



# **Third Annual National Symposium on Recent Advances in Pollutant Monitoring of Ambient Air and Stationary Sources**

**Agenda  
Abstracts  
Attendee List**

**Raleigh, North Carolina  
May 3 through  
May 6, 1983**





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National Symposium  
on Recent Advances  
in Pollutant Monitoring  
of Ambient Air and  
Stationary Sources**

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Raleigh, North Carolina  
May 3 through May 6, 1983**

Some abstracts are from EPA funded programs. These abstracts have not been peer reviewed and do not reflect EPA policy. However, a symposium publication will be peer reviewed and approved by the agency at a later date. This publication will be sent to attendees after review and preparation.

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## **AGENDA**

AGENDA FOR  
THIRD ANNUAL  
NATIONAL SYMPOSIUM

RECENT ADVANCES IN POLLUTANT MONITORING  
OF AMBIENT AIR AND STATIONARY SOURCES

TUESDAY, MAY 3, 1983

- 8:30 to 8:45 a.m. WELCOME AND INTRODUCTION  
Thomas R. Hauser, Director, Environmental  
Monitoring Systems Laboratory, EPA/RTP
- 8:45 to 9:30 a.m. KEYNOTE ADDRESS  
Courtney Riordan--Acting Assistant  
Administrator, Office of Research and  
Development, EPA

SESSION I - INORGANIC POLLUTANTS

Session Chairman: Donald H. Stedman, University of Michigan

- 9:30 to 9:50 a.m. EVALUATION OF DENUDER TUBES FOR THE RAPID  
MEASUREMENT OF AMBIENT AMMONIA AND SULFATES  
David E. Layland, University of  
North Carolina
- 9:50 to 10:10 a.m. A SEQUENTIAL AND SPECIFIC HOLLOW TUBE  
SYSTEM FOR TRACE NITROGEN COMPOUNDS IN AIR  
Robert S. Braman, University of  
South Florida
- 10:10 to 10:30 a.m. BREAK
- 10:30 to 11:00 a.m. A LUMINOL-BASED NITROGEN DIOXIDE DETECTOR  
OF AMBIENT STUDIES  
Donald H. Stedman, University of  
Michigan
- 11:00 to 11:30 a.m. MEASUREMENTS OF TRACE TROPOSPHERIC  
CONSTITUENTS USING A MOBILE TUNABLE DIODE  
LASER SYSTEM  
Gervase I. Mackay, Unisearch Associates,  
Concord, Ontario

(TUESDAY, MAY 3, 1983 - CONTINUED)

11:30 to 12:00 noon     AIRBORNE DIAL MEASUREMENTS OF TROPOSPHERIC  
                             GASES AND AEROSOLS  
                             Arlen F. Carter, NASA Langley Research  
                             Center

12:00 to 1:30 p.m.     LUNCH - END OF SESSION I

SESSION II - PARTICULATE POLLUTANTS

Session Chairman: Andrew R. McFarland, Texas A & M University

1:30 to 2:00 p.m.     OVERVIEW  
                             Andrew R. McFarland

2:00 to 2:30 p.m.     CALIBRATION AND TESTING OF INHALABLE  
                             PARTICULATE SAMPLERS  
                             Walter John, California Department of  
                             Health Services

2:30 to 3:00 p.m.     PARTICULATE SAMPLING EFFICIENCY DEPENDENCE  
                             ON INLET ORIENTATION AND FLOW VELOCITIES  
                             Klaus Willeke, University of Cincinnati

3:00 to 3:30 p.m.     BREAK

3:30 to 4:00 p.m.     A METHOD TO IMPROVE THE ADHESION OF  
                             PARTICLES ON TEFLON FILTERS  
                             Thomas G. Dzubay, EPA

4:00 to 4:30 p.m.     DESCRIPTION OF A STATE-OF-THE-ART FUGITIVE  
                             EMISSION SAMPLING METHOD  
                             Kevin J. Kelley, TRC Environmental  
                             Consultants

4:30 to 5:00 p.m.     DEVELOPMENT OF FEDERAL REFERENCE METHOD  
                             FOR PM<sub>10</sub> (PARTICULATE MATTER LESS THAN  
                             10 MICROMETERS)  
                             Madhav B. Ranade, Research Triangle  
                             Institute

5:00 p.m.             END OF SESSION II

WEDNESDAY, MAY 4, 1983

SESSION III - GENERAL AND SOURCE-ORIENTED MONITORING

Session Chairman: Robert S. Braman, University of South Florida

8:30 to 9:00 a.m.        SOME THEORETICAL CONSIDERATIONS ON THE  
APPLICATION OF THE HOLLOW TUBE TECHNIQUE  
FOR GASEOUS DIFFUSION COEFFICIENT STUDIES  
Robert S. Braman

9:00 to 9:30 a.m.        METAL FOIL COLLECTION/FLASH VAPORIZATION/  
FLAME PHOTOMETRY AS APPLIED TO AMBIENT  
AIR MONITORING OF TOTAL GASEOUS SULFUR  
Richard A. Kage, University of Idaho

9:30 to 10:00 a.m.      MEASUREMENT OF MERCURY EMISSIONS FROM A  
MODIFIED IN-SITU OIL SHALE RETORT  
Martin J. Pollard, Lawrence Berkeley  
Laboratory

10:00 to 10:30 a.m.    BREAK

10:30 to 11:00 a.m.    COMPARISON OF TRANSMISSION AND SCANNING  
ELECTRON MICROSCOPE TECHNIQUES FOR  
MEASUREMENT OF AIRBORNE ASBESTOS FIBERS  
Daniel Baxter, Science Applications

11:00 to 11:30 a.m.    OPTIMIZATION OF ELECTRON MICROSCOPE  
MEASUREMENT OF AIRBORNE ASBESTOS  
George Yamate, ITT Research Institute

11:30 to 12:00 noon    A PILL FOR THE ASSESSMENT OF POLLUTION  
MEASUREMENT METHODS  
R. K. M. Jayanty, Research Triangle  
Institute

12:00 to 1:30 p.m.    LUNCH

1:30 to 1:50 p.m.      DYNAMIC IMPINGER - APPLICATION TO THE  
ANALYSIS OF HALOGENATED HYDROCARBONS FROM  
SOURCE EMISSION  
Jimmy C. Pau, Environmental Monitoring  
Systems Laboratory, EPA/RTP

1:50 to 2:15 p.m.      SAMPLING AND ANALYSIS OF INCINERATION  
EFFLUENTS WITH THE VOLATILE ORGANIC  
SAMPLING TRAIN (VOST)  
Gregory A. Jungclaus, Midwest Research  
Institute



(WEDNESDAY, MAY 4, 1983 - CONTINUED)

2:15 to 2:40 p.m.	COMPUTER ASSISTED, REALTIME DETECTION OF CHLOROPICRIN, CHLOROFORM AND CARBONYL CHLORIDE DURING WASTE DISPOSAL OPERATIONS Michael E. Witt, Department of the Army, Rocky Mountain Arsenal
2:40 to 3:00 p.m.	APPLICATION OF API-MS/MS FOR STUDIES OF HAZARDOUS AIR POLLUTANT REACTIONS Chester W. Spice, Battelle Columbus
3:00 to 3:30 p.m.	BREAK
3:30 to 4:00 p.m.	AIR MONITORING DURING INCINERATION OF PESTICIDE CONTAMINATED MILITARY SMALL ARMS AMMUNITION Gaydie Connolly, Department of the Army, Rocky Mountain Arsenal
4:00 to 4:30 p.m.	ANALYSIS OF PCB'S BY CAPILLARY GC/ECD FOR DETOXIFICATION STUDIES Alston L. Sykes, TRW
4:30 to 5:00 p.m.	ALDEHYDE EMISSIONS FROM WOOD-BURNING FIREPLACES Frank Lipari, General Motors
5:00 p.m.	END OF SESSION III

SESSION IV - PERSONAL MONITORING

Session Chairman: John D. Spengler, Harvard University

8:30 to 9:00 a.m.	WHAT HAVE WE LEARNED FROM EXPOSURE STUDIES? John D. Spengler
9:00 to 9:30 a.m.	VALIDATION OF A PASSIVE SAMPLER FOR DETERMINING FORMALDEHYDE IN RESIDENTIAL INDOOR AIR Alfred F. Hodgson, Lawrence Berkeley Laboratory
9:30 to 10:00 a.m.	PASSIVE SAMPLING DEVICES FOR ORGANIC VAPORS IN AMBIENT AIR Robert G. Lewis, EPA
10:00 to 10:30 a.m.	BREAK

(WEDNESDAY, MAY 4, 1983 - CONTINUED)

10:30 to 11:00 a.m.	LABORATORY STUDIES OF TEMPERATURE RESPONSE OF THE PALMES PASSIVE NO <sub>2</sub> SAMPLER J. Girman, Lawrence Berkeley Laboratory
11:00 to 11:30 a.m.	CHARACTERIZATION OF PERSONAL EXPOSURE TO NO <sub>2</sub> AND SO <sub>2</sub> AND THEIR INDOOR CONCENTRATIONS IN OTTAWA, CANADA Tahir R. Khan, Department of Health and Welfare, Ottawa, Canada
11:30 to 12:00 noon	NIOSH DEVELOPED SYSTEM FOR MONITORING EQUIPMENT EVALUATION Mary Lynn Woebkenberg, NIOSH
12:00 to 1:30 p.m.	LUNCH
1:30 to 2:00 p.m.	RELATIONSHIPS OF MEASURED NO <sub>2</sub> CONCENTRATIONS AT DISCRETE SAMPLING LOCATIONS IN RESIDENCES D. P. Miller, Washburn University of Topeka
2:00 to 2:30 p.m.	ESTIMATED DISTRIBUTIONS OF PERSONAL EXPOSURE TO RESPIRABLE PARTICLES R. Letz, Harvard University
2:30 to 3:00 p.m.	EMPIRICAL MODELS FOR ESTIMATING INDIVIDUAL EXPOSURES TO AIR POLLUTANTS IN A HEALTH EFFECTS STUDY Charles F. Contant, University of Texas
3:00 to 3:30 p.m.	BREAK
3:30 to 4:00 p.m.	CO EXPOSURES IN WASHINGTON, D.C. AND DENVER, COLORADO DURING THE WINTER OF 1982-1983 Gerald G. Akland, EPA
4:00 to 4:30 p.m.	MODELS OF HUMAN EXPOSURE TO NO <sub>2</sub> USING PERSONAL MONITORING DATA James J. Quackenboss, University of Wisconsin
4:30 to 5:00 p.m.	COMPARISON OF PERMEATION AND DIFFUSION TYPE PASSIVE SAMPLERS VERSUS CHARCOAL TUBE COLLECTION OF SELECTED GASES Philip W. West, West-Paine Laboratories
5:00 p.m.	END OF SESSION IV

THURSDAY, MAY 5, 1983

SESSION V - ACID DEPOSITION

Session Chairman: John Miller, NOAA

8:30 to 9:15 a.m.	OVERVIEW Ellis B. Cowling, North Carolina State University
9:15 to 9:45 a.m.	INTENSITY WEIGHTED SEQUENTIAL SAMPLING OF PRECIPITATION J. K. Robertson, Department of the Army, West Point
9:45 to 10:15 a.m.	AUTOMATION OF AN ION CHROMATOGRAPH J. K. Robertson
10:15 to 10:45 a.m.	BREAK
10:45 to 11:15 a.m.	DESIGN AND TESTING OF A PROTOTYPE RAINWATER SAMPLER/ANALYZER Richard J. Thompson, University of Alabama in Birmingham
11:15 to 11:45 a.m.	MEASUREMENT OF WEAK ORGANIC ACIDITY IN PRECIPITATION FROM REMOTE AREAS OF THE WORLD William C. Keene, University of Virginia
11:45 to 1:30 p.m.	LUNCH
1:30 to 2:00 p.m.	AERIAL INPUT OF TOXAPHENE TO THE SOUTH CAROLINA COASTAL ZONE WITH RESIDUE ANALYSIS BY CAPILLARY GAS CHROMATOGRAPHY Mark T. Zaranski, University of South Carolina
2:00 to 2:30 p.m.	A FIELD INTERCOMPARISON OF PARTICLE AND GAS DRY DEPOSITION MEASUREMENT AND MONITORING METHODS Donald A. Dolske, Illinois Department of Energy and Natural Resources
2:30 to 3:00 p.m.	A COMPARISON OF AMBIENT AIRBORNE SULFATE CONCENTRATIONS DETERMINED BY SEVERAL DIFFERENT FILTRATION TECHNIQUES Donald A. Dolske
3:00 to 3:30 p.m.	BREAK

(THURSDAY, MAY 5, 1983 - CONTINUED)

3:30 to 4:00 p.m.	COMPARISON OF SURROGATE SURFACE TECHNIQUES FOR ESTIMATION OF SULFATE DRY DEPOSITION J. J. Vandenberg, Duke University
4:00 to 4:30 p.m.	DRY DEPOSITION OF SULFATE WITHIN A HARDWOOD FOREST CANOPY K. R. Knoerr, Duke University
4:30 to 5:00 p.m.	COLLECTION AND MEASUREMENT OF THE CHEMISTRY OF DEW Brent E. Smith, Mitre Corporation
5:00 p.m.	END OF SESSION V

SESSION VI - ORGANIC POLLUTANTS

Session Chairman: Hanwant Singh, Stanford Research Institute

8:30 to 9:00 a.m.	OVERVIEW Hanwant Singh
9:00 to 9:30 a.m.	EVALUATION OF SOLID SORBENTS FOR COL- LECTION OF ORGANIC VAPORS IN AIR L. J. Hillenbrand, Battelle Columbus
9:30 to 10:00	AN AUTOMATED INTEGRATED CAPILLARY-PACKED CHROMATOGRAPHIC SYSTEM FOR MOBILE AMBIENT AIR MONITORING OF VOLATILE ORGANICS Hans Plugge, Ecological Analysts
10:00 to 10:30 a.m.	BREAK
10:30 to 11:00 a.m.	ENVIRONMENTAL TRACE GAS ANALYSIS USING A LOW COST PORTABLE MASS SPECTROMETER Graham Gibson, VG Instruments
11:00 to 11:30 a.m.	GC/FTIR AND GC/MS: COMBAT OR CONCERT J. W. Brasch, Battelle Columbus
11:30 to 12:00 noon	DETECTION OF ENVIRONMENTAL POLLUTANTS USING PIEZOELECTRIC CRYSTAL SENSORS Matt H. Ho, University of Alabama in Birmingham
12:00 to 1:30 p.m.	LUNCH
1:30 to 2:00 p.m.	A MINIATURE GAS CHROMATOGRAPH UTILIZED IN A PORTABLE GAS ANALYSIS SYSTEM David A. Hawker, Microsensor Technology

(THURSDAY, MAY 5, 1983 - CONTINUED)

2:00 to 2:30 p.m.	TUNABLE ATOMIC LINE MOLECULAR SPECTROSCOPY BENZENE MONITOR Donald R. Scott, EPA
2:30 to 3:00 p.m.	REDUCED TEMPERATURE PRECONCENTRATION OF VOLATILE ORGANICS FOR GAS CHROMATOGRAPHIC ANALYSIS; SYSTEM AUTOMATION William A. McClenny, EPA
3:00 to 3:30 p.m.	BREAK
3:30 to 4:00 p.m.	SAMPLING AND ANALYSIS OF POLYNUCLEAR AROMATIC HYDROCARBONS IN AIR USING SOLID ADSORBENTS Dori Karlesky, Emory University
4:00 to 4:30 p.m.	ANALYSIS OF POLYCHLORINATED BIPHENYLS IN AMBIENT AIR SAMPLES E. Singer, Ministry of the Environment, Ontario, Canada
4:30 to 5:00 p.m.	INFLUENCE OF VOLATILITY ON THE COLLECTION OF PAH VAPORS WITH POLYURETHANE FOAM Feng You, University of South Carolina
5:00 p.m.	SECTION VI TO BE CONTINUED

FRIDAY, MAY 6, 1983

SESSION VI - ORGANIC POLLUTANTS (CONTINUED)

