

# PROCESS MEASUREMENTS REVIEW

INDUSTRIAL  
ENVIRONMENTAL  
RESEARCH  
LABORATORY



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## DEVELOPMENT OF A MASTER ANALYTICAL SCHEME FOR VOLATILE ORGANICS

The development of a master scheme for the analysis of organics in water was discussed during a January 26, 1979, workshop held in Atlanta, Georgia. Approximately 75 people attended the workshop, representing analytical personnel from government agencies, private industry, academic institutions, and research organizations.

The first generation of the analysis scheme is intended to be applicable to volatile organics in industrial, energy, or municipal effluents and in surface or drinking waters. Volatile organics, for the purposes of this scheme, include any that will elute from a gas chromatographic column at 300° C in 1 hour or less or any that may be derivatized to do so. Lower detection limits for the scheme have been set at 0.1 µg/L for drinking water, 1 µg/L for surface water, and 10 µg/L for effluents. Users of the scheme will be given guidance concerning sampling, addition of internal standards, handling, preservation, extraction, concentration, and cleanup of the sample. Detection and quantification will be by gas chromatography-mass spectrometry.

The project originated at EPA's Environmental Research Laboratory in Athens, Georgia. Work on the scheme is being carried out under EPA contract 68-02-2704 with Research Triangle Institute (RTI). A. W. Garrison is EPA's Project Officer; Edo Pellizzari of RTI is the Principal Investigator. The project is scheduled for completion in the spring of 1980.

## SYMPOSIUM ON ADVANCES IN THE SAMPLING AND MEASUREMENT OF PARTICULATE MATTER

During the past few years, through the efforts of both industry and government, advancements have been made in the technology of sampling and sizing of particulate matter in various industrial and ambient environments. In order to gather together the experts in these areas, EPA's Industrial Environmental Research Laboratory-RTP is sponsoring its second symposium on measurement of particulate matter. Speakers will discuss the status of current research and development in the areas of:

- Inertial, optical, diffusional, and electrical particle sizing
- Control device evaluation
- Aerosol characterization
- Quality assurance
- Data reduction techniques
- Real-time monitoring of mass concentrations and size distribution
- Inhalable particulate matter.

The number of speakers will be limited so that symposium attendees will have ample time to ask questions and to discuss their own experiences in these fields. The symposium will be held October 7-10, 1979, at the Daytona Hilton, Daytona Beach, Florida. Coordinator of the symposium is Wallace B. Smith, Southern Research Institute, 2000 Ninth Avenue, South, Birmingham, Alabama 35205.

The views expressed in the *Process Measurements Review* do not necessarily reflect the views and policies of the Environmental Protection Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by EPA.

# SULFURIC ACID MEASUREMENT METHODOLOGY

The present interest in sulfur oxide emissions from flue gas desulfurization (FGD) units has stimulated increased sampling and analysis for sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and particulate sulfate. Though unsubstantiated at this time, there is reason to believe that FGD units emit sulfate aerosols while removing  $\text{SO}_2$ . Part of the problem in confirming this theory is the lack of proven methodology to quantitatively separate and efficiently collect the various sulfur oxides emitted by stationary sources. EPA's Industrial Environmental Research Laboratory at Research Triangle Park (IERL-RTP) has established several programs to define the present state of the art and to develop new methods for sulfate and sulfuric acid sampling and analysis. This article summarizes some of the highlights of that research and provides background information on the sampling problems associated with  $\text{H}_2\text{SO}_4$ /sulfate aerosols.

## $\text{H}_2\text{SO}_4$ /SULFATES AS A HEALTH HAZARD

The toxicity of  $\text{H}_2\text{SO}_4$  in gross quantities is well known, but until recently the effect of  $\text{H}_2\text{SO}_4$  aerosol had not been investigated. In recent tests, laboratory animals were exposed to various amounts of  $\text{H}_2\text{SO}_4$  aerosol. Results were narrowed air passages and mucosal swelling or increased secretion. Consequently, people who already have trouble breathing (the aged, asthmatics, or heart patients) can be affected not only by the directly corrosive qualities of  $\text{H}_2\text{SO}_4$ , but also by the respiratory strain that the aerosol places on them. As the particle size was decreased, an increase in the flow resistance to respiration was noted in the animals. Other sulfates produced similar results. A comparison of zinc ammonium sulfate, zinc sulfate, and ammonium sulfate showed zinc ammonium sulfate to be the worst irritant. When zinc ammonium sulfate and  $\text{H}_2\text{SO}_4$  aerosol of equal particle size were compared,  $\text{H}_2\text{SO}_4$  was the greater irritant.

## $\text{H}_2\text{SO}_4$ /SULFATE SAMPLING PROBLEMS

Experience has shown that the  $\text{H}_2\text{SO}_4$ /sulfate content of typical combustion gas from oil- or coal-

fired sources ranges from 1 to 5 percent of the total sulfur burned. The bulk of the sulfur ends up as  $\text{SO}_2$ . Any  $\text{H}_2\text{SO}_4$ /sulfate method must prevent small interferences from  $\text{SO}_2$  to avoid a large positive error in the  $\text{H}_2\text{SO}_4$ /sulfate quantification.

