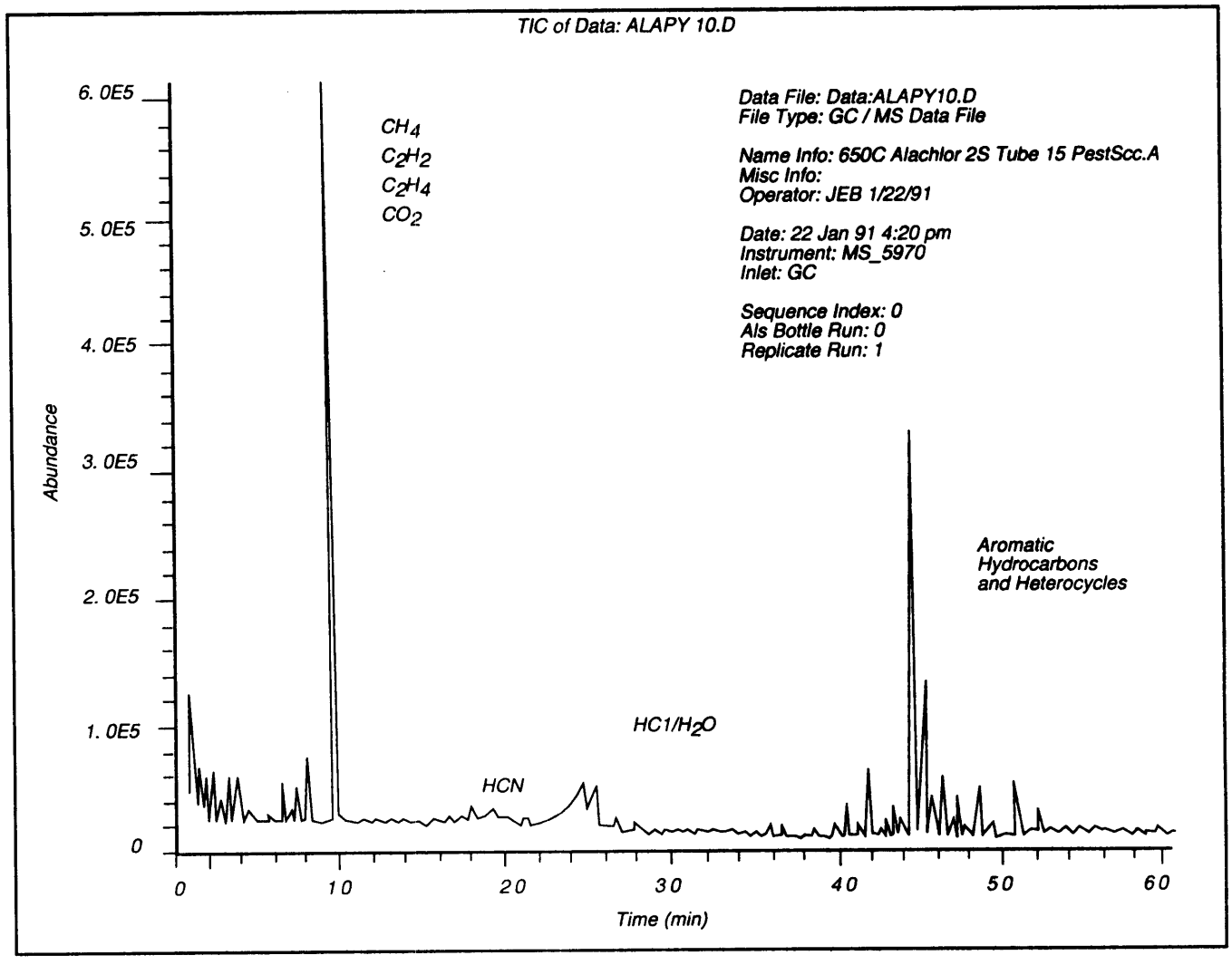

Figure 2. *Example chromatogram generated for Alachlor pyrolysis, phi=10, 4000 ppm, 2 sec residence time, 1.23 atm.*

the maximum temperature in this study, 1000°C. The most stable organic intermediates were primarily nitrogen-containing compounds (i.e., nitriles and cyanides).

5. Relatively good mass balances were obtained for three of the pesticides; this suggests a reasonably complete set of product identifications.
6. Polyethylene bag oxidation intermediates did not appear to be as environmentally significant as the pesticide intermediates.

Table 3. *Number of Decomposition Byproducts Observed*

Pesticide	*phi = 10*	*phi = 0.4 to 0.5*
Aldicarb	*39 (23)**	*22 (17)*
Phorate	*31 (19)*	*25 (17)*
Atrazine	*63 (50)*	*47 (36)*
Alachlor	*86 (59)*	*29 (23)*

* *( ) indicates the number identified by the mass spectra, remainder listed as unidentified.*

Recommendations

Because of the numerous byproducts and complex chemistry observed as the results of the thermal degradation of pesticides, we make the followings recommendations:

1. Additional laboratory testing of other pesticide products suspected to be environmentally sensitive should be performed. Results of these studies can be used to evaluate the environmental effect of pesticide burning and to guide larger scale evaluation programs.
2. The analytical protocols associated with the monitoring of decomposition products from pesticide materials should be further developed. Many of the byproducts observed in our

Table 4. *Pesticide Stability*

	$T_{99}(2)$ (°C)	
Pesticide	*phi = 10*	*phi = 0.4 to 0.5*
Aldicarb	*<200°C*	*200°C*
Phorate	*<400*	*<275*
Atrazine	*510*	*475*
Alachlor	*620*	*525*