Session Chairman: Ralph M. Riggin, Batelle Columbus

8:30 to 8:55 a.m.	METHOD FOR DETERMINATION OF SUB-PART PER BILLION CONCENTRATIONS OF PHOSGENE AND ACYLCHLORIDES IN AMBIENT AIR Ralph M. Riggin
8:55 to 9:20 a.m.	ADVANTAGES AND OPERATING CHARACTERISTICS OF A REFRACTIVELY SCANNED FOURIER TRANSFORM SPECTROMETER BASED AMBIENT AIR MONITORING SYSTEM J. W. Mohar, Analect Instruments
9:20 to 9:45 a.m.	A COST-EFFECTIVE PROCEDURE TO SCREEN AIR SAMPLES FOR POLYAROMATIC POLLUTANTS T. Vo-Dinh, Oak Ridge National Laboratory

(FRIDAY, MAY 6, 1983 - CONTINUED)

9:45 to 10:10 a.m. ANALYSIS OF POLYCYCLIC AROMATIC HYDRO-  
CARBONS IN AMBIENT AIR AND RAIN  
William T. Foreman, University of  
South Carolina

10:10 to 10:30 a.m. BREAK - END OF SESSION VI

SESSION VII - PANEL DISCUSSION

10:30 to 12:00 noon POLLUTANT MEASUREMENTS NEEDED TO MEET  
EPA'S REGULATORY RESPONSIBILITY

Panelists - Thomas R. Hauser, EPA, Office of Research  
and Development; Discussion Leader

David R. Patrick, EPA, Office of Air  
Quality Planning and Standards

David Friedman, EPA, Office of Solid  
Waste

Frederick Kutz, EPA, Office of Toxic  
Substances

12:00 noon ADJOURNMENT

**SPEAKERS' ABSTRACTS\***

**\*The name of the presentor is underlined**

SESSION I  
INORGANIC POLLUTANTS

Donald H. Stedman  
Session Chairman



EVALUATION OF DENUDER TUBES FOR THE MEASUREMENT OF  
SULFUR AEROSOL COMPOSITION

David E. Layland and Donald L. Fox  
Department of Environmental Sciences and Engineering  
University of North Carolina at Chapel Hill  
Chapel Hill, NC

The need for measurement techniques which do not rely on filtration and which provide real time measurement of the composition of ambient sulfur aerosols has been recognized. Much interest has been placed on exploiting the thermal volatilization of sulfuric acid to determine the acid fraction of sulfur aerosols. However, this technique does not provide information on the composition of the more abundant atmospheric aerosols having bulk  $\text{NH}_4/\text{SO}_4$  ratios in the range of 1 to 2. For aerosols in this range, thermal deammoniation of the ammonium sulfate component has been proposed as a technique for providing the desired compositional information.

The purpose of this investigation was to evaluate whether thermal deammoniation could be employed to differentiate among the various ammonium salts of sulfuric acid using a series of diffusion denuder tubes. In particular, real time measurements of the ammonia released by thermal deammoniation were to be made by a recently developed technique which utilizes tungsten (VI) oxide-coated tubes as denuder/collectors.

The experimental apparatus consisted of two parallel sample manifolds. Along one manifold, the sample stream was first passed through a tungsten oxide-coated tube, stripping out ambient ammonia, and then through a heater tube. A second tungsten oxide-coated tube collected ammonia released from the heated aerosol. After the desired sampling interval, the tungsten denuder/collector tubes were heated and the desorbed ammonia was eluted by a helium carrier and converted to NO by a heated gold catalyst. The NO was measured with a commercial chemiluminescent analyzer, appearing as a well-defined peak. Along the second manifold,  $\text{SO}_2$  was removed by a lead dioxide denuder tube, then sulfate was determined by FPD, either directly or via an NaCl thermodenuder tube which removed sulfuric acid. Test atmospheres were generated in an outdoor smog chamber by the homogeneous oxidation of  $\text{SO}_2$  in a propylene/ozone reaction system. The resulting  $\text{H}_2\text{SO}_4$  aerosol was then neutralized to  $(\text{NH}_4)_2\text{SO}_4$  by injection of excess  $\text{NH}_3$ .

Experimental data taken over a range of heater tube operating temperatures showed a large degree of scatter in the amount of ammonia released by thermal deammoniation of the ammonium sulfate aerosol. If the simple first order reaction  $(\text{NH}_4)_2\text{SO}_4 \rightarrow \text{NH}_3 + \text{NH}_4\text{HSO}_4$  held true, then the fraction of aerosol  $\text{NH}_4$  released as ammonia ( $\text{NH}_3/\text{NH}_4$ ) would be simply 0.5. However, at no operating temperature did the experimental data cluster about this value. Instead, ammonia fractions ( $\text{NH}_3/\text{NH}_4$ ) ranged from less than 0.1 to more than 0.6 and were independent of temperature.

A partial explanation may be had by examining simple thermodynamic equilibria for the three component solid phase  $[(\text{NH}_4)_2\text{SO}_4(\text{s}), \text{NH}_4\text{HSO}_4(\text{s}), \text{H}_2\text{SO}_4(\text{s})]$  and two component vapor phase  $[\text{NH}_3(\text{g}), \text{H}_2\text{SO}_4(\text{g})]$  system. Two nomograms were constructed over the range of heater tube operating temperatures, one for the vapor phase equilibria between  $\text{NH}_3$  and  $\text{H}_2\text{SO}_4$  and one for the solid phase equilibria with  $\text{NH}_3$  as a function of bulk aerosol ratio,  $\text{NH}_4/\text{SO}_4$ . Knowing the rate of diffusion of  $\text{NH}_3$  to the walls of the denuder, and the temperature profile along the length of the denuder, the nomograms permit estimates of the fraction of ammonia released from the heated aerosol and collected on the tube,  $\text{NH}_3/\text{NH}_4$ .

While the thermodynamic calculations can only be regarded as qualitative, they do indicate that the aerosol would not be expected to behave according to the simple thermal deammoniation reaction  $(\text{NH}_4)_2\text{SO}_4 \rightarrow \text{NH}_4\text{HSO}_4 + \text{NH}_3$ . Rather, the extent of aerosol decomposition upon heating is expected to depend on the aerosol concentration itself. Moreover, as the gas stream cools after leaving the hot zone of the heater, heteromolecular homogeneous nucleation is expected to occur. The onset of nucleation is highly sensitive to the rate of temperature drop and the rate of ammonia absorption in the denuder, which in turn control the vapor pressures of  $\text{NH}_3$  and  $\text{H}_2\text{SO}_4$ . This suggests that the ammonia fraction observed ( $\text{NH}_3/\text{NH}_4$ ) will also depend on sample time; absorption sites on the warmer upstream surfaces of the denuder are occupied first, increasing the importance of nucleation later in the sampling interval.

To avoid nucleation at higher operating temperatures, the tungsten oxide tube could be utilized directly as a thermodenuder tube. However, peaks eluted from such tubes were found to be highly degraded. An alternative solution would be to replace the heater tube with another NaCl thermodenuder to remove the sulfuric acid vapor. The fraction of ammonia released would nevertheless be expected to depend on the aerosol concentration, because higher aerosol concentrations equilibrate at lower ammonia fractions ( $\text{NH}_3/\text{NH}_4$ ). It should, however, be possible to completely decompose the aerosol at

high temperatures and collect the total aerosol  $\text{NH}_4$  burden, thereby avoiding this effect.

In summary, neither experimental measurements nor theoretical considerations provide support for the feasibility of measuring the ammonium sulfate component of ambient aerosols by means of thermal deammoniation. However, the tungsten oxide denuder/collector appears to be a convenient and reliable technique for the measurement of gaseous ammonia. Therefore, total thermal decomposition of the aerosol at high temperatures with in situ removal of sulfuric acid to avoid nucleation, followed by ammonia collection on tungsten oxide tubes, may well offer the possibility of real time measurement of bulk  $\text{NH}_4/\text{SO}_4$  ratios in the future.

A SEQUENTIAL AND SPECIFIC HOLLOW TUBE SYSTEM FOR  
TRACE NITROGEN COMPOUNDS IN AIR

Robert S. Braman and Maria Trindade  
Department of Chemistry  
University of South Florida  
Tampa, FL 33620

Qing Xiang Han  
Fushun Petroleum College  
People's Republic of China

Metal and metal oxide interior coatings for hollow tubes have been investigated for specificity of chemisorption of reactive nitrogen compounds in air. Coatings studied include: Au, Ag, Pt, Cu,  $\text{MoO}_3$ ,  $\text{WO}_3$ ,  $\text{Ag}_2\text{O}$ ,  $\text{CuO}$ , C, Ni and  $\text{NiO}$ . Nitrogen compounds studied include:  $\text{NO}_2$ ,  $\text{HNO}_3$ ,  $\text{NH}_3$ ,  $(\text{CH}_3)_2\text{NH}$ ,  $(\text{CH}_3)_3\text{N}$ ,  $\text{HCN}$ , and  $(\text{CN})_2$  and PAN. A combination of, in order,  $\text{WO}_3$ ,  $\text{Ag}_2\text{O}$ ,  $\text{NiO}$ , and  $\text{Cu}$  lead to preconcentration and analysis for:  $\text{HNO}_3$  and ammonia ( $\text{WO}_3$ ),  $\text{HCN}$  ( $\text{Ag}_2\text{O}$ ),  $\text{NO}_2$  ( $\text{NiO}$ ) and  $(\text{CN})_2$  ( $\text{Cu}$ ). Preconcentrated components are desorbed by heating and are detected using a chemiluminescence-type  $\text{NO}_x$  detector fitted with an oxidizing catalyst bed. Detection limits are on the order of 1 mg/sample. Selectivity of the specific surfaces will be discussed and results of application to ambient air analysis will be presented.

A LUMINOL-BASED NITROGEN DIOXIDE DETECTOR  
FOR AMBIENT AIR STUDIES

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Department of Chemistry and  
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Denver, CO 80025

An instrument for the continuous detection of  $\text{NO}_2$  in the sub-ppb range is described. The instrument is based upon the chemiluminescent reaction between  $\text{NO}_2$  in air and luminol (5-amino-2, 3-dihydro-1, 4-phthalazine dione) in alkaline solution. The present detector exhibits a 2 Hz response speed to changes of +20 ppb and a field detection limit of 30 ppt. The instrumental technique has been expanded to measure NO by the catalytic oxidation of NO to  $\text{NO}_2$  using  $\text{CrO}_3$  on Silica Gel as the oxidizing agent; however, at low ambient NO concentrations some drift in the NO zero is observed. Interference from ambient  $\text{O}_3$  is eliminated by modification of the inlet system and luminol solution. Expansion to the measurement of  $\text{O}_3$  by NO addition is also feasible.

MEASUREMENTS OF TRACE TROPOSPHERIC CONSTITUTENTS  
USING A MOBILE TUNABLE DIODE LASER SYSTEM

H.I. Schiff and G.I. Mackay  
Unisearch Associates Inc.  
Concord, Ontario L4K 1B5

Infrared absorption in the 2-15  $\mu\text{m}$  region offers a number of advantages for atmospheric trace gas measurements. It is a passive technique, well adapted to in situ, real time measurements. While virtually every trace constituent absorbs in this spectral region, the  $\text{N}_2$  and  $\text{O}_2$  do not. In fact, the absorption spectra for the trace gases are so high that resolution comparable to the line width is usually required to avoid mutual interferences.

Diode lasers have extremely narrow line widths and sufficient power to permit absorptions as low as  $10^{-5}$  to be achieved. We have combined the high selectivity of these diodes with the sensitivity provided by a long-path White cell to achieve sensitivities in the fractional part per billion range.

A mobile system has been constructed for measurement in real air. Sampling and calibration procedures have been developed for  $\text{NO}$ ,  $\text{NO}_2$  and  $\text{HNO}_3$ . These gases were measured in ambient and captive Los Angeles air. Comparisons were made with chemiluminescence instruments.

Measurements were also made in central Ontario over a two-week period and comparisons made with filter and tungstic oxide techniques.

The system has recently been upgraded to permit the near simultaneous measurements of more than one tropospheric constituent.

Sampling and calibration procedures are now being developed for the measurements of other gases of interest in urban and regional tropospheric air.

AIRBORNE DIAL SYSTEM FOR MEASUREMENTS OF  
TROPOSPHERIC GASES AND AEROSOLS

Arlen F. Carter and Edward V. Browell  
NASA Langley Research Center  
Hampton, VA 23665

A multipurpose airborne differential absorption lidar (DIAL) system has been recently developed at the NASA Langley Research Center to remotely measure the profiles of various gases and aerosols in the troposphere. The system has the capability to make measurements of ozone or sulfur dioxide in the UV, nitrogen dioxide in the visible, and water vapor, atmospheric temperature (using water vapor or oxygen absorption lines), and pressure (using oxygen lines) in the near-IR. Aerosol backscatter measurements in the UV, visible, and near-IR are made simultaneously with the DIAL measurements.

The DIAL technique will be discussed and the airborne DIAL system will be described. The first remote measurements of ozone profiles from an aircraft will be presented. Results from a major field study with the Environmental Protection Agency (EPA) to investigate elevated pollution episodes will be discussed. Results from other flight studies of ozone measurements and water vapor measurements will also be presented. Potential airborne DIAL measurements of sulfur dioxide, nitrogen dioxide, and simultaneous water vapor and temperature measurements will be briefly reviewed.

SESSION II  
PARTICULATE POLLUTANTS

Andrew R. McFarland  
Session Chairman



CALIBRATION AND TESTING OF  
INHALABLE PARTICLE SAMPLERS

Walter John and Stephen M. Wall  
Air & Industrial Hygiene Laboratory  
California Department of Health Services  
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The Environmental Protection Agency has been preparing a new inhalable particle standard for the past several years. New size-selective samplers have been developed to support the standard. In the course of testing these new samplers in the laboratory, test methodology has been evolving. Since the drafts of the new standard call for the acceptance of samplers according to performance criteria, it is necessary to adopt standard testing methods. Progress thus far has provided a basis for a uniform approach to sampler testing, but some problems remain to be resolved.

The sampling effectiveness of an inlet is determined in a wind tunnel equipped with an aerosol generator. Liquid aerosol is used to calibrate the particle size cutoffs and to measure wall losses with the aid of the fluorescent tracer. It is also necessary to test samplers with solid particles to determine the amount of excess penetration to the after-filter resulting from particle bounce and re-entrainment. Methods for these measurements currently in use by several laboratories are similar, but there are some significant differences. These will be discussed and illustrated with data on the Dichotomous sampler and the Size-Selective Inlet. The principal uncertainty remaining concerns possible effects of turbulence in the wind tunnel on the test results. Suitable methods for the generation of solid particles of known aerodynamic size are still under development.

PARTICULATE SAMPLING EFFICIENCY DEPENDENCE ON  
INLET ORIENTATION AND FLOW VELOCITIES

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New standards on particulate air sampling for the protection of human health will have upper particle size cutoffs specified. This creates a demand for extensive testing of particulate sampling inlets. A wind tunnel has been designed and built that incorporates a new method for determining sampling efficiencies. The inlet under study is integrated into a modified optical single particle counter which records the aerosol concentration penetrated through the inlet. The penetrated aerosol concentration is thus measured dynamically and quickly for various particle sizes, sampling velocities, wind velocities and sampling angles. All measurements are related to the sampled aerosol concentration at isokinetic conditions, for which the aerosol depositions on the inner wall are also determined, so that the aerosol concentration upstream of the inlet is known for all sampling conditions.

By means of this technique extensive measurements of the overall sampling efficiency of a thin-walled sampling tube of 0.565 cm inner diameter and 20 cm length were done. The sampling efficiency of the inlet tube was studied at wind velocities of 250 to 1000 cm/sec, inlet velocities of 125 to 1000 cm/sec and angles of 0 to  $\pm 90^\circ$ .

The sampling efficiency was found to be significantly reduced when sampling was performed at an angle to the flow. When the sampling velocity in the inlet differed from the ambient wind velocity, the sampling efficiency was significantly increased or decreased. Differences in sampling efficiency were found

for particles above 10  $\mu\text{m}$  in diameter when the aerosol was sampled  $15^\circ$  upward vs  $15^\circ$  downward from the horizontal, downward sampling giving the higher sampling efficiency. At an angle of  $30^\circ$ , a smaller difference between upward and downward sampling was found than at a  $15^\circ$  angle. No difference in sampling efficiency was found between upward and downward sampling at a  $90^\circ$  angle to the horizontal wind direction. For  $\theta = 30$  to  $90^\circ$  the sampling efficiency was found to be approximately a function of Stokes number  $Stk$  with the ratio,  $R$ , of wind velocity to inlet velocity as a parameter. At  $\theta = 90^\circ$  the sampling efficiency was approximately a function of  $Stk \cdot \sqrt{R}$ . About half or more of the particle deposition in the inlet occurred with the first 1 cm of the 20 cm long inlet tube.

