

ENVIRONMENTAL Science & Technology

Emphasizing

Water,

Air, &

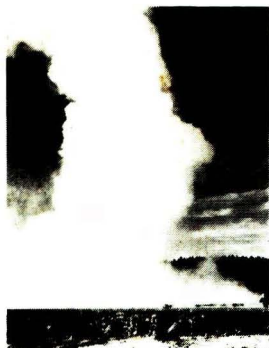
Waste

Chemistry

DECEMBER 1967

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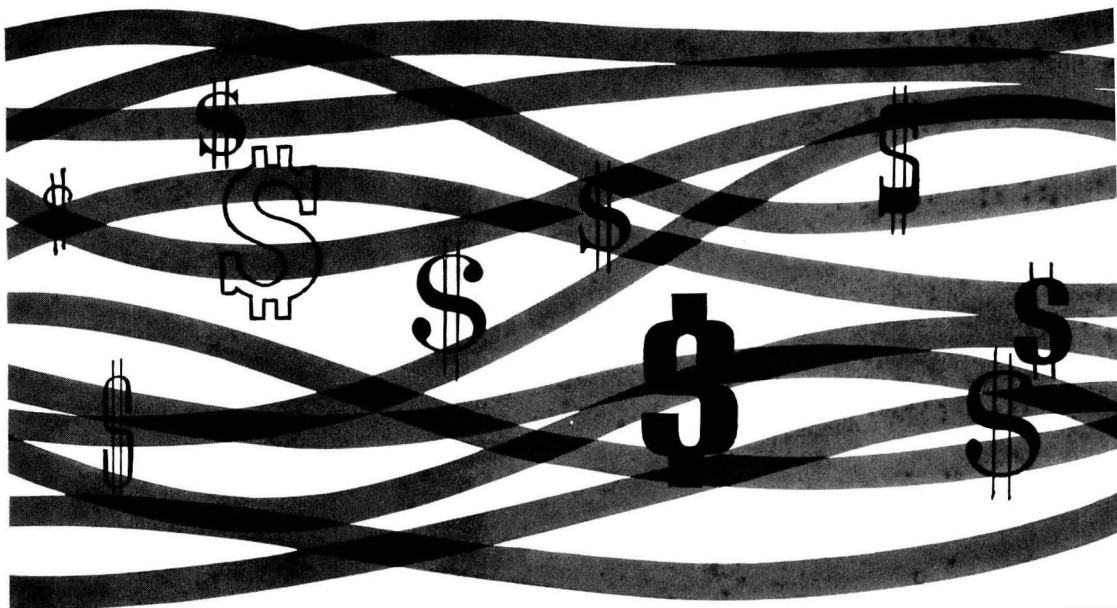


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Editorial

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Hyperfiltration. Processing of pulp mill sulfite wastes with a membrane dynamically formed from feed constituents 991

J. J. Perona, F. H. Butt, S. M. Fleming, S. T. Mayr, R. A. Spitz, M. K. Brown, H. D. Cochran, K. A. Kraus, and J. S. Johnson, Jr.

Although all the variables affecting performance of dynamically formed membranes in the hyperfiltration (reverse osmosis) of spent liquors of the sulfite process are not known, results of recent studies indicate that a production facility operating at 60° C. can attain production rates of nearly 30 g.p.d. sq. ft. at pressures less than 500 p.s.i.g. Such processing rates can be maintained for at least two weeks. The dynamically formed membranes build on porous bodies when the spent liquors circulate, under pressure, past the bodies. These membranes reject at least 90% of the colored matter in the feeds and substantial but somewhat lower percentages of chemical oxygen demand and total dissolved solids. Under optimum conditions, production rates three times as high, with equivalent rejection levels, might be achieved.

Phenolics by aqueous-injection gas chromatography 997

R. A. Baker and B. A. Malo

Many, but not all, mono- and dihydric phenolic materials, which constitute an important class of water pollutants, can be separated and identified by gas-liquid chromatography without preliminary sample concentration. The procedure, which uses a flame-ionization detector, gives the best resolution of aqueous solutions (1 mg./l.) of these materials with a 5 to 10% loading of FFAP (a reaction product of Carbowax 20M and 2-nitroterphthalic acid) on Chromsorb T. The ortho-substituted phenolics are eluted ahead of the meta- and para-substituted compounds. The meta and para isomers are not completely separated so that supplemental separation and identification schemes are necessary for complete identification.

Continuous monitoring of organophosphorus compounds in air with an alkali metal-dual flame ionization detector 1008

M. J. Prager and Benjamin Deblinger

Instrumentation is available for monitoring air-borne organophosphorus compounds continuously for longer than one month. Key to the instrumentation is a two-stage dual-flame ionization detector. The upper burner of the device responds only to organic phosphorus and halogen compounds provided other substances are efficiently burned in the lower flame. With this instrumentation airborne organophosphorus compounds at concentrations of 0.1 to 7 µg. per liter of air are routinely monitored without interference from airborne nonphosphorus organic compounds at higher concentration levels.

Influence of hydrogen fluoride fumigation on acid-soluble phosphorus compounds in bean seedlings 1011

M. R. Pack and A. M. Wilson

Bean seedlings can tolerate, without apparent adverse effects, higher concentrations of fluorides than those found in the ambient atmosphere. These seedlings can tolerate atmospheres containing hydrofluoric acid at concentrations of 14 µg. of fluoride/m.³ for four days without showing any significant inhibition of any enzymatic reactions involving acid-soluble phosphorus compounds. The lack of inhibition of phosphoglycerate is at odds with the commonly accepted view of enolase inhibition as a probable effect of hydrofluoric acid on plants.

Communication

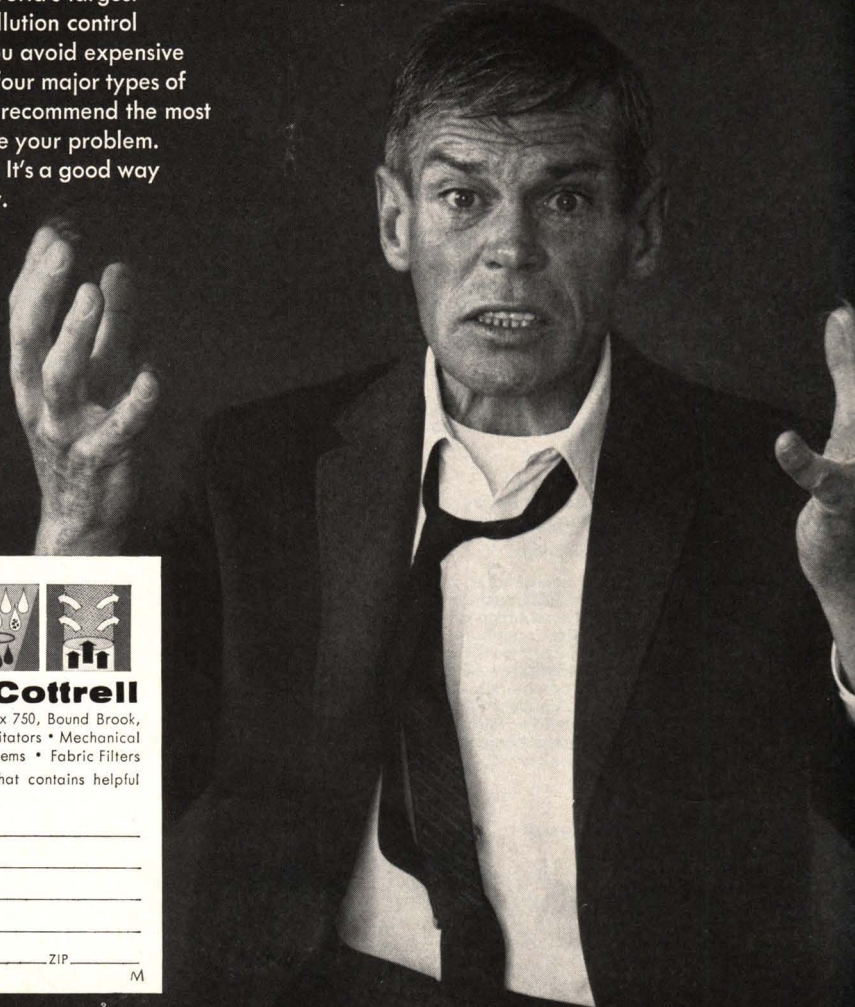
Further observations on the ferrous ammonium thiocyanate reagent for ozone 1014

I. R. Cohen and J. J. Bufalini

No other known colorimetric method for low levels of ozone is as sensitive (0.1–2.0 p.p.m.) and as stable (color development after 14 days) as the ferrous ammonium thiocyanate method. Highly accurate results can be obtained by measuring the molar absorptivity at 480 mµ if a calibration curve for low levels of ozone is prepared and if both bubbler frit size and the rate of collection are properly taken into account in developing the calibration curve. The method does not follow Beer's law with an oxidant such as ozone, although it does so with such oxidants as *n*-butyl hydroperoxide, *t*-butyl hydroperoxide, and hydrogen peroxide. The reason for this inconsistent behavior is not known.

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Earth's environment is a closed, integrated system

ES&T's continuing goal is to present an integrated view of the complex system

Stock taking at this time of year is hardly a novel event. And usually no one is very surprised at what is said. Nonetheless, a brief look at ENVIRONMENTAL SCIENCE AND TECHNOLOGY's brief history may serve to show where we have been, where we are, and where we might be going.

In ES&T's first issue (January, page 101) and again in this one (page 1015), we note that the new publication places special emphasis on reporting original chemical research, engineering developments, and technico-economic studies in fields of science directly related to man's environment.

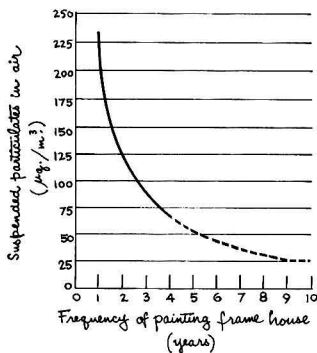
With the encouragement of the ACS Board of Directors and our rapidly increasing body of contributors and readers, we have striven to push the perimeter of ES&T's coverage as far and in as many directions as practicable. To this end we have carried articles, staff written and by outside contributors on a variety of subjects, stressing the interrelationship and inter-

dependence of scientific understanding, technological skills and application, economic restraints and pressures, and legal and legislative influences and controls.

The Earth's environment is a closed, albeit huge, integrated system. What happens in one sector of the world one day is related—whether or not the relationship is apparent—to a further happening in another part of the world on another day. As an author once noted, a Chinese sneezes in Manchuria and a month later a major storm hits California.

In the field of environmental studies, ecology per se and human ecology, in particular, play special roles. Environmental sciences and technologies are mere exercises unless at some point they are joined with social, political, and humanistic philosophies and considerations in an effort to promote the welfare of man. Our continuing goal is to give our readers an integrated view of the complex system known as the environment.





Cheaper to Clean Up Than Not to Clean Up

The cost of living in a metropolitan area that has polluted air is greater, in terms of increased costs of common cleaning and maintenance operations, than the cost of living in an average rural area with unpolluted air. Yet, the extra household costs of living in polluted areas are so subtle that most city dwellers are unaware of them. Household cleaning and maintenance costs in Washington, D. C., for example, reach about \$100 per person per year, considerably higher than the national average of \$70. Annually, the average family head in Washington spends \$335 above the national average to maintain his household in proper repair, according to testimony by Environmental Health and Safety Research Associates at the National Capital Metropolitan Area Interstate Air Pollution Abatement Conference in Washington.

The total economic penalties paid by Washington residents for extra cleaning and maintenance come close to \$250 million annually. However, the same witness noted, a survey made last year by the Department of Commerce showed that the cost of reducing pollution in Washington by two thirds is only \$20 million annually—one tenth the extra cost of living in a polluted air environment. Cost figures, based on data from surveys on the frequency with which residents of various areas found it necessary to paint the exteriors of their frame homes and to clean draperies within the homes, show a direct correlation between household costs and level of suspended particulates in the atmosphere.

Air Tracer—Keener Than Most Other Systems

Knowledge of air movements and the ability of the atmosphere to dilute whatever is dumped into it is no longer a matter only of scientific curiosity. With the recent passage of the Air Quality Act of 1967 (ES&T, November, page 884), the movement of air masses and the entrained materials is a matter of law and government concern. Now, scientists at HEW's National Center for Air Pollution Control in Cincinnati have developed a highly sensitive gaseous tracer system. The system is so sensitive that the gas can be detected in concentrations of 1 part per 100 trillion parts of air. The gas, sulfur hexafluoride, is nontoxic and has been used successfully in experiments that required detection of the gas as far as 70 miles from the originating source.

From Slop Oil to Salable Oil

A process for the recovery of salable oil from slop oil, a nonsalable emulsified crude oil, has been developed by Conservation Process, Inc., a division of Phillips Petrochemicals, Inc. The process, called the Conservoil System, separates slop oil into its components and thus avoids the development of slop oil pits. The equipment, which is portable, is on stream at the Barber's Hill Salt Water Co., Mont Belview, Tex., and comprises four stages of treatment—heating (190° F.), removal of solids (screening and the like), chemical treatment (demulsifiers and the like), and high speed centrifugation.

Respiratory Diseases and Air Pollution



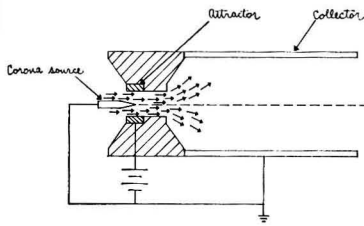
Lawther

“Based on a 15-year study of the problem of air pollution, we have found that emphysema, bronchitis, and other respiratory diseases are not caused by increased air pollution but simply because people are living longer,” noted Dr. P. J. Lawther, Director of Air Pollution Research at St. Bartholomew’s Hospital Medical College, London England and an authority on the problem. At the recent symposium at Hazelton Laboratories, Inc., in metropolitan Washington, D. C., Dr. Lawther said, “Based on a study of 5000 British school children, we have found evidence to show that infections in the lower respiratory tract are much higher in children of working class parents than those of upper class parents.” In his studies Lawther also found that lung cancer mortality for groups of people such as chimney sweeps, bus garage employees, and the like was only twice as high as average, although the group had been exposed to nearly 10,000 times more air pollution than the average citizen. “Since the Clean Air Act was passed in London in 1956, the number of smoke particles in the air have been reduced from 450 particles per million to about 100 particles per million. However, the respiratory disease rate continues to rise,” he said. “It is interesting to note that the two areas of the world with the highest lung cancer rate are the Channel Islands and Finland, both of which have no air pollution at all,” Lawther commented. Air pollution is not necessarily the cause of lung cancer and respiratory diseases, although a particular agent in air pollution might be a factor, he cautioned.

The Sky Above and the Water Below

Apparently satisfied that its edict “No sewage treatment device, no boat permit” worked successfully this year on Norris Lake, the Tennessee Valley Authority now says that it plans to extend the restriction to other TVA lakes in North Carolina, Tennessee, and Georgia (ES&T, May, page 369). The problem in TVA country is acute, the authority notes, because of a marked increase in the size of the boating community—from 10,000 boats in 1947 to more than 50,000 today, plus more than 9000 houseboats and boathouses.

Showing equal concern about the atmosphere, TVA also revealed plans for a major research project to test a method for reducing the quantity of sulfur dioxide emitted from the stacks of its fossil fuel power plants. The project will test the efficacy of dry powdered limestone injected into fuel combustion chambers in controlling SO₂ stack emissions (ES&T, January, page 9). Involving two large power generating units, the tests will begin on an intermittent, experimental basis. Then, at least one unit will be run under test conditions for six months. Sulfur dioxide, normally bypassing the stack equipment used to trap ash particles, combines with limestone in the combustion chamber and forms solid particles that can be collected by the ash collection system. The project is a quid pro quo deal—TVA supplies the boilers, manpower, and other supporting services. PHS pays the direct costs involved, estimated at \$4.2 million.



Precipitators for Vehicle Exhaust Control

Two prototype electrostatic (EGD) precipitators for vehicle exhaust are currently being evaluated by the National Center for Air Pollution Control in Cincinnati. Developed by Gourdine Systems, Inc. (Livingston, N. J.), these precipitators remove solid particulates and aerosol droplets from the exhaust gases of diesel and gasoline internal combustion engines. They remove from vehicle exhaust gases micron and submicron particles—oil and smoke particles from diesel engine exhaust, lead from gasoline engine exhaust. The diesel unit is rated for 800 c.f.m., and the gasoline unit is rated for 100 c.f.m. The compact precipitators resemble conventional mufflers in both size and cost. They are installed in the existing tailpipe after the muffler and connected to a solid state power supply.

In the EGD precipitators a corona ion source converts heat directly into electricity. As the particulates and droplets pass by this source they become charged and are attracted to—then deposited on—a collector plate. According to Gourdine, analyses of the deposits in single precipitators on gasoline engines that have completed 6000 miles of road operations reveal that 25% of the precipitate is the lead from tetraethyllead, a common gasoline additive. At Cincinnati, the EGD units will be evaluated under road conditions to determine their efficiencies, to measure the effect of the units on vehicle performance, and to determine the quality and quantity of the collected precipitate. Test results should be available in mid-February. (For more on EGD units see ES&T, January, page 17.)

New Men for Water Pollution Control



Edwards



Moore

Suggestive of what appears to be major changes in the Water Pollution Control Administration are two personnel changes that involve departing key agency men: DiLuzio and Quigley. *Max N. Edwards* is the new Assistant Secretary of the Interior for Water Pollution Control and Desalination. Formerly, he was Assistant to the Secretary and Legislative Counsel for the Department of Interior. He succeeds Frank C. DiLuzio who left government service to become president of Reynolds Electrical Engineering Co. (Las Vegas, Nev.).

Joe G. Moore, Jr. is candidate for FWPCA commissioner. He is executive director of the Texas Water Development Board and chairman of the Texas Water Quality Board. Moore has spent all of his professional career serving Texas—research assistant to the Texas Legislative Council, executive director of the Texas Industrial Board, research director and secretary of the State Finance Advisory Commission, finance examiner for Texas Commission on Higher Education, and chief budget examiner for the governor. Moore is the only candidate in sight to replace James M. Quigley, former commissioner, who left government service this month to accept a position as a vice president of U. S. Plywood-Champion Paper Co. (New York City).

Keeping Park Visitors Out of Hot Water

The roads in Yellowstone National Park must permit a close view of the geysers. Yet, these roads interfere with the free passage of the hot water run-off from the geysers. So park authorities place culverts under the roads. The hot water from the geysers contains large quantities of highly corrosive materials that quickly destroy culverts. In a search for replacement materials, park authorities settled on two—Type 316 stainless steel and vitrified clay tile. Because of its greater strength, the stainless steel, from Republic Steel Corp., looks like the winner.



Geyser run off

Watching an Environmental Watchdog Watch

Winding up its annual meeting, held late this month in New York City, the American Association for the Advancement of Science said it established a new committee to study the consequences of environmental alteration. And so another chapter was added to an involved story that started, publicly at least, 12 months ago. Meeting at the close of 1966 in Washington, D.C., AAAS, in response to an expression of concern by some of its members for a study of the long-term consequences of the widespread use of defoliants in Vietnam, resolved to establish a committee to advise the parent group of studies concerning chemical and biological agents that alter the environment. The advisory committee, appointed in March, was chaired by Dr. Rene Dubos (Rockefeller University). By summer the Dubos committee filed a report that, among other things, recommended a permanent AAAS committee to make a broad and continuing study of the consequences of environmental alterations. A second proposal was that AAAS request the National Academy of Sciences to undertake a special study of the effects of chemical and biological warfare agents on ecology and human health.

The broad, civilian inquiry was assigned to the AAAS Committee on Science and Human Welfare headed by Dr. Margaret Mead. The Mead committee, in turn, recommended that AAAS set up a permanent committee to study the consequences of environmental alteration. This is the committee that AAAS president, Dr. Don K. Price (Harvard), revealed this month in New York. Headed by Dr. David R. Goddard (University of Pennsylvania), the committee includes Dr. Rene Dubos, Dr. Barry Commoner (Washington University), and Dr. Athelstan F. Spilhaus (president of the Franklin Institute).

Meanwhile, AAAS approached the National Academy of Sciences to undertake the special, military oriented study. However, NAS did not have the funds to pursue the study. So, AAAS asked the Department of Defense for assistance. DOD agreed and farmed the job out under contract to Midwest Research Institute. The MRI report, now complete, is being studied by an NAS committee headed by Dr. Geoffrey Norman (University of Michigan). After NAS scrutiny the report will go to AAAS for review by the newly established Goddard committee. Thus the effort has gone full circle—watcher and watchee watching each other.

Big Things Ahead for Air Pollution Research

The National Center for Air Pollution Control ends its first full year of operation with a new idea in research grants, plus a record number of dollars to parcel out to research organizations



Dalmat

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Steigerwald

Tapping private sector expertise

The old adage that money begets more money is never truer than when applied to the research grants field—at least in the opinion of the young research worker. He sees the big names, particularly from the big universities, seemingly experiencing little difficulty in getting substantial support. Yet, at the same time, his proposals, modest though they be, face considerably tougher sledding.

Herbert T. Dalmat, chief of the National Center for Air Pollution Control's (NCAPC) Office of Research Grants, is inclined to agree: "Almost the first thing said when a proposal comes up for review is 'What is this person's research experience? What has he published?' These are impossible criteria for a young researcher just getting started. Or take the case of an engineer who, after a few years' experience in industry, decides he wants to do research. There has to be a better way of getting such people started when they have good ideas. We hope our new Exploratory Grants Program will provide this way."

Dalmat's research grants operation is one of several programs by which

NCAPC will pay out an ever increasing number of research and training dollars in FY 1968. Universities, research institutes, and commercial outfits will share in the bounty. And the Air Quality Act of 1967 (ES&T, Nov., page 884) authorizes still more research dollars—as much as \$125 million in the next two years, probably mostly as contracts—for those groups ready and able to get into the national research effort to control air pollution.

Early start

Dalmat and his staff plan on having their new program under way by early 1968. "At this time we are setting no definite figures on how many of the grants, which will not exceed \$10,000, we'll award. We're going to wait and see what kind of proposals we get. If necessary, we'll request supplemental funds from Congress to support worthy projects."

NCAPC is putting no restrictions on these grants. They can range across the entire field of air pollution—its causes, effects, extent, prevention, and control. A grant, which will run for no more than two years, can be used to

establish feasibility, to exploit an unexpected research opportunity, to analyze data previously collected, or to carry out pilot studies. However, NCAPC has singled out some areas in which it feels such short term research might be particularly fruitful. These targeted areas are:

- Behavioral toxicology.
- Development of mathematical and statistical bases.
- Exploration of new physical concepts.
- Life cycle of important pollutants.
- Public attitudes and acceptance of aesthetic insults.
- Relationship between air quality in living spaces and air pollution.
- Study of relationship between air pollution damage and market parameters.

