

**SUMMARY OF ANALYTICAL METHODS
WITH THE
POTENTIAL TO DETECT PESTICIDES IN AIR**

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Environmental Hazards Assessment Program



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By

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**SUMMARY OF ANALYTICAL METHODS WITH THE
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I. INTRODUCTION

This compilation is intended to give an overview of instrumental methods that can be applied to the determination of pesticide concentrations in air. Techniques that have been used already as well as recently developed approaches that seem to be promising are covered. The emphasis will be on checking the feasibility of the novel techniques for this application. Because no data about the detection of pesticides are available in these cases, I will sometimes refer to the detection of air pollutants in general instead of pesticides.

The need for new techniques that can identify a broad range of compounds in environmental samples has been expressed, for example, by Gurka and Hiatt working at the US EPA lab in Las Vegas:¹

"The large number of potentially hazardous analytes in environmental samples coupled with deficiencies in currently available organic analytical techniques make routine environmental analysis of gas chromatographicable (GC) volatiles extremely difficult. To optimize analytical procedures, while minimizing the possibility of false identifications in situations of regulatory significance, the United States Environmental Protection Agency (USEPA) usually employs a target compound approach which is sometimes expanded to include a limited number of nontarget compounds. Such an approach would not be expected to utilize much of the available information in sample extracts. In addition, due to the nonavailability of suitable analytical methods the target compound

identifications are generally not confirmed by independent spectral methods. This has led to some criticism of regulatory agencies from external sources".¹

To emphasize let me briefly address the two basic analytical approaches implied in the above citation. These approaches are based upon different expectations about the information gained from the analysis: one is "what is the concentration of compound X in this sample" (target compound approach), and the other one "which compounds of a given class are in this sample" (multicomponent analysis approach). In the former case, separation of the (potential) hundreds of chemical compounds present in a sample is used just to discard the unwanted ones and retain the single compound of interest. In the latter case, after separation, all components are subjected to an identification process. Thus, while in the first case the analytical method should be as specific as possible just for one compound, in the second case the method has to be general enough to detect a wide range of compounds while still being specific for each one of them simultaneously. Unfortunately, the gain in application range usually goes together with a loss in sensitivity.

This summary, does not cover methods for specific compounds but rather mentions some instruments that can potentially be used for most analytical problems related to the detection of pesticides. Other techniques presented here are more limited and are covered only because their novel approaches theoretically provide very useful information.

An extensive literature survey about the analysis for pesticides is given in review articles from 1985² and 1987³. This summary is more informal and does not have an extensive literature reference section. Each method has just one or two references which refer to either typical applications or a good

description of the procedures. It is also outside the scope of this summary to provide much information about the basic principles or specific operating conditions of these methods. For the former, I recommend general texts about analytical chemistry, like ref. (4), and for the latter the original research papers.

There are four different methodologies for the collection and analysis of air pollutants: a) collect a sample at the site, then send the sample to a laboratory for analysis; b) collect and analyze the sample at the site; c) analyze the air at the site without subjecting it to a sampling mechanism; d) analyze the air at a given site from a distance. Methods from categories c) and d) are applicable to gaseous compounds only; the techniques that require sample collection and preparation can determine pesticides in soil, water and on aerosols as well.

The next sections subdivide the array of methods according to the amount of sample preparation needed (i.e., in basically reverse order of the methodologies listed above). Before covering methods that require major sample preparation, a section about air sampling methods (IV) has been inserted.

II. IN-SITU MEASUREMENTS

In-situ refers to measurement techniques that do not involve sample taking, as sampling implies a removal of the substance from its original surroundings. The most useful technique for the in-situ detection of gaseous compounds in ambient air is absorption spectroscopy, specifically differential absorption spectroscopy. The techniques used are usually subdivided by the wavelength region of the measurement, ultraviolet/visible (UV/vis) and infrared (IR), and

by the type of instrument used, dispersing or non-dispersing spectrographs. Two of the four combinations are used currently: dispersing grating spectrometers in the UV/vis region and non-dispersing Fourier transform (FT) spectrometers in the IR region. Dispersing IR spectrometers have been replaced in most applications by FTIR instruments because of the improved performance of the FTIR systems. The analogous step in the the UV/vis range has not happened yet because FTUV systems are technically much more demanding, and the first commercial instruments are just appearing on the market.