A problem specific to  $\text{H}_2\text{SO}_4$  sampling is the difficulty in separating  $\text{H}_2\text{SO}_4$  vapor (or aerosol) from alkaline particulate matter. TRW research results, reported at the 70th annual APCA meeting, indicate that the current EPA Federal Reference Method 5 recommended filter temperature of  $121^\circ\text{C}$  is too low to prevent collection of  $\text{H}_2\text{SO}_4$  on the filter. When this happens, differentiation of  $\text{H}_2\text{SO}_4$  and particulate sulfate is impossible. In fact, temperature control at all locations along the train is important. Formation of  $\text{H}_2\text{SO}_4$  aerosols from the vapor phase is highly probable due to the nucleation sites provided by the fine particles present in the flue gas. Surfaces that these aerosols might contact must be hot enough to revaporize the  $\text{H}_2\text{SO}_4$ . TRW's investigation of this problem has shown that gas temperatures above  $250^\circ\text{C}$  are required to prevent  $\text{H}_2\text{SO}_4$  fallout in sampling trains.

## $\text{H}_2\text{SO}_4$ SAMPLING METHODOLOGY

The best approach to  $\text{H}_2\text{SO}_4$  sampling methodology would be to design a comprehensive sampling system so that  $\text{SO}_2$ ,  $\text{H}_2\text{SO}_4$ , and particulate sulfate ( $\text{SO}_4^{2-}$ ) are separated. Currently available methodology for sampling particulate sulfate is adequate provided it is not important if small amounts of  $\text{H}_2\text{SO}_4$  are included in the final value. Numerous attempts have been made to design systems capable of separating  $\text{H}_2\text{SO}_4$  from sulfur dioxide. Almost all the techniques fall into one of two types of sampling strategies—selective absorption or controlled condensation.

Selective absorption uses 80 percent 2-propanol (isopropyl alcohol) to collect the sulfur trioxide ( $\text{SO}_3$ ) and to pass the  $\text{SO}_2$ . The  $\text{SO}_2$  is collected in 3 percent hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). This method has been used extensively and is the basis of EPA's compliance test (*Federal Regulations*, 41, 111, 1976) for acid mist from sulfuric acid plants. The major problem with utilizing the EPA procedure for other types of sources is the lack of a prefilter to effectively prevent particulate matter from reaching the alcohol impinger. The particulate matter in the impinger can act either as a direct interferent by contributing  $\text{SO}_4^{2-}$  or as an indirect interferent by promoting the

oxidation of  $\text{SO}_2$  in the liquid phase through action of trace elements such as iron, copper, or vanadium. The method also assumes that all  $\text{SO}_2$  can be purged from the alcohol impinger. Recently, oxidizing agents have been found in reagent grade 2-propanol. This finding partially explains the high values normally found when selective 2-propanol absorption methods are used. Because of the particle separation and  $\text{SO}_2$  purging problem, this procedure is not recommended for coal or oil combustion sources.

The controlled condensation approach for  $\text{H}_2\text{SO}_4$  was first proposed by B. P. Knol in a 1960 issue of *Rivista dei Combustibili* and was further developed by Goksoyr and Ross and reported in the *Journal of the Institute of Fuel* in 1962. The Goksoyr-Ross system is the basis of an American Society for Testing and Materials (ASTM) procedure for sulfur oxides ( $\text{SO}_x$ ) (Part 26, ASTM Method D3226-73T, 1974). In the controlled condensation approach,  $\text{H}_2\text{SO}_4$  is separated from the gas stream by reducing the temperature of the flue gas below the dewpoint for  $\text{H}_2\text{SO}_4$  but above the dewpoint for  $\text{H}_2\text{O}$  (Figure 1). The resulting aerosol is collected either on the walls of the cooling coil or on a backup frit. Controlled condensation has been studied in the laboratory and found to have a precision and accuracy of  $\pm 6$  per-

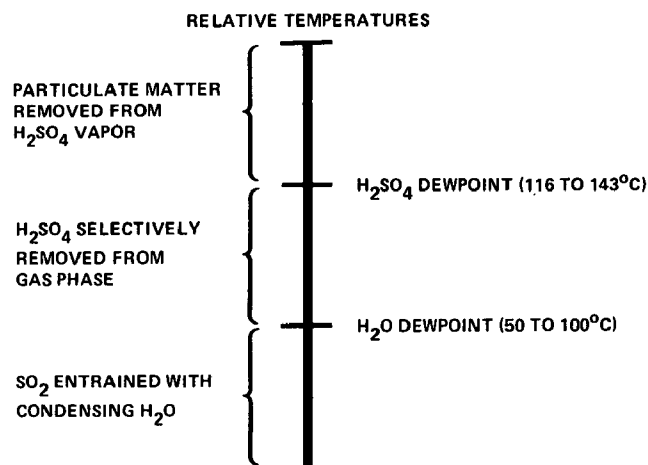


Figure 1. Principle of controlled condensation.

cent in synthetic gas streams. However, the laboratory systems have neither adequate temperature control nor particle removal systems for field use.

TRW, under EPA Contract 68-02-2165, adapted the controlled condensation procedure to particle-laden streams and developed the system shown in Figure 2. This system uses an all quartz probe to collect the gas from the stack and a quartz filter holder