A METHOD TO IMPROVE THE ADHESION OF AEROSOL PARTICLES  
ON TEFLON FILTERS

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The adhesion of atmospheric coarse particles (2.5 to 15  $\mu$ m) collected with dichotomous samplers on coated and uncoated Teflon filters was tested under conditions intended to simulate the jarring that might be encountered by samples that are shipped by mail. The coated filters contained  $23 \pm 5$   $\mu$ g/cm<sup>2</sup> of mineral oil that was applied before aerosol samples were collected. The test consisted of packaging filters containing ambient aerosol and laboratory-generated mineral dust aerosol samples in well-padded boxes and analyzing them after they were dropped from various heights with deposit-sides oriented down. Analyses by  $\beta$ -gauge and X-ray fluorescence indicated that when the samples were dropped once each from heights of 0.3 and 0.9 m, the average losses of coarse particles were less than 4% for the oil-coated filters and ranged from 6 to 22% for uncoated filters. After being dropped five times from a height of 2.3 m, the average losses were less than 5% for oil-coated filters and ranged from 19 to 56% for uncoated filters. Oil deposits were not applied to filters used to collect fine particles (<2.5  $\mu$ m), and no losses of fine particles were detected.

DESCRIPTION OF AN ISOKINETIC SAMPLING SYSTEM  
FOR THE MEASUREMENT OF FUGITIVE EMISSIONS

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During the past decade, research has shown that particulate matter emissions from fugitive sources contribute significantly to the particulate matter levels in many areas. In many instances, contributions from fugitive sources are the principle cause of nonattainment of particulate matter air quality standards. The specific fugitive emission contribution is, however, frequently unknown because of the lack of a practical and reliable measurement system for measuring the emission rates from fugitive sources.

Because of this lack of an accurate measurement system, TRC Environmental Consultants, Inc. (TRC) has developed MEDUSA (Multi-Sample Emission Determination Unit for Source Assessment) an automatic isokinetic sampling system for the quantification of particulate emissions from fugitive sources. Using a technique generally known as exposure profiling, sampling heads are distributed over a vertical matrix positioned downwind from the source. This technique involves sampling the cross section of a pollutant cloud to establish the flux of pollutant through the sampling plane per sampling period.

A major sampling requirement for this technique is that the extraction of the sample from the plume is done isokinetically. MEDUSA is superior to other exposure profiler units in this respect because the system is presently capable of utilizing up to 16 individually controlled isokinetic samplings heads. Each sampling head is connected to an Apple II plus computer which logs all test data and can provide real-time screen display of ambient air flow and velocity through each nozzle. Additional improvements over previously developed exposure profiler units include: (1) the ability to sample at an increased height (up to 16 meters), (2) the use of gimbal-mounted samplers to reduce sample loss by keeping filters horizontal, and (3) the use of nested nozzles which allow sampling over a wide range of wind speeds.

MEDUSA is capable of sampling both line and point sources, and is adaptable to several particle sizing techniques. To date the MEDUSA system has been used in several field testing programs to measure highway particulate emissions and in the development of fugitive particulate emission factors.

DEVELOPMENT OF A FEDERAL REFERENCE METHOD FOR  
PM-10 (PARTICULATE MATTER LESS THAN 10  $\mu$ m)

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The Environmental Protection Agency (EPA) has recognized that the current national ambient air quality standard for particulate matter is non-specific in relation to human health. EPA is considering promulgating a new size-specific standard for particles less than 10  $\mu$ m in aerodynamic diameter (PM-10). This PM-10 standard would regulate only those particles that have a high probability of being deposited deep in the respiratory system. EPA's Environmental Monitoring Systems Laboratory (EMSL) has been charged with the responsibility of establishing a reference method for measuring PM-10 concentrations in the atmosphere. Research Triangle Institute (RTI) has been assisting EMSL in reaching this goal.

RTI coordinated two workshops concerned with the development of the reference method as well as advances in particle measurement technology. As a result, the approach taken was to base the reference method on standards of the sampler as measured in a wind tunnel. Wind tunnel testing procedures have been drafted and are based on a factorial design incorporating various wind speeds, particle sizes, and particle types. Present efforts center on establishing the workability of these procedures in the RTI wind tunnel (90 cm x 90 cm cross section). A commercially available PM-10 sampler inlet has been tested with monodisperse liquid and polydisperse solid particles. Also, an existing EPA wind tunnel has been modified recently with a 1.2 m x 1.8 m cross section, and comparison between results obtained at the two facilities is made.

SESSION III  
GENERAL AND SOURCE-ORIENTED MONITORING

Robert S. Braman  
Session Chairman

SOME THEORETICAL CONSIDERATIONS ON THE  
APPLICATION OF THE HOLLOW TUBE TECHNIQUE  
FOR GASEOUS DIFFUSION COEFFICIENT STUDIES

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A number of preconcentration and denuding techniques have been developed for analysis of trace components in air. The most prominent of these have been methods for ammonia and nitric acid in air. The technique is extendable to analysis of any reactive gaseous compounds for which a chemisorbing surface can be found. The Gormley-Kennedy equation describes absorption efficiency of perfectly chemisorbing analytes in laminar flow gas streams. Physical characteristics in this equation include gas flow rate, absorbing surface length and diffusion coefficient of the analyte. Consequently, by determining the efficiency of tube absorption at a known flow rate and for a known tube length, a diffusion coefficient value can be calculated. This model is satisfactory only for monomolecular, non-hydrated or single particle sized detected entities. If a mixture of detected entities is present, complications arise.

A computerized model has been developed for generating tube efficiency data for one to three detected entities having different diffusion coefficients. This model can be used for tube-tube or tube-particle collector combinations. Experimental data analysis programs have also been developed to deconvolute overlapping Gormley-Kennedy exponentials for multiple components in both tube-tube and tube-packed tube combinations. The deconvolution technique is used on flow rate study data and separates components into  $D_a$  and  $D_b$  in which  $D_a$  is the large diffusion coefficient and  $D_b$  is the mean diffusion coefficient of all other components.

For mixed components, the simplistic use of the Gormley-Kennedy equation will result in obtaining an experimental mean diffusion coefficient  $\bar{D}_{ex}$  which is flow rate dependent and thus only partly reflective of the real state of affairs. By deconvolution analysis of a flow rate data set, it is possible to develop a plot of  $D_a$  vs  $D_b$  value combinations which best fits the experimental data. At  $D_b = 0$  one obtains a value of



$\bar{D}$ , the mean diffusion coefficient of a mixture, i.e.:  $\bar{D} = (m.f.)_a D_a + (m.f.)_b \cdot D_b + \text{---} \text{etc.}$ , where  $(m.f.)_a$  is the detected mole fraction of component a.

If  $D_a$  is known, a single study will permit calculation of  $D_b$  and the mole fraction of components a and b in a sample. Otherwise, it is necessary to perform analyses under conditions in which the mole fraction of a and b are different.

The flow rate study and mathematical analysis is best applied to detection of monomers, dimers, hydrates and other reasonably small aggregates or their combination with particulates for which  $\bar{D}$  is more than  $0.001 \text{ cm}^2/\text{sec}$ . The technique has been applied to  $\text{HNO}_3$  in air and  $\text{Hg}^0$  in air.

An error function for the Gormley-Kennedy equation has also been derived and is of a form similar to that developed for use in spectrophotometry. This aids in selecting a range of experimental conditions for minimum effect of experimental error on  $\bar{D}_{\text{ex}}$ .

METAL FOIL COLLECTION/FLASH VAPORIZATION/FLAME PHOTOMETRY  
AS APPLIED TO AMBIENT AIR MONITORING OF TOTAL GASEOUS SULFUR

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The concentration of sulfur-containing vapors ( $\text{SO}_2$ , COS,  $\text{CS}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{CH}_3\text{SH}$ ,  $\text{CH}_3\text{SCH}$ ,  $\text{CH}_3\text{SSCH}$ , etc.) in rural atmospheres rarely exceeds one or two parts per billion total sulfur (ppbS-W/W), yet the state of the art in sulfur-selective ambient air monitoring systems allows a practical detection limit of no better than 10 ppbS-W/W. Preconcentration schemes have been reported which do provide sub-ppb detectability; however, these previous methods have typically included such drawbacks as poor overall precision, long sampling times (i.e., no real time monitoring capability), and/or poor field sampling compatibility).

Silver nitrate-impregnated filters as well as metal-coated glass beads and metallic foils have been shown to collect, via ambient temperature chemisorption, a number of gaseous sulfur-containing compounds present at low and sub-ppb levels in air. Recently, the collection of volatile sulfur species on a metallic foil followed by resistively heated flash desorption has been shown to be analytically useful when coupled to a flame photometric sulfur-selective detector. This technique, termed metal foil collection/flash vaporization/flame photometric detection (MFC/FV/FPD), can be used to yield a total gaseous sulfur response since certain foils show nearly identical collection efficiencies and response curves toward the sulfur gases of major interest. This system has distinct advantages in terms of sensitivity, repeatability, ease of automation, and sample throughput over methods which use relatively slow, conductive thermal desorption of metallic collectors. The response of the flame photometric sulfur-selective detector is mass-flow rate dependent; therefore, a large detectability enhancement is realized by flash injection of the sample (i.e., less than 100 millisecond desorption duration). A precisely controlled ( $\pm 0.05\text{V}$ ) constant voltage capacitive discharge system allows extremely repeatable desorption ( $\pm 1\%$  of response). Since the metallic foil collector returns to its original collection characteristics after "flashing," sampling cells can be flash analyzed and prepared for the next sampling event in less than ten seconds.

The MFC/FV/FPD approach to ultratrace airborne sulfur measurements has been incorporated into a "quasi-continuous" air monitoring system capable of time-programmed sampling periods and flash desorption events via an onboard microcomputer. The programmable flash desorption system when coupled, as an inlet manifold, to a commercially available FPD sulfur monitor allows unattended operation. In this particular configuration, a pump in the FPD sulfur monitor draws the air sample through the collection cell at 200 mL/minute. Data points can be taken automatically every minute (for sample concentrations ranging from 25 ppb to 0.75 ppb [ $W_{\text{total-S}}/W_{\text{air}}$ ]), every five minutes (for levels from 10 ppb to 150 ppbtrillion) or every fifteen minutes (for levels from 2.5 ppb to 50 ppbtrillion). For remote field sampling application, the sampling flow rate is increased to 5 L/minute and sampling cells can be sealed, transported to the laboratory, and "flash analyzed" several weeks later. These conditions provide a detection limit of 10 ppbtrillion DMDS (V/V) for a five-minute sampling period. The precision of these measurements is approximately  $\pm 2\%$  over the entire LDR of  $10^3$ .

This presentation will focus on the characterization of a MFC/FV/FPD system for the determination of seven sulfur gases of main interest (listed above) at concentrations ranging from 20 ppb to 50 ppbtrillion (V/V) in air. The collection efficiencies and analytical response curves for these sulfur compounds collected on several different metals will be discussed. Collection efficiency versus sampling flow rate, inter-cell variance, sampling cell storage, and interference study results will be included.

MEASUREMENT OF MERCURY EMISSIONS FROM A  
MODIFIED IN-SITU OIL SHALE RETORT

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Mercury concentrations in Colorado oil shale are on the order of several hundred nanograms per gram, which is typical for sedimentary materials. Although these concentrations are low, there is the potential for the mobilization of most of the Hg contained in shale during shale oil production due to the high volatility of Hg and its compounds. A laboratory investigation of Hg mobilization during retorting resulted in the development of two methods for the quantification of total Hg in a simulated oil shale offgas stream. The processing of a large modified in-situ retort ( $4 \times 10^4 \text{ m}^3$ ) from June to December 1981, at the Rio Blanco Oil Shale Company lease tract in Colorado provided a unique opportunity to apply these analytical techniques to the measurement of Hg emissions from an actual semi-commercial scale retort.

A continuous, on-line, gas monitor based upon the principal of Zeeman atomic absorption spectroscopy was the primary method used for the measurement of offgas Hg concentrations. This instrument is capable of accurately quantifying Hg concentrations from 0.1 to  $20 \mu\text{g}/\text{m}^3$  with up to 90% extinction of the analytical and reference beams due to particulates, water, and organics. Forty-two hours of on-line data were obtained with the gas monitor over a 35-day period during the latter half of the retort burn. Mercury emission rates in grams per day were calculated from the Hg concentration data and offgas flow rates. The concentrations and the calculated emission rates were highly variable both within and between days. This variability demonstrates the importance of having continuous measurement capabilities for the determination of Hg emissions resulting from shale oil production.

The performance of the on-line gas monitor was compared with the performance of an independent reference method for the quantification of offgas Hg concentrations. The reference method collects gaseous Hg by amalgamation on columns containing gold-plated glass beads. Analysis is by two-stage thermal desorption and atomic absorption spectroscopy detection. Both

the gas monitor and the amalgamation method produced comparable results. The amalgamation method was easy to use; however, only a limited number of samples could be processed.

COMPARISON OF TRANSMISSION AND SCANNING ELECTRON  
MICROSCOPE TECHNIQUES FOR MEASUREMENT OF  
AIRBORNE ASBESTOS FIBERS

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Electron microscopy has become the accepted technique for determination of asbestos in air. Although the transmission electron microscope (TEM) is the preferred instrument for measuring airborne ambient asbestos, analysis of "workspace" environments may be accomplished using the scanning electron microscope (SEM).

Selection of any analytical method must consider the sample matrix that is being tested. Ambient air and "workspace" environments are commonly sampled and tested for asbestos. Asbestos fibers found in the ambient environment are too small for positive identification using the SEM. Limitations of the scanning electron microscope analysis, when compared to transmission electron microscopy, include lower spatial resolution and image contrast.

In lieu of these apparent shortcomings, the SEM has distinct advantages over the TEM for analysis of air samples containing a large concentration of fibers in the "occupational" (>5.0 m) size range. In addition, sample preparation for SEM is not as complex or time consuming as that required for TEM.

In order to evaluate the advantages and limitations of fiber measurement using the SEM, airborne asbestos samples were collected for analyses by both SEM and TEM. Fibers were counted using the Environmental Protection Agency's "Provisional Methodology for Airborne Asbestos" (EPA-600/2-77-178). Over 95% of the particulate emissions collected in these samples were asbestos fibers, thus minimizing any potential matrix effects from other fibrous materials normally present in air.

Differences in fiber count and dimensions using both SEM and TEM instruments were evaluated using the following criteria: total fiber concentration, fiber dimensions (length and diameter) and calculated mass. The results of these analyses and a discussion of the applications of both instruments to various matrices will be presented.

## OPTIMIZATION OF ELECTRON MICROSCOPE

### MEASUREMENT OF AIRBORNE ASBESTOS

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Asbestos is recognized as a health hazard. Asbestos may be present in:

- air samples (ambient air; emissions from the degradation of end-use materials--insulation, textiles etc.; emissions from mining and processing activities; from naturally occurring sources; etc.);
- water samples (drinking, lakes, rivers, effluents from industrial plants, etc.);
- biological or clinical samples (organs, tissue body fluids, etc.); and
- other miscellaneous bulk samples (ores, food, alcoholic beverages, etc.).

These various types of samples require different collection methodologies and very diverse preparation techniques.

To minimize contact with this naturally occurring mineral, analytical methods to determine its presence (identification and possible size) and concentration (number and/or mass) are essential. These various types of source materials must be analyzed in order to ascertain the present and potential effects of asbestos on the general population.

There are several asbestos analysis methodologies. Each has its advantages and limitations. These methods may be categorized into those providing concentration information (bulk material analysis) and those providing morphology, size distribution, and concentration (single fiber analysis). Electron microscopy provides particle morphology, size and identification.

The Environmental Protection Agency has a Provisional Methodology (PM) for the measurement of airborne asbestos concentrations, specifically for ambient air and for use as a "screening" procedure. Inherent in such a procedure were problem areas such as: (1) sample collection and transport; (2) sample preparation; (3) a more exacting identification of asbestos, especially of amphibole; and (4) proper use of the methodology.