Long pathlength absorption spectroscopy, in the form of an UV/vis differential optical absorption spectrometer (DOAS) system and an FTIR system, has been applied successfully to determine trace pollutants like nitric acid and nitrous acid in the troposphere.⁵ Note that 'differential absorption spectroscopy' is a general technique, and 'differential optical absorption spectrometer' is a specific instrument. Both DOAS and FTIR, as applied to ambient measurements, use differential absorption spectroscopy.

Small, portable systems can be built with mirrors 2.5 m apart for total path lengths of about 100 m. The larger systems used so far^{5,6} need a few days to set up and calibrate, but with their 25 m base path, they achieve total path lengths of 1000 m or more. Both DOAS and FTIR measurements have been used by the California Air Resources Board as reference methods for the detection of gaseous nitric acid, nitrous acid and formaldehyde in ambient air during recent intercomparison studies.^{5,6}

At 1000 m the detection limits of the FTIR instrument range typically from 1 to 20 ppb for a wide range of organic and inorganic compounds. The number of compounds detectable in the UV/visible region with the DOAS system is much

more restricted. However, the detection limits are generally in the sub-ppb range, occasionally even dropping to sub-ppt levels.

In the infrared region water vapor causes strong interference so that the detection of pollutants is limited to relatively small wavelength regions (referred to as 'windows') with reduced water absorption. This is of no concern in the UV/visible region. In fact, a compact long pathlength DOAS system can conceivably be used to determine in-situ concentrations of some pollutants in water.

There is another in-situ detection method that goes one step further than the ones described so far. Light detection and ranging (LIDAR) has the added advantage over the other spectroscopic techniques that the detector can be a few hundred meters away from the actual measurement site. The light source is in this case backscattered light from a laser pulse. The light attenuation is measured at various times after emission of the pulse, and concentration vs. distance data are then derived from the absorption vs. time data. Mobile LIDAR systems have been used so far to determine atmospheric trace pollutants from a van⁷ and from an airplane⁸. Further background information and various applications were presented at a workshop on optical and laser remote sensing.⁹

III. METHODS THAT DO NOT REQUIRE SAMPLE EXTRACTION

I am aware of two systems that fit into this category: a tunable diode laser spectrometer (TDLS) and photoacoustic spectroscopy (PAS).

The TDLS system can monitor two gaseous components simultaneously using infrared absorption.¹⁰ It has been applied to the detection of air pollutants

like nitric acid and formaldehyde in the sub-ppb range. In contrast to the in-situ FTIR system mentioned before, this instrument pulls air through a sampling line into a low pressure cell where the compounds are monitored. Because of some special features used during data acquisition and data processing, the detection limits of this system are generally at least an order of magnitude lower than the ones for the conventional FTIR.

Photoacoustic spectroscopy can be used to detect compounds adsorbed onto a substrate. When molecules absorb light, part of the energy increase can be transferred into translation. A change in translational energy equals a change in temperature and causes a change in density. Thus, rapid changes in light intensity will cause rapid changes in air density or, in other words, soundwaves. For example, if a substance is irradiated with monochromatic light chopped at a frequency of 400 Hz a resulting audio signal can be detected at the 400 Hz frequency whose magnitude is proportional to the absorption strength of the compound at the irradiated wavelength and to the amount of the compound present in the sample. However, even though this technique has been used to make measurements on skin¹¹, it would require major development work to confirm the possibility to detect pesticides on particulate matter or on plant surfaces.

IV. AIR SAMPLING METHODS

For the analytical methods mentioned later, the sampling process is separated from the analysis. As, in principle, each sampling method can be used with any of the analysis procedures, these two steps are treated here in separate sections.