Another goal of the exploratory grant program is to get grants money out and research under way more quickly. "Under our regular research program, six to eight months might elapse between the time a proposal is submitted and the money ends up in the scientist's hand," according to Dalmat. "Exploratory grant proposals

will not have to come in at our three annual deadlines. As they come in, we'll expedite our normal reviewing procedures. It might be possible in some cases to have money in the researcher's hands within four to six weeks," he notes.

"We welcome more grant applications," Dalmat encourages. And, to this end, he invites anyone interested in more information on exploratory grants to write to Chief, Office of Research Grants, National Center for Air Pollution Control, 8120 Woodmont Ave., Bethesda, Md. 20014.

More to spend

Besides the problems of launching a new program in FY 1968, Dalmat and his staff will have the added responsibility of disbursing a record appropriation of \$8.6 million for research grants—a \$2.4 million increase from the preceding year.

Dalmat sees the grants program becoming more mission oriented. "This is not to say that we're going to do away with long-range programs. That would be foolhardy. But we do want to program more, to direct our efforts more to the areas where bigger pay-offs are possible."

One area needing a lot more effort is that which deals with the health effects of air pollution. However, Dalmat feels that the National Institutes of Health's new Environmental Health Sciences Division may do much of the basic research in this area. This would free his grants program to spend more effort on gross health effects, as well as on epidemiological studies and on studies of the psychological effects of air pollution. Social and political implications of air pollution are still other areas he feels need more attention.

Review by PHS method

In selecting these grants, Dalmat uses the so-called Public Health Service reviewing method. The first review is by study sections based on disciplines. Composed of 13-15 scientists

(mostly from outside Government), a study section assigns a priority based on scientific merit. Close to 85% of NCAPC's proposals are reviewed by a study section NCAPC set up when none of PHS's existing sections covered the disciplines involved. This section reviews proposals in atmospheric chemistry, analytical techniques, meteorology, botany, and toxicology, all as they relate to air pollution.

The second review is by NCAPC's advisory council, also composed largely of outside scientists. The review considers applicability of a proposal to air pollution, as well as scientific merit.

The House Government Operations Committee recently criticized this method, charging that PHS relies year after year on a small group that advises on grants, as well as receives grants. And the Federal Water Pollution Control Administration is using more in-house review (ES&T, Sept., page 688).

"In my experience, I've sensed little of the 'I'll scratch your back if you'll scratch mine' philosophy," Dalmat maintains. "More often I've heard 'He ought to know better than submit such a sloppy proposal.' We are turning down about 60% of the proposals we receive—both new and renewals—and the PHS system seems to be working for us."

Biggest gains in contracts

Most of the research grants go to universities, although foundations, state agencies, hospitals, and even foreign groups can and do get grants. Research contracts, on the other hand, usually end up in commercial firms or research institutes. This area of NCAPC research, from a modest \$2 million in FY '67 could grow to \$100 million by FY '69—if Congress appropriates funds for what it has authorized.

The original appropriation for FY '68 calls for \$9.6 million in research contracts—\$2.5 million for federal, or interagency, contracts, and \$7.1 million for nonfederal contracts. The Air Quality Act of 1967 authorizes \$35

million during FY '68 and \$90 million in FY '69 for development of control technology of combustion by-products. Most of this development money will be in the form of research contracts. According to Dr. B. J. Steigerwald, head of the office of program planning and evaluation, a sizable appropriation is possible in the final half of FY '68 under this section of the act.

The key to effective use of these new funds is tied in with organizing the private sector. "The big, tough problems in air pollution won't be solved completely by in-house research," is the way Steigerwald puts it. "In the past two years, we've negotiated a lot of research contracts in our program to control sulfur dioxide emissions from fossil fuel combustion. Our experience has been gratifying.

"We find that when we define our problems specifically, we can tap the expertise of the private sector. We have approximately 30 contracts active in our sulfur program; they run the gamut from a \$5000 contract for minor consulting services to a \$1.5 million contract for design and construction of a demonstration plant for control of sulfur oxides. We hope for the same kind of participation in the big new programs being developed in the next few years."

In soliciting contracts, Steigerwald expects NCAPC to go more and more to publicizing its specific needs. "Some of our advertisements in the *Commerce Business Daily* have brought in replies from as many as 80 interested organizations. We request detailed proposals from the best, then make our choice from their proposals. But we still can—and do—fund unsolicited proposals, particularly those with unique or innovative ideas."

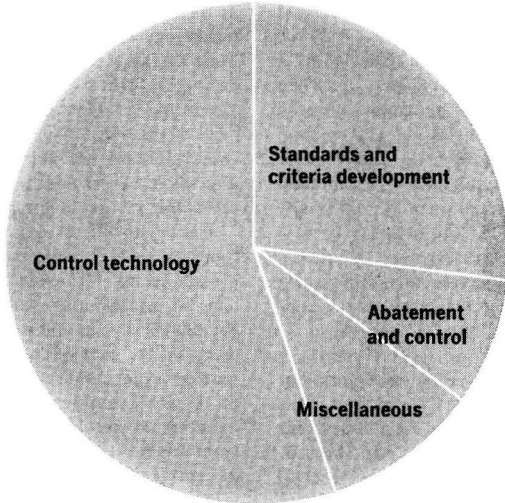
The contracts will originate principally with NCAPC's three major programs:

- Control technology research and development.
 - Standards and criteria development.
 - Abatement and control.
- Steigerwald sees the entire con-

National Center for Air Pollution Control

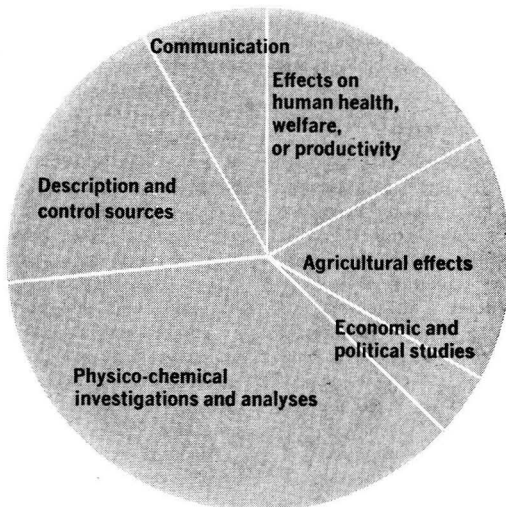
Estimates for fiscal year 1968

Research and development contracts by subject area



Total \$7.1 million

Research grants by subject area



Total \$8.6 million

tracts area growing faster than NCAPC in general, and the control technology area growing the fastest of all the contracts. Control technology is budgeted for close to \$4.4 million in the current fiscal year; of that, about \$3 million is going to study industrial processes as part of the search to find ways to minimize production of pollutants.

Sulfur oxides are getting the major attention at present, but NCAPC also wants to start doing the groundwork for the other pollutants it expects to attack with the increased expenditures anticipated for FY '69. Foremost among these pollutants slated for attack are the nitrogen oxides. Design of many types of combustion units will probably have to be modified to minimize nitrogen oxides production. From the larger sources, nitrogen may even be recovered.

Another major effort in control technology contracts will be on improving instruments for air sampling. "In these contracts, we hope to tap the technology developed by NASA and DOD to help solve air monitoring and source monitoring problems," Steigerwald says. Current monitoring stations leave much to be desired. NCAPC's director John T. Middleton put it this way recently at a meeting of the New Jersey Council for Research and Development: "The fact is that these sta-

Federal grants for air pollution research

Fiscal Year	Number	Millions of Dollars
1961	71	\$ 1.8
1962	78	1.9
1963	97	2.9
1964	107	3.9
1965	130	4.6
1966	149	5.3
1967	162	6.2
1968 (est.)	205	8.6
1969 (est.)	241	9.2
1970 (est.)	269	11.0
1971 (est.)	321	12.0
1972 (est.)	369	14.0
1973 (est.)	426	16.6

tions are too expensive. They require an air conditioned building, a supply of wet chemicals, and an operator. Surely, when we can analyze the dust on the moon, we can do better than this. What we need is a continuous monitor for the major pollutants, the size of a desk, needing no chemical supplies, easy to maintain, not requiring an operator, having telemetering capacity, and inexpensive. Can you make one? Can you make a thousand?"

Almost \$1 million in control technology contracts are due to be awarded for motor vehicle projects in FY '68. They will be for two general purposes:

- *Development of standard test methods.* "What we need here," says Steigerwald, "are base line data on emissions and driving habits. What are emissions for freeway driving versus stop-and-go driving, for example? From these, we can develop better standard laboratory methods, simple and reproducible, that will tell us what exhaust control devices will do and how they'll hold up in practice."

- *Development of new concepts to control vehicle emissions.* There is a continuing interest in development of new, more effective methods for control of hydrocarbons and carbon monoxide. Areas of emphasis will include control of particulate emissions, diesel odor, and oxides of nitrogen.

Pollution effects

A big new contract effort is scheduled this year on the economic effects of pollution. This area takes on increasing importance as the air pollution control program grows. Commenting on this, Steigerwald says, "One question frequently asked is 'How much damage would occur were there no control?' So we're setting out to determine in more detail damage functions for all major pollutants on all major receptors—people, plants, paints, fabrics."

In the health effects area, NCAPC plans to start studies on behavioral toxicology, an area as yet almost untouched. How do low levels of carbon monoxide affect a driver's responses? Diesel odor is now considered largely a nuisance, but what of the general well being of those affected?

NCAPC also wants to develop more data on the synergistic effects of a wide variety of things and pollution. Particularly important are data on infectious agents, as well as data on the effects of pollution on those whose defenses are lowered—the very young, the very old, the sick.

NCAPC this year initiates an effort that will take several years to complete: compiling an inventory of atmospheric emissions, industry by industry. Working jointly with industry, NCAPC wants to find out what each

industry puts out, how it is controlled, and at what cost.

Development of improved computer models to simulate urban atmospheres and control plans is another project NCAPC will push this year. Such models would permit control officials to evaluate the cost and effectiveness of alternative control strategies using computer gaming techniques.

More for training grants

The training grants and fellowship activity, too, are expanding. The initial appropriation in FY '63 was \$0.4 million. In FY '68 it is budgeted for \$3.2 million; this includes \$2.7 million for grants to institutions and \$0.5 million for fellowships to individuals.

Ten training grants were made in the first year of the program. The total number NCAPC now supports is 23. Carl Lindstrom, head of NCAPC's training program, hopes to be able to start three or four new programs with this year's funds. Training grant applications are evaluated with the help of the air pollution Training Advisory Committee.

Meanwhile, Lindstrom is going to re-evaluate the existing programs. "In looking at these programs, we will have to consider a couple of things—how well a program is doing the job it set out to do, and how this job fits into our national program. We anticipate being able to maintain particularly strong programs even beyond the seven years that has been set as the maximum project period. We may find that some, with greater support, could become really strong programs. And, frankly, there is a chance we will have to phase out some of the programs. We feel we must target our resources according to priority need so we can build a stronger national training program."

Another job Lindstrom's group will be tackling in the coming year is a survey to determine the training required to meet the growing manpower needs. This survey was spelled out in the Air Quality Act of 1967; it is due to be presented to Congress by July 1969.

NCAPC research appropriations

	FY '68	FY '67 (millions of dollars)	FY '66
In-house research	\$ 8.7	\$ 6.6	\$ 5.3
Research grants	8.6	6.2	5.3
Research contracts			
Federal	2.5	1.8	1.5
Nonfederal	7.1 ^a	7.8	2.1
Training programs			
Grants and fellowships	3.2	2.5	1.7
Direct training	1.4	0.7	0.6
Grants for nonfederal pollution control programs	20.3	7.0	5.0
Survey and demonstration grants	2.0	2.0	1.9
Other technical assistance	5.2	2.6	1.5
Abatement program	3.8	1.8	1.3
Control of motor vehicle emissions	1.4	1.1	0.5
Totals	\$64.2	\$40.1	\$26.7

^a Does not include supplemental request

Aircraft noise

unrelenting, unremitting, intolerable

"... This country . . . can no longer afford to turn a deaf ear to the 20th century problems of noise."

Most of the witnesses heard so far at hearings on proposed legislation for aircraft noise control favor some form of legislation. But what form the legislation should take is not so obvious. Nor, according to witnesses at the hearings, which began in mid-November, are any solutions imminent for instantaneous control and abatement of aircraft noise.

The problem of aircraft noise exists at every major airport in the world. It became aggravated in 1958 with the introduction of the first jets and increased travel loads. And it will continue to be bothersome until legislation and technology, either separately or collectively, control, reduce, or eliminate the noise. "For those who must live surrounded by the din of normal city life, the whine of an increasing number of jet aircraft can make life almost intolerable for many who live below or near the path of flight," said Alan S. Boyd, Secretary of Transportation.

Although the problem of noise is not unique to O'Hare, the Chicago airport is of special interest because of its size and aircraft volume.

"O'Hare International Airport, the busiest airport in the world, routinely handles more than 1200 flight operations daily," according to Rep. Roman C. Pucinski (D.-Ill.) whose congressional district borders O'Hare.

At the hearings early this month before the House Subcommittee on Transportation and Aeronautics, Congressman Pucinski said, "On an average day, O'Hare copes with a minimum of 1000 jet operations. During peak

travel hours, this means a jet into or out of O'Hare at a rate of one every 40 seconds. To the 23,500,000 passengers who use the facilities at O'Hare each year, this usually means prompt and efficient service. To the hundreds of thousands of persons clustered in homes, hospitals, and apartment buildings within a 15 mile radius of the airport, it means far more. It means noise—unrelenting, unremitting, intolerable noise."

"Students have their classroom dis-

cussions shattered. Telephone conversations cease. Television viewing is impossible. Countless thousands of my constituents spend endless nights unable to obtain a moment's respite from the unbearable noise. Patients in hospitals must endure the constant roar and scream of jet engines. Elderly people in nursing homes have nowhere to flee the boom and whine of tidal waves of sound," Pucinski continued.

At the hearings witnesses testified to a very real concern about the wors-

Interior launches sonic boom study

This month a broad study of sonic booms and other noise pollution has been initiated by Stewart L. Udall, Secretary of Interior. Although the effects of sonic booms on parklands, man-made structures, and the like have been documented to some extent, the overall effect of sonic booms on our environment is not known.

Included in the list of prominent scientists who participated in the recent meeting for this study are the following:

John C. Calhoun, vice president, Texas A & M College

H. Stanley Bennett, University of Chicago

Rene Dubos, Rockefeller Institute

Joseph L. Fisher, president, Resources for the Future, Inc.

Roger Revelle, Harvard Center for Population Studies

Athelstan F. Spilhaus, president, Franklin Institute

In addition, other prominent scientists who, according to Interior, will join in the study are the following:

Harrison Brown, National Academy of Sciences

M. Barry Commoner, Washington University (Mo.)

Leonard Duhl, special assistant to the Secretary of Housing and Urban Development

Gordon J. MacDonald, executive vice president, Institute for Defense Analyses

Milner B. Schaefer, science advisor to the Secretary of Interior

John L. Buckley, Office of Ecology, Department of Interior

S. Fred Singer, deputy assistant secretary for Water Pollution Control

The report of this study will become available in midyear 1968.

ening noise levels that are coming simply through the growth of aviation.

"Aviation is still a fast growing industry in the U.S. In 1960 only 16 airports serviced jets; today there are 150 airports that receive jets," said E. Thomas Burnard, executive vice-president of the Airport Operators Council International, a voluntary, nonprofit association of more than 120 organizations and public agencies which own or operate more than 500 public airports in the 50 states and in other countries around the world. "It is estimated that nearly 350 airports will service jets in 1970 and more than 500 airports will service jets by 1975," he added.

A correlation in terms of total aircraft and aircraft movements yields the following table:

Year	Jet aircraft	Aircraft movements (millions)
1960	224 (turbojets)	26
1966	896	47.8
1974 (est.)	2240	95.6

Source: E. Thomas Burnard in a statement before House Subcommittee on Transportation and Aeronautics

Attesting to the public awareness of the relentless noise, citizens in the U.S. have urged their legislators to attack the problem. "The widespread interest

in Congress is evident by the 29 bills pending in the house, all seeking to solve the aircraft noise problem," Burnard noted.

Congressman Theodore R. Kupferman (R.-N.Y.) warned, "The idea that noise is a necessary price of industrial and economic progress is as antiquated as the belief that contaminated waters and a polluted atmosphere must accompany civilization's advances. . . This country, and the entire world for that matter, can no longer afford to turn a deaf ear to the 20th century problems of noise."

The three essential component parts of aircraft noise are as follows:

- Reducing aircraft noise at the source.
- Developing noise abatement flight techniques.
- Fostering the compatible use of land adjacent to the airports.

The three, which are additive, go hand in hand. But H.R. 3400, introduced by Rep. Harley O. Staggers (D.-W.Va.) provides for control of noise at the source—the aircraft.

Features of H.R. 3400

H.R. 3400 would authorize federal regulation and control of jet noise.

Features of other noise bills

As of the latest count some 30 bills, endorsed by different legislators, are being considered by the House Subcommittee on Transportation and Aeronautics, under the chairmanship of Rep. Samuel N. Friedel (D.-Md.). The proposed 30 bills amend 49 U.S.C. 1301 and 49 U.S.C. 1421-30 and are essentially alike. Associated bills include HR. 13846 (introduced by Rep. Donald M. Fraser (D. Minn.)); H.R. 7266 (introduced by Rep. Joseph P. Addabbo (D.-N.Y.)); H.R. 2819 and H.R. 2820 (introduced by Rep. Theodore R. Kupferman (R.-N.Y.)); H. R. 14146 (introduced by Rep. Samuel N. Friedel (D.-Md.)); and others.

Congressmen Fraser's H.R. 13846 and Kupferman's H.R. 2819 would establish an Office of Noise Control in the Office of the Surgeon General. According to Kupferman, "The primary function of the Office of Noise Control would be to act as a national clearing house for general and specific noise information, and could, upon request, disseminate the wealth of its accumulated knowledge to the states and local governments to help them control noise at its point of origin." The proposed legislation is designed to make the Office of Noise Control responsible for controlling other types of noise such as those associated with traffic, subways, and the like.

One title of Congressman Fraser's H.R. 13846 permits certain FAA reimbursements to airlines that conform to proposed federal noise regulations. This includes the following:

- As much as a 30% reimbursement of the cost of modifying aircraft to conform with federal noise regulations.
- As much as 30% reimbursement to airports making the changes.
- A 90% reimbursement for cities acquiring land near the airports or along flight lines in an effort to reduce the effects of noise.

Stephen's substitute bill, endorsed by ATAA, would limit the application of noise measurement standards to aircraft type certification.

Basically, the bill gives federal officials a new type of regulatory power of noise—in addition to their present regulatory power in the area of safety. The bill would amend the Federal Aviation Act of 1958 to give the Secretary of Transportation power to prescribe aircraft noise abatement and control measures, including sonic booms.

The effect of H.R. 3400 would be to include noise control and safety as considerations in issuing or revoking aircraft certificates. The federal authority for certification with respect to safety would continue to be vested in the FAA Administrator. But the new federal authority for certification with respect to noise would be vested in the Secretary of Transportation.

Support for control

Support for aircraft noise control resides not only in the myriad, regional groups of irate citizens and most of the witnesses heard so far, but also in the high governmental offices.

"We regard enactment of the bill (H.R. 3400) as a most important step in our efforts to alleviate the noise problem," said Gen. William F. McKee, FAA Administrator, before the House Interstate and Foreign Commerce Committee in early March. "Perhaps of greatest significance is the authority it provides for the application of noise standards in the certification of aircraft. We believe this authority is essential to the achievement of any real progress in the reduction of aircraft noise at its source," he continued.

Also, Dr. Donald F. Hornig, Presidential Science Adviser, noted, "Concerning noise standards: I regard the passage of legislation authorizing the Secretary of Transportation to set aircraft noise standards to be critically important. In the long run it is the only way I can see to protect the public interest in this respect."

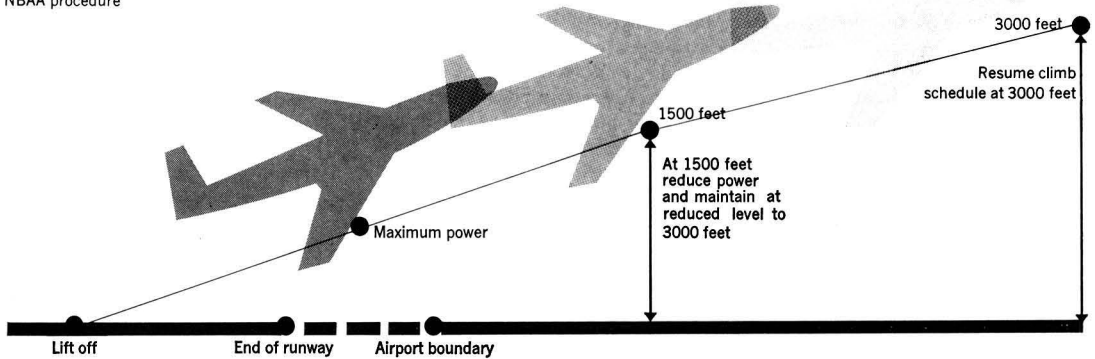
Contradictory solutions

Potential solutions for the 20th century problems of aircraft safety and noise contradict one another. For example, the safest jet approach is a long low approach. It is also the most noisy.

Several existing techniques reduce the noise but not to such a low decibel level to be socially acceptable or en-

NBAA Jet Aircraft Noise Abatement Department Procedures

NBAA procedure



endurable by the public. These have included efforts on the part of the airlines to reduce noise at the source, flight patterns, land use, and control procedures.

Climb-out profiles

The FAA, with assistance from NASA, tested and developed climb-out profiles which significantly reduce aircraft noises prior to the creation of Department of Transportation, according to DOT Secretary, Alan S. Boyd. The climb-out profile is a three segment noise abatement takeoff profile which substantially reduces noise in terms of flyover time.

The profile consists of, first, a maximum takeoff climb to a specific altitude; second, a reduction in power and a lower rate of climb to an altitude where the generation of noise is not as annoying; and, third, thereafter a resumption in the normal climb procedure. These climb-out profiles were acceptable to the industry and consistent with the safe and reasonable operation of the aircraft.

Glide slopes for landing

The aviation community, by way of its National Aircraft Noise Abatement Council (NANAC), was also instrumental in securing wide acceptance and application of the increased visual glide slope, converted to a six degree landing approach from the former three degree approach.