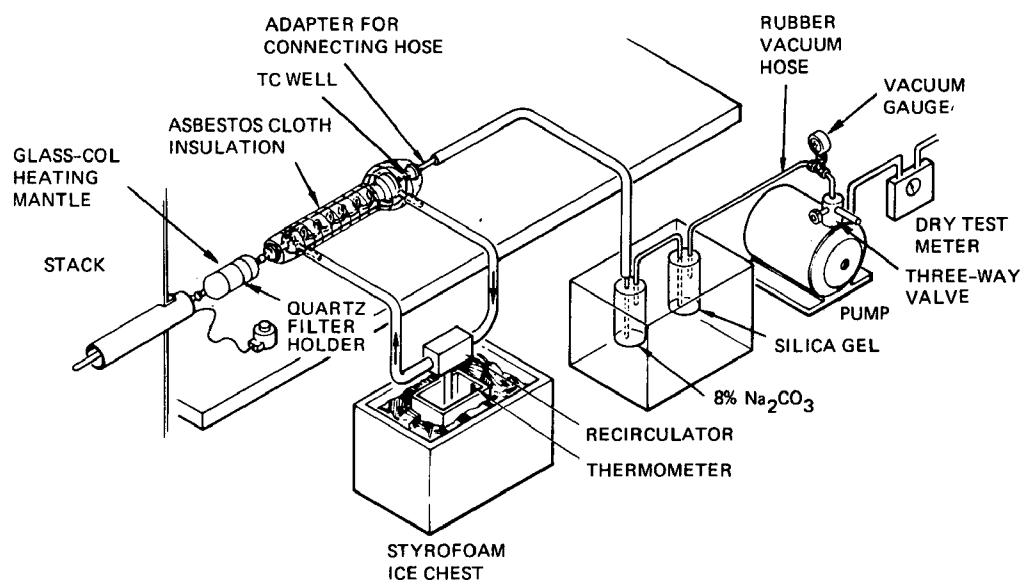


Figure 2. Controlled condensation system field setup.

and filter to remove the particulate matter. Prior to the controlled condensation coil, the gas temperature is maintained above 250° C to prevent H<sub>2</sub>SO<sub>4</sub> fallout and to ensure the separation of H<sub>2</sub>SO<sub>4</sub> from particulate matter. Using this system during an intensive 30-day test program at the TVA/EPA Shawnee FGD Test Facility, the average inlet H<sub>2</sub>SO<sub>4</sub> concentration was 8.3 ppm and the average outlet concentration was 3.1 ppm. Both values, based on daily morning and evening tests, were found to be highly variable. It became apparent that a continuous H<sub>2</sub>SO<sub>4</sub> monitor was needed to record the fluctuations in process conditions. Such an instrument was designed using the controlled condensation approach and was built by TRW under the supervision of the Process Measurements Branch of EPA's IERL-RTP. This automated version of the controlled condensation system is de-

signed to produce a H<sub>2</sub>SO<sub>4</sub> concentration value every 15 minutes. It will operate continuously for 24 hours under mass loading conditions up to 13 µg/m<sup>3</sup>. A typical output from this monitor is shown in Figure 3.

## Summary

Sampling for H<sub>2</sub>SO<sub>4</sub> requires the quantitative separation of SO<sub>2</sub>, particulate matter, and H<sub>2</sub>SO<sub>4</sub>. To date, the best approach for this task is the controlled condensation system. By automating this system, semicontinuous H<sub>2</sub>SO<sub>4</sub> measurements can be attained and a direct correlation with on-line process information is possible.

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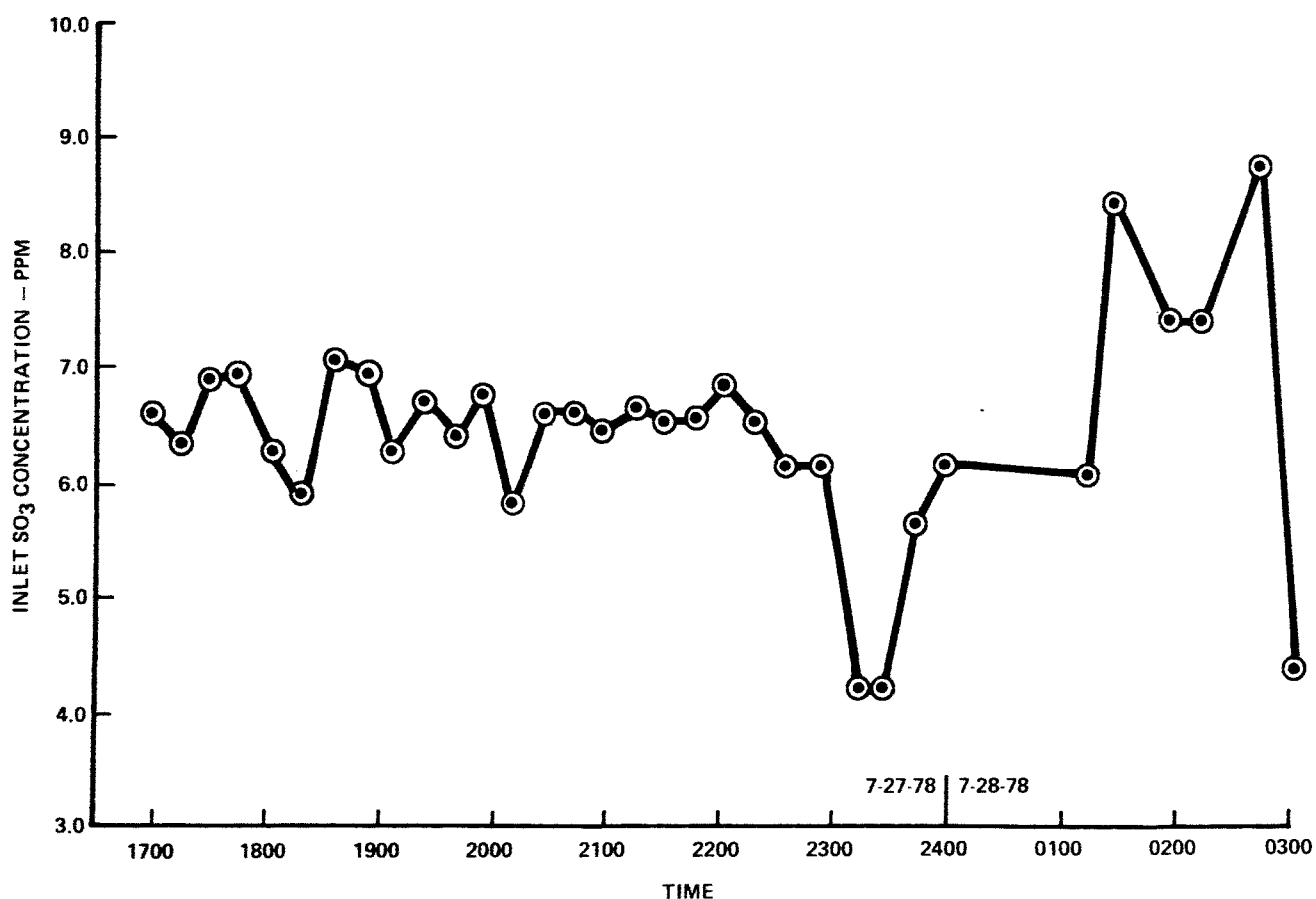