With the exception of photoacoustic spectrometry, all methods described so far could only detect gases. The air sampling techniques mentioned in this section also sample compounds adsorbed on particles suspended in air. In fact, care has to be taken to be able to distinguish between gaseous compounds and adsorbed compounds in the gas phase. Each sampling method has different collection efficiencies for the two categories. The methods have to be selected carefully depending on the entity to be measured: gaseous concentration, adsorbed concentration or total amount in the gas phase.

The general requirements for sampling of gas phase components are: the method has to retain the compound of interest quantitatively (or at least at a known and constant ratio); and the concentration should not change due to physical or chemical processes during sampling or storage.

One approach is to draw air into a sampling volume (glass or steel bulbs, teflon bags, etc.). This is a convenient way to get a large number of samples from widely separated areas. For instance, polished steel sampling tanks have been sent to all continents to establish data bases about global background concentrations in the troposphere. The disadvantages of this technique are that some compounds can react with the container surface and that the volume (a few liters) is not sufficient to detect most trace components.

More commonly, the air is pulled over a surface that can retain and thus accumulate the compounds of interest. Three variations of this method are: passive, low volume and high volume samplers.

Passive Samplers rely on turbulent air flow and/or diffusion processes to carry the compound of interest to the collection surface. For gaseous

components, a substrate is chosen that can adsorb the pollutant; for particulate matter or droplets, it is a surface that can retain these particles. For example, in air pollution studies sampling tubes filled with adsorbant were used for gases like nitrogen dioxide¹² and formaldehyde¹³ or sampling surfaces were set up for condensates like dew.¹⁴ Advantages of these devices are simplicity and low cost. A major drawback is that they rely on external air movement to transport the compound of interest to the sampling substrate. To collect sufficient amounts of material for chemical analysis, long collection times of half a day to one week are typical.^{12,13}

The low volume sampler increases the efficiency of the passive sampler by use of a small vacuum pump that pulls the air over an absorbing sampling surface or through a liquid.¹⁵ Besides adsorbing trace components on a solid surface, the air can be pulled through a liquid that can retain, through adsorption or chemical reaction, the compounds of interest. Another possibility would be to freeze out the compounds on a cold surface. Using a lo-vol, air sampling rates of up to a few liters per minute are common with exposure times of about 30 min to 12 hrs.^{6,15} Sampling time is critical as the sampling medium can retain only a limited amount of material, as discussed later.

The high volume sampler further lowers the detection limit by increasing the flow rate by about two to three orders of magnitude over the lo-vol. There is no clear cut flow setting for the air samplers. The recommended value for hi-vols is 40 SCFM (approximately $1.1 \text{ m}^3/\text{min}$). On the other hand, the Statewide Air Pollution Research Center at the University of California, Riverside, has an ultra-high volume sampler with a flow of 640 SCFM (about $18 \text{ m}^3/\text{min}$). An important point is that the collection surface has been adjusted so that the

face velocity of the air hitting the filter is similar to the one for a standard hi-vol.

Using hi-vols, particulate matter is sampled usually on surfaces like glassfiber (GF) filters or teflon impregnated glass fiber (TIGF) filters, gaseous components are collected on polyurethane foam (PUF) plugs or granular adsorbant.^{6,16}

Major problems encountered with both high and low volume samplers are volatilization off the substrate and saturation of the substrate resulting in breakthrough of the compound. One way of checking for breakthrough is to add a second sampling surface right after the first one. If there are only small amounts found on the second surface (compared to the first one), a good approximation for the total amount is the sum of the concentrations. However, if the amount on the second sampler is larger, the data have to be discarded.

In the case of the low volume sampler, a better approach is to use a number of sampling tubes in parallel with different flow rates, for example, 5, 10, 20, 40 l/min.¹⁷ A plot of the concentration versus the sampling volume will have a linear slope if no breakthrough occurred; otherwise a curvature will be noticeable at the larger sampling volumes.

When checking for volatilization, combinations of sampling surfaces may be necessary. For instance, up to three PUF plugs mounted after a TIGF filter have been used to check for volatilization of compounds off the TIGF filter.⁶

A more sophisticated sampling train consists of three stages. The first stage, a denuder, adsorbs the gaseous components while letting the

particulates through. The second stage is a filter to collect the particulate matter, followed by a PUF plug to check for volatilization.