NANAC, organized in early 1960, represents the aviation community's move to coordinate nationwide planning and procedures for the reduction of aircraft noise. The organization comprises the Aerospace Industries As-

sociation (representing the principal airframe and engine manufacturers); Air Line Pilots Association (comprising 15,000 licensed commercial airplane pilots); Air Transport Association of America (consisting of the 36 scheduled certificated route carriers of the U.S.); Airport Operators Council International (consisting of 107 member organizations representing more than 500 public airports in the 50 states of the U.S. and other countries); and American Association of Airport Executives (representing the managers and directors of more than 400 airports throughout the U.S.).

Although the primary concern of those interested in noise reduction generally centers on a reduction of noise at takeoff, DOT is investigating noise abatement approach profiles. Presently in the experimental phase, the approach profile calls for an increased rate of descent. Unfortunately, the increased decent rate is inherently less safe than existing approach procedures.

"Aviation is not in and of itself inherently dangerous. But like the sea, it is terribly unforgiving of mistakes, and for this reason we, the pilots, will continue to resist the application of operational noise abatement techniques which inhibit the safety margins we find to be necessary," cautioned Capt. Vernon C. Brunelle at this month's hearings.

Existing procedures

In spite of the fact that some form of noise abatement and control procedure is in operation at nearly every U.S. airport, the aircraft noise problem remains unsolved.

One of the best known (at least to

those involved in aircraft noise control) and probably the best example of existing noise control procedures in effect at airports is the 112 PNdb Rule of the Port of New York Authority. This rule is enforced against carriers operating at the New York airports, under the right of the Port Authority, as operator of its airports, to control the conditions of airport use. Indeed, it was only after noise measurements had been made on the Soviet Il-62 at Dulles International Airport that the Il-62 was permitted to land and take off from Kennedy International Airport.

The noise abatement procedures in effect at Washington National Airport serves as yet another example of moderately effective noise control procedure. The procedure specifies the following:

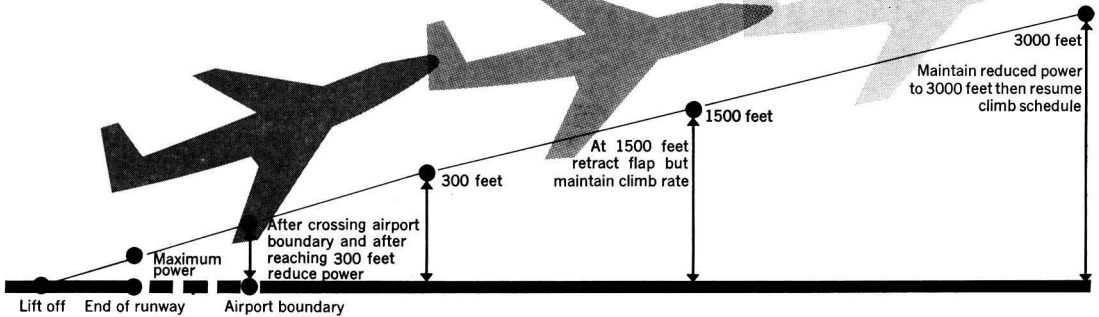
- A prescribed course over the Potomac River on departure and arrival.
- A thrust-control departure procedure comprising instructions to reduce power at 1500 feet, climb at 500 feet per minute to 3000 feet, and continue normally thereafter.

Predicting aircraft noise

DOT is developing a computerized method for predicting aircraft noise exposure at airports, according to Secretary Boyd. Already the methodology has been applied to three principal, international airports—Kennedy, O'Hare, and Los Angeles; and plans are under way to apply it to 29 additional airports. When the model is available, it will enable the Department of House and Urban Development to inventory land use at all 32 of the airports. The resulting noise exposure

Close-in procedure

(Use when the noise sensitive areas are within 10,000 feet of brake release)



forecast-land use inventory in the vicinity of these airports will then be used to achieve compatible land use and to assist planners in developing communities free from aircraft noises.

NANAC is also taking a systems approach to the total airport-environment complex, according to John E. Stephen, general counsel for Air Transport Association of America. "The output of the operations research model will be the optimum combination of aircraft configuration, flight procedures, and compatible land measures, to produce maximum noise abatement for expenditures made," he claimed. Also, he noted, a \$200,000 contract for the performance of Phase I has been let by the Aerospace Industrial Association. Stephen expects completion of Phase I within the next six to eight months. If Phase I establishes a feasible computer model, then Phase II will be carried out at a further cost to the industry of approximately \$1 million, he predicted.

Noise at Washington

Last year FAA measured noise at Washington National Airport at 846 data points for departing aircraft and another 231 for arriving aircraft. The data were recorded by six two-man teams of electronic technicians operating from mobile units. The measurements include noises produced by jets such as Boeing 727, Douglas DC-9, BAC-111, and Caravelle; piston aircraft such as Douglas DC-3, DC-6, and DC-7, Lockheed Constellation, Convair 440, Martin 202 and 404; and turboprop aircraft such as the Lockheed Electra, Vickers Viscount, Fairchild F-27, and Convair 580. During

the measurements, the Washington National noise abatement plan was in effect.

The ambient sound pressure level was 60 decibels. About 25% of the aircraft recordings were masked by surface noises and did not exceed the ambient neighborhood sound pressure levels. Significantly, arriving aircraft produced less noise than departing aircraft. As a category, departing aircraft of the intermediary range type delivered less noise to the community than did old four-engine piston-type air transports.

For example, departing four-engine piston aircraft delivered, as a group, higher noise than did jet aircraft, both in terms of overall sound pressure levels and perceived noise levels. As a total group, these aircraft delivered to the sites a greater sound pressure level in decibels (90 db.) than did the jet aircraft (87 db.). The computed mean PNdb. was higher for all four-engine piston aircraft (94 PNdb.) than for all jets (92 PNdb.).

A typical departing Boeing 727, one half mile from the end of the runway and at an altitude of approximately 400 feet, produced ground noise levels of 112 db. and 116 PNdb. On the average, Electras produced noise levels of 103 db. and 103 PNdb. Viscounts, noise levels of 106 db. and 112 PNdb.

Pilots' position

"Washington National Airport is no doubt the most convenient downtown airport of any major capital in the world. Yet the ridiculous noise abatement procedures which were adopted for use at Washington National—such as following the narrow Potomac River

in its snake track course northwestward—were not only operationally unfeasible, but, additionally, provided more noise for the persons living in the immediate vicinity of the river," complained Capt. Vernon C. Brunelle, a line pilot for one of the nation's certified air carriers and chairman of the Air Line Pilots Association's (ALPA) standing committee on noise abatement. ALPA has more than 23,000 air line flight deck crew members and has been involved in aircraft noise abatement for the past 20 years. "The adoption of a noise maximum for airworthiness certificates, however, is not in and of itself the total answer," Capt. Brunelle observed.

Subjective reactions

The oft-used term aircraft noise is seldom defined, according to Stephen. "But essentially it refers to complaints," he noted. "If anything has been learned from the exhaustive studies made of noise complaints, it is that—no matter how much the noise level is reduced—there will remain an in-eradicable hard-core group of complainants. Both British and American studies indicate that this constant hard-core group of complainants constitutes about 30% of the population near airports. In this sense, the aircraft noise problem will probably never be solved," Stephen continued.

Support for the above outlook has also been demonstrated in studies of the Society of Automotive Engineers. SAE committee A-21, comprising numerous representatives from the aviation industry, demonstrated that the overall sound pressure levels of various aircraft noises do not correlate well

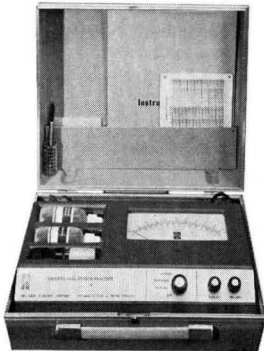
Pollution control calls for accurate data on water quality

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with subjective reaction to these noises. At the recent 22nd meeting of A-21 at Orly Airport, a study group told of its efforts, using panels of subjects, to develop a method for the measurements of subjective reaction to aircraft flyover noise.

This study on the effects of pure tones in the spectrum compared calculated pure tone corrections with pure tone corrections determined by direct panel ratings of noise flyover cycles. The results show that for a series of takeoff and landing flyovers of Boeing 707 and DC-8 aircraft, the calculated pure tone corrections were on the order of one half of the actual values. The work is continuing to obtain information on the confidence level of the results.

Cost of reducing noise

The airlines' cost for noise control and abatement are masked because such costs are usually included in other airline costs. Although the actual cost of noise control is not readily obvious, some assessment of it was provided by Air Transport Association of America's Stephen's testimony.

He said, "Predating the airline's introduction of the turbofan engines more than \$50 million was spent on R&D to perfect in-flight noise suppressors, for the first jet engines. By 1962, the airlines had installed suppressors on 325 jet aircraft at a cost of \$250,000—a total of \$73 million for the industry. By 1965, the airlines had invested nearly \$150 million in the installation of noise suppressors. In addition to this original cost, it cost the airlines approximately \$10,000 per aircraft per month to operate the suppressors, because of increased weight and drag and reduced speed—a total for the industry of \$36 million per year," Stephen revealed.

Industry's contribution

Continuing his discussion, Stephen noted that the airlines have made considerable investments to alleviate the noise. "Specifically designed and developed for the express purpose of reducing noise, the fanjet engine was substituted for the turbojet engines in aircraft orders of all U.S. airlines. One airline even replaced all turbojet engines in its existing aircraft at a cost of

\$1 million per aircraft," Stephen said.

Stephen also observed that the airlines serving Kennedy International Airport have agreed to pay more than \$11 million at the airport alone for required runway extensions—not for operational reasons, but solely for noise abatement. One airline, alone, estimated its cost at more than \$4 million per year. Another carrier estimates that its economic penalty for complying equalled 5% of the annual gross operating revenue of each of its affected aircraft operated at Kennedy International.

Technological promises

As part of the national program on aircraft noise, established within the Office of Science and Technology in 1966, the National Aeronautics and Space Agency has some technological programs directed toward reduction of aircraft noise. NASA's short-range retrofit program will incorporate sound absorption techniques into engine nacelles. According to NASA, this technology promises a 6 db. noise reduction and should be ready late next year.

Its longer-range retrofit program aims at reducing the noise an additional 9 db., when the technology becomes available in late 1969. NASA's quiet engine program is an attempt to design an entirely new engine that would afford a 20 db. reduction from present noise levels—perhaps by 1972.

NASA has demonstrated that it is possible to reduce noise levels by operating commercial and military aircrafts having turbofan engines at a bypass ratio of 5:1 or 8:1 (the bypass is the ratio of bypass cold air to combustion gas). This possibility has an added disadvantage. Now the whine of the turbines prevails to such an extent that the remaining overall noise is still annoying and objectionable.

Limits of proposed legislation

Testifying in opposition to H.R. 3400, Stephen called attention to several deficiencies in the basic legislation and offered a substitute bill for consideration by the House subcommittee. Stephen said that ATAA does not support the bill's proposed authority for noise certification of airmen, airports, air carriers, and air agencies.

He also pointed out the following additional inconsistencies:

- H.R. 3400 is of little consequence in the attack upon the problem of compatible land use adjacent to airports. (Many noise abatement experts believe that solving airport noise problems in such areas is the most promising approach to the solution of the overall airport noise problem.)

- H.R. 3400 is concerned with only the reduction of aircraft noise at the source.

- Confinement of the noise problem to civil, not public, aircraft only. (Stephen says, "Among the public aircraft not covered by H.R. 3400 are military aircraft. Military jets are probably the noisiest of all aircraft, and they are substantial contributors to the aircraft noise problem." The bill covers only civil aircraft, which do not create sonic booms, but excludes military aircraft, which do. This anomalous situation should be corrected, Stephen says.)

- H.R. 3400 is deficient in its review and hearing procedure after certification. (Under the present safety certification procedure, certification cannot be amended, modified, suspended, or revoked without giving the certificated holder an opportunity to answer the charges made by FAA. H.R. 3400 does not afford the certificate holder the right to answer the DOT Administrator and receive a hearing.)

Private business aircraft

Presently, there are about 500 turbo-jet business aircraft in operation, according to Mr. John P. Woods, spokesman for the National Business Aircraft Association (NBAA). The association comprises nearly 800 U.S. corporations that own and operate aircraft Association (NBAA). The association publishes a noise abatement program in mid-November. The NBAA program includes the following:

- Flight procedures.
- Air traffic control procedures.
- Jet engine design.
- Land use.

According to Woods, NBAA staff specialist on aircraft noise abatement, NBAA does not believe that legislative action is required in the areas of

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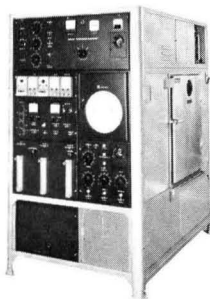
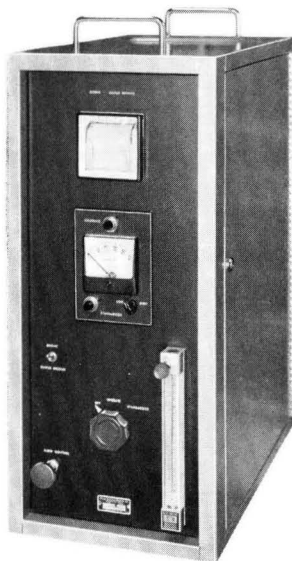
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flight and air traffic control procedures. "Unless land use planning in the vicinity of the airports is completed and effected, all other noise abatement efforts will fail to produce the desired results," he warned.

"Although flight procedures are only one of several parts of an effective noise abatement program, they are the only part over which NBAA members, as aircraft operators, have direct control," he continued. "NBAA urges their pilots to utilize these flight procedures whenever, in their judgment, beneficial community relations will result," Woods concluded.

Assessment

Factors such as frequency of operation, time of day, ambient noise level, and the like—all in addition to aircraft noise levels per se, complicate the total noise picture with respect to community reaction.

"Future generations will likely be better adjusted to aircraft noise," Capt. Brunelle observed. "Only four generations ago, our forefathers resisted the steam locomotive, and a bit later the automobile, as an unnecessary noise adjunct to the transportation system," he remarked.

In the same vein is Boyd's remark last month. "I do not believe there will ever be such a thing as a quiet airplane," Boyd said. "Despite our far longer experience with the problems of truck noise and railroad noise, we have not been able to produce quiet vehicles in those modes of transportation. But I am convinced that we will be able, by technological and regulatory means, to reduce the impact of aircraft noise exposure for the majority of Americans who are now, or will potentially be, subject to excessive aircraft noise exposure," Boyd concluded.

"In all events, certification for noise will afford no magic results," Stephen noted. "Certification can yield no more than technology can produce." And he added that the extent of noise reduction through technological improvement of aircraft and engines is limited by our present knowledge.

No short term or instantaneous areas of substantial improvement are likely. On the contrary, at most locations aircraft noise nuisance will become even more aggravated before the condition starts to improve.

QUOTE...

We will no longer abide pollution

To conserve our resources and to maintain our capability to meet not only our domestic but also our international food commitments, we must move ahead boldly and decisively to halt the sweeping deterioration of our environment and the senseless squandering of our natural resources.

No nation on earth has been more abundantly blessed with bountiful natural resources than the United States. No nation on earth has abused and squandered its resources at such an astonishing pace as we have. Our country was once covered with beautiful forests and rolling prairies; many of our states were dotted with beautiful fresh, clear lakes, and a myriad of streams meandered across the land.

Much of this is gone. A major portion of our forests was carelessly if not wantonly harvested by the lumberman, much of our fertile soil was allowed to erode away, and our fabled, priceless streams and lakes are rapidly becoming polluted. Even the quality of our air has deteriorated to the point that metropolitan areas like New York and Los Angeles are constantly flirting with a major environmental disaster.

Perhaps the best known, and certainly the most widely discussed, resource problem is that of water pollution. Every major river system in this country is polluted to some degree—most of them seriously. We have all stood by and watched the virtual destruction of Lake Erie and are now seeing Lake Michigan in the early stages of its death throes. Municipalities and industries along the shores of these lakes and on the rivers which feed into them are dumping each day millions of gallons of untreated wastes into their waters.

This nation is losing the equivalent of 400,000 acres of good land each year from erosion and other forms of soil deterioration. Flood damages in upstream watershed areas still amount to \$1 billion every year. It is estimated that the accumulation of sediments in reservoirs and ponds throughout the country totals at least 850,000 acre feet annually. Each year it costs us an estimated \$250 million to remove soil deposited in stream channels, harbors, and reservoirs.

We are told that air pollution does some \$12 billion worth of damage each year. Damages to crops alone amount to about \$325 million a year, while depressed growth of livestock due to air pollution costs an estimated \$175 million a year.

Today, we have at our disposal the technology required to stem the tide of pollution in this country. We know how to improve the quality of our water and our air.

All of this is not enough. If we are ever going to win this war against the deterioration of our environment, we must mount a massive effort that has the support of everyone from the citizen on the street to the local, state, and federal officials right up to the White House. The decision must be made at all levels of government that we will no longer abide pollution in any way.

GAYLORD NELSON

U. S. Senator (D.-Wis.) at the annual meeting of the American Society of Agronomy, Washington, D.C., Nov. 7, 1967

Telemetered Air Quality Network Helps Chicago

Automatic sensing and relaying of environmental data around the city help to create an effective level of awareness in responsible public officials and the public

Austin N. Heller, New York City Department of Air Pollution Control, and Samuel G. Booras, Chicago Department of Air Pollution Control

A brief history of Chicago's Department of Air Pollution Control is necessary in order to view its Telemetered Air Monitoring Program in proper perspective.

The nation's pioneer municipal ordinance against dense smoke emissions from industrial and commercial establishments was enacted in Chicago in 1881. A separate city department was organized in 1907, when permits were first required for the installation of fuel-burning equipment. Ten years later the department codified the first definitions concerning the use of high and low volatile coals. In 1938 the nation's first large-scale program for the inspection of fuel and refuse burning equipment was inaugurated.

Scientific advancement in the areas of medical knowledge and technology has greatly increased over the years. As knowledge about the nature, cause, effects, and control of air contaminants became more refined, so, too, did the city's efforts to utilize the information.

Recognizing the urgency of combatting the increasing problem of air contamination in a large industrial complex, Chicago reorganized the original Department of Smoke Abatement in 1952, renaming it the Department of Air Pollution Control. The new name was indicative of a widening of the department's jurisdiction and reflective of its expanded activities in the monitoring and analysis of various pollutants.

A landmark in the advancement of the cause for cleaner air in Chicago was the enactment of a more comprehensive municipal ordinance in 1958, which became effective in May 1959. The new ordinance regulates industrial, manufacturing, and chemical processes; steam and diesel locomotives; the handling, storage, and transportation of materials; open burning; the sale and use of high volatile fuels; and the sale of refuse-burning equipment.

Table I. Some statistics on the concentrations of three major pollutants in Chicago (1966)

Period	Number of observations	Average	Average lows	Average highs
Sulfur dioxide (p.p.m.)				
Annual	1766	0.053	0.009	0.143
Tuesdays	868	0.055	0.009	0.143
Thursdays	898	0.050	0.010	0.143
Winter	443	0.098	0.017	0.242
Spring	463	0.051	0.009	0.145
Summer	460	0.021	0.004	0.057
Fall	400	0.043	0.008	0.116
Suspended particulates ($\mu\text{g./m.}^3$)				
Annual	3059	144	73	270
Sundays	1008	139	73	277
Tuesdays	1020	148	72	273
Thursdays	1031	146	75	261
Winter	749	151	64	292
Spring	803	143	69	274
Summer	800	137	80	245
Fall	707	147	80	272
Total dustfall (tons/square mile/month)				
Annual	237	36.9	17.7	64.2
Winter	60	43.8	19.9	71.8
Spring	59	45.2	23.4	81.5
Summer	60	24.6	14.2	39.9
Fall	58	34.5	13.1	63.8

Table II. Summary of suspended particulates in Chicago

December 1963–November 1964

	Station	Number of samples	X	Med.	Correlation
I	A 1	134	90	76	r 3.1 .98
	C 3	136	95	85	
II	B 2	138	128	120	r20.2 .90
	T 20	132	118	110	
III	D 4	131	175	155	r 4.6 .85
	F 6	130	173	150	r 4.7 .90
	G 7	128	158	150	r 4.9 .71
	I 9	122	154	140	r 6.9 .73
IV	L 12	134	170	175	r12.15 .53
	J 10	135	140	125	r10.11 .84
V	K 11	132	123	115	r11.14 .85
	N 14	133	116	115	
	O 15	138	126	118	
	P 16	126	121	100	
VI	Q 17	137	136	115	r17.18 .77
	R 18	135	120	100	
VII	H 8	134	136	125	
VIII	M 13	131	111	100	
	All Stations	132			

Subsequent amendments in 1962 against open burning of debris from demolition (effective in July 1964) and in 1963 against all forms of polluting emissions have strengthened the control of noxious atmospheric contaminants. Moreover, the exclusion of certain industries from the norms of the ordinance—for example, coffee-roasting and steel industries—was corrected when the “research clause” was deleted in the revised code.

In October 1962, the Department of Air Pollution Control entered a technical assistance project with the U.S. Public Health Service to plan a dynamic air management program for the city. The time had arrived once more for Chicago to broaden its concept of air pollution control and to devise more modern methods of assuring clean air for its citizens.

The outgrowth of the planning was a five-year air resource management program, inaugurated in 1963, consisting of a comprehensive eight-point program involving an integrated team of scientists, engineers, technicians, enforcement officers, and administrative personnel. The eight objectives constituting the program summarize the operations of the department:

- Provide the public with a thorough understanding of air pollution.
- Determine the effect of air pollution on the health of Chicagoans.
- Inventory all potential sources of air pollution.
- Improve the monitoring of pollutants.

- Improve the analysis of pollutants.
- Determine the transport and dispersion of pollutants.
- Recommend effective legislation for air pollution control.
- Increase metropolitan activity in the control of air pollution.

This, then, is the basis for the department's activities in the organization and operation of a total air monitoring program that led to the establishment of its telemetered network.

Site selection

After a careful review of the geographic distribution of the population, commercial and industrial operations, and meteorological considerations, a total of 20 sampling sites was selected to provide a synoptic profile of the atmosphere. But site selection is more complicated than simply saying it was done. Let us, then, consider the methodology of site selection.

The population of Chicago is concentrated on the lakeshore and follows an east-west line through the center of the city; the concentration of older multiple-dwelling units follows a similar pattern.