These problem areas were evaluated and the results were utilized in refining the Provisional Methodology. The optimized methodology has three levels of effort, each requiring a more sophisticated instrumentation and a more highly trained operator. Procedural continuity is maintained from level to level.

The levels are:

- Level I     a screening methodology utilizing morphology and visual selected area electron diffraction (SAED) pattern recognition
- Level II    a regulatory methodology utilizing morphology plus visual SAED plus elemental analysis
- Level III   a confirmatory methodology utilizing morphology plus visual SAED, plus a selected number of SAED micrographs of zone axis patterns plus elemental analysis.



## A PILL FOR THE ASSESSMENT OF POLLUTION MEASUREMENT METHODS

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Accurate and precise assessment of the relative risks of man-made environmental hazards is essential for the establishment of sound regulatory policies. Monitoring of various sources of hazardous emissions is an important component of this assessment. In order to assure acceptability of the monitoring data, the monitoring activities must be closely scrutinized by means of a rigid program of quality control and quality assurance. One generally appropriate quality assurance practice is the performance audit, which involves challenging the methods and/or analysts in question with test samples. If the performance audit is conducted concurrently with source emission testing, an assessment of the measurement accuracy can be made. Currently, the USEPA is providing standards in the form of gases or liquids as audit materials for certain EPA source reference methods. For example, liquid sulfate and nitrate standards are available as audit materials for Source Reference Methods 6 and 7, respectively. These audit materials are useful for the evaluation of the analytical procedures but not for the sampling procedures of the method. Hence, a need exists for a simple method or device that can be used to audit both the sampling and analytical phases of the EPA reference methods.

Recently, Research Triangle Institute (RTI) under contract to the U.S. Environmental Protection Agency (USEPA) developed an audit material for the evaluation of both the sampling and analytical aspects of EPA Method 6. The method uses a known amount of a chemical compound in the form of a tablet or pill (or placed in a capsule) to generate sulfur dioxide quantitatively by reaction with an acid. The reaction takes place in a compact glass impinger system which can be taken to the field. The SO<sub>2</sub> generated in test runs was collected and analyzed using the method 6 procedure. The SO<sub>2</sub> generation was quantitative and the recoveries were found to be 94 + 5 percent. The time required to complete the chemical reaction was less

than 15 minutes at a flow rate of 1 liter/minute but 45 minutes sampling time is recommended.

The technique was evaluated both in intra- and interlaboratory studies. The results of these studies, experimental details, percision and accuracy data and finally tablet stability data will be presented. The pill technique offers a simple, inexpensive and accurate means for evaluating and/or auditing sampling and analytical phases of the EPA Source Emission Reference Methods.

DYNAMIC IMPINGER - APPLICATION TO THE ANALYSIS OF HALOGENATED  
HYDROCARBONS FROM SOURCE EMISSIONS

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A countercurrent flow gas sampling cell (dynamic impinger) was designed for source emission measurements. The absorbing solution can be withdrawn from this dynamic impinger continuously and analyzed by a specific analytical technique, or be fed into an autoanalyzer/continuous monitor for analysis. The design of this dynamic impinger is such that the temperature of the absorbing solution, the flow rate of the gaseous sample, and the flow rate of the absorbing solution can be adjusted before or during the sampling process to accommodate the sensitivities of the end analytical techniques used for analysis.

This dynamic impinger has been evaluated, in the laboratory, for the analysis of halogenated hydrocarbons. Iso-octane was used as the absorbing solution for the halogenated hydrocarbons. The absorbing efficiencies of halogenated hydrocarbons at different sampling temperatures, gas sample flow rates, and absorbing solution flow rates were determined. A gas chromatograph with electron capture detector and fused silica capillary column was used for the analysis of the halogenated hydrocarbons.

A dual sampling train which consists of one sample gas inlet from the source with a 50/50 splitter to two identical dynamic impingers was designed for the quality assurance program. This will allow duplicate samples to be taken for analyses. An injection port was also built into one side of this dual sampling train that would allow the addition of standard gas for the recovery efficiency study.

SAMPLING AND ANALYSIS OF INCINERATION EFFLUENTS  
WITH THE VOLATILE ORGANIC SAMPLING TRAIN (VOST)

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MRI has conducted trial burns at several different types of hazardous waste incinerators throughout the country. Regardless of the design of the incinerators, volatile principal organic hazardous constituents (POHCs) are generally important components of the stack effluent. The traditional method of collecting samples of analysis of volatile organics is integrated gas bags. Gas bags are used with varying degrees of success by many different laboratories. However, the integrated gas bag technique generally suffers several drawbacks, including the need to position the gas bag in a bulky evacuated sample box, bag valve leakage problems, adsorption losses of sample components, contamination problems, and low sensitivity when the bag is analyzed directly using a gas-tight syringe sampling technique.

In response to the need to develop a better sampling system for volatile POHCs from stack effluents, EPA funded MRI's development of the VOST. As reported previously, the VOST has undergone a successful laboratory evaluation and has been used by MRI, along with integrated gas bags, at several hazardous waste incineration trial burns. The purpose of this paper is to present details of the design of the field version of the VOST and also present information on how the VOST is used in the field for sampling and analysis including adsorbent trap preparation procedures, procedures for preventing contamination, sampling parameters, trap analysis procedures, data showing the distribution of collected compounds on the front and backup adsorbent traps, and comparison of VOST data with integrated gas bag data.

#### Design of the VOST

The VOST basically consists of a system designed to draw sample gas at a flow rate of 0.1 to 1 liter/min through two adsorbent traps connected in series. The front trap contains Tenax and the backup trap contains a section of Tenax and a section of charcoal. The purpose of the second (backup) trap is to collect very volatile POHCs (e.g. vinyl chloride and methylene chloride) which may partially break through the front adsorbent trap during sampling.

The field version of the VOST, which folds up inside a portable case for easy transport, attaches to a section of glass tubing which is inserted into the stack to collect the sample. The hot, wet stack gases, which are drawn into the VOST by a small air pump, are cooled in a spiral condenser preceding the front trap. The bottom portion of the open case is filled with ice water which is continually circulated by a small water pump which sits at the bottom of the case. The condensed water and stack gas then pass down through the front Tenax trap where most of the organics are adsorbed except those with very low breakthrough volumes, such as vinyl chloride. The condensed water collects in an Erlenmeyer flask-shaped impinger and is continually purged by the sampled gas. Any volatile POHCs which pass through the front Tenax adsorbent trap are then carried up through a Teflon<sup>R</sup> connecting tube, down through a second spiral condenser, and through the backup Tenax/charcoal trap where they are adsorbed. The gas is then dried in a silica gel tube and passes into the dry gas meter for volume measurement. The Tenax and charcoal are held in 10 cm x 1.6 cm ID glass tubes with a fine-mesh screen supported by a C-clip. Both materials are made from stainless steel. These supporting materials hold the adsorbents more uniformly inside the tubes than glass wool. This results in a lower likelihood of channeling and lower retention of water in the trap. The glass tube containing the adsorbents is held within a larger diameter outside tube using Viton O-rings. The purpose of the outside glass tube is to protect the outside of the adsorbent-containing tube from contamination. The outer glass tubes are held in stainless steel carriers which provide easy removal from and insertion into the VOST during sampling.

#### Trap Preparation and Contamination-Prevention Procedures

During development and evaluation of the field VOST, it was discovered that the traps were sometimes severely contaminated with volatile organic compounds. Several possible sources of contamination were identified, such as the hostile ambient environment, contaminated metal carriers, O-rings, and the adsorbents. In order to prevent contamination, a series of stringent trap preparation procedures was tested and adopted which has proved very effective in eliminating the contamination for field sampling with the VOST. These procedures will be discussed during the presentation.

#### Sampling Parameters

During field sampling the VOST trap pairs are generally replaced with fresh traps at selected intervals (i.e., every 20 min or 20 liters of sample) over a 2-hr sampling period. These are two basic reasons for changing the traps at selected intervals:

-At sample volumes of greater than 20 liters, some of the very volatile POHCs may break through both the front and backup adsorbent traps.

-The changing of the traps allows an initial analysis of one pair of traps. Analysis of a single pair of traps lowers the possibility of collecting too much sample and overloading the GC/MS system. However, if the POHCs are not detected or are present at low levels in the single pair, the option exists of combining the contents of the remaining pairs of traps onto one pair of traps with a concomitant increase in sensitivity.

A "SLOW VOST" is also being evaluated during which only one or two pairs of traps are used for sample collection. The slow VOST, which generally samples only 5 liters of stack gas over the 2-1/2 to 3-hr sampling period, has the following advantages:

-The 25 ml/min sampling rate reduces the likelihood of breakthrough and serves as a check on breakthrough for the regular VOST.

-A more integrated sample is obtained. This is very advantageous in situations where the stack gas composition changes during the incineration test.

The main disadvantage of the slow VOST is the decreased sensitivity.

#### Trap Analysis Procedures

Prior to GC/MS analysis, all Tenas and Tenax/charcoal adsorbent trap samples and standards are spiked with 50 ng of D<sup>4</sup>-1,2,-dichloroethane and D<sup>6</sup>-benzene internal standards using the flash vaporization technique in which the spiking solution is vaporized and carried onto the trap with a carrier gas.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, and calibration standards) are thermally desorbed using a stream of carrier gas into a water column (1 to 5 ml) which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The sample trap is dropped into the desorption chamber and desorbed at a flow rate of 100 ml/min for 10 min at 180 C. The desorbed compounds pass into the bottom of the water column, are purged from the water, and then are collected on an analytical adsorbent trap which also contains Tenax and charcoal. The compounds are then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

COMPUTED ASSISTED, REAL TIME DETECTION OF CHLOROPICRIN,  
CHLOROFORM AND CARBONYL CHLORIDE DURING WASTE  
DISPOSAL OPERATIONS

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In order to ensure worker and environmental safety during Army disposal of chemical waste, analytical instrumentation was selected and methodology developed to provide sensitive, accurate and rapid testing of chloropicrin, chloroform and carbonyl chloride. The instrument utilized is a programmable, single beam infrared spectrophotometer fitted with a twenty meter gas cell to enhance sensitivity. Selected wavelengths for individual compounds from 2.5 to 14.5 microns ( $4000 - 690 \text{ cm}^{-1}$ ) were used for quantitative determination of the compounds at parts-per billion level. The integral micro-processor allowed rapid analysis and reporting of concentrations with corrections for interferences. Calibration was performed using a permeation standards generator simulating actual environmental sampling techniques. A concentration versus absorption matrix was developed using data from all pertinent analytical wavelengths.

APPLICATION OF API-MS/MS FOR STUDIES OF HAZARDOUS  
AIR POLLUTANT REACTIONS

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The fate of hazardous air pollutants (HAPs) is of concern due to the possibility that even more hazardous products may result from reaction of these materials in air. We have applied the relatively new technique of Atmospheric Pressure Ionization MS/MS to this problem in an effort to identify the major reaction products of selected HAPs.

The experiments make use of a Sciex, Inc. Trace Atmospheric Gas Analyzer (TAGA 6000) coupled directly to a 17 m<sup>3</sup> irradiated reaction chamber. The volume of the reaction chamber is sufficient to permit sampling at the high inlet flow rates of the TAGA (~20 lpm) for extended periods, as required for study of compounds of very low reactivity. The large volume of the chamber also permits detailed characterization of the reaction by more conventional monitoring techniques (e.g. for O<sub>3</sub>, NO, NO<sub>2</sub>, SO<sub>2</sub>, HNO<sub>3</sub>, PAN, CO, etc.). The Sciex instrument employs a point to plane corona discharge in a high volume atmospheric sampler. In a typical chamber experiment, the mass range from 40 to 250 AMU was scanned once every 30 minutes using the instrument in the traditional MS single analyzer mode. This procedure required sampling from the chamber for only 5 minutes out of every 30 minutes, thus minimizing the volume of sample withdrawn from the chamber. At the end of the experiment, the instrument was switched to the MS/MS mode of operation and the first mass analyzer was tuned sequentially to the masses of the prospective reaction products (generally those peaks which grew during the experiment). Argon was used to induce collisional decomposition and the third analyzer was then employed to scan the fragment or daughter ions.

The HAPs examined thus far include epichlorohydrin, trichloroethylene, propylene oxide, acrylonitrile, benzene, toluene and p-dichlorobenzene. The three aromatic compounds represent a special class of HAPs which has been investigated in more detail. Aromatic hydrocarbons make up a considerable fraction of urban NMOC concentration, and their reactions contribute significantly to photochemical smog formation. Attempts to



incorporate aromatic hydrocarbons into mathematical models of photochemical air pollution have been thwarted by a lack of definitive information on the mechanisms of aromatic hydrocarbon reactions and the nature and fate of the major reaction products. The API-MS/MS environmental chamber combination is providing information on the formation and subsequent decay of aromatic hydrocarbon oxidation products such as dicarbonyls, alcohols, acids, and nitration products. Studies with the various nonaromatic HAPs indicate the formation of compounds such as phosgene, chloroacetyl chlorides, ketones and dicarbonyl compounds.

AIR MONITORING DURING INCINERATION  
OF PESTICIDE-CONTAMINATED MILITARY SMALL ARMS AMMUNITION

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Thermal destruction is an attractive alternative for the disposal of hazardous wastes. To protect environmental quality and operator health and safety, and to meet regulatory guidelines, a program must be developed to monitor stack gases and workplace air. We recently incinerated 1000 tons of military small arms ammunition contaminated with DDT, DDE and pentachlorophenol (PCP) at the grams per ton level. Monitoring for the contaminants, partial and complete oxidation products, copper and lead was accomplished by absorber sampling tubes and cellulose filters and/or bubblers. This is followed by solvent desorption and GC analysis of acid digestion and AA or colorimetric determination, respectively. These methods are reliable and reproducible for concentrations below currently mandated permissible levels.

## ANALYSIS OF PCB'S BY CAPILLARY GC/ECD FOR DETOXIFICATION STUDIES

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Recent efforts have been undertaken to determine the best practical methods for disposing of hazardous materials such as polychlorinated biphenyls (PCB's). Disposal in approved solid-waste landfills and incineration have been the most used and studied methods. Questions concerning long-term problems of landfills have brought about a need for alternate methods of disposal. One method being studied is chemical detoxification.

One common need in all of the methods used and under study is documentation of the presence and amount of PCB's. This determination is needed to calculate efficiencies of destruction or combustion of the PCB compounds. Therefore, the most critical part of these studies is the analysis. PCB's were analyzed by gas chromatography with electron capture detection utilizing a SE-54 fused silica capillary column for the purposes of determining PCB detoxification efficiency. A demonstration was conducted for EPA by Acurex Waste Technologies, Inc. of Mountain View, California to show the detoxification efficiency of their process. Samples of the PCB spiked transformer oil were taken before and after the detoxification process for each batch of oil treated. In the first batch of untreated oil, Aroclor 1260 was found at a concentration of 5800 ppm. The treated sample from this same batch contained less than 0.5 ppm of Aroclor 1260. Another batch of untreated oil was analyzed to contain a mixture of Aroclor 1254 and Aroclor 1260 at concentrations of 525 and 170 ppm, respectively. Results of the treated oil were less than 0.5 ppm for both Aroclor 1254 and 1260. The analysis of these samples by fused silica capillary was very simple and required only an initial water extraction to remove sodium salts present in the treated samples. Appropriate dilutions were made of the untreated oil for the PCB's to be within the range of the standard curve. Use of the fused silica column also increased resolution of the isomers of the Aroclors, thus allowing a much easier confirmation of the Aroclors and their quantity than from using packed columns. Interferences such as pesticides will not create as much of a problem on the fused silica capillary column as is a common problem with the Aroclors or bias results as much as 50 to 100%.

## ALDEHYDE EMISSIONS FROM WOOD-BURNING FIREPLACES

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Aldehyde emissions from wood-burning fireplaces were measured. Total aldehydes ranged from 0.6 to 2.3 g/kg wood burned based on tests with cedar, jack pine, red oak, and ash. Formaldehyde, acetaldehyde, and p-tolualdehyde were the major aldehydes emitted with formaldehyde comprising 21-42% of the total. Aldehyde and particle emissions were inversely correlated with burn rate and may also be related to wood type.