Figure 3. Shawnee power plant venturi scrubber No. 2 inlet SO<sub>3</sub> concentration.

## COMPUTER-BASED CASCADE IMPACTOR DATA REDUCTION PROGRAM

Cascade impactors have gained wide acceptance in the measurement of particle size distribution. These devices are regularly used in a wide variety of environments ranging from ambient conditions to flue gas streams at 500° C (950° F). Specially fabricated impactors can be used for more extreme conditions.

Because of their usefulness, research has been funded under EPA Contract 68-02-2131 and performed by Southern Research Institute to explore the theoretical and practical aspects of impactor operation. As part of this research, an effort has been made to design a comprehensive data reduction system that will make full use of cascade impactor measurements.

The cascade impactor data reduction system is designed to automatically reduce data taken with any one of four commercially available round-jet cascade impactors: the Andersen Mark III Stack Sampler, the Brink Model BMS-11 (as supplied and with extra stages), the University of Washington Mark III Source Test Cascade Impactor, and the Meteorology Research Inc. Model 1502 Inertial Cascade Impactor. With modification the computer programs can accommodate any round-jet impactor with an arbitrary number of stages. Provision is not presently made in the system for slotted jet impactors. However, with more extensive revision the system could also handle data from these impactors.

The original computer programs comprising this data reduction system are written in FORTRAN IV language. The plotting subroutines used were written specifically for the Digital Equipment Corporation PDP-15/76 computer and are not compatible with other plotting systems. However, these programs are in the process of being revised for use with other systems.

The overall system incorporates six programs: MPPROG, SPLIN1, GRAPH, STATIS, PENTRA, and PENLOG. Impactor design, particulate catch information, and sampling conditions from single impactor runs are used to calculate particle size distributions. MPPROG and SPLIN1 perform data analyses and make curve fits. GRAPH is totally devoted to various forms of graphical presentation of the calculated distributions. The particle size distributions can be delivered in several forms. STATIS averages data from multiple impactor runs under a common condi-

tion, and PENTRA or PENLOG calculates the control device penetration and/or efficiency.

Two reports describing this data reduction system are available from NTIS. A brief overview of the program, including several examples, is given in *A Data Reduction System for Cascade Impactors*, EPA-600/7-78-132a, July 1978. The detailed program description with program listings can be found in *A Computer-Based Cascade Impactor Data Reduction System*, EPA-600/7-78-042, March 1978.

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Southern Research Institute

## A MASSIVE VOLUME SOURCE SAMPLER FOR HEALTH EFFECTS STUDIES

A critical need exists for information pertaining to the health effects of the particulate pollution emitted from emerging alternative energy sources. A portion of this information can be obtained from bioassays and animal inhalation studies conducted on samples of these emissions. However, the extended time periods needed to perform these studies require large quantities (1 kg or greater) of particulate sample. The purpose of this task, sponsored under EPA Contract 68-02-2131, was to design, fabricate, and test a sampling system that would collect sufficient quantities of particulate samples for health studies in relatively short time periods. A sampling rate of 340 normal cubic meters per hour (Nm<sup>3</sup>/hr) was estimated to be adequate. Other design considerations were the need to separate the sample into two size fractions—coarse and fine—and minimization of wall deposition within the system. Because the sampler would be used at various sites, it had to be readily transportable and conveniently adaptable to different site conditions.

The ultimate goal of this task was to ensure that the biological impact of the collected sample would be an accurate representation of the emissions produced by the energy source. Fulfilling the particle size distribution aspect necessitated the building of a traversing sampler capable of near-isokinetic sampling. Accomplishing the biological impact goal required a sampler that would prevent or minimize contamination of the sample. This was accomplished by using special construction materials to minimize contamination by the walls of the sampler and by maintaining source gas temperatures in the sampler, thus

eliminating condensation of organic and inorganic vapors.

The prototype system (see Figure 4) consists of a probe, a cyclone dust collector, a fabric filter, a flowmeter, a blower, and a sampling/interconnecting line. The probe is 2.1 m (7 ft) long and has an adjustable opening at its inlet to establish isokinetic sampling. The probe is capable of traversing and, although it is designed for a 9-cm (4-in.) port, it can be easily adapted to fit larger ports.