In view of possible site locations, use was made of a study conducted by the Department of City Planning entitled “Locational Patterns of Manufacturing Industries.” This study indicated the distribution of population and areas where industrial establishments are located in the city (Figure 1).

Meteorological conditions and site choices

An important consideration in defining the meteorological effects of an area is local topography. Chicago is eight to 10 miles wide and extends north-south for about 25 miles along the western shore near the southern end of Lake Michigan. The entire area is essentially a level plain slightly higher than the surface of Lake Michigan, which is 580 feet above mean sea-level.

The surface features have too slight an effect on air movement to cause any noticeable drainage or channeling of winds. The city's buildings are a much more prominent landscape feature than its topography, particularly true in the central business area with its many high-rises reaching 500–600 feet above street surfaces.

An important effect of local winds is caused by temperature differences between Lake Michigan and the adjacent land area. Not infrequently parts of the city near the lakeshore are cooled by easterly breezes off the lake, extending several miles on shore.

Any reasonable geographic distribution of sampling sites would provide the desired synoptic profile of the atmosphere over the city because there are no significant topographical features other than buildings and because the lakefront parallels the eastern border of the city.

Another significant factor in site selection was uniformity of elevation for sampling stations. To attain the optimum level of 50 to 60 feet above ground level, public schools, most of which are three or four stories high, were selected. The 358 structures available afforded an adequate horizontal selection, ready accessibility, security for our equipment as well as cooperation for a public agency.

One final determination in site selection aside from population density, locations of industrial and commercial firms, and meteorological conditions was an adequate number of sampling stations. An analysis of Chicago's 220 square miles indicated that 19 sites would be sufficient to establish a city-wide profile of air pollution. An additional site was selected in a predominantly rural area outside the

Table III. Monthly averages of sulfur dioxide in Chicago

Month	1964	1965	1966
January	0.13	0.09	0.11
February	0.10	0.06	0.11
March	0.07	0.06	0.07
April	0.06	0.06	0.05
May	0.03	0.03	0.04
June	0.03	0.02	0.03
July	0.03	0.02	0.01
August	0.02	0.03	0.02
September	0.04	0.03	0.03
October	0.06	0.05	0.05
November	0.06	0.07	0.05
December	0.10	0.08	0.07
Averages	0.06	0.05	0.05

Source: Department of Air Pollution Control, Technical Services Division

Population distribution patterns

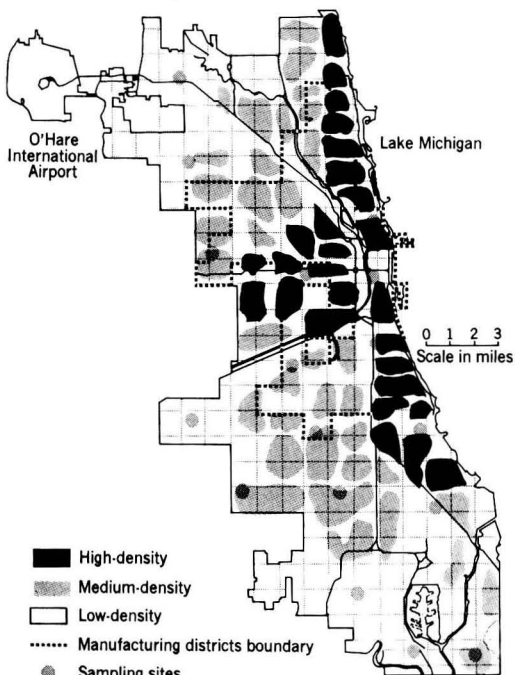


Figure 1

Suspended particulate monitoring network (1966)

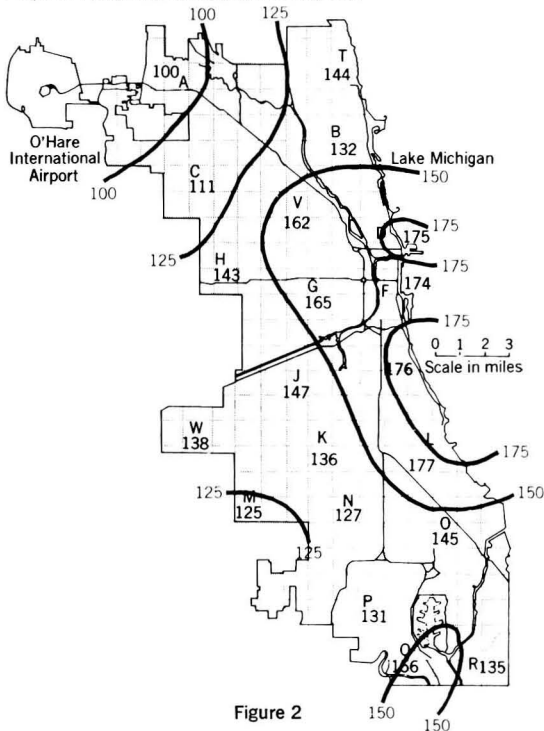


Figure 2

Sulfur dioxide monitoring network (1966)

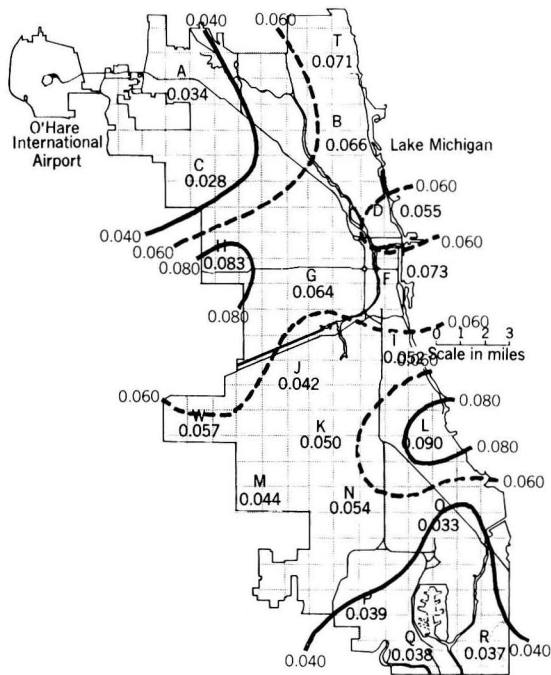


Figure 3

Dustfall monitoring network (1966)

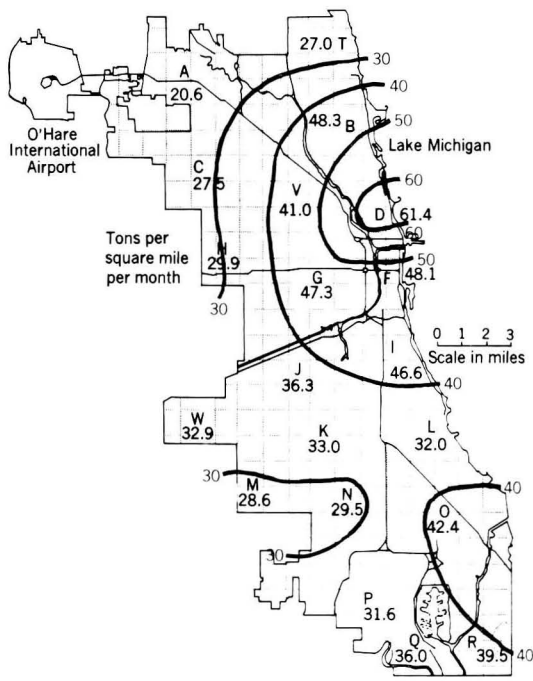


Figure 4

Chicago's air sampling network

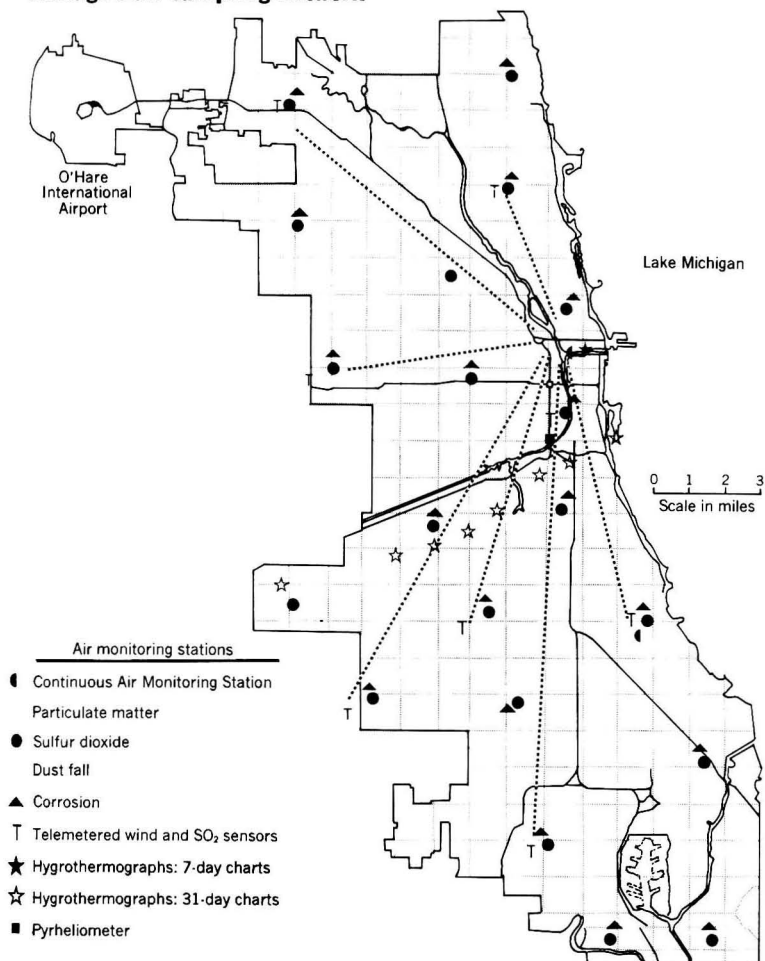


Figure 5

city to determine the concentration of pollutants in a relatively clean area. Argonne National Laboratory was selected as this control site (Figure 5).

Since the activation of the 20 air monitoring stations in 1963, data have been provided on three major types of pollution—sulfur dioxide, suspended particulates, and dustfall.

Testing and sampling procedures

Sampling for suspended particulate concentrations consists of passing about 2400 cubic meters of air (or 84,000 cubic feet) through a 7-inch \times 9-inch area of flash-fired glass mat filter in a 24-hour period. Starting at midnight of the previous day, sampling is conducted on Sundays, Tuesdays, and Thursdays. The filters are

weighed before and after the sampling. Before each weighing the filters are stored at constant temperature and humidity for 16 hours. Filters are analyzed at the department's laboratory for concentrations of sulfate, nitrates, and metals. Concentrations are reported in micrograms per cubic meter ($\mu\text{g./m.}^3$).

The West-Gaeke analytical procedure is used for sulfur dioxide. It consists of bubbling air through 20 ml. of 0.10 normal sodium tetrachloromercurate solution for 24 hours. Samples are collected in duplicates on Tuesdays and Thursdays. On the average, 240 liters of air are passed through each bubbler in a 24-hour period. However, the exact volume of air sampled is determined during

the calibration procedures used in maintaining the sampling equipment.

Dustfall measurements are made by collecting the settled material in glass jars containing 1–3 inches of water. The jars are 6 inches in diameter ($1/5$ square foot area) and are set approximately 54 inches above roof level at the test site. The jars are exposed for 25–31 days and then replaced with new ones while the contents of the exposed jars are being analyzed. The sample is analyzed for materials soluble and insoluble in the collecting medium, and further sub-classifications are made for chloroform soluble, hydrochloric acid soluble, combustible, and inert components.

Suspended particulate matter

Of the 20 stations originally planned for suspended particulate sampling, only 18 provided data, as scheduled, from March 1963 through December 1963. Uninterrupted data have been available on suspended particulates throughout (1964–66). Concentrations have been recorded on punch cards and analyzed statistically by computer.

A detailed summary of all suspended particulate data for 1966 is presented in Table I. This summary contains the inclusive dates for which data were obtained from each station as well as the number of samples and cumulative frequency distributions giving the per cent of all samples equal to or less than stated concentrations in $\mu\text{g./m.}^3$. On the basis of this information it is now possible to determine the geographic variability of suspended particulate matter in Chicago.

The median concentrations of suspended particulates for the 20 stations during 1966 are plotted on a map of the city (Figure 2). For example, on the basis of median concentration it is apparent that station A ($100 \mu\text{g./m.}^3$) ranked as the least polluted area of the city, while station L ($177 \mu\text{g./m.}^3$) was the most heavily polluted from the standpoint of total suspended particulate matter. The median concentration for all 20 stations was $145 \mu\text{g./m.}^3$.

As part of this analysis, data were summarized by day of the week for all 20 stations; no significant difference is noted in levels of suspended particulates between Tuesdays and Thursdays throughout 1966. Similarly, data

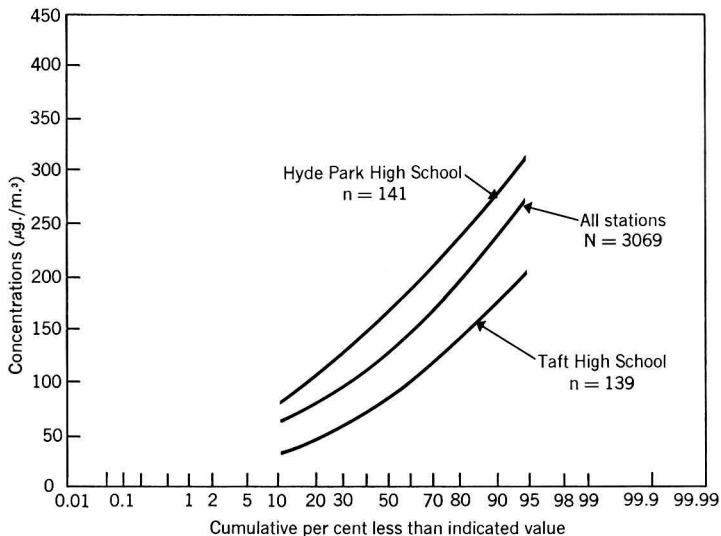


Figure 6

Table IV. Financial breakdown of telemetered network vs. existing stations for SO₂ Networks

All figures below are based on three years of experience from operating a network which could provide only 24-hour-mean concentrations.
 All figures are based on five-year depreciation of equipment.
 In order to obtain daily 24-hour-mean concentrations, the following personnel and equipment are needed:

Equipment	Cost (1 year)	Cost (5 years)
24 Bubbler boxes plus glass, \$136 each for 8 stations.	\$ 3,264	\$ 3,264
24 Gelman pumps, \$68. each	1,632	1,632
1 Technician, instrument, \$7700 for analysis of SO ₂	7,700	7,700
8 Aerovanes, \$412.50 each		3,300
8 6-Bladed propellers, \$10 each		80
8 Recorders, dual chart, \$1525 each		12,200
Supplies		
Chemicals not necessary plus glassware, plus strip charts and miscellaneous		3,250
Personnel		
3 Air sampling and equipment technicians, \$6024 each	18,072	90,360
1 Chemist III	6,900	34,500
Mileage, miscellaneous travel, 8 cents per mile	2,500	12,500
TOTAL		168,786

Eight-Station Automatic Telemetered System		Cost (5 years)
8 Beckman SO ₂ Analyzers, plus Bristol Strip charts		
8 Aerovanes plus strip charts		
8 Masts		
8 Remote stations with associated electronics		
1 Central station, equipped with electronics, teletype, and five-channel punch tape		
Total cost		\$128,000
1 Electronics equipment technician (70%)	\$4830	24,150
Rental of Teletype lines, \$276 per month (1 year)	\$3312	16,560
Strip charts		750
TOTAL		\$169,460

were analyzed according to season of the year, revealing that concentrations are rather uniform on a seasonal basis.

The annual isopleth for suspended particulates (Figure 2) corresponds fairly well with the population and industry concentration, particularly with population. The concentrations reduce westward, rapidly on the north and near south, slowly at the center. The far south (the location of the heavy industries) shows a higher concentration than the far southwest.

Sulfur dioxide

A graphic summary of sulfur dioxide data from all 20 stations during 1966 has been plotted on a map of Chicago (Figure 3). It should be noted that in relation to the range of average levels of suspended particulates, the range of average levels of sulfur dioxide is quite large. For example, the average concentration of sulfur dioxide for the year ranged from 0.028 p.p.m. for station C to 0.090 p.p.m. for station L. Station L, it should be noted, also recorded the highest level of suspended particulates.

Sampling of pollutants has indicated that meteorological factors play a significant role in concentrations. Daily analysis of data reveals that the levels of pollutants vary inversely to wind speed. Heavier concentrations of pollutants are recorded with light wind speeds rather than with stronger winds.

In the case of sulfur dioxide, there is a very pronounced seasonal cycle in Chicago, indicative of a relationship with heating demands. The correlation coefficient between these two variables was 0.97. Generally speaking, the average levels during the winter are more than three times those observed during the summer. Again, station L is most outstanding with levels in the summer of 0.027 p.p.m. and in the winter of 0.197 p.p.m. The maximum 24-hour average concentration of sulfur dioxide recorded at station L was 0.249 p.p.m. By referring to the cumulative frequency distribution for all stations for the year, it can be seen that 1% of the total of 1766 samples exceeded a concentration of 0.26 p.p.m.

In the isopleth map (Figure 3) the sulfur dioxide lines tend to parallel the lakeshore (the high population zones), indicating a slower decay of

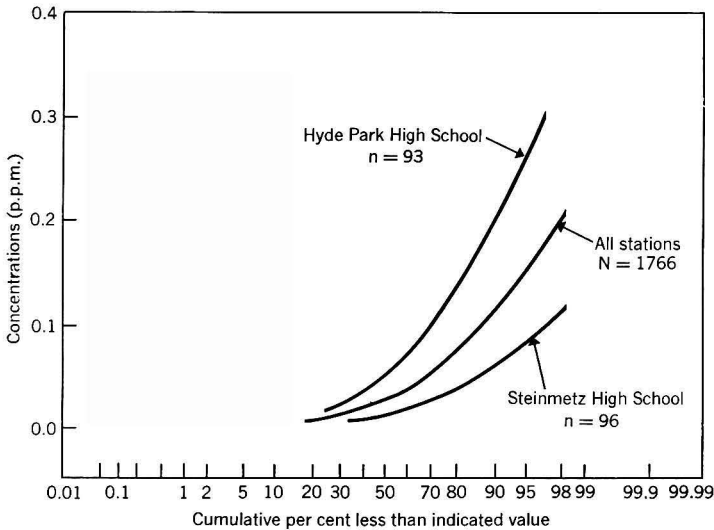


Figure 7

concentration toward the northwest and southwest. The highest concentrations are in the Hyde Park-Kenwood area, the location of station L, and the central business area, the location of station F.

Dustfall

While dustfall is not as important as either sulfur dioxide or suspended particulates, from a public health point of view, it is included here for the sake of completeness. At the 20 dustfall stations that compose the network, the yearly average was 36.9 tons/square mile/month (Figure 4).

The dustfall concentrations follow the pattern of density of population and industrial concentration as well as reflect seasonal variation because of heating, particularly due to low grade fuels. During the heating season, solubles and combustibles are particularly high. The insolubles and non-combustibles appear to follow wind patterns, showing higher concentrations in spring and summer months.

Locations of CAMP stations

As the department's five-year air resource management program progressed, it became apparent because of (1) health studies conducted in the city, (2) data needed to assist in formulating a diffusion model, and (3) testing this model, that *continuous* air quality data must be available. Bi-weekly or triweekly information gathered by air sampling technicians was simply not adequate.

A statistical analysis of two years of data on suspended particulates and sulfur dioxide was made to determine the adequate number of stations and their location throughout the city. The stations had to meet two criteria:

- They must be truly representative of the geographical coverage of the city.
- They must provide an estimate of air quality not significantly different from the data acquired from the 20-station network.

Statistical analysis of the 20 stations revealed an adequate grouping of eight subsets, with the stations in each subset part of the same regional area. The readings of pollutants were reasonably similar at the different stations located within the same subset (Table II). For example, stations A and C, located in the northwest portion of the city, had median concentrations of suspended particulates of 100 and 111 $\mu\text{g./m.}^3$, respectively. In addition, the correlation coefficient between measurements made on the same day at these stations was $r = 0.98$, indicating nearly a one-to-one correspondence. For all practical purposes, either one of these stations could be selected to represent that general area of the city.

By June 1965, seven months of SO_2 data at 19 stations had become available. A check was made on the adequacy of the chosen sites based on the correspondence between the average SO_2 concentration for these stations and that for the total network. In addition, eight stations were inde-

pendently selected, using a graphic method that was based on the correspondence of graphed data of the individual stations with those of averages of the network. Table 3 shows the results of these checks. Though the alternate network thus obtained revealed a somewhat better correlation coefficient, it was not as adequate geographically. Thus, the original eight-station choice was retained; however, station P was substituted for Q.

Finally, two additional criteria were used in selecting one station from each of the eight subsets:

- Health studies under the direction of Dr. Mark Lepper, executive vice president, Presbyterian-St. Lukes Hospital, required broad socio-economic distribution.
- Wide geographic distribution was desirable to obtain suitable meteorological coverage.

When the scope of the optimum continuous monitoring system had been determined on the basis of these criteria, then, a comparison of costs could be made between the existing stations and the proposed network. The analysis showed that the cost of equipping and operating either system over a five-year period is nearly the same. The cost figures for the manual network are based on three years of operating experience in the city of Chicago. The cost figures for the telemetered network are based on known costs for equipment and supplies and estimated costs for maintenance personnel. In both cases a five-year depreciation period for equipment has been assumed. Table IV shows the costs breakdown for the two systems.

Continuous air monitoring telemetered network

In evaluating various approaches to a data-acquisition system the following factors had to be considered: cost, reliability, simplicity for ease of maintenance, data handling, availability of service, and manufacturers with proved experience in all phases of the system requirements. Specifications for this system were developed with these general considerations in mind and by evaluating the technical aspects of a number of systems available.

To obtain reliability of data telemetered to a certain location and ease of maintenance, the equipment at re-

remote sites was digitized by installing a shaft encoder on a strip chart recorder which is an integral part of each chemical analyzer and meteorological sensor. Solid state design electronics was specified because of the inherent reliability of solid state equipment, in addition to the fact that such equipment lends itself to modular plug-in construction, making maintenance a matter of merely replacing a plug-in module and repairing the defective unit at a more convenient time.

Teletype transmission of data to the central station offered the use of the lowest cost leased line available from



Heller



Booras

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the telephone company. At the central station the transmitted data are printed on a page printer in addition to being recorded on a punched paper tape for subsequent computer handling. The remote stations utilize solid state distributors in lieu of the electromechanical units normally used for teletype transmission. They are controlled by a precision clock at the master control panel and should require virtually no maintenance or adjustment.