Based on our measurements, nationwide aldehyde emissions from residential wood-burning were estimated to be between  $14-54 \times 10^6$  kg/yr. This value is comparable to both power plant and automotive aldehyde emission sources. It is likely that residential wood-burning is a major source of primary aldehydes during the winter.

SESSION IV  
PERSONAL MONITORING

John D. Spengler  
Session Chairman

## WHAT HAVE WE LEARNED FROM EXPOSURE STUDIES?

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The need to improve epidemiologic studies of air pollution and the cost of controls for stationary and mobile sources has raised concern about the representativeness of ambient fixed site monitoring. In addition, concerns about population exposures to hazardous pollutant emissions has increased the demand for improved exposure assessments. Both modeling and measurement studies have been undertaken. This paper focuses primarily on a series of direct exposure studies in an effort to extrapolate the fundamental findings and the remaining uncertainties.

Personal monitoring in the field of air pollution is still relatively new. Urban exposures to particles and lead was reported by Fugas, et al., in 1972. Studies of CO exposures to bicyclists and commuters were first published as recently as the mid-1970's. Binder, et al., 1976, used personal epidemiologic study to contrast home factors leading to different exposures among children. Since the early 1970's there has been an increasing number of studies reporting personal exposures to CO, sulfates, NO<sub>2</sub>, respirable particles, SO<sub>2</sub>, lead, aluminum, iron, and several volatile organic compounds.

This paper reviews a number of these studies that can be categorized as having one of four objectives:

1. Determining the representativeness of ambient fixed site monitoring.
2. Characterization of population exposure groups.
3. Parameterization of activity/concentrations for model development.
4. Source contributions to total exposure (individuals/populations).

VALIDATION OF A PASSIVE SAMPLER FOR DETERMINING  
FORMALDEHYDE IN RESIDENTIAL INDOOR AIR

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The recent development of passive sampling devices for the determination of low concentrations of formaldehyde in air have made large-scale investigations of the magnitude and extent of chronic formaldehyde exposure in the residential indoor environment feasible. A passive sampling device based on the principle of diffusion has been developed and tested specifically for this application. The device, which is inexpensive and easy to use, is capable of measuring one-week time weighted average concentrations of formaldehyde from as low as 0.018 ppm to over 1 ppm. The one-week sampling interval is ideally suited for the quantification of formaldehyde in residences since formaldehyde concentrations vary in response to environmental factors such as temperature, humidity, and ventilation which are influenced by occupant activity cycles.

This paper presents the results of laboratory validation experiments conducted with the formaldehyde passive sampler, as well as the results of a field evaluation in which the performance of the passive sampler was compared to that of a reference pump/bubbler sampler in occupied residences and an office. The parameters evaluated in the laboratory and field experiments were: sampling rate; detection limit; relative humidity effects; face velocity effects; chemical interferences; shelf life; sample stability; overall precision; and overall accuracy. The performance of the passive sampler compares favorably to that of a reference pump/bubbler sampler while offering many practical advantages. Formaldehyde concentration data obtained with the passive sampler in a variety of housing types are presented.

## PASSIVE SAMPLING DEVICES FOR ORGANIC VAPORS IN AMBIENT AIR

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Several commercially available passive sampling devices for volatile organic compounds were evaluated for potential use as ambient air monitors (0.1 to 50 ppbv levels). Laboratory studies were conducted to examine the effects of background interference, humidity and air velocity on sampling efficiency. One commercial device, which uses a charcoal strip collector, was found to be sensitive enough for ambient air use when special precautions were taken by the manufacturer to limit blank levels. Chamber exposures were conducted and demonstrated that device performance was independent of normal ambient air concentrations. However, high relative humidities (above 80%) and insufficient ventilation greatly impaired performance.

In a parallel study, a high-performance passive monitor was developed for short-term, low-level monitoring applications. The small, stainless steel device is simply designed and inexpensive. It has a high equivalent sampling rate, is reusable and rechargeable, and is amenable to thermal desorption. Laboratory and field tests with Tenax GC as the sorbent have shown that the monitor compares very favorably with active (pump-based) samplers. The device was found to be useful for monitoring at a hazardous waste site.



LABORATORY STUDIES OF THE TEMPERATURE DEPENDENCE  
OF THE PALMES NO<sub>2</sub> PASSIVE SAMPLER

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The use of passive samplers for air pollution measurement has become widespread. The samplers are employed in many different ways, e.g., in assessing personal exposures to pollutants and determining area concentrations and in comparing indoor and outdoor pollutant concentrations. In some studies, passive samplers have been used under conditions for which they have been inadequately tested. One factor that can affect the sampling rate is temperature. Diffusion theory predicts only a 1.7% change in the sampling rate with a 10°C change in temperature at 21°C; however, triethanolamine, the NO<sub>2</sub> absorbent employed in the Palmes NO<sub>2</sub> sampler, has a liquid-solid phase transition at 21°C. Because this phase change occurs at a temperature typical of indoor and outdoor temperatures, the effect of temperature upon the performance of the Palmes NO<sub>2</sub> passive sampler was investigated. This report describes the procedure used to generate and control exposures of passive samplers along with the analytical results and a discussion of error. During these tests the NO<sub>2</sub> concentration, exposure time, and face velocity were held constant while the temperature of the sampled air was varied from 7°C to 38°C. The collection efficiency of the Palmes NO<sub>2</sub> passive sampler decreased by 15% when the temperature decreased from 27°C to 15°C. This study illustrates the need for careful evaluations of passive samplers under controlled conditions that closely approximate actual use conditions.

CHARACTERIZATION OF PERSONAL EXPOSURE TO NO<sub>2</sub> and SO<sub>2</sub>  
AND THEIR INDOOR CONCENTRATIONS IN OTTAWA, CANADA

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A study designed to determine the health effects of airborne pollutants must attempt to measure personal exposure levels. The confronting problem, however, is that the personal monitoring device must be sensitive to the concentration levels found generally in an indoor setting, where people spend 95% of their time, without interfering with the daily activities of the volunteers. The most simple device which can be adapted to both personal and site monitoring, is the Palmes tube. The device has been successfully applied to the measurement of a single component (NO<sub>2</sub>) only. We initiated the present study with a threefold purpose; first, to field test the Palmes tubes for monitoring personal exposure and site concentrations of NO<sub>2</sub>, second, to develop the device for the simultaneous measurements of two gases SO<sub>2</sub> and NO<sub>2</sub>, and finally to use it for measuring personal exposures of a selected population to these gases in the city of Ottawa, Canada.

This report summarizes the work completed under the first phase of the study; the latter two are underway and will extend to the winter months of 1982-83. It is, however, anticipated that the study would be finished by April 1983, and a complete report will be available for presentation at the national symposium.

The performance of Palmes tubes was field tested in a selected sample of 51 staff members of this Directorate residing in Ottawa. Each respondent wore one tube for a week and placed another in the kitchen of his/her home for the same period in June 1982. Average personal (p) exposure levels of NO<sub>2</sub> were higher in all cases than the average kitchen (K) levels, irrespective of the type of heating in volunteers' homes with the highest values found in oil-heated homes (P = 7.2, range 3 to 16 ppb; K = 3.8, range 2 to 13 ppb) followed by electric heated homes (P = 5.4, range 3 to 9 ppb; K = 3.2, range 2 to 9 ppb) and gas heated homes (P = 5.0, range 3 to 8 ppb; K = 3.2, range 1 to 6 ppb). The analysis of variance indicates that the difference was significant (p>0.02). Results of regression analysis, however, indicate a significant correlation between P

and K readings for gas heated homes ( $r = 0.58$ ) only;  $r = 0.50$  for electric homes and 0.11 for oil homes. Overall, indoor levels of  $\text{NO}_2$  as measured by both personal and kitchen monitors were found to be low.

## NIOSH-DEVELOPED SYSTEMS FOR MONITORING EQUIPMENT EVALUATIONS

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The National Institute for Occupational Safety and Health (NIOSH) has an active program in the evaluation of workplace monitoring devices and personnel exposure monitors. In addition, NIOSH frequently writes evaluation criteria, performance specifications and testing protocols for instruments and monitors. Specialized systems are sometimes needed to carry out the exacting experimentation required for both the evaluations and to demonstrate the efficacy of developed specifications and protocols.

This paper will describe a specialized system consisting of a gas and vapor generation system with programmed automatic cyclic output, a recirculating exposure chamber and a multiple, variable sampler. The generation system can use vapor pressure, syringe injection, permeation/diffusion tubes or gas cylinders as the contaminant source while automatically repeating a preprogrammed generation cycle over a time period from a few minutes to several days. The exposure chamber, with a 1 M<sup>3</sup> internal volume, can house portable instruments as well as personal monitors (e.g. sorbent tubes and passive monitors) for evaluations. Six paired samplers allow up to twelve samples of equal or variable time and contaminant loading to be obtained on the multi-sampler. This total system can be used for the generation and sampling of complex atmospheres down to the sub-parts-per-million range.

RELATIONSHIPS OF MEASURED NO<sub>2</sub> CONCENTRATIONS AT DISCRETE  
SAMPLING LOCATIONS IN RESIDENCES

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The average concentration of NO<sub>2</sub> was measured using NO<sub>2</sub> diffusion dosimeters (Palmer tubes) in 140 private homes in Topeka, Kansas. Each home was sampled eight times throughout a one-year period, and each home was sampled in at least three locations: the kitchen, the child's bedroom, and outside. Half the homes used gas for cooking, and half used electricity. Consistent with previous studies, the homes with gas cooking stoves had measured NO<sub>2</sub> concentrations significantly higher than homes with electric cooking stoves. The results will be presented, including correlations of the measured NO<sub>2</sub> concentrations with a number of home descriptors, the adequacy of a single sampler location within a home as representative of the exposure within the home, and the variation of results at sampler locations over an annual period of time.

ESTIMATED DISTRIBUTIONS OF PERSONAL  
EXPOSURE TO RESPIRABLE PARTICLES

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Historically, attempts to estimate exposure to airborne pollutants have used data collected at centrally-located outdoor monitoring stations as the starting point. It has been assumed that outdoor pollutant concentrations represent a good measure of the amount of pollutant to which the population is exposed. However, significant spatial variation in pollutant concentrations with a community have been documented in many cases. Simple examples of this phenomenon may include elevated particulate levels near dirt roads or high concentrations of SO<sub>2</sub> downwind of a coal-fired power plant.

This spatial variation represents only one component of the total exposure variance, but remains indicative of the problems associated with single-point measurements used for exposure estimates. Perhaps more significant from the point of view of exposure estimation are the differing pollutant levels found in differing microenvironments in which people spend their time. Recent monitoring programs have shown that pollutant levels far in excess of outdoor concentrations can be found in microenvironments containing a source of pollutant contamination.

The combination of spatial variation in outdoor pollutant concentration and differences in pollutant levels in various microenvironments, coupled with the expected variation in daily activities of individuals, results in measured personal exposures which differ markedly from and are only weakly correlated with the pollutant concentrations at centrally-located monitors. Thus, we are left with the problem of trying to extract personal monitoring information from single-point observations in cities without personal monitoring programs.

In this paper, we describe preliminary efforts to estimate mean exposures and distributions of these exposures based on the microenvironment concept. The approach is to develop mean concentrations and distributions for these concentrations for several microenvironments using existing area monitoring data.

These data are then combined with activity data to form estimated mean exposure:

$$E = \sum_i f_i c_i$$

where:  $f_i$  = fractional time in the  $i^{\text{th}}$  microenvironment, and  $c_i$  = concentration in the  $i^{\text{th}}$  microenvironment. The estimated distributions are determined by two methods: Gauss' law of error propagation as applied to equation (1); and simulation using appropriate distributions associated with each of the parameters in equation (1). The mathematical form of the exposure distribution is left open for the present.

In this paper, we report the results of our estimation of exposure to respirable particles for the six cities in the Harvard Air Pollution/Lung Health Study. Within each city, we have categorized the population as cigarette-smoke-exposed or non-cigarette-smoke-exposed. The results presented are for children's annual average exposure. The children are assumed to spend all of their time in one of three microenvironments: inside their home, at school and outdoors.

Results of the exposure estimation suggest that although mean outdoor concentrations of respirable particles vary by a factor of four over the six cities, exposures within an category vary by less than a factor of two. Furthermore, the exposure variance is quite large, resulting in overlap among seemingly distinct populations. For example, smoke-exposed children in Portage, Wisconsin, suffer similar exposures as non-smoke-exposed children in Steubenville, Ohio.

The preliminary conclusion of this report is that the variance of the distribution of exposures within a population is affected by many factors. The use of simple dichotomous categorizations such as exposure to smoke may result in significant mis-classification of exposure levels with concomitant loss of power in epidemiologic studies for determining health effects associated with ambient air pollution.

EMPIRICAL MODELS FOR ESTIMATING INDIVIDUAL EXPOSURE  
TO AIR POLLUTANTS IN A HEALTH EFFECTS STUDY

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A panel study of 52 asthmatics was carried out in the summer of 1981 to evaluate the acute health effects of air pollution in the Houston area. It was recognized that accurate estimates of individual exposures were required for this evaluation. Data from a three-tiered monitoring network (fixed-site ambient monitoring stations, a mobile van employed for indoor and outdoor monitoring at residences, and personal monitoring) were used to develop a set of house-specific exposure models which were then used with personal activity patterns to derive individual exposure estimates.

A preliminary step established the level of complexity of data representation needed to provide an adequate model. Simple linear models were then constructed relating the data from the fixed-site station to the mobile van outdoor data. Similar models were constructed for outdoor to indoor relationships at each of the twelve locations the mobile van was used. Estimates of exposures were determined for individuals for whom personal monitoring data were available. These estimates were then compared to the observed values recorded. Differences among models were examined employing differences among the locations and houses monitored. Results of this estimation process will be presented for ozone, nitrogen dioxide, respirable particulates, aeroallergens and formaldehyde.



CO EXPOSURES IN WASHINGTON, D.C. AND DENVER, COLORADO  
DURING THE WINTER OF 1982-83

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A study of exposures to carbon monoxide (CO) using personal exposure monitors (PEMs) was conducted in Washington, D.C. and Denver, Colorado during the winter of 1982-83. The primary objective of the study was to validate a methodology for measuring the distribution of CO exposures in a representative sample of an urban population so that risk to the entire population can be estimated. The methodology for selecting the participants, measurement of CO, and other study procedures will be discussed. Preliminary CO PEM measurements from each of the cities will be compared with fixed-site CO measurements. Recommendations for methodological improvement will be discussed.

MODELS OF HUMAN EXPOSURE TO NITROGEN DIOXIDE  
USING PERSONAL MONITORING DATA

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Human exposures to air pollutants arise from several sources and are experienced in various "microenvironments." Nitrogen dioxide ( $\text{NO}_2$ ) formation is closely related to high temperature combustion. Automobiles and fossil fuel power plants are major outdoor sources; indoor sources include gas appliances, cigarette smoking, and unvented space heaters. Traditionally, most concern regarding the health effects of air pollution has been based on outdoor air quality. However, Americans spend nearly 90% of their time indoors, thus making pollutant concentrations in indoor locations -- especially inside the home -- a major influence on total personal exposure. These indoor concentrations should be incorporated into estimates of group or individual exposure used by epidemiological studies to permit accurate assessment of the health risks of air pollution, both from indoor and outdoor sources.

As part of a longitudinal air pollution/health study (Harvard Six Cities study), personal exposure to  $\text{NO}_2$ , time spent in various locations and household concentrations were measured for nearly 350 individuals residing in 82 homes in the Portage, Wisconsin area for one week during both the summer and winter of 1981-1982. Average levels of  $\text{NO}_2$  measured outside these homes were  $13.55 \text{ ug/m}^3$  (S.D. 5.64) during the summer, and  $13.55 \text{ ug/m}^3$  (S.D. 6.15) during the winter. Kitchen concentrations in homes with gas stoves averaged about  $30 \text{ ug/m}^3$  higher in the summer and  $70 \text{ ug/m}^3$  in the winter than the outdoor levels. Non-kitchen areas in these homes were about

12 ug/m<sup>3</sup> higher in the summer and 40 ug/m<sup>3</sup> in the winter. Participants spent more than 65% of their time at home in the summer compared with about 15% outside; nearly 70% of the average day was spent at home during the winter compared with less than 10% spent outside. Personal weekly average exposures for individuals from gas stove homes were greater in both magnitude and availability in the winter with mean 45.11 and S.D. 19.20 ug/m<sup>3</sup>, than in the summer with mean 25.00 and S.D. 8.55 ug/m<sup>3</sup>. The seasonal means for individual exposures of participants from homes with electric stoves were 15.44 (S.D. 9.41) ug/m<sup>3</sup> and 17.89 (S.D. 7.32) ug/m<sup>3</sup> for winter and summer, respectively. These measures of exposure and time allocation suggest that there is a wide range of variability in personal exposure to NO<sub>2</sub> that may not be adequately accounted for by simple stratifications based on cooking fuel type.