A Fisher-Klosterman XQ-5 cyclone with a calibrated 50 percent efficiency cut point ( $D_{50}$ ) for a  $2.5\text{-}\mu\text{m}$  aerodynamic diameter at  $340\text{ Nm}^3/\text{hr}$  ( $200\text{ stdft}^3/\text{min}$ ) is the initial particle collector. The cyclone, with its heated enclosure, is approximately 2.1 m high  $\times$  0.6 m wide  $\times$  0.6 m long ( $6 \times 2 \times 2\text{ ft}$ ) and weighs 91 kg (200 lb). Immediately following the cyclone is a single chamber fabric filter approximately 2.1 m high  $\times$  0.9 m wide  $\times$  0.9 m long ( $7 \times 3 \times 3\text{ ft}$ ) and weighing 113 kg (250 lb). The filter chamber can accommodate from 1 to 20 envelope bags. The filtration surface of the bags is composed of Gore-Tex porous Teflon laminate backed by Nomex. Each bag has  $0.5\text{ m}^2$  ( $5.0\text{ ft}^2$ ) of collection surface. The fabric filter is equipped with a manual shaker and can be easily modified to a double-chambered, automatic shaker design.

An orifice plate flowmeter is used to monitor the

$340\text{ Nm}^3/\text{hr}$  flow rate selected for the sampler. The flow rate must be stable to maintain the  $2.5\text{-}\mu\text{m}$   $D_{50}$  cut point of the cyclone. A hand-operated damper attached to the outlet of the blower is used to adjust the flow rate. The blower is a centrifugal pressure blower powered by a 3-phase, 7.5 HP motor and weighs approximately 453 kg (1,000 lb). At the designed flow rate, the sampler requires about 2.5 days of continuous sampling to collect 1 kg of particulate matter from the outlet of an efficient control device with a particulate mass concentration of  $0.05\text{ g}/\text{Nm}^3$ .

The entire sampler is heat traced from the probe to the outlet of the fabric filter. All of the surfaces are either 304 or 316 stainless steel, Teflon, or glass. Heating is controlled from three heater control panels, each panel handling a separate heater load. The individual heaters can be connected in various arrangements to accommodate different sampling site voltages and heating requirements. Design operating temperature of the sampler is  $204^\circ\text{C}$  ( $400^\circ\text{F}$ ).

Construction of the sampler has been completed and a preliminary field test conducted. A full field test will be run in June 1979.

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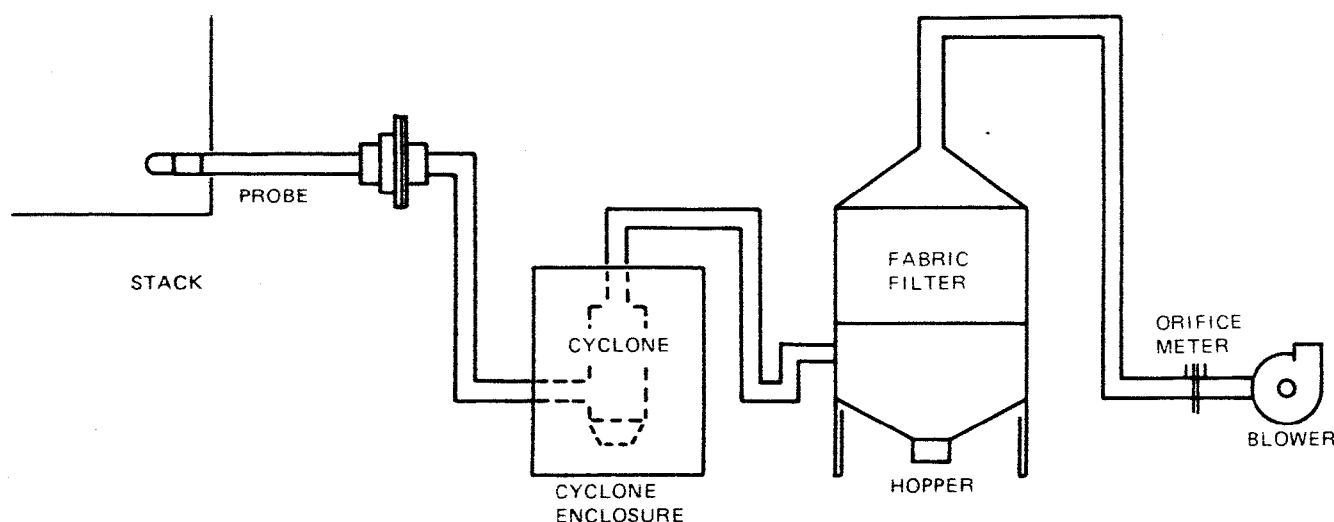


Figure 4. Schematic of high volume particle sampler.

## THE USE OF SORBENT RESINS IN ENVIRONMENTAL SAMPLING

Research in many laboratories over the past few years has shown that macroreticular (sorber) resins have desirable characteristics for environmental sampling. The resins tend to have good collection efficiency and also provide good recovery of the collected sample. While most of the reported work has focused on the sampling of organic species from air (source and ambient), these resins are also finding application in water sampling and in sampling of species such as organometallics.

A research program is underway at Arthur D. Little, Inc., to obtain a detailed quantitative understanding of the behavior of sorber resins in sampling systems. Most of the work to date has been applicable to the sampling of air. This work is described in the documents *Selection and Evaluation of Sorber Resins for the Collection of Organic Compounds* (EPA-600/7-77-044) and *Characterization of Sorber Resins for Use in Environmental Sampling* (EPA-600/7-78-054).