Systems operation

The air quality monitoring system finally purchased consists of eight remote stations, each having a sulfur dioxide analyzer and an aerovane. The system is designed for recording data at 15-minute intervals by transmitting sequentially from each remote station to the central station. Data gathered at each remote site include sulfur dioxide in parts per million, an integrated wind direction to the nearest degree, and an integrated wind speed to the nearest one tenth knot (Figures 6 and 7).

Fifteen-minute intervals were selected because of their representativeness over a 24-hour period, thereby precluding the likelihood of excessive highs and lows occurring without being detected. A new and perhaps unique meteorological method is employed in the recording of 15-minute intervals inasmuch as wind speed and direction are integrated. Readings on wind speed and direction are taken every five seconds and are stored in the electronic memory; the information is then averaged by the computer for 15-minute readings and recorded. The problem of eliminating ambiguity of readings of wind speed and direction was solved by using two Selsyn motors counterclockwise.

At preselected intervals the central station clock signals the beginning of a recording cycle. The programmer starts the printer and tape punch and initiates the recording of the date-time group and parameter data which are utilized by the computer for the analysis assigned to the block of data. The programmer then interrogates the first station, and it immediately transmits its three-digit identification followed by the air quality and wind data. This sequence is repeated for all stations

with a total transmission time of 40 seconds.

The central station control panel allows for automatic or manual selection of signals for zero checks, calibration checks, and changes of sampling and recording intervals. Further, it contains provisions for bypassing a station in the event of line or equipment difficulties, initiation of demand recording cycles, and indicators to show functions taking place. The central console incorporates a graphic display representing the metropolitan Chicago area with indicators that illuminate each station location when the station is transmitting data.

Advantages of the system

The system is so constructed that it can be expanded to include 59 remote stations by the addition of simple plug-in modules. Each remote station will be able to accept inputs from as many as five different air quality analyzers.

Encoders allow the operator to utilize any of the available analyzer instruments by tying onto the shaft of the recorder which in most cases is an integral part of the analyzer. It also presents linearized and scaled output and no data interpretation is required. This approach introduces no errors to the analyzer accuracy. Digital transmission precludes any degradation of system accuracy. Further, minimal modification is required for tying the system to an on-line computer for complete pollution monitoring.

The Telemetered Air Monitoring Network information will be used to do the following:

- Advise the public of the air quality in the city of Chicago.
- Provide the back-up information with which health studies will be correlated.
- Provide the information to determine the effectiveness of the enforcement program in reducing sulfur dioxide.
- Provide an enforcement tool to pinpoint large sulfur dioxide emitters.
- Provide the information to support the design and operation of a diffusion model for the city of Chicago.
- Establish air quality standards as an aid to enforcement.
- Develop an air pollution climatological for the city of Chicago.

Hyperfiltration. Processing of Pulp Mill Sulfite Wastes with a Membrane Dynamically Formed from Feed Constituents

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■ Liquors, generated in the sulfite process of the pulp and paper industry, form "membranes" dynamically on porous bodies when the liquors are circulated under pressure past the bodies. In hyperfiltration (or reverse osmosis), these membranes, in favorable cases, reject 90% or more of the colored matter in the feeds and substantial (though somewhat lower) fractions of the chemical oxygen demand and total dissolved solids. By proper selection of conditions, processing rates of 30 gallons/day sq. foot of membrane at 500 p.s.i.g. apparently can be maintained for at least 2 weeks, and much higher rates may be possible. The economic usefulness of this technique has not yet been established. Judgment regarding its practicability must await more detailed and longer testing, engineering development, and an economic analysis.

One of the principal chemical methods used in manufacture of wood pulp is the sulfite process, in which wood is cooked in sulfurous acid and a salt of this acid, often the calcium salt. The spent calcium-base liquor cannot be reused, and disposal of this liquor and the dilute wash liquor from pulp washing operations poses a serious problem.

Economical processes for concentrating wash liquors to 10% solids (where methods in limited current use for treatment of digester strength spent liquor, such as evaporation followed by burning, could be used) or alternative processes for concentrating spent liquors to greater than 35% solids (where the residue can be burned) would be attractive improvements in current practice, since a source of stream pollution would be eliminated, and the water recovered could be reused.

Hyperfiltration, or reverse osmosis, has undergone intensive development during the past 10 years, primarily as a method for desalting water. Separation is achieved by forcing the solution under pressure through a membrane which passes solvent more readily than solute. Most of the work on desalination has been done with cellulose acetate membranes, which are cast as films and then installed in pressure vessels with suitable support structures.

The Pulp Manufacturers' Research League has investigated hyperfiltration with cellulose acetate membranes for processing of spent sulfite liquor (Wiley, Ammerlaan, *et al.*, 1967). They found excellent separation of solutes and water, with rejection of 97.5 to 99% of total dissolved solids. However, product fluxes were rather low, usually less than 10 gallons per day per sq. foot of membrane surface. Even at these production rates, they felt that the technique merited serious consideration for practical application.

Dynamically formed "membranes," under study at Oak Ridge National Laboratory (Kraus, Shor, *et al.*, 1967), have the potential advantage of high production rates and reject neutral organic solutes (Kuppers, Marcinkowsky, *et al.*, 1967) as well as salts. Usually, these membranes are formed by circulating, under pressure, feeds containing additives of a colloidal nature past bodies having pores in the micron range. The additive tends to concentrate at the interface, and a purified solution permeates through the porous body. In cases where rejection of additive has been measured, additive has been more highly rejected than the salts in the feed. Examples of such self-rejecting systems are Th(IV), Fe(III), and humic acid in aqueous solutions (Marcinkowsky, Kraus, *et al.*, 1966; Kraus, Shor, *et al.*, 1967).

Calcium-base sulfite liquors form self-rejecting membranes of possible practical interest, and some observations of their hyperfiltration properties are reported here.

Experimental

Solutions. The sulfite liquors were prepared by dilution of a concentrate containing 50% total dissolved solids. Calcium lignosulfonate constitutes about 60% of the total dissolved solids. A considerable fraction is carbohydrate material as 5- and 6-carbon sugars and hemicelluloses; the solution also contains resins and other minor constituents. The concentrate had been prepared by evaporation and did not contain some of the volatile components present in the original liquor. These components presumably tend to be of lower molecular weight than the solute in the concentrate, and therefore probably would be less efficiently rejected in the hyperfiltration process.

Also, a few tests with two other solutions were carried out, used as supplied: a first-stage (chlorination) bleach waste

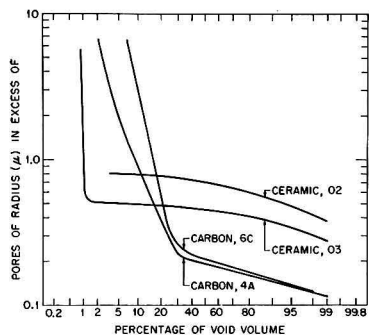


Figure 1. Pore size distribution of porous tubes

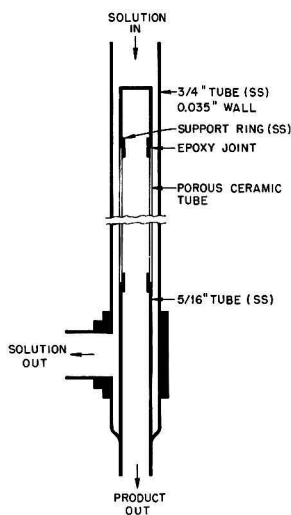


Figure 2. Mounting of typical tube for hyperfiltration

Pressurized feed outside

solution, which contained about 0.15% total dissolved solids and 350 p.p.m. chloride; and a second-stage (alkaline extraction) bleach waste, containing 0.5% total dissolved solids and 175 p.p.m. chloride.

Analytical Procedures. In most cases, observed rejections

$$R_{obs} = 1 - (c_w/c_f)$$

(where c is concentration, w indicates the effluent or product solution, and f the feed) were calculated from the optical densities of the solutions at 281 $m\mu$, measured with a spectrophotometer. Absorption at this wavelength is considered representative of the lignin concentration. In some cases, rejection was checked by evaporating and weighing the residue, or by analyzing for chemical oxygen demand by the dichromate-sulfuric acid method (Standard Methods, 1965); typically, values of R_{obs} were 10 to 20% less than by light absorption.

Apparatus. Most experiments were done in pressurized hyperfiltration loops of the type described by Shor, Kraus, *et al.* (1968). With these, porous tubes supported the dynamic membranes. Two circulation loops were used, both constructed of stainless steel tubing and components; one also contained some high-pressure rubber hose and the other some reinforced Teflon hose. In each loop, a makeup pump brought the feed up to the experimental pressure, and a second pump circulated it. There were also heat exchangers, appropriate valves, and instrumentation to permit temperature, pressure, and flow rate control.

The porous tubes were carbon of two different types (manufactured for use as electrodes) and three grades of ceramic (porcelain) tubes, sold commercially as polishing filters. Porosimetry measurements by mercury intrusion revealed that the carbon tubes had a much broader pore-size distribution than the ceramic tubes (Figure 1). The pertinent physical characteristics of the tubes are given in Table I.

The tubes were usually mounted so that the outer surface was the membrane-forming surface. The pressurized solutions flowed through an annular space between the outer surface of the porous tube and the inner surface of a stainless steel tube (Figure 2). This arrangement was always used with the ceramic tubes because of their low strength when pressurized on the inside. The carbon tubes were strong enough to allow membrane formation on either surface. For pressurization

Table I. Characteristics of Porous Tubes

Tube Type	Median Pore Diameter, μ	Porosity, %	Diameter, Cm.	
			Inside	Outside
Carbon, 4A	0.38	27	0.5	1.1
Carbon, 6C	0.41	33	0.6	1.0
Ceramic, 03 ^a	0.90	38	1.2	1.6
Ceramic, 02 ^a	1.40	45	1.0	1.6
Ceramic, 01 ^a	3.0	65	1.0	1.6

^a Selas Flotronics.

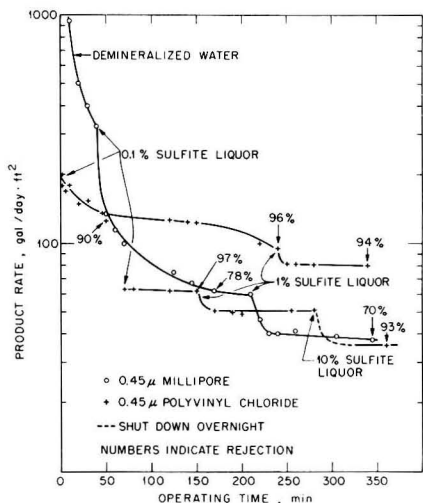


Figure 3. Short-term permeation rate of porous plastic membranes showing the effect of spent sulfite liquor concentration

Pressure 500 p.s.i.g.

on the inside they were mounted by attaching tubing fittings to the ends with epoxy cement. The solutions were circulated inside the tubes, and the product was collected from the outer surface in trays. (See Figure 1, Shor, Kraus, *et al.*, 1968.)

In addition to the tubular porous supports, some work was done with flat cellulose acetate (Millipore) and poly(vinyl chloride) (Gelman) membranes (nominal pore size, 0.45 micron) supported by porous titanium disks of 5-micron nominal pore diameter. A sketch of the cell which held these disks is given by Baldwin, Holcomb, *et al.* (1965). The circulation loop for this cell was constructed entirely of titanium components, except for a rotameter, which had stainless steel parts.

Results and Discussion

Initial Formation of Membrane. The rejecting membranes are formed on the porous supports by passing the feed solutions over them at appropriate pressures and circulation velocities. To illustrate the behavior of the system, several typical experiments are described, one with a Type 4A carbon tube (flow inside) and the others with flat Millipore and poly(vinyl chloride) membranes.

With the carbon tube installed in the loop, permeability measurements were carried out with circulating demineralized water. Over a period of about 1 hour, the pressure was varied from 60 to 400 p.s.i.g., giving flow rates through the tube wall of 13.7 to 99.0 cc. per minute. Dividing these flow rates by the surface area of the tube and the pressure difference, 0.083 to 0.090 cm./minute atm. are obtained for the permeabilities Φ of the porous carbon. These permeabilities are equivalent to 800 to 870 gallons/day sq. foot at 400 p.s.i.g. (Permeability is defined as the superficial velocity through the membrane divided by the pressure drop across the membrane, taken here as the feed pressure.)

The system was drained and refilled with 1% spent sulfite liquor. The pumps were started, and the system was brought to 400 p.s.i.g. The circulation flow rate was 5 gallons per minute, which corresponds to an average linear crossflow velocity of 46 feet per second and a Reynolds number of 75,000; the flow was thus well in the turbulent regime. The permeation rate decreased almost immediately, and color rejection became apparent. After half an hour, the observed rejection was 75% as determined spectrophotometrically, and the product flux had decreased to 103 gallons/day sq. foot; after about 2 hours, the observed rejection and product flux had leveled off at 80% and 87.5 gallons/day sq. foot. They remained constant until the experiment was altered after 7 hours by adding hydrous zirconium oxide to the sulfite liquor, making it $10^{-3}M$ Zr(IV). The observed rejection then rose to 93%, and the flux decreased to 47.5 gallons/day sq. foot. Hydrous Zr(IV) oxide membranes are known to give good rejection of sodium chloride in water solutions (Marcinkowsky, Kraus, *et al.*, 1966) and reasonable organic rejection (Kuppers, Marcinkowsky, *et al.*, 1967).

Different porous supports give different fluxes and rejections, and sometimes supports which are supposed to be the same give different results under similar conditions, for reasons not yet clear. Examples of very different early transient behavior obtained with the high-pressure cell are illustrated in Figure 3. In one, with a Millipore membrane, distilled water which had been passed through a demineralizing column was initially circulated in the titanium loop. Permeation rates were very high at first, but fell rapidly during the first 40 minutes. Enough concentrate was then injected to bring the sulfite liquor concentration to 0.1% by weight. Introduction of this material caused a discontinuity in the log permeation rate-time curve, with a faster decrease in the rate. Later, injection of concentrate to bring the feed to 1% caused a similar discontinuity, the rate reaching about 37 gallons/day sq. foot after 6 hours. Rejections were about 78% for 0.1% liquor and 70% for 1% liquor.

In the two other examples of Figure 3, the films were exposed from the beginning to 0.1% sulfite feed, and the differences in behavior indicate the extent of irreproducibility of the observations. Raising the liquor concentration caused breaks in the permeability curves similar to those found with the water-exposed film. In both these cases, the permeabilities leveled out more quickly than those of the membrane which had first been exposed to water; the rates for 1% liquor (though different for the two samples) and the rejections were both higher than for the water-exposed. This effect of water circulation appears to conflict with the carbon-tube experiment just cited, and, although similar behavior has been observed in several other cases with plastic film supports, the authors are not completely confident that these differences do not arise from as yet unidentified experimental variables, whose existence is implied by the scatter.

Long Term Experiment. In one test, the Type 03 ceramic tube and Type 6C carbon tube both pressurized on the outside were operated in series continuously with 1% liquor for

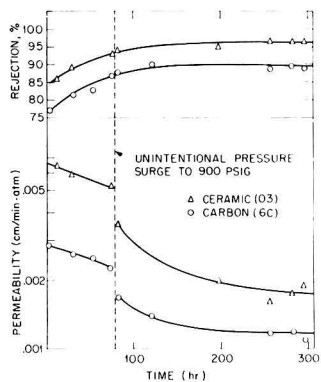


Figure 4. Permeability and rejection vs. time

33° C., 1% sulfite waste liquor, ca. 400 p.s.i.g.

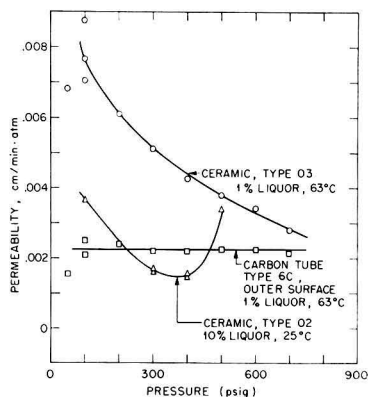


Figure 5. Variations of permeability with pressure in hyperfiltration of pulp mill wastes

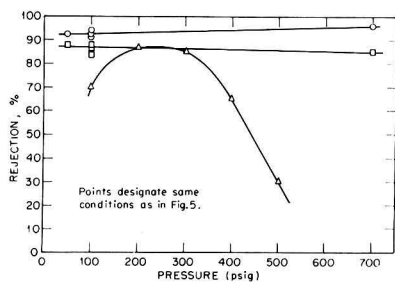


Figure 6. Variations of rejection with pressure in hyperfiltration of pulp mill wastes

340 hours. Permeabilities decreased and rejections increased significantly during this period (Figure 4). There was a noticeable decrease in permeability accompanying a pressure surge to 900 p.s.i.g. For the ceramic tube, the permeability and rejection after 4 hours were 6.5×10^{-3} cm./minute atm. and 85% and after 300 hours appeared to level off at 1.80×10^{-3} and 96%. For the carbon tube these values at the same times were 2.8×10^{-3} cm./minute atm. and 77% and leveled off at 1.25×10^{-3} and 90%. The experimental conditions under which the points of Figure 4 were taken were 400 p.s.i.g., 33° C., and a circulation rate of 3.2 gallons per minute, corresponding to a Reynolds number of 6900 for the ceramic tube and 11,200 for the carbon. Data at these conditions were taken from time to time, interspersed with measurements made under many other conditions during the run; pressure, temperature, and velocity excursions were carried out, and some of the results are described below.

Comparison of the behavior of these two tubes indicates that a low permeability is not a necessary condition for high rejection, and that other support structures giving better performance than either of these can probably be found or made.

Dynamic Aspects of the Membrane. The membrane is formed by the radial flow of the solution through the porous wall, with the water passing through preferentially, leaving a region of concentrated solutes near the porous surface. Any change in operating conditions which affects the boundary layer near the tube wall—e.g., pressure, axial velocity, or turbulence intensity—probably would affect the permeability and rejection of the membrane.

Pressure Effects. Three different kinds of effects of pressure on permeability were observed. With the Type 03 ceramic tube and 1% sulfite liquor at 63° C., the permeability decreased by a factor of 2.3 as the pressure increased from 100 to 700 p.s.i.g. (Figure 5). This behavior seems reasonable, because the product flux increased by a factor of 3.1, and the increased radial flow probably caused a thickening of the membrane, resulting in decreased permeability. This behavior could be produced starting at any place in the pressure range, and the permeability changed as quickly as the controls could be adjusted—namely, about 2 minutes per pressure point. At lower temperatures, the permeation rates did not reach such high values, and the decrease of permeability with pressure was less pronounced, if it occurred at all. The observed rejection was nearly constant at 93 to 96% (Figure 6). These observations were made after the membrane had been used for more than 150 hours of operation. When the membrane was fresh, operation at 63° sometimes caused a sharp increase in permeation rate and decrease in rejection, behavior similar to the third type described below. The membrane properties were rapidly restored on decreasing the temperature. These instabilities disappeared after long-term operation.

Type 6C carbon tubes exhibited a second type of behavior. Permeability was unaffected by pressure when the sulfite liquor was passed over the outer surface. This behavior might

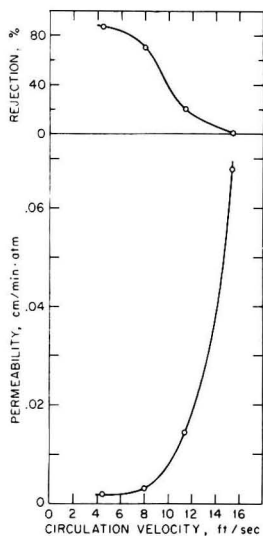


Figure 7. Variation of hyperfiltration properties observed with Type O2 ceramic tube

10% sulfite liquor, 33° C., 400 p.s.i.g.

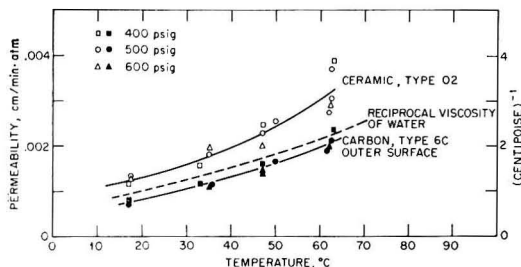


Figure 8. Effect of temperature on hyperfiltration properties of pulp mill wastes

occur if the pores of the carbon were irreversibly plugged—e.g., by corrosion products—and the principal flow resistance were across this layer; the dynamic membrane would then not be rate controlling. The observed rejection was nearly constant at 85 to 88% (Figure 6).

The third type of behavior is also illustrated in Figure 5 by the Type O2 ceramic tube, with a median pore diameter of 1.4 microns and 10% liquor at 25° C. A dynamic membrane was probably the controlling resistance in the pressure range of 100 to 300 p.s.i.g., but at higher pressures permeability increased. Apparently, the membrane was pushed in part through the larger pores, and feed leaked through these “pinholes.” The rejection fell from 86% at 300 p.s.i.g. to 30% at 500 p.s.i.g. (Figure 6).

Stable membranes could not be formed with Type O1 ceramic tubes, which have a median pore diameter of 3.0 microns. Experiments with 10% liquor and Type O1 tubes at 25° C. resulted in rejections of only 24%, but even then the

rejection decreased with no apparent change in operating conditions. Apparently, near 1.4 microns is a critical pore diameter, and with higher pore diameters, sulfite liquor membranes are unstable without additives.

A leakage effect was also observed with Type 6C carbon tubes and 1% liquor at 25° C. when the feed was passed through the inside of the tube. The permeability increased from 0.0017 to 0.0031 cm./minute atm., and the rejection fell from 49% to 27% as the pressure was increased from 350 to 450 p.s.i.g. This behavior is different from that shown in Figure 5 for membrane formation on the outer surface.

Circulation Velocity, Temperature, and Concentration Effects. Qualitatively, a dynamic model of membrane formation suggests that increasing the circulation velocity should decrease the boundary layer thickness and cause an increase in permeability. However, at velocities high enough to give Reynolds numbers above ~2000 (near the transition point from laminar to unstable flow), permeabilities and rejections were independent of velocity for all systems except the Type O2 ceramic tube–10% liquor system. Here, velocities greater than 4.5 feet per second (Reynolds number 1920) caused increasing permeability and decreasing rejection (Figure 7). At 15.4 feet per second, no rejection was obtained. This effect could be demonstrated starting from either end of the velocity range. The independence of rejection and circulation velocity with smaller pore sizes indicates that the results reported here are not seriously distorted by concentration polarization.