Predictive models of personal exposure to NO<sub>2</sub> are examined for different population groupings in relation to actual personal exposure measurements. Comparisons are made between estimates of exposure that utilize only central station outdoor measurements and those derived from classifications based on indoor sources (e.g. stove type), those that use both indoor and outdoor concentrations, and those that incorporate activity information.

Finally, this paper discusses the implications of these results to epidemiological investigations of the health effects of air pollution, both from indoor and outdoor sources.

COMPARISON OF PERMEATION AND DIFFUSION TYPE PASSIVE SAMPLERS  
VERSUS CHARCOAL TUBE COLLECTION OF SELECTED GASES

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A study has been made of the performance characteristics of permeation type samplers, (REAL, Inc.), diffusion type devices (3M and DuPont) and charcoal tube collection of four gases of interest. The gases studied were Freon 113, trichloroethylene, 1,1,1-trichloroethane, and perchloroethylene. Concentrations of the gases were ten, fifty, one hundred, and one hundred fifty percent of the respective TLV's with face velocities of 50 and 500 ft. per minute. Temperatures were at 20°C and 30°C and relative humidities of 30% and 80% were included.

Passive monitors are to be recommended. However, the diffusion type devices were found to show errors of as much as 95% when used in humid atmospheres.

SESSION V  
ACID DEPOSITION

John Miller  
Session Chairman

## INTENSITY WEIGHTED SEQUENTIAL SAMPLING OF PRECIPITATION

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Tremendous interest in the chemistry of rain has been generated by the often heated discussion of the problem of acid precipitation. Both long- and short-term effects of the precipitation are questions which need to be addressed. To address the question of short-term effects, the question of changes of chemistry within an individual storm need to be quantified. To focus upon a partial answer to this question, the Science Research Laboratory has built a sampler with which to obtain samples of precipitation based upon the intensity of the storm. The sampler obtains a discrete sample for each 0.01" of precipitation. The design of the sampler will be described.

In addition to the design, some of the results of the chemistry of several storms will be discussed. The samples obtained using the sequential sampler are analyzed for  $H^+$  by autoanalyzer, and for  $SO_4^{=}$ ,  $NO_3^-$ ,  $Cl^-$ ,  $NH_4^+$ ,  $K^+$  and for selected samples,  $Ca^{+2}$ ,  $Mg^{+2}$  using ion chromatography. This paper will also focus upon some of the methods of interpretation of the data. Time-series concentration and deposition records are correlated with concurrent synoptic meteorological settings. Concentration variations of an order of magnitude or more have been observed in time spans as short as an hour or two. Air mass trajectory analysis is utilized to characterize air mass approach direction and speed. Time series deposition rates will also be presented for several case studies for the dominant ions of  $H^+$ ,  $SO_4^{=}$ ,  $NO_3^-$ , and  $NH_4^+$ .

## AUTOMATION OF AN ION CHROMATOGRAPH

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The considerable interest generated in the past few years for the topic of acid precipitation has led the Science Research Laboratory to design an instrument to sample precipitation events based upon the intensity of a storm. The large number of samples produced by such an instrument requires that an efficient and rapid means of analyzing the samples be available. The ion chromatograph is capable of rapidly, accurately and precisely analyzing a sample for a large number of analytes. The automation of an ion chromatograph via the addition of an autosampler and an interface to a laboratory microcomputer aided in greatly enhancing the already powerful technique of ion chromatography.

A set of programs, in DEC-RT/Fortran IV, has been written and tested which allows for automated acquisition, filtering and reduction of ion chromatographic data. Analog data is acquired and converted to digital data using a preamplifier and an analog-to-digital converter. Data acquired from one or two channels of the ion chromatograph is written to a disc for later use. Since the autosampler is used on the ion chromatograph, insufficient time exists after data acquisition of one sample and before injection of the next sample to allow for data analysis of the first sample. Thus communication routines have been written to allow for the reduction of the data in the background while acquiring data in the foreground. The background processing smoothes the data, finds the peaks, and calculates such peak parameters as height, width, area, etc. Data is smoothed using a moving box car average and peaks are found using standard system software. It is intended that the peak and retention time data will be sorted for transmission via a local area network to a larger computer for storage and further processing. Provisions are made to allow for varied analysis and run times.

DESIGN AND TESTING OF A PROTOTYPE  
RAINWATER SAMPLER/ANALYZER

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A device for the collection and analysis of rainwater has been conceptualized; component parts are undergoing testing. The sampler will record the temperature, pH, conductivity and volume of collected rain samples and note the time of capture/analysis. Ions from samples of known volume will be trapped for subsequent laboratory analysis.

Performance characteristics will be presented which are designed to meet the short-term needs of meteorological modeling and atmospheric chemists as well as the long-term needs of soil scientists.



MEASUREMENT OF WEAK ORGANIC ACIDITY IN PRECIPITATION  
FROM REMOTE AREAS OF THE WORLD

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The Global Precipitation Chemistry Project collects precipitation by event to determine the composition of precipitation and processes that control it in five remote regions. Ion balances based on major inorganic species revealed consistent anion deficits at certain sites. This and other evidence suggested that weak organic acids contributed to free acidity. Accurate and precise techniques were developed to measure organic anions and total acidity in precipitation. Twelve samples from a remote site were analyzed for major organic and inorganic chemical constituents. Formic and acetic acids were found in all aliquots which had been treated with a biocide. The disappearance of these acids from untreated aliquots corresponded to a proportionate decrease in free acidity. Weak organic acids contributed 64 percent of free acidity and 63 percent of total acidity to precipitation during part of the 1981-82 wet season at Katherine, Australia. Unmeasured proton donors contributed 21 percent of total acidity during the period.

AERIAL INPUT OF TOXAPHENE TO THE SOUTH CAROLINA COASTAL ZONE,  
WITH RESIDUE ANALYSIS BY CAPILLARY GAS CHROMATOGRAPHY

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Until its recent ban (November 1982), toxaphene had enjoyed heavy usage throughout the southeastern U.S., in particular on cotton and soybean crops. Toxaphene, a complex chlorinated bornane mixture, is a widespread contaminant in the aquatic environment. Residues have been identified in fish from such diverse areas as the South and North Atlantic, Alpine lakes, and the Antarctic Ocean (Zell and Ballschmiter, 1980), Sweden and the Baltic Sea (Jansson et al., 1979), and the Great Lakes (Ribick et al., 1982). Atmospheric transport appears responsible for dispersion of toxaphene and other chlorinated hydrocarbons. Toxaphene in rainfall has been reported over the Baltimore area by Munson (1976) and over coastal South Carolina by Harder et al. (1980). These previous analyses employed packed-column gas chromatography. Capillary-column gas chromatography reveals many more details of the toxaphene residue. This report will discuss aerial input of toxaphene and other chlorinated hydrocarbons to the South Carolina coastal zone from September 1981 to September 1982. Special emphasis will be given to capillary column analysis and computer-assisted chromatogram comparisons of rain and air samples with technical standards and laboratory-weathered standards.

Rain samples were collected by two methods: bulk samples (precipitation plus dry deposition) were collected in stainless steel funnels continuously exposed for 2-3 weeks; precipitation only samples were collected with an Aerochem Metrics automated wet/dry sampler fitted with glass collection vessels. Airborne organics were sampled by drawing 500 - 1500 cu. m of ambient air through a glass fiber filter and polyurethane foam plugs. After extraction, cleanup, and fractionation, sample residues were analyzed on 30-m non-polar capillary columns using electron capture detection. Chromatograms of air and rain were compared with those of technical and vaporized toxaphene. These comparisons were made using Euclidean distances between points in  $n$ -dimensional space, each point representing a chromatogram. The value in each dimension is the normalized and auto-scaled peak height at a particular relative retention time, and  $n$  is the number of peaks employed.

The results of this study show that the main chlorinated hydrocarbons in rain over the South Carolina coast were toxaphene and  $\alpha$  and  $\gamma$ -hexachlorocyclohexane, with minor amounts of chlordane, DDE, and PCB. Over the one-year sampling period, 1021 grams of toxaphene was rained out into a 26 sq. km salt marsh. Most of this input occurred during the late summer of 1982. Rain toxaphene patterns are often enriched in the intermediate molecular weight components relative to the lighter and heavier isomers. Possibly this arises from a two-step fractionation procedure. Rain might be expected to be depleted in the heavier isomers, as these have the lowest volatility and would not evaporate from treated fields as readily as the lighter isomers. In fact, air samples and laboratory-vaporized toxaphene show an enrichment of the lighter toxaphene components. Once in the atmosphere, the higher molecular weight components are preferentially adsorbed to particles and selectively transported downward by rain and fallout. Thus the heavies do not evaporate readily and the lights remain in the air as vapors, leaving the middle-weights to accumulate in rain.

# A FIELD INTERCOMPARISON OF PARTICLE AND GAS DRY DEPOSITION

## MEASUREMENT AND MONITORING METHODS

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From 3 through 30 June 1982 concurrent dry deposition measurements were performed using a variety of currently available techniques. Researchers from twelve U.S. and Canadian institutions gathered at an 800 m by 400 m grass-covered sampling site in rural Champaign County, Illinois. Methods employed to measure dry deposition included micrometeorological techniques such as eddy correlation, eddy accumulation, particle concentration variance, and concentration profiles/modified Bowen ratio computations. Deposition flux collection methods, utilizing surrogate surfaces such as polyethylene buckets, Teflon plates, and polyethylene funnels, filtration methods, and vegetation washing procedures were also used. The principal species for which dry deposition was measured were particles, particulate sulfate, sulfur, and nitrate, and gaseous sulfur dioxide, nitric acid, and ozone. Each deposition measurement method was executed as normally done in the field, except that an effort was made to synchronize sampling activity and standardize heights of measurement, where possible, in order to maximize intercomparability of the results. An interlaboratory cross analysis of shared aliquots were performed to reduce uncertainties due to chemical analyses. Preliminary results from a few of the experiments suggest sulfate deposition velocities on the order of a few tenths of a centimeter per second, and a possibly significant contribution of large (diameter > 2.0 microns) particle associated sulfate to the total flux. A mean deposition velocity of about 3.0 cm/s was found for nitric acid vapor, and the estimated nitric acid vapor flux to the grass field was 1.4 kg per hectare per month. A comprehensive ambient conditions data set, consisting of meteorological and atmospheric chemistry information, was compiled throughout the study period. These data provided an additional basis for evaluation and comparison of the deposition results.

A COMPARISON OF AMBIENT AIRBORNE SULFATE CONCENTRATIONS  
DETERMINED BY SEVERAL DIFFERENT FILTRATION TECHNIQUES

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Ambient airborne concentrations of sulfate, total sulfur, and sulfur dioxide were monitored at a rural Champaign County, Illinois site from 3 through 30 June 1982. Several different filtration techniques were used concurrently. 1.0  $\mu\text{m}$  Zefluor PTFE and 0.8  $\mu\text{m}$  Nuclepore filters in open-face holders were exposed on a 30-minutes-on, 30-minutes-off schedule with daily filter changes at 0700 CDT. Flow rates were about 35 l/min. Two-stage filter packs consisting of a 1.0  $\mu\text{m}$  Zefluor PTFE prefilter and a potassium carbonate/glycerol impregnated Whatman 41 second stage were exposed at about 30 l/min and also changed daily at 0700 CDT. Three-stage filter packs consisting of a 2.0  $\mu\text{m}$  Zefluor PTFE prefilter, 1.0  $\mu\text{m}$  Nylasorb second stage, and similarly treated Whatman 41 third stage were exposed at 25 l/min and changed twice daily, at 0700 and 1900 CDT. The three stage packs and their analyses were provided by the Ontario Ministry of the Environment. Two Beckman dichotomous samplers with 10  $\mu\text{m}$  inlets, using type 504 Teflon filters, were operated throughout the period. One of the samplers changed filters daily at 0700, while the other changed twice daily at 0700 and 1900. All of these filters sampled air at 1.5 m above ground. Two streaker samplers with 0.4  $\mu\text{m}$  Nuclepore filter rings were operated at 0.8 l/min continuously during the month. The streakers were either colocated at 1.5 m above ground, or one sampler was raised to a height of 6.0 m. The variety of extraction and analytical methods applied to the various filter samples will be presented, together with a comparison of the resultant airborne concentration data. These observations were performed in support of a dry deposition measurement methods intercomparison, and were therefore used to define a single set of concentrations covering the entire month. The computations used to synthesize that data set will also be described.

# COMPARISON OF SURROGATE SURFACE TECHNIQUES FOR ESTIMATION OF SULFATE DRY DEPOSITION

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Natural vegetation is thought to be effective in removing atmospheric pollutants through dry deposition as well as wet deposition processes. However, difficulties related to the leaching of internal plant sulfate have limited the accurate assessment of the dry deposition component of pollutant removal by natural surfaces. Although a number of researchers have relied upon surrogate deposition surfaces to estimate the flux rate of many materials, little work has been done to intercompare the surfaces. In our study, the dry deposition rates of sulfate particles to artificial surfaces within and above a hardwood forest were measured over an annual range of synoptic weather conditions. Artificial surfaces representing both rough and smooth textural types included deposition buckets, petri dishes, filter paper, Teflon configurations and polycarbonate membranes. Ambient concentrations of sulfate and sulfur dioxide were also monitored.

The dry deposition rates of sulfate to the artificial surfaces were evaluated on the basis of their magnitude and precision. Correlations between techniques and the magnitude of the flux rates were used to identify technique similarities. For diverse reasons, many of the techniques were found to have limited reliability. The petri dish and filter plate surfaces were found to represent the best devices for the estimation of dry deposition to smooth and rough artificial surfaces, respectively. Seasonal averages for these samplers were 12.8 to 71.2  $\mu\text{g SO}_4^{2-}/\text{m}^2/\text{hr}$ , respectively. USEPA high volume samplers and Huey sulfation plates provided the best sampling accuracy and precision for the measurement of ambient concentrations of sulfate and sulfur dioxide, respectively. Ambient concentrations of the sulfur oxides and the deposition rates were not well correlated.

The wide range of surface deposition rates estimated from the variety of deposition surfaces emphasizes the uncertainty of the individual measurement techniques as well as the dependency of sulfate dry deposition on surface characteristics. In spite of these limitations, the use of surrogate surfaces provides at least an approximate estimate of sulfate flux rates not currently obtainable from natural surfaces. A critical research need is studies emphasizing surface deposition on natural vegetation. Such studies should provide a relationship between the deposition to surrogate surfaces in this and other studies to that on natural surfaces.

## DRY DEPOSITION OF SULFATE WITHIN A HARDWOOD FOREST CANOPY

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Vegetation provides a major surface area available for the deposition of atmospheric gaseous and particulate pollutants and may therefore provide an important sink for sulfur oxides. Direct measurements of sulfate dry deposition to natural surfaces have been hindered by technique difficulties and the leaching of foliar sulfate. We estimated deposition rates and dry deposition velocities to a hardwood forest through the use of petri dish and filter plate surrogate surfaces. The choice of these surrogate surfaces were examined in a previous presentation by the authors.

Data from the surrogate surfaces gave an estimate of the dry flux of sulfate to the forest as 5.0 and 27.9 kg  $\text{SO}_4^{2-}$ /ha/(foliated season from the petri dish and filter plate surfaces, respectively. These values were calculated on the basis of a leaf area index of 5.5. Seasonal differences in dry deposition were also observed. Deposition rates decreased through the canopy during foliated periods, while they were similar at all canopy levels during non-foliated periods. Ambient sulfur dioxide profiles, estimated from Huey sulfation plate measurements, also indicated the effectiveness of the forest canopy in removing this gaseous pollutant.