In addition to chemical surface properties, important physical parameters in resin selection are particle size, pore volume, and surface area. Resins commonly considered for environmental studies are Chromosorb 101, Chromosorb 102, XAD-2, XAD-4, Tenax-GC, and Poropak. Particle size of the resins has a practical impact on their use in sampling trains because the size range utilized will affect the pressure drop across the sorber bed. The pressure drop of several resins tested in a 4.5-cm diam  $\times$  9-cm holder at a velocity of 30 cm/sec (1 ft<sup>3</sup>/min flow rate) is shown in Table 1. Thermal stability of the resins is also important. The decomposition temperature of several resins, as measured by differential scanning

calorimetry, is given in Table 2. Actual use temperature should be maintained well below these values when the resins are being used for analysis of trace level compounds. XAD-2 has been found to be the preferred resin when solvent extraction procedures are to be used in sample preparation. If thermal desorption is to be used, Tenax-GC is the sorber of choice. Thermal desorption methods should generally be reserved for small-scale sampling apparatus.

While XAD-2 and Tenax-GC have comparable collection characteristics at 20° C, XAD-2 generally has slightly better volumetric capacity and has substantially greater (10X) weight capacity than Tenax-GC. These two basic factors, weight capacity and the volumetric capacity ( $V_g$ ) for each compound with its specific volatility (boiling point) and polarity, control the quantitative behavior of the resins in a sampling system. The capacity of the resin for a pollutant is a complex function of concentration, volatility, and polarity in streams with high pollutant levels. Generally speaking, XAD-2 will have an upper weight sorption capacity of 1 to 10 percent of the resin weight. Tenax-GC weight capacity is generally about 10 percent of that of XAD-2, or 0.1 to 1 percent of the resin weight.

The collection of vapors in most environmental assessment studies occurs when the pollutant concentration is quite low (e.g., 1 to 100 mg/m<sup>3</sup>). In these cases, the principal characteristic influencing resin quantity and sampling conditions is the volumetric capacity ( $V_g$ ) for the specific pollutant. The volumetric capacity describes the breakthrough capacity of the resin (in units of milliliters per gram) and is the maximum volume of sample stream that can be pulled through the resin while still quantitatively retaining the pollutant.

Table 1. Comparison of Pressure Drop  
of Various Sorbers

Sorber	Mesh range	$\Delta P$ (cmHg)
XAD-2	20-50	6.4
	30-40	6.4
	30-50	8.9
Chromosorb 102	48-60	15.2
Tenax-GC	35-60	8.6

Table 2. Sorber Thermal Decomposition  
Characteristics

Sorber	Onset of Decomposition
XAD-2	210° C
Chromosorb 102	200° C
Tenax-GC	400° C

The general behavior of the resin/pollutant interaction can be conveniently summarized in terms of an empirical correlation observed between elution volume (actually  $\log V_g$ ) and boiling point, as shown in Figure 5. As is indicated in Figure 5, good correlations were observed between  $\log V_g$  and the boiling point for each compound category studied. There are some significant differences in  $V_g$  values for compounds that have the same boiling point but are in dissimilar compound categories.

These data can be used in two basically different ways starting with either the resin quantity or the volume to be sampled as the fixed quantity. The following examples describe the procedures used in designing the sampling approach. Suppose a source were to be sampled for chlorobenzene and a typical set of Method 5 conditions were to be used, i.e., 28 L/min for 1 hr. The  $V_g$  for chlorobenzene on XAD-2 is  $2.4 \times 10^5$  mL/g. Because 1,680 L are to be sampled, a minimum of 7 g of XAD-2 would be required for complete collection.

A different type of question would be whether the SASS train, with its predetermined resin bed volume, will quantitatively collect benzyl amine, for example. The sorbent cartridge in the SASS train contains about 130 g of XAD-2 and a  $30\text{-m}^3$  sample is normally collected. These values can be used to calculate a cutoff  $V_g$  value for the SASS train of  $2.3 \times 10^5$  mL/g or  $\log V_g = 5.4$ ; this value is indicated by the dashed line in Figure 5. In order for a compound to be collected completely by the sorbent module, its  $V_g$  would have to be greater than  $2.3 \times 10^5$ . The  $V_g$  for benzylamine is  $7.9 \times 10^6$ ; thus, benzylamine would be completely collected. A comparison of several sampling trains relative to vapor collection is given in Table 3.

The compound categories studied, as represented in Figure 5, cover each of the different types to be encountered well enough so that one can estimate a  $V_g$  ( $\log V_g$ ) value for new compounds by careful interpolation and extrapolation.

Philip L. Levins  
Arthur D. Little, Inc.

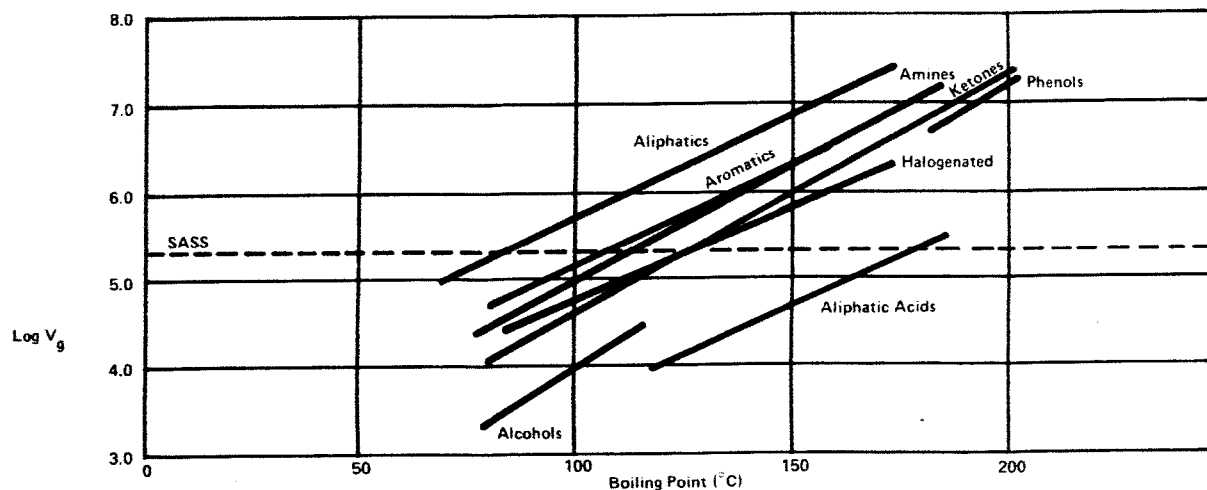


Figure 5.  $\log V_g$  vs. boiling point for individual adsorbate groups on XAD-2.