Over the temperature range of 17.5° to 62.5° C., the permeability increased approximately in inverse proportion to the viscosity of water for the two systems studied, the Type O3 ceramic and Type 6C carbon (flow outside) with 1% liquor (Figure 8). This would be expected from the Carman-Kozeny equation for laminar flow through porous media provided the properties and thickness of the membrane do not change. The increase is slightly greater for the ceramic tube than can be explained by viscosity decrease, perhaps indicating a thinning of the membrane for this system. Permeabilities at 60° C. in Figure 8 correspond to 29 gallons/day sq. foot for the ceramic tube and 19 gallons/day sq. foot for the carbon tube at 400 p.s.i.g. These rates were obtained after 300 hours of operation.

The rejection decreased only slightly as the temperature increased, from 97 to 95% for the ceramic tube and from 90 to 85% for the carbon tube. The higher permeabilities found at high temperatures are of practical interest, particularly since the waste liquor might be received from the pulp mill at an elevated temperature.

Rejections were about the same with 1% liquor and 10% liquor for three tubes studied, Type O2 and O3 ceramic and Type 6C carbon (flow outside). Permeabilities at 10% concentration were decreased 30 to 40% from their values at 1% concentration. This is the magnitude of the difference in viscosity of 1% and 10% solutions (0.93 and 1.44 cp., respectively). If the boundary layer were affected little by radial flow, the thickness of the laminar sublayer would be proportional to viscosity and therefore would increase about the right amount to account for the decreased permeability. In addition, the membrane probably was thicker because the radial flow, though reduced in velocity, was carrying six or seven times as much solute toward the wall. That the permeability with 10% liquor is as high as observed is surprising.

Independence of Rejection and Permeability. Figure 4 demonstrated for tubular supports that improvement in rejection is not necessarily paid for by lower permeability. Data in Figure 9, obtained with high-pressure cells, support this conclusion. Rejections are given as a function of permeability for many different experiments with porous plastic film supports. These points were obtained under various conditions and by various procedures, some of them indicated in the figure; most were taken on the first or second day of operation with the membrane in question. As with Figure 3, the authors are not sure that differences in the observations arise from known, as distinguished from as yet unidentified, variables. However, scatter does not obscure lack of any consistent correlation of low permeability with high rejection. For the point of highest permeability, for example, rejection of 0.1% waste [0.45-micron poly(vinyl chloride) support] was 94% at 230 gallons/day sq. foot (500 p.s.i.g.).

Results with First- and Second-Stage Bleach Wastes. A few scouting runs with other pulp mill waste solutions also gave encouraging rejections. A membrane was formed with 1% spent liquor on Type 02 and 03 ceramic tubes in series. After 15 hours of operation, typical rejections were obtained, but permeation rates were rather low, at 7.5 and 9 gallons/day sq. foot for the 02 and 03 tubes, respectively, at 500 p.s.i.g. The loop was drained and filled with the chlorination bleach waste. At 400 p.s.i.g. and 30° C., the Type 03 tube immediately gave 90% rejection, based on optical density, and 22% by chloride analysis (7 gallons/day sq. foot). Rejection for the 02 tube rose over 4.5 hours to 67% by color and 18% of chloride (9 gallons/day sq. foot).

The loop was drained, rinsed with demineralized water, and filled with alkaline extraction bleach waste. Rejection based on optical density for the 03 tube was 90% (8 gallons/day sq. foot), and for the 02, 51% (14 gallons/day sq. foot). Chloride rejections were 32% and 16%, respectively.

In experiments with the high-pressure cell, membranes were formed directly with the chlorination bleach waste on 0.1-micron Millipore supports. After about 5 hours of operation, rejection based on absorbance was 95% and of chloride, 34%; the permeation rate was 10 gallons/day sq. foot at 500 p.s.i.g. In a similar test, second-stage alkaline bleach waste formed a membrane rejecting 94% of color and 43% of chloride at 15 gallons/day sq. foot.

Conclusions

Although irreproducibility shows that all variables affecting the performance of these membranes are not yet under control, these results indicate that a production facility operating at 60° C. could attain product rates of about 30 gallons/day sq. foot and rejections greater than 90% at pressures less than 500 p.s.i.g. If factors which have given optimum observed behavior could be identified and controlled in practical operation, production rates three times as high, with comparable rejection, might be reached.

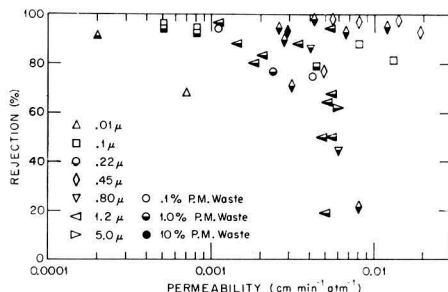


Figure 9. Rejection of sulfite liquor as a function of permeability

Poly(vinyl chloride) and Millipore supports; high pressure cell

Acknowledgment

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Phenolics by Aqueous-Injection Gas Chromatography

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■ Monohydric phenolic compounds in water were determined by direct aqueous injection gas-liquid chromatography and flame-ionization detection. Various combinations of steric substrates on diatomaceous and synthetic supports were evaluated. Silanization of diatomaceous supports decreases sensitivity and increases peak tailing. FFAP, a reaction product of Carbowax 20M and 2-nitroterephthalic acid, on Teflon 6 packing was the best column tested. Substrate loadings of 5 to 10% provided efficient separation of components and reasonably long column life. The hydrophobic Teflon support is limited to a maximum operating temperature of about 200° C. Many phenolics are readily quantified at 1 to 10 mg. per liter, although certain separations, such as meta and para forms of cresols and monochlorophenols and certain dichlorophenols, are not possible. Addition of steam to the carrier gas reduces but does not eliminate ghosting and accelerates substrate decomposition.

Mono- and dihydric phenolic materials of natural and man-made origin constitute an important class of water pollutants. Because chlorination of these hydroxybenzenes often produces substituted reaction products of greater taste and odor potential than the original material (Baker, 1963), the U. S. Public Health (1946) Service adopted 1 μ g. per liter as a limit for phenol. The term "phenol" or "phenolic" is generally defined by an analytical test. It embraces all hydroxybenzene materials which react with 4-aminoantipyrene to form a complex detectable colorimetrically in a standard test procedure (American Society for Testing Materials, 1962; American Public Health Association, 1965). Although the 4-aminoantipyrene test is sensitive for phenol per se, it lacks specificity and is relatively insensitive to many substituted phenolics—for example, those substituted in the para position are not measured. Phenolics at the microgram per liter concentration in water cannot be identified without preliminary concentration with present technology. All existing analytical techniques are deficient in some respect. Infrared and ultraviolet spectroscopy fail to distinguish closely related phenolics in mixtures and require greater concentrations than are commonly present in water. Thin-layer and paper chromatography allow characterization and in some cases estimation of quantity.

Gas-liquid chromatography (GLC) combined with sensitive ionization detectors offers a means of achieving a considerable degree of separation and identification of complex mixtures of organic components. The merits and disadvantages of GLC analyses of water have been reviewed (Baker, 1962). Most organic analyses have been made after transfer of the organic solutes to a solvent. Phenolics have generally been measured from solvents after chromatographic separation on nonpolar columns—for example, Fitzgerald (1959) determined relative retention times for the elution of many substituted monohydric and some dihydric phenols on both polar and nonpolar columns. He concluded that nonpolar Apiezon L on a diatomite support was satisfactory because of its stability and nonvolatility, although it does not separate the meta and para forms of such components as cresols. Certain other components are also poorly resolved. von Rudloff (1964) demonstrated eugenol-type phenol separation on seven substrates. Low-loaded columns (1%) were necessary to overcome difficulties in separation of polyphenols at high temperature. He also reported that SE-52, a phenyl-substituted silicone polymer, gave somewhat better resolution than that obtained previously (Narasimhachari and von Rudloff, 1962) on SE-30, a silicone polymer column.

Pillion (1965) separated less volatile phenols on XE-60, a silicone nitrile polymer, and on SE-30. Adlard and Roberts (1965) used tri-2,4-xylene phosphite to separate C₆, C₇, and C₈ monohydric phenols on packed and capillary columns. Best results were obtained when the phenols were converted to their trimethylsilyl ether derivatives and analyzed by capillary GLC. The derivatives are resolved better than the parent phenols and more theoretical plates are obtained by the capillary mode of operation. Narasimhachari and von Rudloff formed phenolic derivatives by methylation or silylation to improve chromatographic response in their study of polyphenols. Nelson and Smith (1966) used GLC and infrared spectroscopy in a study of the trimethylsilyl ethers of phenols relative to lignin and ellagitannins.

Chromatographic columns prepared from a mixture of two substrates that do not interact chemically have been demonstrated to be equivalent in properties to those of a two-stage column prepared in the same ratio (McFadden, 1958). Duvall and Tully (1963) used this concept in mixing a nonpolar silicone oil 550 and a polar Carbowax 4000 to obtain a substrate for determination of phenol and its five *o*- and *p*-*tert*-butylphenols. The ratio of the substrates is very important,

Table I. Phenolic Retention and Calibration Values

Substrate	15% STAP ^b		10% STAP		10% Carbowax 1540		20% Carbowax 4000 TPA		20% Carbowax 20M TPA		20% Carbowax 20M TPA		15% UCON 50LB550X		15% UCON 50LB550X		
	70/80 W	70/80 T	60/80 HMDS W	60/80 W	60/80 HMDS W	60/80 W	60/80 HMDS-W	60/80 W	60/80 A/W	60/80 A/W	60/80 A/W	60/80 W	60/80 HMDS W	60/80 W	60/80 DMCS-W	60/80 W	
Chromosorb support	70/80 W	70/80 T	60/80 HMDS W	60/80 W	60/80 HMDS W	60/80 W	60/80 HMDS-W	60/80 W	60/80 A/W	60/80 A/W	60/80 A/W	60/80 W	60/80 HMDS W	60/80 W	60/80 DMCS-W	60/80 W	
Stainless steel column, 0.125-in. diam., length, feet	10	5	10	10	10	10	10	10	10	10	10	10	5	5	5	5	
Aerograph Model No.	600D/328	204-1B	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	600D/328	204-1B	204-1B	
Recommended max. temp., ° C.	255	255	200	200	200	200	250	250	250	250	250	250	200	200	200	200	
Compound	Boiling Point, ° C.	r ^a	c	r	c	r	c	r	c	r	c	r	c	r	c	r	c
Phenol	182	1.0	43	1.0	44	1.0	49	1.0	32	1.0	45	1.0	29	1.0	48	1.0	45
<i>o</i> -Cresol	192	1.0	39	1.0	38	1.0	45	1.0	31	1.0	51	1.0	27	1.0	42	1.0	39
<i>m</i> -Cresol	203	1.3	43	1.3	38	1.3	49	1.3	37	1.3	52	1.3	28	1.3	45	1.3	45
<i>p</i> -Cresol	202	1.3	41	1.3	38	1.3	54	1.4	34	1.3	51	1.3	28	1.3	41	1.3	46
<i>o</i> -Chlorophenol	176	0.7	67	0.7		0.6	67	0.6	46	0.8	87	0.7	39	0.6	54	0.6	65
<i>m</i> -Chlorophenol	214	3.9	96	3.9		^b				3.6	106	4.1	48	3.8	88		
<i>2</i> -Chlorophenol	217	3.9	88	3.9		^b				3.6	125	4.1	50	3.8	84		
<i>2,3</i> -Dichlorophenol	...	1.8	85	1.9		2.1 ^c				1.8	77	1.8	53	1.8	76	1.9	1.9
<i>2,4</i> -Dichlorophenol	210	1.8	89	1.8		2.0 ^c	138			1.8	117	1.8	77	1.8	95	1.9	1.9
<i>p,5</i> -Dichlorophenol	210					2.1 ^c				1.8	133	1.8	68				
<i>2,6</i> -Dichlorophenol	220	1.5	60	1.5		1.5 ^c				1.6	72	1.5	52	1.5	83	1.5	1.5
<i>3,4</i> -Dichlorophenol	254	^b															
<i>o</i> -Nitrophenol	215	^b				^b		0.5	148								
Thymol	233					1.9 ^c	35					1.8	25				
Guaiacol	205	0.7	56	0.6	56	0.6	92	0.7	49			0.7	44	0.7	66	0.8	69

^a r = relative retention; c = calibration, nanogram/sq. in. at 90 inches/hour chart speed, 1-mv. response with range 1, attenuation 1. ^b No peak. ^c Smear estimated.

since it alters the elution order of the components. Crouse *et al.* (1963) employed two columns, a polar, polyethylene glycol succinate (DEGS) and a nonpolar Apiezon N to determine various phenols contained in steam-distilled cigarette smoke volatiles. The phenolic content of the distillates was higher than the levels of interest in most water quality surveys. However, this is an excellent example of the advantages in achieving component resolution by pairing dissimilar GLC columns. The above references represent typical GLC analytical applications which are particularly suited to higher boiling, more complex phenolic materials. Goren-Strul *et al.* (1966) recently summarized most of the phenolic analytical procedures now employed in water technology. They stressed ether extraction of phenolics from water and subsequent GLC analyses on the tri-2,4-xylenyl phosphate column.

There has been an increasing interest in direct aqueous injection GLC combined with flame-ionization detection (FID) for determination of organics in water. Improved electrometers and advances in column supports and substrates have increased the potential of the procedure. Direct aqueous injection GLC eliminates pretreatment or extraction in many cases and assures that molecular structure and relative distribution of complex mixtures are not altered. The principles of the technique, including an explanation of some parametric effects using phenolics as illustrations, have been

reported (Baker, 1966a). Despite the advantages of direct aqueous injection, developments have been slow in this field of GLC because of inherent difficulties such as tailing of the water peak into the solute peaks, ghosting, and other problems. Lechner *et al.* (1966) recently concluded that phenolic analysis by aqueous injection was not possible. They suggested ether extraction and nonpolar column GLC analyses.

A preliminary study based on aqueous GLC phenolic analyses using selected polar substrates has been reported by this laboratory (Baker, 1966b). It was demonstrated that milligram per liter concentrations of monohydroxy-substituted phenols could be separated and measured. The GLC procedure was shown to determine more phenolic material than the commonly accepted 4-aminoantipyrine procedure if complex mixtures of substituted phenols were present. All monohydric components are not resolved—for example, *m*- and *p*-cresol and certain dichlorophenols are not separated. The substrates reported as most suitable were Carbowax 20M-terephthalic acid terminated and FFAP, a condensation reaction product of Carbowax 20M and 2-nitroterephthalic acid.

The objective of this study was to examine various column substrate and support combinations and GLC operating parameters to determine the most suitable procedure for phenolic analyses by direct aqueous injection.

Experimental

Apparatus. The chromatographs used were a Varian Aerograph (Varian Aerograph, Walnut Creek, Calif.) single-column Hy-Fi Model 600D equipped with a Model 328 isothermal temperature controller and a Varian Aerograph dual-column, linear temperature-programmed Model 204-1B, each equipped with flame-ionization detectors. Highest purity tank nitrogen and helium were used as the carrier gases. Flame hydrogen was obtained from a Varian Model A650 hydrogen generator and from tank supplies. Glass inserts were used at the chromatographic injection port to facilitate cleaning. Columns were preconditioned in a Varian Model 550 chromatographic oven, which was also used in conjunction with the 600D electrometer to facilitate column screening. Leeds & Northrup (Leeds & Northrup, Philadelphia, Pa.) single-channel and dual-channel variable-

speed Speedomax recorders with 1-mv. full scale response were used to trace the chromatograms.

Procedure. Precautions were taken to maximize noise-free, stable operation. Details of the GLC analytical practices were recently described (Baker, 1966a). All columns were packed in 0.125-inch diameter stainless steel tubing and were preconditioned at 30° to 50° C. above expected operating conditions if substrate or support properties permitted. Generally, conditioning required less than 24 hours. Reagents were prepared from the highest purity reagent grade chemicals available. Some were recrystallized or redistilled. Aqueous solutions were prepared fresh daily by dissolving precisely weighed quantities of the phenolics in distilled water. The standard solutions were checked for purity and calibrated using the GLC procedure. This study emphasized the monohydric phenolic compounds listed in Table I.

The stability of the electronic systems was carefully checked and maintained. The electrometer is the heart of the GLC process, and analytical sensitivity depends in large part on its sensitivity. Many of these tests were made at maximum electrometer sensitivity settings of range 0.1, attenuation 1. This required that electronic components, particularly tubes, be in excellent operating condition. A small waffle-type blower was mounted on the 204-1B electrometer to facilitate air circulation and to minimize possible thermal effects of the electronics.

Phenolic components were determined at approximately 1 and 100 mg. per liter concentrations, either singly or as mixtures. The higher concentrations were used for convenience during screening and do not reflect the sensitivity of the technique. Sample sizes of 3 μ l. were generally injected. Similar-sized water injections were made between runs to minimize ghosting and to keep the injection port and flame tip clean. The base line stability and presence of ghosts or artifacts were checked by making the water injections at maximum electrometer sensitivity setting regardless of the chromatographic analytical conditions. The limit of analytical detection was estimated by the quantity of phenolic component necessary to produce a measurable peak. The minimum measurable peak is defined as the smallest area which may be reproduced within 10% at maximum electrometer sensitivity for the chart speed being used. Signal-noise ratio was not considered as an adequate definition in this study. To provide a common basis of comparison, all calibration factors are reported as nanograms per square inch peak area (ng. per sq. inch), and based on a chart speed of 90 inches per hour, 1-mv. full scale response with electrometer range 1, attenuation 1 (Table I). These sensitivity values are presented for comparative purposes only and vary with columns and chromatographic instruments. The elution interval of the various phenolic materials relative to phenol is reported in Table I. The actual elution interval of phenol corresponding to the data of Table I is reported in Table II. Column performance was judged by peak symmetry and lack of tailing, component resolution,

Table II. Representative Phenol Elution Intervals

Column ^a	Temp., ° C.	Carrier Flow, Ml./Min.	Time, Min.
10-ft. 15% STAP on 70/80 W	190	20	10
5-ft. 10% STAP on 70/80 T	190	20	6.5
5-ft. 10% STAP on 60/80 W	190	20	6.5
10-ft. 10% Carbowax 1540 on 60/80 W-HMDS	170	40	18.5
10-ft. 20% Carbowax 4000-TPA on 60/80 W-HMDS	170	40	12.5
10-ft. 20% Carbowax 20M-TPA on A/W 60/80 W-HMDS	210	20	8.0
10-ft. 20% Carbowax 20M-TPA on 60/80 W	210	20	10.9
10-ft. 20% Carbowax 20M on A/W 60/80 W	210	20	18.6
5-ft. 15% UCON 50LB550X on 60/80 W-HMDS or DMCS	170	20	7.2
10-ft. 10% FFAP on 60/80 T	170	60	7.5
5-ft. 10% FFAP on 60/70 T	170	30	7.5
5-ft. 5% FFAP on A/W 70/80 W-DMCS	150	40	8.0
5-ft. 10% FFAP on A/W 60/80 W-DMCS	170	20	6.2
5-ft. 2% FFAP on A/W 60/80 W	170	20	3.0
5-ft. 5% FFAP on A/W 70/80 W	150	40	8.0
5-ft. 10% FFAP on A/W 60/80 W	170	20	8.5
5-ft. 10% FFAP on 60/80 W	190	20	4.8
10-ft. 10% FFAP on 60/80 W	170	20	18.5
5-ft. 5% FFAP on JM porous polymer	190	20	12

^a All columns 0.125-inch diam. stainless steel with Chromosorb supports.

elution interval, freedom from base line noise, and useful life. The initial response recorded for passage of the water matrix through the hydrogen flame was also examined. If water is adsorbed on the column substrate or support there is a significant tailing effect as it is desorbed. This may interfere with determination of rapidly eluting components.

Supports

Substrate supports for packed columns should not exhibit adsorptive tendency for gas-phase components. Adsorption leads to peak distortion of varying degree. Few supports are completely inert and most possess some measure of polarity. If there is a break or vacancy in the substrate coating, adsorption and peak tailing will occur, resulting in lack of resolution. Lightly loaded columns (less than 2%) are particularly subject to such problems. To minimize this effect, diatomaceous earth packings such as Chromosorb W (Johns Manville, New York, N. Y.) are chemically treated to deactivate surface sites. The treatment may involve acid- or base-washing which removes iron, aluminum, and other trace cations and/or treatment with liquids containing specific functional groups. Typical treatment materials are dimethyldichlorosilane (DMCS) and hexamethyldisilazane (HMDS). These surface silanol groups to silyl ethers. Acid-washed (A/W), silanized diatomite supports are frequently recommended for aqueous injection gas-liquid chromatography, since the polar matrix can itself produce severe tailing through adsorption on open sites. The diatomaceous earths are excellent supports for high-temperature operation, but this property is not important with steric substrates used in aqueous analyses, which are generally limited to operation at 250° C. or lower. Therefore, hydrophobic synthetic supports such as fluorocarbon resins have proved useful (Kirkland, 1963). Chromosorb T (Teflon 6, Johns Manville) has been used by this laboratory with considerable success for aqueous analyses, although it has a practical operating limit of 200° C. or less.

Frederick *et al.* (1962) and Sawyer and Barr (1962) have evaluated various column support characteristics and noted that establishing a basis for direct comparison is complicated and is particularly difficult for low substrate loadings and small sample size. Urone and Pecsok (1963) and Urone and Parcher (1966) show that support treatment greatly affects the specific retention volumes of polar compounds. These and other studies recommended support deactivation by chemical treatment.

The effect of the support and its treatment on a phenolic chromatogram is demonstrated in Figure 1. The substrate is 5 to 10% FFAP (Varian Aerograph) (polyester-type free fatty acid phase). All columns are 5-foot \times 0.125-inch stainless steel tubing packed with 60/70 Chromosorb T, 60/80 Chromosorb W, 70/80 A/W Chromosorb W, and 70/80 A/W Chromosorb W-DMCS. The peaks for *o*-chlorophenol and phenol are symmetrical in each case. The water tailing effect is the smallest for the fluorocarbon support, Chromo-

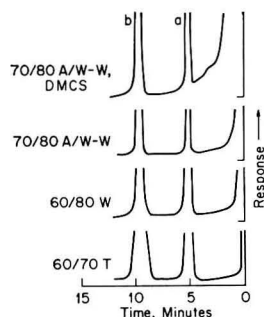


Figure 1. Effect of column support treatment

Aerograph 600D/328. 5-foot \times 1.125-inch stainless steel, 5 to 10% FFAP on Chromosorb packings. N_2 60 ml./min. H_2 25 ml./min. Chart. 12 inches/hr., 1 mv.; *R*/1, *X*/1; 1- μ l. sample of \sim 100 mg./liter of (a) *o*-chlorophenol and (b) phenol T_{col} , 150° to 170° C. T_{inj} , 210° to 250° C.