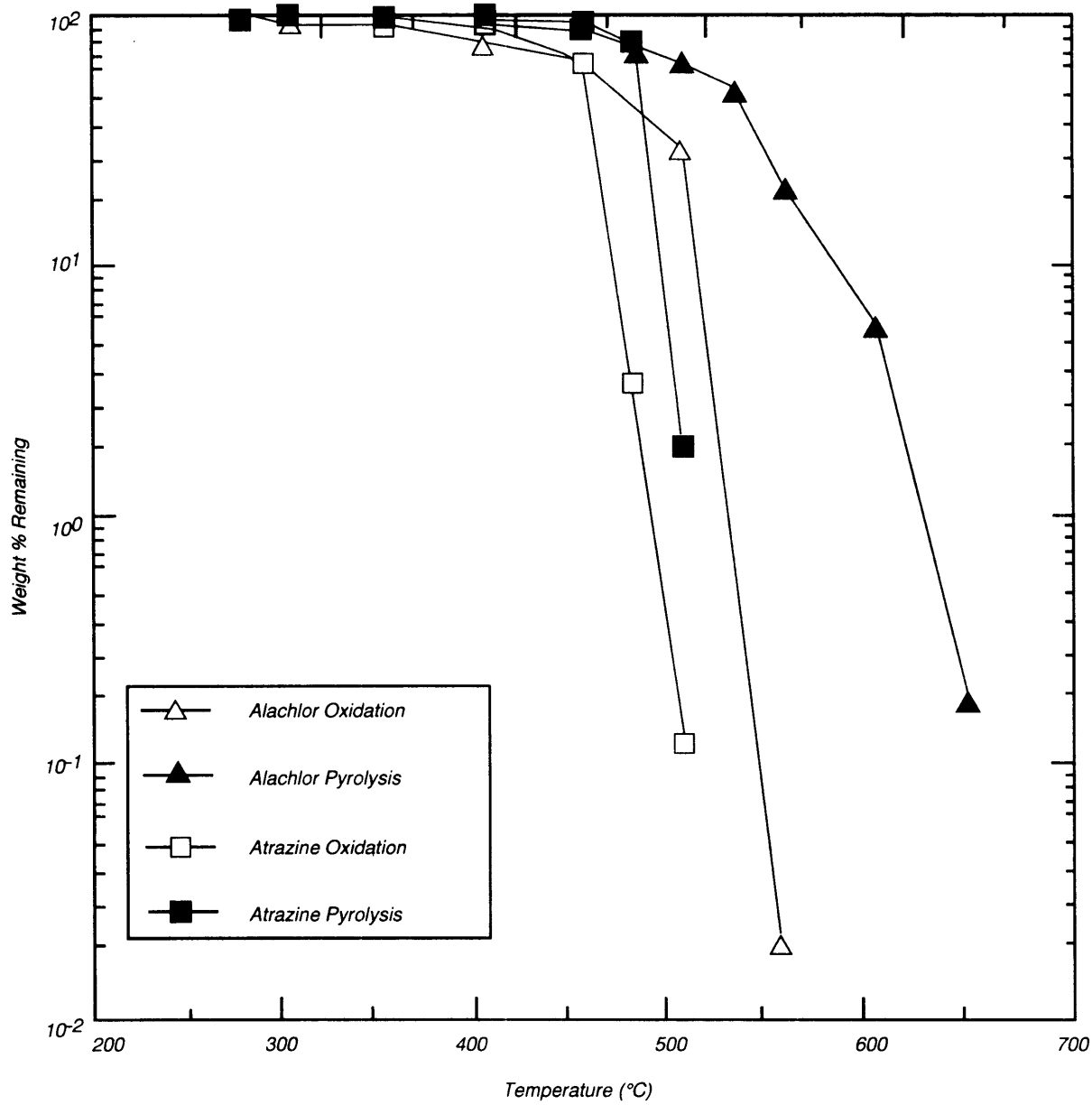

Figure 3. *Weight % remaining curves for parent materials, Alachlor and Atrazine, generated under oxidative (phi = 0.5) and pyrolytic (phi= 10.0) conditions, 2.0 sec residence time, 4000 and 3000 ppm respectively, and 1.23 atm.*

laboratory evaluations are polar and may be water soluble thus complicating their analysis. Standardized analytical techniques are not available for many compounds that may be environmentally significant.

3. Thermal decomposition chemistry and kinetics of pesticides should be the subject of further research so that the open burning and incineration behavior of pesticides may be better understood. Organic nitrogen and sulfur combustion chemistry are largely unexplored fields of apparent environmental significance.
4. Close coordination between laboratory researchers and field test researchers should be attempted to ensure that the products identified in the laboratory are targeted for measurement in the field.
5. Toxicological evaluation of the observed byproducts should be done to aid in determining the environmental risk associated with open burning and incineration of pesticides.

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D. A. Tirey, B. Dellinger, W. A. Rubey, and P. H. Taylor are with the University of Dayton Research Institute, Dayton, OH 45469-0132.
***D.A. Oberacker and P.C.L. Lin** are the EPA Project Officers (see below).*
The complete report, entitled "The Thermal Degradation Characteristics of Environmentally Sensitive Pesticide Products," (Order No. PB93-201127; Cost: $19.50, subject to change) will be available only from:
National Technical Information Service
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