Analysis of variance demonstrated that the data variability associated with vertical position and sampling data factors was highly significant to the deposition rate and similar in magnitude. Thus measurements of dry deposition must carefully characterize the location in time and space from which averages are calculated.



## COLLECTION AND MEASUREMENT OF THE CHEMISTRY OF DEW

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Previous materials corrosion and dry deposition studies have suggested that the presence of dew and other forms of surface wetness may play a significant role in the removal of contaminants from the atmosphere and the activation of previously dry deposited aerosols. MITRE, under its Independent Research and Development program, initiated field experiments in the summer of 1981 at Sterling, Virginia and Champaign, Illinois to investigate the interaction of dew and dry deposition more specifically. The objectives of the study were:

1. to develop and construct an appropriate collection device satisfying various functional requirements such as chemical inertness, and the ability to collect dew in amounts sufficient for analysis
2. to collect and analyze samples of dew representing various surface exposure times and investigate influences on the various chemistry of dew, including length of collector exposure time, air flow changes, site location, and atmospheric particulate concentrations.

The greatest hinderance to the study of surface wetness chemistry has been the small sample volumes (typical dew volumes amount to about  $0.01 \text{ ml cm}^{-2}$ ). To overcome this problem, the collectors were designed to have a large surface area ( $0.6 \text{ m}^2$ ) which would yield about 50 ml per dewfall. They were constructed of Teflon film bonded to aluminum sheet metal and supported horizontally by a frame one meter from the ground.

The collector plates were exposed from one to several days and allowed to accumulate varying amounts of dry deposition. In the morning after a significant dewfall, some or all of the plates would be sampled. Sampling involved inclining the surfaces  $45^\circ$  and scaping the dew droplets into a Teflon-lined trough which drained into a sample bottle. Dew damples were then analyzed for dissolved species using ion chromatography and atomic absorption spectroscopy.

Analysis of the data obtained in both experiments lead to the following findings:

- Dew deposition to the collectors averaged about 0.10 mm at Sterling and about 0.16 mm at Champaign. This amounted to an average of about 58 to 93 ml of dew per collector and was more than enough for chemical analyses.
- Dew amounts collected on different, but identically exposed collectors generally agreed to within five percent. Mass loadings calculated from concentration data, generally agreed within 20 percent for the major ions.
- The predominate anions found in dew at both sites were sulfate, nitrate, and chloride, in decreasing order of abundance.
- Predominate cationic species varied between the sites and could be related to differences in local particulate sources.
- Hydrogen ion as, generally, a minor constituent in most of the dew samples collected. The acidity associated with sulfate and nitrate compounds was found to be essentially neutralized by alkaline soil and/or dust particles. Mean pH values at Sterling and Champaign were 6.3 and 6.2, respectively.
- Indirect evidence seems to suggest that certain ions have higher loading rates to the collector surface in the presence of dew relative to dry conditions.
- Changes in air flow patterns had a significant effect on dew chemistry characteristics.

SESSION VI  
ORGANIC POLLUTANTS

Hanwant Singh  
Session Chairman

EVALUATION OF SOLID SORBENTS FOR  
COLLECTION OF VOLATILE ORGANICS IN AMBIENT AIR

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The problem of sampling atmospheric organic vapors by adsorption at parts per billion concentrations imposes several special requirements on the sorbent to be used. Tenax GC resin has been widely employed because of its thermal stability, its high capacity, and rapid kinetics for adsorption and desorption of the vapor. Nevertheless, some field problems have arisen implying difficulties with complete desorption of some vapors, and candidate replacement resins have been suggested. This is a progress report on the demonstration of a protocol suitable for qualifying such resins, and for prediction of performance for adsorption of new vapors that have not yet been tested.

The procedure is based on the equilibrium adsorption model of Dubinin-Radushkevich and the adsorption kinetics model of Wheeler and Robell. The method has had some success in correlating and predicting the relative adsorbability of various organic species on charcoal and has been used to describe the adsorption of hazardous vapors at low concentrations in air.

The method of procedure is a simple one:

1. A reference resin (Tenax GC) is characterized using a few vapors that also will serve as references in later work. The characterization involves adsorption of the vapor on resin columns of different lengths (weights) so that a correlation of breakthrough time and bed weight can be produced for a specified breakthrough concentration.
2. Following each adsorption trial, the vapor is desorbed by a programmed temperature rise, and the completeness of desorption is tested following various periods of storage for the sorbed vapor.
3. The full adsorption capacity of the reference resin is tested by several experiments in which the resin bed weight is held constant and vapor concentration is varied.

The very low vapor concentrations being employed, i.e., parts per billion, extend the application of the model to very low sorbent loadings and also tend to emphasize calibration and reproducibility uncertainties arising from the high-gain detector sensitivities that must be employed. From the plots of breakthrough time-versus-sorbent weight, the sorbent capacity and sorption kinetics are obtained.

When adsorption is completed, a programmed temperature rise is used to desorb the vapor. A complete and facile desorption of the vapor almost certainly implies that the vapor adsorption is limited to physical processes and the prediction procedure for the adsorption of a new vapor is based on this fact. Once a few reference vapors have been tried, the adsorption of the others may be predicted.

For the study of vapor sorption by Tenax GC and Chromosorb 101, resin bed depths of 1.0, 3.0, 5.0, and 6.5 inches were tried. The 1.0-inch bed depth provided to be too close to the critical value, for which breakthrough begins immediately, and so was abandoned. For the remaining three bed depths, initial benzene vapor concentrations of 120 and 580 ppb were employed and the breakthrough times were measured at  $C_0/C_b$  equal to 20, 10, 5, and 2, corresponding to 5, 10, 20, and 50 percent vapor concentration breakthrough. The multiplicity of inlet and breakthrough concentrations permitted several analyses of the adsorption kinetics for improved precision of the kinetic constants. Benzene and 1,2-dichloroethane vapors showed complete desorption, and no abnormalities were experienced either during adsorption or desorption of the vapors from the resin. For acetone on Tenax GC, the desorption peaks averaged only 78.0 percent of the size of the adsorption peaks, and extraneous high molecular weight species appeared in the desorption curve. It is believed that these artifact peaks represent species desorbed from the resin by acetone and in proportion to the amount of acetone taken up.

The results indicate that a detailed evaluation of a candidate sorbent can be obtained through the method investigated in this program.

AN AUTOMATED INTEGRATED CAPILLARY-PACKED CHROMATOGRAPHIC  
SYSTEM FOR MOBILE AMBIENT AIR MONITORING OF VOLATILE ORGANICS

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As a result of growing public concern regarding hazardous chemicals present in ambient air, a need has developed for a mobile air monitoring system capable of measuring low and sub PPB levels of organic compounds. Ecological Analysts has configured such a system consisting of two gas chromatographs with flame ionization (FID) and halide specific (HSD) detectors, a microcomputer controller, two dual trap inletting systems, a calibration gas diluting/blending system and data storage/presentation equipment including two tape drives, two printer/plotters and one printer. The system is highly flexible and can be reconfigured to meet changing priorities.

The system at present collects ambient air on two separate multicomponent trapping systems (one for packed and one for capillary column chromatography), desorbs the sample, and simultaneously monitors column effluents with both detectors. Through a BASIC computer program FID/HSD response ratios can be calculated. The program also compares results of dissimilar columns for confirmation. The entire system will automatically calibrate itself at predetermined intervals and has multiple level calibration capabilities. The calibration standard contains most of the volatile priority pollutants, several ketones, and some commonly found non-halogenated hydrocarbons. The combination of capillary and two dimensional packed column chromatography provides good resolution of all compounds in the calibration standard. The system can be operated on line power unattended for seven days. Automated operation on generators is limited to gasoline supply.

Other features of the system allow on-site purge and trap analysis, the analysis of sorbant tubes collected remotely, and direct thermal desorption of soil or other solid samples.

Topics to be discussed will include packed column vs. capillary column chromatography for sorbant tube analysis, thermal desorption of sorbant tubes, and problems encountered during development of this system.

REAL TIME (ppb) POLLUTION MONITORING WITH A LOW COST  
PORTABLE MASS SPECTROMETER

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Introduction

PETRA, Personnel and Environmental Trace gas Analyzer, is a mobile or fixed-station pollution monitor. It can identify, detect and measure air or breath-borne pollutants down to sub ppb levels in a typical analysis time of 1 second.

These features allow the system to be used in applications for monitoring factory work space areas, chemical leak detection, non-invasive screening of process workers for critical body pollution levels, effluent monitoring, and contamination of drinking water by chemical dumping.

The system is transportable, has low power consumption, and can therefore be used in the field with very few requirements. Data storage is available so that both recorded as well as real time data can be obtained. In the fixed-station mode the levels of each pollutant monitored can be recorded as hard copy for giving the time-weighted averages (TWA), maximum values, mean values and standard deviations on each of the sample lines being monitored. A multipoint sampling system is available.

The Personnel breath analyzer can record the levels of each pollutant on the breath of an exposed subject. Correlation with blood-borne levels is such that tedious, time consuming blood and urine tests may be replaced.

System Description

The instrument is based around a high sensitivity quadrupole gas analyzer with an enrichment membrane inlet device which gives a factor of 500 times sensitivity over the atmosphere gases such as O<sub>2</sub> and N<sub>2</sub>. The mass analyzer is a triple filter assembly and is made so as to give high abundant sensitivity and freedom from contamination. The abundance sensitivity is particularly important when compounds such as HCN and methanol are being monitored because the peaks for these compounds are adjacent to nitrogen and oxygen, the main constituents of air.

The temperature of the whole system including automatic calibration loop is critical and is elevated to increase transmission through the membrane, and retain the compounds being monitored in a volatile phase resulting in low memory effects and fast responses.

The vacuum system required for the operation of the mass analyzer is an ion pump and has the qualities of being oil free and of low power consumption, aiding portability.

The mass spectrometer control and data acquisition system is provided by an integral microcomputer, VDU and floppy disk, and is designed to provide direct real time data.

Calibration of the system is easily carried out by using a standard and takes approximately 60 seconds to complete.

A sample can be taken from many systems including a 500 ft. sample probe, head space sampler, thermal desorber, sparging system, gas chromatograph and in some cases an HPLC system.

### System Performance

The detection level for most halogenated hydrocarbons, alkanes, alkenes and aromatics is in the 1-10 ppb range. Some compounds are worth specifically mentioning, viz., dimethylsulphate at 10 ppb, CS<sub>2</sub> at 0.7 ppb and pcb's at 0.5 ppb.

These compounds can be monitored in a speed of 250 milliseconds and can therefore follow the breath profile in breath-by-breath analysis. All compounds measured to date have a linear response with concentration which is typically +4% and over 5 decades from 1 ppb upwards. The reproducibility is better than +2% for compounds measured in the 100 ppb range and the heated Inlet reduces memory to less than 10 ppb for a 200 ppm injection in less than 1 minute.

### Applications

As well as the direct monitoring of the atmosphere in both portable or fixed station modes Petra can be used for medical breath analysis, measurement of toxic gases in simulated fires, toxicology inhalation tank monitoring, chemical leak detection, and evaluation of protective materials used in handling toxic chemicals.

In the fixed-station system up to 96 lines with a 500 ft. radius can be used with an analysis time of around 35 s/line. Total control of the sampling system and the associated medical



reporting is carried out by the microcomputer. Time weighted averages, maximum and mean values as well as trend analysis can be obtained easily as a hard copy printout. A programmable alarm level is also available.

## GC/FI-IR AND GC/MS: COMBAT OR CONCERT?

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Until very recently, publications and presentations describing GC/FT-IR techniques began or ended with a defense of the technique in comparison to GC/MS techniques. The unfortunate inference of competition was promulgated in the early development stages of GC/IR techniques, and remains implicit in much ongoing research.

Latest developments in hardware and software, particularly FT-IR interfaces for capillary column chromatography, permit reliable evaluation of FT-IR and MS data obtained on the same mixtures with similar, if not identical, chromatographic separation. In this paper, we will examine data on real-world samples obtained separately by GC/FT-IR and by GC/MS techniques. Rather than contrasting or comparing the data, we will instead elaborate the interplay between sensitivity, selectivity, and information content of the data; how these impact on analytical requirements and constraints; and guidelines in choosing the most effective measurement scheme.

DETECTION OF ENVIRONMENTAL POLLUTANTS  
USING PIEZOELECTRIC CRYSTAL SENSORS

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In recent years, coated piezoelectric crystal sensors have become of increasing interest for detection of trace amount of pollutants from ambient air. Not only are they highly sensitive detectors, but they are also simple, inexpensive, low power consumption, light-weight and portable devices.

The principle of the detector is that the frequency of vibration of an oscillating crystal is decreased by the adsorption of a gaseous sample onto its coated surface. The decrease in frequency is a measure of the amount of gas adsorbed. This linear relationship between frequency change and added mass enables a piezoelectric crystal to be used as a sorption analytical detector with a detection limit of about  $10^{-10}$ g. The selectivity of detector can be achieved by coating the crystal with a substance which selectively adsorbs the pollutant one want to detect.

In this presentation, we report new methods and coatings for the specific detection of mercury, organophosphorus pesticides, and formaldehyde. The effect of flow rate, amount of coating, cell configuration, and practical instrument suitable for use as personal monitor will be presented. The use of immobilized enzymes as coatings for sensitive and specific detection of air pollutants will also be described.

A MINIATURE GAS CHROMATOGRAPH  
UTILIZED IN A PORTABLE GAS ANALYSIS SYSTEM

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A new technology based on "micromachining" of silicon wafers like those used to make microelectronic chips has been applied to the miniaturization of a gas chromatograph. A three-inch wafer forms the heart of a complete G.C. system that will fit into the palm of the hand. Gas channels are formed by etching grooves into the silicon and bonding the wafer to a glass plate. Square holes etched completely through the wafer allow the gas to flow from the system components etched into the front of the wafer to those mounted on the back. Two valve seats are etched into the back of the wafer. The sample to be analyzed is drawn into one of the gas channels where it is compressed by a tiny piston. The pressure in the channel is monitored by a silicon sensor mounted on the wafer. On command the pressurized sample is injected onto both a 100 micron I.D. fused silica open tubular column which is interfaced to the wafer and a reference channel on the wafer. Both the separation and reference columns lead to a gas channel on the back side of the wafer, over which is mounted a thermal conductivity detector fabricated on a silicon chip. The complete G.C. including carrier gas supply solenoid actuators and analog electronics board measures only 2x4x4-1/2 inches. It uses very little carrier gas, requires only nanoliters of injected sample and performs most analyses in less than 30 seconds. Five of these G. C modules have been combined with a powerful microcomputer in a fully self-contained, portable gas analyzer.

The internal microcomputer has been pre-programmed with information about each gas in the instrument's internal library. When a particular gas is selected, the microcomputer activates one or more appropriate G. C. modules within the instrument. As with other G.C.'s, gases are identified qualitatively on the basis of the expected retention time for a gas peak generated by the detector signal. Since the temperature of each column is not controlled, the expected retention time will be a variable with temperature. However, each G.C. module incorporates a temperature sensor which enables the microcomputer to monitor the exact temperature of the column at the start of each analysis. Two parameters for each gas selected from the

internal library are stored in the microcomputer's memory to enable it to predict retention time over the entire operating temperature range. Using this information and a reference peak time from the previous analysis, the microcomputer opens up a retention time window to look for a peak in the detector signal. If a peak is found within that window that satisfies certain peak shape and width criteria stored in the microcomputer's memory, the peak is then identified as the gas selected and the peak is then further analyzed by the microcomputer's quantitation software programs to calculate the concentration of the selected gas.

To allow quantitation over a wide dynamic range of concentrations three analog detector signals are generated at low, medium, and high amplification. Within the retention time windows for the gases selected, the microcomputer monitors all three signals, converting them to digital form (raw data) so that, depending on the peak size, any of the three signals may be used to calculate the concentration of the selected gas.