Table 3. Relationship of SASS and Modified Method 5 Trains to Specific Retention Volume ( $V_g$ ) Data

	Sampling time (hr)	Sampling volume ( $\text{m}^3$ )	Sorbent trap volume (mL)	Resin cap (g)	XAD-2	
					Breakthrough $V_g$ (mL/g)	$\log_{10} V_g$
SASS	4	34	445	130	260,000	5.42
141 L/min (5 $\text{ft}^3$ /min)	1	8.5			65,000	4.81
Modified Method 5	4	6.8	100	29	234,000	5.37
28.3 L/min (1 $\text{ft}^3$ /min)	1	1.7			59,000	4.77



## REVISIONS TO IERL-RTP PROCEDURES MANUAL: LEVEL 1 ENVIRONMENTAL ASSESSMENT BIOLOGICAL TESTS FOR PILOT STUDIES, EPA-600/7-77-043

### Change 1: "Marine Algal Bioassay" Chapter 3, beginning on page 65

The old procedure, while effective, required revisions in order to simplify the assay and provide for both stimulation and inhibition of growth caused by Level 1 samples. The new, simplified procedure provides a combination of nutrients and minerals necessary to allow both growth inhibition and stimulation to be evaluated for Level 1 samples.

### Change 2: "Daphnia Bioassay" Chapter 3, page 50

On page 52 of the current manual, *Daphnia pulex* is recommended. *Daphnia magna* should replace *Daphnia pulex* as the test animal throughout the procedure because it is much larger, is easier to work with, and has a much stronger toxicity data base.

### Change 3: "Chinese Hamster Ovarian Clonal Growth Use for Cytotoxicity Testing" Chapter 3, pages 35-36

The existing procedure recommends the use of the cell line WI-38 for cytotoxicity testing of liquid samples (i.e., aqueous and organic liquids). The cost of this test has increased markedly since its inclusion in the protocol and alternate cytotoxicity tests exist that provide comparable results at much less expense. The Chinese Hamster Ovarian (CHO) clonal assay (Chapter 3, page 36) should be used as the cytotoxicity test in the Level 1 biological procedures for liquid samples.

NOTE: Revisions appear in condensed form. For complete change notices, contact Ray Merrill, PMB, IERL-RTP (919-541-2557).

## DEVELOPMENTS IN SAMPLING TECHNIQUES FOR INHALABLE PARTICULATE MATTER

In support of a reassessment of the total suspended particulate standard now underway by EPA's Office of Air Quality Planning and Standards, three laboratories in EPA's Office of Research and Development—the Health Effects Research Laboratory (HERL-RTP), the Environmental Sciences Research Laboratory (ESRL), and the Industrial Environmental Research Laboratory (IERL-RTP)—are examining potential sampling requirements. The HERL-RTP has recommended a 15- $\mu$ m upper cut size for inhalable particulate matter and a second division at 2.5  $\mu$ m for fine particulate matter. Current particulate matter sampling techniques do not provide data at these cut sizes for either ambient or source samples. At a workshop of leading aerosol scientists sponsored by the Process Measurements Branch of IERL-RTP, a measurement development program was recommended. The program considers short-term modifications for existing techniques and a longer term effort to fully investigate the require-

ments for more information, including data on stack condensable matter.

A number of developments have been made in this program. Extrapolation techniques have been developed to estimate the 15- $\mu$ m particulate loading using existing data on loadings up to 10  $\mu$ m. A 15- $\mu$ m cyclone has been designed and is being tested for use with a Method 5 train. Horizontal elutriators are being investigated and have shown good laboratory agreement with theory. A prototype elutriator is being built for use with the Fugitive Air Sampling Train (FAST) system for fugitive emission measurements. ESRL is investigating particle losses in nozzles; preliminary data indicate significant collection (up to 90 percent) for many particle sizes below 15  $\mu$ m.

D. B. Harris  
EPA/IERL-RTP

## ENVIRONMENTAL ASSESSMENT MEASUREMENT SYMPOSIUM

The second symposium on "Process Measurements for Environmental Assessment" is scheduled for February 25-27, 1980, in Atlanta, Georgia. The symposium will consist of sessions on sampling techniques, chemical analysis, and biological assays of samples from energy and industrial processes. Major emphasis will be placed on measurement method de-

velopment and application to complex samples. Procedures for both initial screening and detailed analysis will be included in the presentations. Authors wishing to propose papers for the symposium should submit titles and brief abstracts to Philip L. Levins, Arthur D. Little, Inc., Acorn Park, Cambridge, Massachusetts 02140.

## RECENT EPA PUBLICATIONS OF INTEREST

R. R. Wilson, Jr., P. R. Cavanaugh, K. M. Cushing, W. E. Farthing, and W. B. Smith

Guidelines for Particulate Sampling in Gaseous Effluents from Industrial Processes, EPA-600/7-79-028, PB 290 899 (1/79).