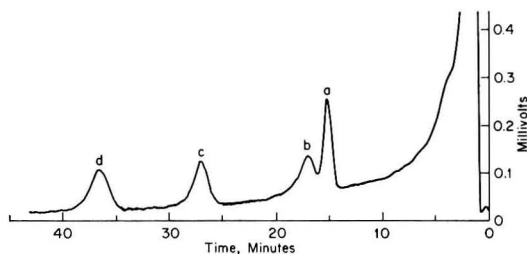


Figure 2. Phenolic analysis, Carbowax 1540

Aerograph 600D/328, 10-foot \times 0.125-inch stainless steel, 10% Carbowax 1540 on 60/80 Chromosorb W-HMDS. N_2 20 ml./min. H_2 25 ml./min. T_{col} , 172° C. T_{inj} , 214° C. Chart. 12 inches/hr., 1 mv., *R* 0.1, *X*/1, 1- μ l. sample of (a) 12.4 mg./liter *o*-chlorophenol, (b) 11.3 mg./liter guaiacol, (c) 9.8 mg./liter phenol, (d) 10.3 mg./liter *m*-cresol

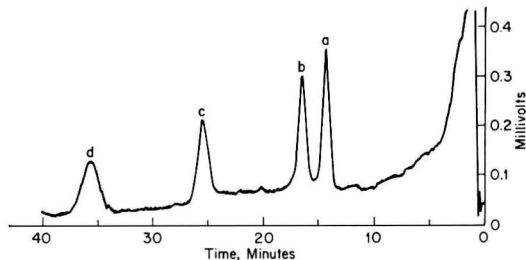


Figure 3. Phenolic analysis, Carbowax 4000 TPA

Aerograph 600D/328. 10-foot \times 0.125-inch stainless steel, 20% Carbowax 4000 TPA on 60/80 ChromosorbW-HMDS. N_2 40 ml./min. H_2 25 ml./min. T_{col} , 150° C. T_{inj} , 200° C. Chart. 12 inches/hr., 1 mv.; *R* 0.1, *X*/1; 1- μ l. sample of (a) 12.4 mg./liter *o*-chlorophenol, (b) 11.2 mg./liter guaiacol, (c) 9.4 mg./liter phenol, (d) 10.3 mg./liter *m*-cresol

sorb T, and the untreated diatomaceous earth, Chromosorb W. The acid-washing of Chromosorb W produced no significant effect on water tailing or peak shape. The acid-washed, silanized column produced the greatest tailing of the water peak, contrary to the behavior expected and generally recommended practice in GLC analyses. This effect has been observed by this laboratory with many steric or polar substrate-loaded columns used for aqueous-matrix analyses. Deactivation of the support is most likely to be important with lightly loaded (1 to 2%) columns where substrate discontinuities will expose active adsorption sites. For routine phenolic analyses, the combination of FFAP and Chromosorb T has proved most suitable. The combination produces one of the best general-purpose columns for analysis of many organic materials by aqueous-injection GLC if temperatures no greater than 200° C. are required. Higher temperatures lead to fusion of the support and decomposition artifacts. In such cases, the support properties may establish the practical upper limit for temperature-programmed or isothermal operation modes.

Substrates

The substrate or stationary phases found most suited for direct aqueous injection GLC for phenolics are of the steric type. These substrates exhibit a specific solute interaction typified by more rapid elution of phenolic compounds substituted ortho to the hydroxy group. Thus, *o*-cresol elutes well in advance of *m*- and *p*-cresol, which are not separable with these substrates and have a longer elution interval (Table I). A characteristic which limits application of these substrates to separation and identification of higher-boiling phenolics is their limiting maximum operating temperature of 200° to 250° C. Higher-boiling phenolics must first be transferred to a solvent matrix and analyzed on nonpolar columns rather than by direct aqueous-injection GLC.

The polyethylene glycols, Carbowaxes 1540, 4000, and 20M, and their chemical modifications have been determined to be among the best of the available substrates for aqueous phenolic analyses (Table I). The modified substrates include FFAP, a reaction product between Carbowax 20M and 2-nitroterephthalic acid and STAP, a steroid analysis phase modification.

The separation characteristics of monohydric phenolics using these substrates are summarized in Table I. Carbowaxes 1540 and 4000 have limiting operating temperature maxima of approximately 200° C., but are suited for phenolic analyses of the lower boiling compounds. The separation of a quaternary mixture of *o*-chlorophenol, guaiacol, phenol, and *m*-cresol, each at approximately 10 mg. per liter, is depicted in Figures 2 and 3 for Carbowaxes 1540 and 4000 TPA, respectively. Preliminary tests with the Carbowax 4000 had shown peak tailing and the TPA modification was used for the screening tests. The TPA treatment sharpens the peaks by reducing adsorption effects and associated peak spreading. It also improves sensitivity as shown for Carbowax 20M (Table I).

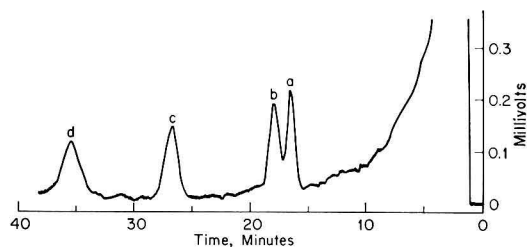


Figure 4. Phenolic analysis, Carbowax 20 M

Aerograph 600D/328; 10-foot \times 0.125-inch stainless steel, 20% Carbowax 20M on 60/80 A/W Chromosorb W
 N_2 , 20 ml./min. H_2 , 25 ml./min.
 T_{col} , 193° C. T_{inj} , 219° C.
 Chart, 12 inches/hr., 1 mv.; R 0.1, X 1; 1- μ l. sample of (a) 12.4 mg./liter *o*-chlorophenol, (b) 11.3 mg./liter guaiacol, (c) 9.8 mg./liter phenol, (d) 10.3 mg. liter *m*-cresol

The chromatograms of Figures 2 and 3 are for HMDS-silanized columns of equal length. Carrier flow rate is twice as great on the Carbowax 1540 column, but the elution interval is approximately the same for the phenolics. In addition, sensitivity is greater for the Carbowax 4000 TPA than the Carbowax 1540 column, phenol-calibrated at 32 vs. 42 ng. per sq. inch. Separation of the *o*-chlorophenol and guaiacol pair is essentially complete on the Carbowax 4000 TPA but only partial on the Carbowax 1540. These differences are attributed chiefly to the TPA treatment.

The higher molecular weight Carbowax 20M is less polar than Carbowax 4000. It has a recommended operating temperature of 250° C., permitting higher-boiling phenolic analyses than the aforementioned substrates. This substrate and its phosphoric acid modification have been used for phenolic analyses. Koloff *et al.* (1963) noted increased efficiency, decreased tailing, and improved sensitivity through phosphoric acid treatment of Carbowax 20M. They determined that an optimum amount of acid is required. Too much H_3PO_4 reduces retention volumes and resolution is lost. In this study, the terephthalic acid treatment was found effective in improving chromatographic analyses with Carbowax 20M. The effect of combined substrate TPA-treatment and support silanization is demonstrated by the separation of a quaternary mixture of approximately 10 mg. per liter each of *o*-chlorophenol, guaiacol, phenol, and *m*-cresol. The analysis on non-TPA-treated 20M supported on a non-silanized Chromosorb W is depicted in Figure 4. The initial water tail extends almost to the phenol peak. The *o*-chlorophenol and guaiacol peaks are reasonably well separated, though not completely. The phenol elution interval at 193° C. and nitrogen flow of 20 ml. per minute is approximately 27 minutes. All Carbowax 20M column variations were tested with 10-foot columns. The effect of column length has been reported (Baker, 1966a). The advantages of 20M with TPA treatment are demonstrated in Figure 5 and Table I. Peaks

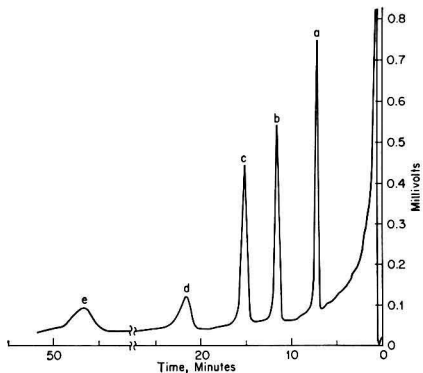


Figure 5. Phenolic analysis, Carbowax 20M-TPA

Aerograph 600D/328. 10-foot \times 0.125-inch stainless steel, 20% Carbowax 20M-TPA on 60/80 Chromosorb W N₂. 40 ml./min. H₂. 25 ml./min. T_{col} . 189°C. T_{inj} . 220°C. Chart. 12 inches/hr., 1 mv.; R 1, X1; 2- μ l. sample of \sim 50 mg./liter each of (a) *o*-chlorophenol, (b) phenol, (c) *m*-cresol, (d) 2,4-dichlorophenol, (e) *p*-chlorophenol

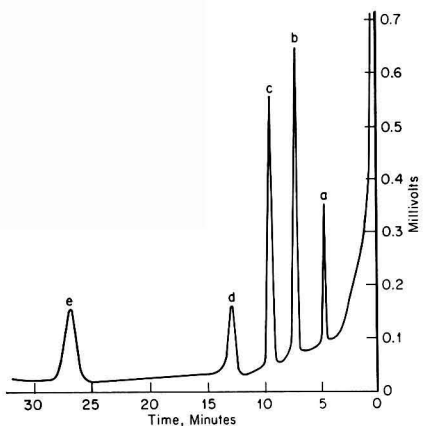


Figure 6. Phenolic analysis, Carbowax 20M-TPA

Aerograph 600D/328. 10-foot \times 0.125-inch stainless steel, 20% Carbowax 20M-TPA on 60/80 Chromosorb W-HMDS. He. 25 ml./min. H₂. 25 ml./min. T_{col} . 210°C. T_{inj} . 250°C. Chart. 12 inches/hr., 1 mv.; R1, X1; 1- μ l. sample of \sim 100 mg./liter each of (a) *o*-chlorophenol, (b) phenol, (c) *m*-cresol, (d) 2,4-dichlorophenol, (e) *p*-chlorophenol

are sharper, elution is more rapid, and sensitivity is markedly improved—i.e., phenol calibration was determined as 29 vs. 48 ng. per sq. inch. However, HMDS-silanzation decreased sensitivity and increased tailing (Figure 6). The calibration factor for phenol is 45 ng. per sq. inch. A similar decrease in sensitivity is noted for the other monohydric phenolics tested. Relative elution values of the longer interval eluters such as the *m*- and *p*-chlorophenols are somewhat greater for the Carbowax 20M on untreated support. TPA treatment obviously improves analytical performance in aqueous matrix GLC.

Various dichlorophenolics were analyzed. Their separation illustrates the effects of ortho-substitution and degree of hydrogen bonding on the elution interval. With the Carbowaxes, as with other steric-type substrates studied, the 2,6-dichlorophenol elutes before the 2,4-dichlorophenol despite a 10°C. higher boiling point. Kolloff *et al.* observed this with a Carbowax 4000-H₃PO₄ column and demonstrated that efficiency decreased with temperature for the 2,4 compound but increased for the 2,6 compound with a decrease in temperature. This is attributed to greater hydrogen bonding with the substrate for the 2,4 than the 2,6 compound. As temperature decreases, the bonding effect increases and peaks tend to spread wider and elute more slowly with these substrates. No attempt was made in this study to establish relative efficiency as a function of temperature, although such an effect has been observed. This is one of many reasons why the analyst must not extrapolate previously determined chromatographic calibrations to other columns or operating conditions. The minimum detectable quantities and the calibration values of Table I will also vary with the sensitivity of the electrometer and the stability of operation the analyst has achieved by minimizing noise.

Another substrate which was found to condition rapidly, produce little tailing, and give good sensitivity (Table I) was STAP. However, STAP deteriorated rapidly after only a few days. Sensitivity decrease was noted within a single day of operation.

The polyester, free fatty acid phase substrate (FFAP) was the best general-purpose substrate tested for aqueous phenolics analyses (Table III). An application of FFAP to phenolic analyses has been reported (Baker, 1966b). The substrate has long life and has been used with synthetic and diatomaceous earth supports with equal success. FFAP columns supported on Chromosorb W and Chromosorb T have been used regularly for several months before sensitivity loss and retention time decrease necessitated replacement. The FFAP columns are easily preconditioned, generally within 24 hours, and the base line produced is relatively noise-free at maximum electrometer sensitivity. Bleed rate and resulting standing current are minimal. Phenol calibration values of approximately 18 ng. per sq. inch are obtained when FFAP is supported on Chromosorb T and the analyses are made using the 204-1B instrument (Figures 7 and 8). Phenol calibration factors with the 600D unit were of the

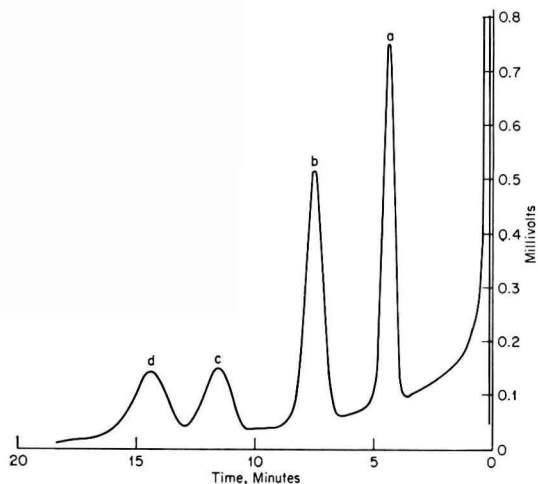


Figure 7. Phenolic analysis, FFAP

Aerograph 204-1B, 5-foot \times 0.125-inch stainless steel, 10% FFAP on 60/80 Chromosorb T
 N₂, 40 ml./min. H₂, 25 ml./min.
 T_{col.} 170°C. T_{inj.} 263°C. T_{det.} 227°C.
 Chart. 24 inches/hr., 1 mv.; R 0.1, X 1, 1- μ l. sample of (a) 12.4 mg./liter *o*-chlorophenol, (b) 9.4 mg./liter phenol, (c) 8.4 mg./liter 2,6-dichlorophenol, (d) 10.4 mg./liter 2,4-dichlorophenol

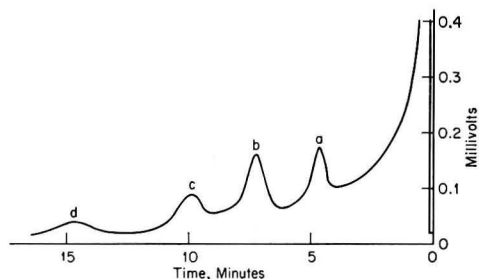


Figure 8. Phenolic analysis, FFAP

Aerograph 204-1B, 5-foot \times 0.125-inch stainless steel, 10% FFAP on 60/80 Chromosorb T
 N₂, 40 ml./min. H₂, 25 ml./min.
 T_{col.} 170°C. T_{inj.} 260°C. T_{det.} 225°C.
 Chart. 24 inches/hr., 1 mv.; R 0.1, X 1; 2- μ l. sample of (a) 1.13 mg./liter guaiacol, (b) 1.22 mg./liter *o*-cresol, (c) 1.03 mg./liter *m*-cresol, (d) 1.30 mg./liter 2,3-dichlorophenol

order of 29 ng. per sq. inch. This difference in calibration, 29 vs. 18 ng. per sq. inch, illustrates the effect of increased electrometer sensitivity. The relative retention of the individual phenolics is unaffected by column length. As expected, elution intervals are extended by using the longer column.

For 10-foot, 10% FFAP columns, Chromosorb T yields phenol calibration of 29 vs. 42 ng. per sq. inch for Chromosorb W with the 600D instrument. The synthetic polymer support

gives longer elution intervals at comparable operating conditions, but peak resolution tends to be better than with the diatomite support. No measurable difference in peak separation efficiency, calibration, or elution interval was indicated for acid-washed vs. non-acid-washed diatomite.

Silanization of Chromosorb W by DMCS did not affect relative elution or calibration factors, although as previously noted for other substrates, tailing, particularly of the water

Table III. Phenolic Retention and Calibration for FFAP Columns

FFAP Loading	10%		10%		10%		2%		5%		10%		5%		10%			
	Chromosorb support		60/80 T		60/80 W		60/80A/W		70/80		60/80		70/80		60/80			
Stainless steel column, 0.125-in. diam., length, feet	10		5		10		5		5		5		5		5			
	Aerograph Model No.		600D/328		204-1B		600D/328		600D/328		600D/328		600D/328		600D/328		600D/328	
Recommended max. temp., ° C.	200		200		275		275		275		275		275		275			
	Compound	Boiling Point, ° C.		r		c		r		c		r		c		r		c
Phenol		182	1.0	29	1.0	18	1.0	42	1.0	65	1.0	46	1.0	33	1.0	47	1.0	37
<i>o</i> -Cresol	192	1.0	27	1.0	18	1.0	43	1.0	60	1.0	53	1.0	32	1.0	57	1.0	32	
<i>m</i> -Cresol	203	1.3	30	1.4	19	1.3	40	1.3	75	1.3	53	1.3	36	1.4	46	1.4	37	
<i>p</i> -Cresol	202	1.3	31	1.4	19	1.3	40	1.3	75	1.3	53	1.3	36	1.4	49	1.4	37	
<i>o</i> -Chlorophenol	176	0.6	45	0.6	23	0.6	62	0.6	104		0.6	48		0.6	48		54	
2,3-Dichlorophenol	...	1.9	47	2.0	39	1.9	79	2.0	140		1.8	65		1.9	67		67	
2,4-Dichlorophenol	210	1.9	55	2.0	38	1.9	98	2.0	137		1.8	68		1.9	76		76	
2,6-Dichlorophenol	220	1.5	50	1.5	38	1.5	78	1.4	130		1.4	69		1.5	74		74	
Guaiacol	205	0.7	56	0.7	26	0.7	55	0.7	95		0.6	44		0.7	45		45	

peak, is more pronounced. At 170° C. and carrier flow of 20 ml. per minute, 10% FFAP on A/W Chromosorb W gave a phenol peak elution in 8.5 minutes *vs.* 6.2 minutes for the comparable DMCS-treated column.

Substrate loadings of 2, 5, and 10% FFAP on 5-foot columns packed with 60/80 A/W Chromosorb W demonstrated that relative retention is essentially the same; calibration values increase as loading decreases because of increased adsorption; phenol retention of 8.5 minutes for the 5 and 10% loaded columns drops to 3 minutes on the 2% column at comparable operating conditions; peak tailing was evidenced at 2%; and the 2% column showed loss of sensitivity and resolution after a few days, although 5 and 10% columns have been used for months without deterioration (Figures 9 and 10). The observation that relative retention of these phenolics did not vary significantly as substrate loading was varied between 2 and 10% is interesting in reference to recent observations by Mitzner *et al.* (1966). These investigators reported that as Carbowax 20M loading decreased from 20% to 5% to 1%, differences in retention of solutes were amplified. The loading range studied here was not as great and differences in relative retention, if they exist, are less likely to be evident. The condensation reaction which produces FFAP from Carbowax 20M obviously results in generally improved performance. Lightly loaded columns give greatest efficiency. Ideally, a thin film of substrate of low bleed rate on uniform-sized small particles gives greatest resolving power. However, per cent loading must be a compromise between longer column life and maximum efficiency. The 5 and 10% FFAP columns have been very satisfactory in day-to-day service.

The polypropylene glycol-type substrates are generally suited to analyses of polar solutes. Of particular interest was the LB product which is insoluble in water and contains an oxidation inhibitor, Ucon 50 LB-550-X. This column (Table I) conditioned rapidly, and exhibited reasonable sensitivity (phenol calibration of 45 ng. per sq. inch), good peak separation, and rapid elution with both HMDS- and DMCS-silanized columns. The Ucon columns deteriorated rapidly, however, in phenolic analytical service, although they have been used successfully over long periods for ketones, aldehydes, and alcohols.

Table IV. Effect of pH in GLC Analyses of 2,4-Dichlorophenol

Column: 5 feet \times $\frac{1}{8}$ -inch SS, 5% FFAP, 60/80 Chromosorb W.

H₂, 25 ml./min. N₂, 25 ml./min. T_c = 176° C. T_i = 205° C. Chart. 90 inches/hr., 1 mv.; R 0.1, X 1; 3- μ l. samples in distilled water; pH adjusted by NaOH; initial dichlorophenolic concentration = 5 mg./l.

pH	4.5	7.7	9.7	10.8	11.7
Peak area, sq. inches	2.88	2.90	2.90	2.93	1.08

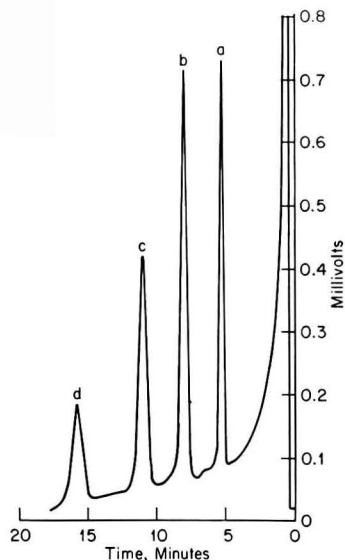
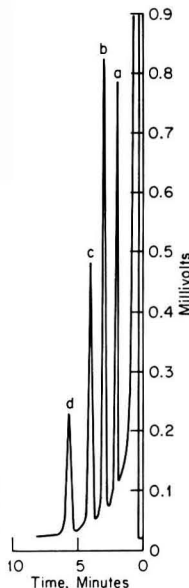


Figure 9. Phenolic analysis, FFAP

Aerograph 600D/328, 5-foot \times 0.125-inch stainless steel, 10% FFAP on 60/80 A/W Chromosorb W
He. 20 ml./min. H₂. 25 ml./min.
T_{col.} 170°C. T_{inj.} 212°C.
Chart. 12 inches/hr., 1 mv.; R 0.1, X 1; 1- μ l. sample of (a) 11.3 mg./liter guaiacol, (b) 12.2 mg./liter *o*-cresol, (c) 10.3 mg./liter *m*-cresol, (d) 10.4 mg./liter 2,3-dichlorophenol

Figure 10. Phenolic analysis, FFAP

Aerograph 600D-328, 5-foot \times 0.125-inch stainless steel, 2% FFAP on 60/80 A/W Chromosorb W
He. 20 ml./min. H₂. 25 ml./min.
T_{col.} 170°C. T_{inj.} 210°C.
Chart. 12 inches/hr., 1 mv.; R 0.1, X 1, 1- μ l. sample of (a) 11.3 mg./liter guaiacol, (b) 12.2 mg./liter *o*-cresol, (c) 10.3 mg./liter *m*-cresol, (d) 10.4 mg./liter 2,3-dichlorophenol



Synthetic porous beads (Hollis, 1966; Hollis and Hayes, 1966) of a highly cross-linked polystyrene, have demonstrated excellent separation of volatile organic components in aqueous solution. They may be used as combined substrate and support or coated with another substrate. These substrates eliminate the problems of support polarity or liquid phase volatility for many analyses. They usually produce sharp, symmetrical peaks and low retention volumes. Uncoated materials were not tested extensively, but a 5-foot \times 0.125-inch stainless steel column of 5% FFAP on porous polymer (Johns Manville, New York, N. Y.) has been tested. This porous polymer is comparable to Poropak Q (Waters Associates, Inc., Framingham, Mass.). This combination was not effective. A solute-substrate reaction took place. Individual phenolic components were not separated but eluted as an unresolved chromatographic hump. This has also been observed in limited tests with the uncoated porous polymer.