For the understanding of long distance transport of pesticides on particulate matter and from a health perspective, it is necessary to know if the pesticides associate primarily with a certain particle size range. To answer this question, additions or modifications for size separation are used. Two examples are a cutoff inlet in front of the hi-vol filter that puts an upper limit on the size of the particles reaching the filter (typically 10 μm), and multistage impactors that give a more detailed size distribution. Reviews about particle size analysis (and other subjects like pesticide analysis) are published regularly in Analytical Chemistry.

V. METHODS REQUIRING SAMPLE PREPARATION

When the samples get to the laboratory the compounds of interest are adsorbed on a surface, dissolved in a liquid (for gaseous components), or were already on particulate matter. Therefore, the first step in analysis is generally an extraction either by boiling the sample under reflux with a proper solvent for a few hours^{6,15} or by sonicating it in a solvent bath.

A variety of methods have been employed for the analysis of organic matter in air. The most widely used include thin layer chromatography (TLC), gas chromatography (GC) and high-performance liquid chromatography (HPLC) for the separation of the mixtures, and UV or IR absorption and mass spectroscopy (MS) for the detection.

There are a variety of detectors available for GC systems that have very general application like thermal conductivity detectors (TCD) and flame

ionization detectors (FID). Some are very sensitive to certain elements, like the thermionic emission detector (TED) for nitrogen and phosphorus (with some residual response to carbon), and the flame photometric detector (FPD) which can be made selective for phosphorus and sulfur. Their strong point is a very low detection limit in the sub-nanogram or sometimes even sub-picogram range. Their disadvantage is that they are not compound specific, at best they are element specific. Peak identifications are made by comparison of peak retention times with reference compounds. The underlying assumption is that the probability that another compound eludes at nearly the same time is negligible. How well that assumption holds for complex environmental samples containing hundreds of chemicals has to be established on a case-by-case basis.

In addition, an identification based solely on an indirect observation is not desirable if the result of this observation can effect the welfare of human beings or the quality of the environment in general. These GC detectors can only prove the absence of a specific chemical (relative to a given detection limit), but cannot uniquely prove the presence of this compound. There is a one-to-one relationship between chemicals and their retention times: for each set of experimental conditions a chemical will always elude at a specific retention time. But the reverse assignment is a one-to-many relationship: a peak at a given retention time can be caused by a number of chemicals. It is just assumed that the probability of an interference is negligible. In order to make sure that the peak observed is truly caused by the assigned chemical, spectroscopic analysis methods have to be used.

A thorough analysis of multicomponent mixtures is done customarily by pre-separation through HPLC into a small set of fractions and subsequent

separation and characterization of selected fractions by GC/MS.⁶ With respect to the detection of pesticides, Alford-Stevens et al. report the determination of PCBs and chlorinated pesticides in Ohio river water using GC/MS with detection limits of approximately 1 µg/L for these chlorinated pesticides.¹⁸ Also using GC/MS, Trehy et al. measured aldicarb in water with a detection limit of 0.3 ng.¹⁹ It is also possible to directly couple the effluent from the HPLC to an MS and achieve sub-microgram detection limits for pesticides.²⁰ See Appendix I for a conversion of absolute amounts into concentrations.

For a relatively quick screening of environmental samples, tandem mass spectrometry (MS/MS) may be quite useful. In the first stage, soft ionization methods are applied that produce molecular ions which are separated according to their mass by the first mass filter (thus replacing the GC as a means to separate the components). The second stage with its hard ionization process produces fragment ions used for identification of the compounds. Using MS/MS, organic compounds in multicomponent environmental samples can be determined semiquantitatively without prior separation in less than 30 min.²¹