*This report lists and briefly describes many instruments and techniques used to measure the concentration or size distribution of particles suspended in process streams. Standard (well-established) methods are described, as well as some experimental methods and prototype instruments. Instruments and procedures are described for measuring mass concentration, opacity, and particle size distribution. Procedures for planning and implementing tests for control device evaluation are also included.*

D. E. Lentzen, D. E. Wagoner, E. D. Estes, and W. F. Gutknecht

IERL-RTP Procedures Manual: Level 1 Environmental Assessment (Second Edition), EPA-600/7-78-201, PB 293 795 (10/78).

*This manual presents revised Level 1 environmental assessment procedures for personnel collecting and analyzing samples from industrial and energy-producing processes. The strategy of the environmental assessment program provides a framework for determining industrial process and stream priorities on the basis of a staged sampling and analysis technique. Level 1 is a screening phase that characterizes the pollutant potential of process influent and effluent streams. The manual is divided into two major sections. Chapters 3-7 discuss sampling procedures for gases, fugitive emissions, liquids (including slurries), and solids. The remainder of the manual is divided into three chapters on procedures for inorganic, organic, and particle analyses. Chapter 11 briefly discusses bioassay procedures. Biological assessment techniques are detailed in a companion procedures manual (EPA-600/7-77-043).*

E. D. Estes, F. Smith, and D. E. Wagoner

Level 1 Environmental Assessment Performance Evaluation, EPA-600/7-79-032, PB 292 931 (2/79).

*This report discusses a two-phased evaluation of Level 1 environmental assessment procedures. Results from Phase I, a field evaluation of the Source Assessment Sampling System (SASS), showed that the SASS train performed well within the desired Level 1 accuracy limit. A Method-5 train was used to estimate the "true" particulate loading. Phase II of the evaluation consisted of three types of control samples to challenge the spectrum of Level 1 analytical procedures: an artificial sample in methylene chloride, an artificial sample on a flyash matrix, and a real sample composed of the combined XAD-2 resin extracts from all Phase I runs. Phase II results showed that when the Level I analytical procedures are carefully applied, data of acceptable accuracy are obtained. Estimates of intralaboratory and interlaboratory precision were made.*

J. C. Harris, J. J. Hayes, P. L. Levins, and D. B. Lindsay

EPA/IERL-RTP Procedures for Level 2 Sampling and Analysis of Organic Materials, EPA-600/7-79-033, PB 293 800 (2/79).

*This manual presents Level 2 procedures for sampling and chemical analysis of organic materials. It represents a step in the development of a general methodology for chemical and biological assessment of environmental effects of industrial effluents. It presents concepts and general guidelines, together with a number of more fully developed, tested, and validated Level 2 procedures. The accumulation of experience in sampling and chemical analysis of environmental pollutants is by no means complete, however. Users of the manual will find a number of areas in which additional research and testing is needed, and in which present knowledge is insufficient to permit hard and fast procedures to be established.*

E. M. Smith and P. L. Levins

Sensitized Fluorescence for the Detection of Polycyclic Aromatic Hydrocarbons, EPA-600/7-78-182, PB 287 181 (9/78).

*This report describes a fluorescent spot test, devised for polycyclic aromatic hydrocarbons (PAH), based on the sensitization of the inherent fluorescence of such compounds. On filter paper, 10 picograms ( $1 \text{ pg} = 10^{-12}$  grams) of PAH in a spot of 0.25 cm diameter can generally be detected when treated with naphthalene. In the case of benzo(a)pyrene, 1 pg has been detected. This method has been shown to be specific for PAH with minimum interference from other compounds. The method may be used to estimate the general level (factors of 10) of PAH in samples to aid in decisions for more specific analyses.*

P. L. Levins, C. E. Rechsteiner, and J. L. Stauffer  
Measurement of PCB Emissions from Combustion Sources, EPA-600/7-79-047, PB 293 360 (2/79).

*Use of gas chromatographic procedures for the determination of polychlorinated biphenyls (PCBs) in solids and water to measure PCBs in combustion source emission has encountered certain difficulties. This report describes a gas chromatographic/mass spectrometric procedure that overcomes these difficulties. The procedure relies on selected mass scanning in restricted regions of the chromatograms. It was developed because the distribution pattern of the individual PCBs changes in the combustion process. This renders invalid the pattern-matching approach used with the gas chromatographic/electron capture detector method.*

W. B. Smith, Compiler

Proceedings: Advances in Particle Sampling and Measurement (Asheville, NC, May 1978), EPA-600/7-79-065, PB 293 363 (2/79).

J. L. Rudolph, J. C. Harris, Z. A. Grosser, and P. L. Levins.

Ferroalloy Process Emissions Measurement, EPA-600/2-79-045, PB 293 171 (2/79).

L. E. Sparks

Cascade Impactor Data Reduction with SR-52 and TI-59 Programmable Calculators, EPA-600/7-78-226, PB 290 710 (11/78).

R. G. Patterson, P. Riersgard, and S. Calvert

Effects of Charged Particles on Cascade Impactor Calibrations, EPA-600/7-78-195, PB 288 270 (10/78).

N. H. Gaskins and F. W. Sexton

Compilation of Level 1 Environmental Assessment Data, EPA-600/2-78-211, PB 286 924 (10/78).

E. A. Burns, Compiler

Symposium Proceedings: Process Measurements for Environmental Assessment (Atlanta, February 1978), EPA-600/7-78-168, PB 290 331 (8/78).

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