After all of the data has been digitized by the microcomputer, a unique peak filtering software program analyzes the data to determine the beginning and end of the peak, the apex of the peak, and the total areas under the peak. Then a baseline is automatically computed by the microcomputer and the actual peak area above the baseline is then calculated. Final quantitation is performed by relating the peak area to both a response factor for the particular selected gas (based on its thermal conductivity) as well as to the area of the reference peak. The reference peak is generated by allowing a small amount of the injected gas sample to bypass the column through a tiny channel in the silicon wafer assembly going directly to the detector. The area of this reference peak is directly proportional to the amount of gas sample injected and thus allows compensation for slight variations in the volume of gas sample injected. The quantitative results of the analysis are then available for display or storage, either in ppm or % concentration. When operating in the automatic mode, statistical software programs are automatically activated which continuously update the minimum and maximum concentrations of each gas selected, as well as mean concentrations, time weighted averages, and standard deviations. A memory board stores the results of at least 1000 analyses for later access.

As with any instrument, there is, of course, the possibility that a gas peak other than the one selected, may appear near enough to the peak of the gas selected to cause an interference. The microcomputer in the instrument utilizes another software program to minimize the effects of interferences by employing a method we call "correlation chromatography." The instrument can include a G.C. module with a nonpolar capillary

column as well as a G.C. module with a polar capillary column. Many organic compounds can be separated on both of these columns, a significant number of them at quite different relative retention times. If one of these gases is selected for analysis, the microcomputer opens up an appropriate retention time window on each module to look for a peak. Only if the microcomputer finds a peak in both windows will it positively identify the gas as detected. Additionally, if one of the peaks is much larger than the other, the concentration will be calculated using the smaller peak.

There are a number of outputs at the back of the instrument. An analog output allows the monitoring of the detector signal from any of the G.C. modules. A printer output is compatible with any serial printer. A two-way RS232C compatible interface allows results to be sent to or the instrument to be controlled by an external computer. Finally an external alarm output provides a contact closure which can be activated by a user selectable upper and/or lower alarm level for any gas selected for analysis.

In summary, the Michromonitor Gas Analyzer is a unique portable, easy to use instrument which allows high resolution, high speed G.C. analyses to be done in almost any field situation where toxic gases may be present.

## TALMS BENZENE MONITOR

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TALMS (Tunable Atomic Line Molecular Spectroscopy) is a high resolution, ultraviolet visible, molecular differential absorption technique for the detection of volatile organic compounds. It exploits the rotational-vibrational fine structure in the electronic absorption bands of organic compounds. A monitor for benzene has been designed and constructed at Lawrence Berkeley Laboratory and is being tested at the Environmental Monitoring Systems Laboratory. It operates with a mercury line at 253.7 nm, is ca. 1 m x .3m x .3m in dimensions, and weighs ca. 75 lbs. It can be calibrated with gas phase injections of benzene. A cryogenic concentrator has been designed for use with the monitor in ambient air. The operating characteristics of the system including detection limit, precision and interferences will be described.

REDUCED TEMPERATURE PRECONCENTRATION OF VOLATILE ORGANICS  
FOR GAS CHROMATOGRAPHIC ANALYSIS: SYSTEM AUTOMATION

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An automated system for unattended, repetitive sampling/analysis of volatile organics in ambient air has been designed and evaluated. The sampling/analysis scheme involves reduced temperature preconcentration with subsequent thermal desorption and capillary column, gas chromatographic analysis. System components and operating procedures are described. Temperature versus time profiles measured at the trapping surface document the stability of the trap temperature during sample collection, as well as the rapid trap temperature changes, i.e., 3.5 minutes for +120°C to -150°C during cooling of the trap, and 1.0 minutes for -150°C to +100°C during thermal desorption. The system will be evaluated as a semi-realtime monitor for volatile organics and as a central system for analysis of air samples collected in small-volume metal containers.



SAMPLING AND ANALYSIS OF POLYNUCLEAR AROMATIC HYDROCARBONS  
IN AIR USING SOLID ADSORBENTS

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Polynuclear aromatic hydrocarbons (PNAs) of great structural variety can be found in the environment. Several of these compounds as well as their derivatives show carcinogenic or mutagenic properties. PNAs can be introduced into the atmosphere from many sources and are produced by incomplete combustion and pyrolysis of fossil fuels. Depending upon their source, they can be found in rather complicated matrices. Consequently, a number of different analytical techniques have been used in the past for sample characterization. Some of these techniques involve extensive sample handling and can be time consuming. Some tend to reduce detectability and analytical reproducibility and often result in the loss of some components of interest.

PNAs have been collected from environmental air by drawing the sample through an adsorbing resin. Currently there are no adsorbents which are particularly selective for PNAs although common practice has been to use Tenax GC, a porous polymer. A previous study by Lindgren and co-workers at the Texas State Air Control Board indicates the great potential of using C<sub>18</sub>, a liquid chromatographic packing material, as a selective adsorbent for PNAs. There are several liquid and gas chromatographic packing materials which might be useful as selective adsorbents for PNAs. We have studied some of these adsorbents which include: charcoal, Tenax GC, Amberlite XAD-2 (a styrene-divinyl-benzene copolymer), Chromosorb 105, (a crossed linked resin) and Chromosorb LC-9 (n-propyl amine bonded to a silica backbone). The ability of each resin to adsorb and desorb PNAs reproducibly has been evaluated.

The PNAs are collected by drawing an air sample through a multichannel gas manifold which allows the same air sample to pass through up to five different resins simultaneously. Adsorbents are packed in glass tubes which can easily be interchanged and replaced. For analysis the adsorbent is removed from the tube and the PNAs and desorbed by ultrasonication with an appropriate solvent. The resulting solution is then analyzed by GC/MS.

This report will compare the performance of each resin for selective absorption/desorption of PNAs. Some of the materials have not been evaluated previously and some have been previously evaluated independently and these latter materials will serve to correlate our work to other studies, since our samples are collected simultaneously. This procedure has been applied to determine vapor phase PNAs in the air in and around an oil refinery in Texas. Examples of the data obtained will also be discussed.

ANALYSIS OF POLYCHLORINATED BIPHENYLS  
IN AMBIENT AIR SAMPLES

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High resolution gas chromatography with simultaneous analysis on two columns of different polarity with computer assisted data reduction and correlation was applied to the analysis of environmental samples.

A method of identification of individual PCB congenors based on retention indices was developed, as well as quantification of congenors, which are unavailable to be used as standards.

A standard, based on a mixture of commercial Aroclors, was synthesized and all major peaks in the mixture were identified and quantified.

A set of a rules for the reduction of data from two columns of different polarity was established. As well, a programme was written for the microprocessor controlled HP 5880 gas chromatograph. The program controls the gas chromatograph, the HP Autosampler 7672A, initiates the recalibration, performs the data reduction, correlation of the peaks, prints the reports and stores all the GC data and report on a magnetic tape.

Ambient air samples from a survey of PCB's across the Province of Ontario were analyzed using methodologies employing packed columns, single capillary columns, and dual capillary columns of different polarity and the results are reported.

INFLUENCE OF VOLATILITY ON THE COLLECTION OF  
PAH VAPORS WITH POLYURETHANE FOAM

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Polycyclic aromatic hydrocarbons (PAH) have received much attention in studies of air pollution because some of these compounds are highly carcinogenic. They occur frequently in the environment and are produced by inefficient combustion of carbonaceous material. Most investigations of airborne PAH have been confined to particles collected on glass fiber filters. However, recent studies have shown that the 3-4 ring PAH are largely in the vapor phase and therefore not retained by filters.

The purpose of our study was to select a collection system suitable for high volume sampling of PAH vapors in ambient air that can retain compounds of interest without breakthrough. This paper presents a study of PAH vapor penetration through thin sections of polyurethane foam (PUF) to determine the relationships between sample breakthrough, PAH vapor pressure, and total air volume.

The sampling train used for the laboratory study consisted of a PUF prefilter, a mixing chamber for PAH vapors, and a collection column consisting of 15 1-cm thick x 7.6-cm diameter PUF plugs. Air was pulled through the apparatus at 0.5 cu. m/min and sample vapors were continuously bled into the mixing chamber by a slow flow of air through a pasteur pipet containing glass beads coated with about 150 mg of the compounds of interest. Experiments were carried out at 20°C using anthracene, fluorene, phenanthrene, and pyrene. At the termination of the experiment, the 1-cm PUF plugs were individually soxhlet extracted with petroleum ether and the extracts were analyzed by GC. The quantity of PAH on each PUF plug was plotted vs. cm of foam, to give a frontal chromatogram. From these fronts, the thickness of foam corresponding to 50 percent breakthrough was obtained for each compound, and this breakthrough point was

related to total air volume. From the frontal chromatograms the number of theoretical plates (N) in the adsorbent bed could also be determined. Knowing the breakthrough volume and N, the collection efficiency of the PUF bed at different air volumes can be calculated.

METHOD FOR DETERMINATION OF SUB-PART PER  
BILLION CONCENTRATIONS OF PHOSGENE AND  
ACYLCHLORIDES IN AMBIENT AIR

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A simple sensitive analytical method has been developed for the determination of compounds containing active halogen groups, especially phosgene and chlorinated acetylchlorides, in ambient air. These compounds are of interest in ambient air monitoring efforts because of their formation from atmospheric halocarbons. However, conventional gas chromatographic methods are generally unsatisfactory because of irreversible adsorption and/or degradation.

The method reported herein involves capture and reaction of the compounds in an impinger containing 2 percent aniline in toluene. The resulting anilides are determined by high performance liquid chromatography (HPLC) or gas chromatography with electron capture detection (GC/ECD). HPLC/UV determination of 1,3-diphenylurea, resulting from the reaction between phosgene and aniline, resulted in a detection limit of approximately 10 parts per trillion of phosgene in ambient air.

Analytical precision and recovery in clean air were determined using a permeation tube/dilution system. Relevant data are tabulated below:

<u>Number of Analyses</u>	<u>Phosgene Level (ppb)</u>	<u>% Recovery + Standard Deviation</u>
4	0.034	63 $\pm$ 13
4	0.22	87 $\pm$ 14
3	3.0	99 $\pm$ 3
3	4.3	109 $\pm$ 12
3	20.0	99 $\pm$ 14
3	200.0	96 $\pm$ 7

A blank level of 0.04 ppbv was observed for clean air.

Formyl chloride is determined in a similar manner, wherein formanilide is determined by HPLC. Recovery of formanilide spiked into impingers was  $89 \pm 12$  percent at the 100 ng level and  $74 \pm 13$  percent at the 12  $\mu\text{g}$  level.

Apparent levels of phosgene in urban air samples were approximately 0.10 ppbv. Experiments are planned that attempt confirmation of these levels using alternate analytical techniques, since this phosgene level is somewhat larger than expected, based on previous studies.

ADVANTAGES AND OPERATING CHARACTERISTICS OF A  
REFRACTIVELY SCANNED FOURIER TRANSFORM INFRARED  
SPECTROMETER BASED AMBIENT AIR MONITORING SYSTEM

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The detection and measurement of hazardous gases and vapors via mid-infrared vibration spectroscopy is an attractive method for the industrial hygienist. The high degree of specificity and the ability to gather quantitative information make it well suited for ambient air analyses. Past instrumentation has been developed around non-dispersive single component monitoring, such as Golay/PAS type cells, and dispersive instrumentation utilizing gratings and/or variable interference filters. Each of these measurement techniques has its limitations, which include lack of sensitivity, stability and specificity.

The design of an ambient air monitoring system around a refractively scanned Fourier Transform Infrared (FTIR) Spectrometer overcomes these problems to various degrees.

The advantages of FTIR spectroscopy over dispersive spectroscopy are quite well known. These include multiplexing and throughput advantages, speed, accuracy and ability to utilize ultra-high sensitivity infrared detectors.

The integral digital computer of the spectrometer is available for data reduction, tabulation, reporting and archival storage of the monitoring system's data.

In this paper we will describe the Analect EVM-60 ambient air monitoring system. This system is designed around the Transsept<sup>R</sup> refractively scanned FTIR spectrometer. Topics will include discussions of the system including sensitivity, accuracy, and environmental stability of such an optical design.

We will also describe the unique data and control architecture of the multiprocessor data system. As an example, the use of a hardware Fast Fourier Transform processor to calculate the spectral information in real time, will be reviewed.



Particular attention will be given to the systems performance and operation software. This software includes routines for analytical method development for the analysis of up to 10 components and storage of multiple user defined methods for future recall. We will also give descriptions of the system's sampling control of up to 20 independent stations, report generation and archival storage of tabulated results at pre-determined intervals. These reports include TWA, raw data results per sampling station and job description.

A COST-EFFECTIVE PROCEDURE TO SCREEN AIR SAMPLES  
FOR POLYAROMATIC POLLUTANTS

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Analytical techniques such as high-performance liquid chromatography (HPLC) and gas chromatography/mass spectrometry (GC/MS) are often employed to analyze complex environmental samples. Although these techniques have demonstrated their usefulness in providing a detailed characterization of polynuclear aromatic (PNA) pollutants in many atmospheric samples, it is generally not cost-effective to apply these methods on a routine and systematic basis to all samples. It is often desirable to have a screening procedure to prioritize these samples for further detailed characterization. This paper reports on the development and field study of a cost-effective procedure based on simple luminescence analyses for screening PNA pollutants in atmospheric samples. The analytical techniques are synchronous luminescence (SL) and room temperature phosphorescence (RTP). The field samples consisted of air particulate samples collected on high-volume filters at various residential locations of two wood-burning communities. After collection, the cyclohexane extracts of the samples were directly analyzed by SL and RTP without any fractionation procedures. The intensities and spectral profiles provided a spectral index for ranking. The procedure and results of the ranking protocol will be discussed in detail. The results of the ranking procedure will be correlated with data obtained

by independent HPLC and GC/MS analyses. The results of this field study demonstrate that a simple and cost-effective screening procedure can be used to obtain PNA spectral profiles as a basis to rank air samples according to their PNA content and/or to determine whether these samples have similar PNA compositions. Use of this screening potential will undoubtedly reduce the total cost of human exposure assessments by reducing the number of unnecessary analyses by more sophisticated and more expensive techniques.

ANALYSIS OF POLYCYCLIC AROMATIC HYDROCARBONS  
IN AMBIENT AIR AND RAIN

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Polycyclic aromatic hydrocarbons (PAH) are emitted by many natural and anthropogenic sources, including forest fires, tobacco smoke, auto exhaust, and emissions from power plants. A number of these compounds are recognized carcinogens, especially benzo(a)pyrene, benzo(c)phenanthrene, the benzo-fluoranthenes, and the dibenzanthracenes. Recently a major concern of those in the health and environmental fields has been the potential contamination of our environment due to "synfuels" development and proposed relaxation of the Clean Air and Water Acts. Since emissions of PAH are usually directly into the atmosphere, it is important to monitor their levels in ambient air. To this end, we have developed sampling techniques to isolate particle and vapor-phase PAH from large air volumes, and have established the ability of polyurethane foam (PUF) to quantitatively collect PAH vapors.

A hi-vol sampler with two 7.6 cm diameter x 7.6 cm thick PUF plugs placed behind a 20 x 25 cm glass fiber filter was used for the collection of all ambient air samples. Analysis of the filter and each plug was performed separately. Total hydrocarbons were analyzed by GC-FID and individual PAH were determined using reversed phase HPLC with fluorescence detection. Selected peaks were trapped from the LC and scanned on a spectrofluorimeter for positive identification.

A comparison of total hydrocarbons and PAH collected by hi-vol air sampling in the city of Columbia and at a rural site (Savannah River Plant) are shown in Table 1. The 3-4 ring PAH were found predominately in the vapor phase, while the heavier ring PAH were particulate.

Of equal importance is the need to understand the mechanisms by which these carcinogens are inevitably returned to earth to contaminate soils, lakes and the oceans. As an extension of our ambient air analyses, we have recently begun an evaluation of PAH levels in rain at Columbia and will report preliminary results.

Table 1

Average total hydrocarbons and PAH in Columbia, SC  
and at SRP, ng/m<sup>3</sup>

	<u>Columbia</u>	<u>SRP</u>
Non-polar total hydrocarbons >C <sub>19</sub>	641	302
Polar total hydrocarbons >C <sub>19</sub>	310	141
Phenanthrene	37.1	11.3
Anthracene	1.2	0.14
Fluoranthene	6.8	1.6
Pyrene	11.7	2.9
Benzo(a)pyrene	0.54	≤0.07
Benzo(ghi)perylene	1.3	≤0.15
Benzo(k)fluoranthene	0.08	≤0.01
Coronene	0.57	N.D.

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N.D. = none detected

SESSION VII  
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