More recent developments involve the combination of GC or HPLC separation with detection by Fourier-transform infrared spectroscopy (FTIR). The US EPA already uses GC/FTIR for environmental monitoring.²² The analogous combination, HPLC/FTIR, is a fairly new technique that has interesting capabilities. Generally, the spectrometer has to scan the eluding compound in the short time it passes through the detector. The method used in this case is called matrix isolation: the eluding liquid is frozen onto a rotating disk, thus preserving the compound at a fixed location on this disk. The FTIR analysis can be done in a subsequent step, and each retention time can be

examined and signal averaged over a much longer time period to lower the detection limit. In addition, the disks may be kept in cold storage for a short time and be re-analyzed if any questions are raised after the first analysis. The combination of a matrix isolation technique with a GC applied to the analysis of PCBs has been reported by Schneider et al.²³

Another promising analytical method is Fourier-transform mass spectrometry (FTMS). This technique can measure all ions simultaneously by trapping them in cyclotron orbits.²⁴ It thus does not need a continuous inflow of ions into the mass filter, an important consideration if the amount of sample available is very small. Using a GC/FTMS system, 10 pg of naphthalene were detected in gasoline.²⁵

Both GC/FTIR and GC/FTMS have been shown to be powerful analytical tools to identify compounds in complex environmental samples. Currently development is under way to combine these techniques to GC/IR/MS systems.^{1,26,27} The main benefit is improved confidence in the identification process. This does not necessarily mean that more compounds are identified correctly, but rather that almost none are assigned incorrectly. To stress this point, I present an excerpt of a table from Wilkins²⁷ where he lists the performance of various automatic computer search algorithms for a group of 45 compounds:

Algorithm ^a	Correct	Incorrect	Rejected ^b
IR	32	13	-
IR/AMM	33	8	4
MS	26	19	-
MS/AMM	30	12	3
IR/MS	32	0	13
IR/MS/AMM	35	0	10

- a. IR or MS alone: best library match is used.
IR/MS: assignment is made only if IR and MS library searches agree.
AMM: accurate mass measurement information used for identification.
- b. If there is no coincidence between the IR and GC search results, no identification is made.

Note that the combination of IR and MS has eliminated all false identifications. Also, so far the retention times of the GC separation step have not been taken into account for the identification. No assumptions have been made about the nature of the compounds, and each peak was matched with all chemicals contained in the database. Adding retention time information will increase the performance of the identification process.¹

Using special matrix isolation FTIR instruments similar to the ones described above, detection limits in the 100 pg range are possible.²⁷ Based on the assumptions explained in the appendix, this corresponds to detection limits of approximately 5 µg/kg soil, 5 µg/L water and 0.01 µg/m³ air.

VI. SUMMARY

Laboratory analysis of environmental samples depends on proper sample taking in the field. For gaseous species, all collections involve trapping the compounds of interest by adsorbing them on a surface or dissolving them in a liquid. Particulate matter is usually collected on a surface. As these samples are acquired over an extended period of time, problems can arise from reaction on the surface, volatilization off the surface, and saturation of the surface followed by a breakthrough of the substance. These problems have been mentioned and some remedies suggested in Section IV.

For volatile chemicals, however, the error levels introduced by sampling, shipping, and storage are hard to assess and can be very large. Wherever possible, in-situ methods (i.e., long pathlength spectroscopy) should be used to monitor gaseous compounds directly. No commercial DOAS systems are available that can be applied for ambient air monitoring. Building such a system would cost approximately \$20,000 to \$40,000, depending on the

sophistication and quality of both the spectrometer and the detector. FTIR systems useful for ambient monitoring can be purchased for about \$35,000 to \$70,000. In both cases, additional optical equipment and mounting structures have to be purchased at a cost of \$5,000 to \$10,000.

For low volatility compounds adsorbed on particulate matter, a combined GC/IR/MS system seems to fulfill the requirements of a general purpose instrument. It is of unequalled value for method validation and quality assurance, and its multicomponent analysis capability makes it a powerful research tool. An integrated GC/IR/MS system incorporating the matrix isolation technique costs about \$200,000 to \$300,000. Because the sample generation and preparation steps are completely independent for the analysis by GC/IR/MS, samples extracted from other matrices like soil and water can be examined too, making this type of instrument a very powerful general purpose detection and identification technique.

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