Analyses

The monohydric phenolics are weak organic acids exhibiting pK values of approximately 10 for phenol and cresols and 7.4 for the dichlorophenols. The effect of sample pH on GLC analysis was determined for the more acidic 2,4-dichlorophenol (Table IV). A 5 mg. per liter solution of the phenolic was adjusted with sodium hydroxide to pH values of 4.5 to 11.7. The chromatographic peak area of 3- μ l. sample injections at fixed operating conditions for an FFAP column did not vary significantly up to pH 10.8. At pH 11.7, however, response was less than 40% of the original value, indicating the effect of the corresponding sodium phenolate formation.

The GLC method has been compared with the 4-aminoantipyrene colorimetric procedure (Baker, 1966b) with 1 mg. per liter concentrations of phenol and the three cresols. This is a difficult quaternary mixture with both methods. Both measure all the phenol. The colorimetric response to the *o*-, *m*-, and *p*-cresols is approximately 63, 38, and 0%, respectively. The GLC method responds quantitatively to all four, but does not resolve by single-column operating mode the pairs phenol and *o*-cresol and *m*- and *p*-cresol (Table I). The composite mixture was measured as 2.4 mg. per liter as phenol by the colorimetric procedure and approximately 4 mg. per liter by GLC. The GLC result could vary ± 0.1 mg. per liter, depending on the calibration values used to calculate the results. Thus, the concentration reported for a peak should specify the basis of calculation.

The milligram per liter concentration level is the practical lower limit of GLC analyses with the equipment and procedure described. Preliminary concentration is necessary for microgram per liter phenolic levels. Current research in this laboratory has demonstrated that phenolics may be concentrated in a nonselective manner by freezing from aqueous solution (Baker, 1967a, 1967b). Combination of freeze concentration and direct aqueous injection GLC has proved to be a very useful analytical procedure in laboratory and field analytical situations (Baker and Malo, 1967).

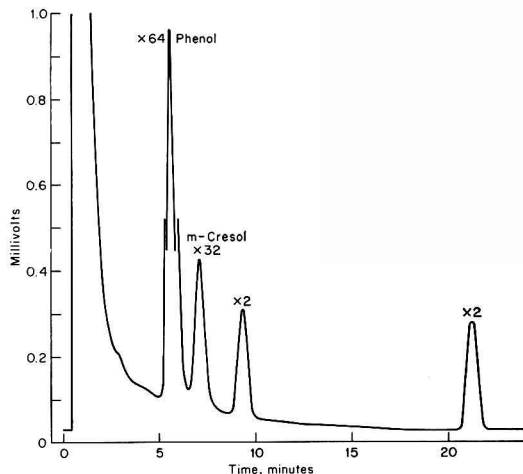


Figure 11. Coke plant ammonia still waste water without dephenolizer in operation

Aerograph 204-1B, 5-foot \times 1/8-inch diameter stainless steel, 10% FFAP on 60/80 Chromosorb W
 N_2 , 20 ml./min. H_2 , 25 ml./min.
 T_{col} , 190°C. T_{inj} , 237°C. T_{det} , 245°C.
 Chart, 24 inches/hr., 1 mv.; R 0.1, 2- μ l. sample

An application of the GLC method and a comparison of results with the 4-aminoantipyrene method are depicted in the analyses of phenolics in a coke-plant, ammonia-still waste water (Figures 11 and 12). This example does not involve operation at maximum sensitivity but does illustrate an application of the technique. The phenol concentration in the sample taken when the dephenolizer was not operating was analyzed as 1360 mg. per liter as phenol by the colorimetric procedure and 1575 mg. per liter as phenol and 300 mg. per liter as *m*-cresol by the GLC procedure. The GLC peak labeled phenol would include *o*-cresol and the *m*-cresol peak would include *p*-cresol if these are present. Two additional organic components were indicated at retention times of 1.8 and 4.1 relative to phenol. These may be estimated at approximately 20 to 30 mg. per liter based on calibration values twice as great as phenol. The phenol content of the waste water with the dephenolizer in operation was measured as 36 mg. per liter by the colorimetric procedures. The corresponding GLC analysis for this water is 27 mg. per liter as phenol, 11 mg. per liter as *m*-cresol, and three minor components each of 3 to 7 mg. per liter concentration. Phenolic reduction by 4-aminoantipyrene is 1360 to 35 mg. per liter or 97.4%. Although over-all reduction is comparable in this case, the GLC method differentiates major components and indicates presence of materials not measured by the colorimetric test.

Temperature Programming

Mixtures of monohydric phenols ranging in boiling points from 176° C. (*o*-chlorophenol) to 254° C. (3,4-dichlorophenol) have been analyzed by linear temperature-programmed GLC to determine the effect on peak separation, speed of analysis, and sensitivity over that attainable by an isothermal operating mode. The columns employed were 5- and 10-foot \times 0.125-inch diameter, 10% FFAP on Chromosorb T and Chromosorb W. Experimental variables included: initial oven temperatures of 50° to 100° C., program rates from 2° to 10° C. per minute at 2° C. intervals, and terminal temperatures of 170° C. for Chromosorb T and 170° to 225° C. for Chromosorb W. After the maximum column temperature was achieved, it was maintained for a period sufficient to effect elution of all phenolics being tested.

Because of the structural similarities and relatively narrow boiling point range of the phenolics examined, no advantage was observed for the temperature program mode over the isothermal mode of GLC analysis.

As the temperature programming rate and/or initial temperature was increased, the tendency for peak overlap increased. Elapsed analytical time was often greater and the measurement of certain peaks was less precise because of increased substrate bleed and associated base line shift. However, if the phenolics occur in complex mixtures with other organic contaminants, temperature programming is necessary to maximize component resolution. An example of such an analysis has been reported (Baker and Malo, 1967).

Carrier Gas Modification

Ghosting, the appearance of peaks of the same elution interval as previously chromatographed components, is a common GLC problem, and is particularly evident at maximum electrometer sensitivity when minimal concentrations are being analyzed. In this laboratory, ghosting is eliminated by water injections between aqueous sample injections. This clears the base line of artifacts or ghosts. The possibility of using steam-enriched carrier gas to effect continuous purging of artifacts and to stabilize operation was of interest. There are few references to such practice in the literature. Hill and Newell (1965) reported the effect of water on flame-ionization detector standing current. Cochran (1966) observed that steam-nitrogen as carrier altered relative retention times of alcohols and benzene-type materials, but furnished no quantitative information. Most laboratories that replied to direct inquiries had avoided such studies because of the difficulty of controlling delivery of low volumes of steam in steady flow or because the steam or steam-nitrogen mixture promoted rapid substrate stripping.

Exploratory tests were made with a modified Aerograph Model 675 steam generator. A voltage regulator was used to stabilize line current; a variable Powerstat was added for precise boiler temperature control; and the flow pattern was adjusted so that incoming nitrogen passed only through the boiler vapor space. The moisture content of the nitrogen was

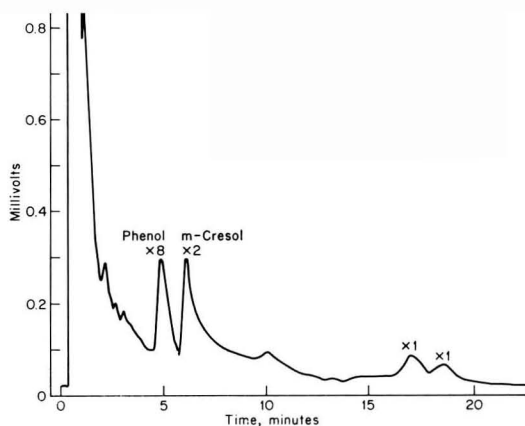


Figure 12. Coke plant ammonia still waste water with dephenolizer in operation

Aerograph 204-1B, 5-foot \times $\frac{1}{8}$ -inch diameter, stainless steel, 10% FFAP on 60/80 Chromosorb W
 T_{col} , 190° C. T_{inj} , 250° C. T_{det} , 265° C.
 N_2 , 25 ml./min. H_2 , 25 ml./min.
Chart, 24 inches/hr, 1 mv.; R 0.1, 2- μ l. sample

precisely controlled as a function of temperature and flow rate. The carrier gas lines from the steam generator to the column oven were electrically heated to preclude water condensation. It was not possible to operate with the steam-nitrogen carrier mixture at higher column temperatures without rapid substrate loss. A 20% Carbowax 20M column deteriorated in less than 6 hours at 210° C. when the carrier contained 1000 mg. of H_2O per mole of N_2 . Phenolic analyses were made with a 5-foot \times 0.125-inch diameter column of 10% FFAP on A/W 60/80 Chromosorb W at 145° C. and 20 ml. per minute carrier flow. The low temperature resulted in extended elution intervals (phenol at 35 minutes), but this was necessary to prevent excessive substrate loss. Replicated chromatograms of the ternary mixture *o*-chlorophenol, phenol, and *p*-cresol with and without a supplemental water loading of 100 mg. of H_2O per mole of N_2 were highly reproducible. The water-enriched carrier produced peaks only about 35% in area of those without water added (Table V). No change in elution interval or relative elution was evident. The initial water peak was significantly reduced but not eliminated. Ghosts were reduced in size but were still evident in the first water-wash injection made after the sample injection. No further tests were made because of the loss of sensitivity.

Summary

A variety of steric-type substrates were evaluated for their suitability as gas-liquid chromatographic column liquid phases for phenolic analyses by direct aqueous injection. These substrates were supported on various diatomite and synthetic packings and tested over a range of GLC operating conditions using flame-ionization detection.

Contrary to general belief, it was shown that silanization of diatomaceous earth supports reduces polarity and leads to

Table V. Effect of Water in Carrier Gas on Phenolic Analyses

Component	Carrier Gas		Ratio of Peak Areas, Wet to Dry
	N ₂	1100 mg. H ₂ O	
		Relative Retention	
<i>o</i> -Chlorophenol	0.52	0.52	0.33
Phenol	1.00	1.00	0.35
<i>p</i> -Cresol	1.40	1.40	0.39

increased peak tailing and decreased sensitivity. Acid washing of diatomites does not seem to affect analyses. Termination with terephthalic acid reduces peak tailing. Synthetic supports of the hydrophobic, fluorocarbon-type such as Chromosorb T were found to be ideally suited to aqueous phenolic analyses at temperatures up to 200° C.

Although many substrates will separate and permit quantification of selected monohydric phenolic materials, Carbowax 20M and FFAP were determined to be the best now available. FFAP is an improvement over Carbowax 20M and is a condensation product of Carbowax 20M and 2-nitroterephthalic acid. Substrate loadings of 5 to 10% FFAP provide reasonably good separation efficiency and long column life. The combination of FFAP supported on Chromosorb T provides maximum separation, highest sensitivity, and symmetrical peaks with minimum tailing. As with all steric substrates, the ortho-substituted phenolics elute ahead of the meta- and para-substituted compounds. The meta and para forms are not separated. No single column is capable of complete resolution of all monohydric phenolics. Supplemental separation and identification by spectrographic or other chromatographic procedures are necessary for more complex separation.

Temperature programming did not improve separation efficiency, sensitivity, or elapsed analytical time over an isothermal operating mode when only monohydroxy phenolics were involved. When these phenolics occur as components of a complex mixture, temperature programming may offer advantages.

Addition of water vapor or steam to the nitrogen carrier gas reduced but did not eliminate ghosting or memory peak formation. The water vapor accelerated substrate loss and reduced analytical sensitivity. The relative order of elution and individual component elution intervals were not affected by the water vapor.

The advantage of the direct aqueous injection GLC pro-

cedure described is that it will separate many but not all monohydroxy phenolic components occurring in mixture. Without preliminary sample concentration, the limiting concentration for analyses is of the order of 1 mg. per liter.

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Continuous Monitoring of Organophosphorus Compounds in Air with an Alkali Metal-Dual Flame Ionization Detector

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■ The Karmen alkali metal flame ionization detector was adapted to continuous monitoring of air for trace quantities of organophosphorus compounds. To achieve selective detection, a two-stage detector was used. Long operating life could be obtained with a sodium silicate-coated electrode coil. After optimizing the operating parameters it was possible to detect 0.1 μg . of organophosphorus compound per liter of air. Much larger concentrations of nonphosphorus organic compounds gave no response under the same conditions.

With the increasing use of pesticides, concern has arisen regarding the effects of the introduction of these toxic materials into the atmosphere. Consequently, some attention has been given to determining the quantities of these materials present in air. Methods have been described for sampling of the atmosphere for organophosphorus pesticide vapors and aerosols and subsequent analysis of collected samples by gas chromatographic (Tabor, 1966) or colorimetric procedures (Lloyd and Bell, 1966).

The purpose of this work was to develop instrumentation and procedures for the direct and continuous monitoring of the atmosphere for the presence of organophosphorus compounds, without prior collection and concentration of samples. The method was desired to be rapid, sensitive, and selective. The authors thought that the thermionic detector (Karmen and Giuffrida, 1964), which, in conjunction with a gas chromatograph, has been used extensively for the analysis of organophosphorus pesticide residues, should be suitable for this purpose. This is a flame ionization detector with an alkali metal salt in the proximity of the flame and exhibits partic-

ularly good sensitivity for organophosphorus compounds. Selective responses to such substances have been achieved with a dual-flame thermionic detector following separation in a gas chromatograph (Karmen, 1964). The upper burner of this device responds only to organic phosphorus and halogen compounds, if other substances are efficiently burned in the lower flame. If such a detector could be operated to sample air directly at relatively high flow rates without requiring a chromatograph for separation of atmosphere components, it should be possible to use it for rapid and reliable detection of trace amounts of organic phosphorus compounds in air.

Experimental

Apparatus. The apparatus is shown schematically in Figure 1. The two-burner detector used was a "stacked thermionic flame detector" (Microtek Instruments, Inc.). It was modified for continuous sampling by attaching to the carrier gas inlet of the bottom burner a $\frac{1}{4}$ -inch Swagelok nut to which was silver-soldered a stainless steel socket joint. Another stainless steel socket joint was silver-soldered to the exit cap of the top burner for connection to a pump to draw samples continuously through the detector. Water produced in the burners was kept out of the pump by inserting a trap between it and the detector. Organic phosphorus compound vapors at low concentrations in air were generated in a gas dilution apparatus previously described (Prager, 1966) and drawn directly into the detector through the modified carrier gas inlet of the bottom burner. A Keithley Model 610B electrometer, was used to monitor the signal from the lower burner. The signal from the upper burner was measured with a Keithley Model 417 picoammeter. The signals were recorded on a Picker X-Ray, 1-ma. dual pen rectilinear strip chart recorder. The electrode coils were heated with Microtek stacked flame power supplies, and B batteries were used to apply polarizing potentials to both detector stages. In some phases of the work, a Varian Aerograph Model 600 HY-FI gas chromatograph was used. Its flame ionization detector was converted into a single-stage thermionic detector by replacing the ignitor assembly with a five-turn Nichrome wire coil coated with an alkali metal salt, and positioned 1 to 2 mm. above the tip of the flame and concentric with it. For the chromatographic experiments a 5-foot by $\frac{1}{8}$ -inch O.D. stainless steel column was used, filled with 15% SE 30 silicone gum rubber and 2% polyethylene glycol 20M on Gas Chrom Z, 60- to 70-mesh.

Procedure. The chromatograph with the single-stage detector was used to compare the useful life of various alkali

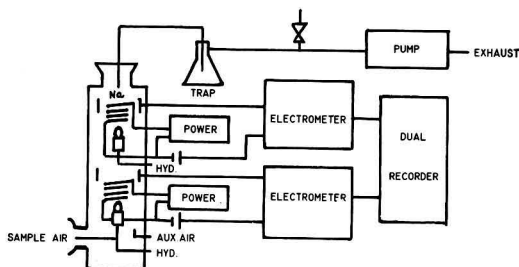


Figure 1. Stacked thermionic detector

metal salt coatings as well as their enhancement of the ionization current from organic phosphorus compounds. Electrode coils were generally coated by dipping them into aqueous solutions of the appropriate salt and letting them dry. Coils were coated with sodium silicate by applying a thick paste of Metso Anhydrous (Philadelphia Quartz Co.), letting them dry first in an oven at 110° C., and finally electrically heating the coated coils in the assembled detector. The various coatings were evaluated by comparing the responses of detectors with coated and uncoated coils to carbon disulfide solutions of dimethyl methyl phosphonate and decahydronaphthalene.

For continuous monitoring, low concentrations of organic phosphorus compounds in air from the gas dilution apparatus were sucked directly into the detector's bottom burner inlet and mixed with hydrogen. Additional air to support combustion was provided in the burner chamber at the point of ignition. The coil surrounding the bottom flame was not coated but served to ignite the flame and also as one electrode. The ion-collecting electrode was situated above the flame. The burned gases from the lower flame flowed into the upper burner, where additional hydrogen and air were supplied. The electrode coil surrounding the flame was coated with an alkali metal salt and the collector electrode was situated above it.

Effects of polarizing potential, hydrogen and sample air flow rates, and alkali metal salt coating were investigated.

Results

Initial experiments with the chromatograph and single-stage detector, with the widely used sodium sulfate coating, showed an enhancement in ionization current from organic phosphorus compounds. On repeated use for several days, the enhancement, however, diminished, and the coating gradually burned off and required replacement. Similar results were experienced with a variety of other salts, making such materials undesirable for use in a field instrument to be continuously operated. The authors thought that a more adherent coating with a longer useful life could be obtained with a glassy substance such as sodium silicate. Results (Table I) indicate little loss in performance when a detector coil coated with sodium silicate was operated for over a month continuously on an 8-hour day, 5 days a week basis.

The electrode coil of the upper burner of the two-stage detector was subsequently coated with sodium silicate, and operating conditions of the detector were optimized for use for continuous air monitoring without a chromatograph.

Electrical heating of the coated coil of this detector has been recommended (Abel, Lanneau, *et al.*, 1966). In this work, heating of the coated coil, which was negatively charged and electrically connected to the burner, resulted in an erratic base line and was therefore avoided. When the detector was operated under optimum conditions without electrically heating the coated coil, the temperature at that coil was measured to be about 700° C. This is in agreement with Karmen's observation that best results are obtained when the coated metal is heated to red heat (Karmen, 1964). Electrical heating of the electrode coil of the bottom burner by passing a current of 3.4 amperes through it improved the response obtained from organic compounds without affecting response in the upper burner. This coil was, therefore, heated electrically during operation of the detector to discriminate better

Table I. Stability and Performance of Sodium Silicate Coating

Coating	Age of Coating, Days	Detector Response to	
		0.08 µg. DMMP ^a	0.4 µg. Decalin ^a
No	...	2	17
Yes	1	425	16
Yes	10	475	20
Yes	18	400	23
Yes	32	360	20

^aLiquid samples dissolved in carbon disulfide.

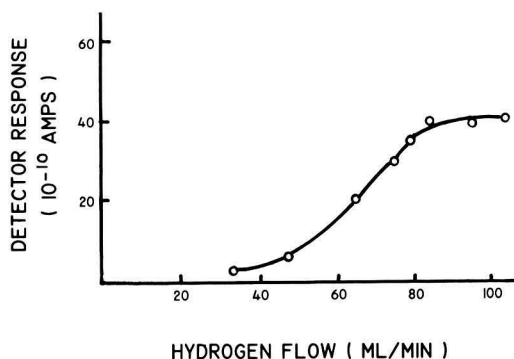


Figure 2. Effect of hydrogen flow of bottom burner on response

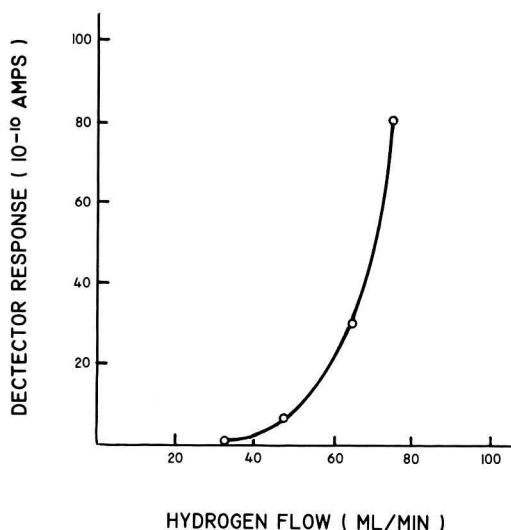


Figure 3. Effect of hydrogen flow of upper burner on response

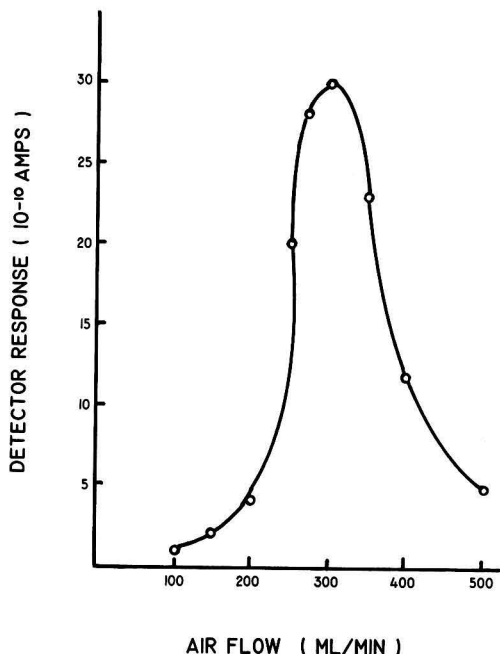


Figure 4. Effect of air flow on detector response

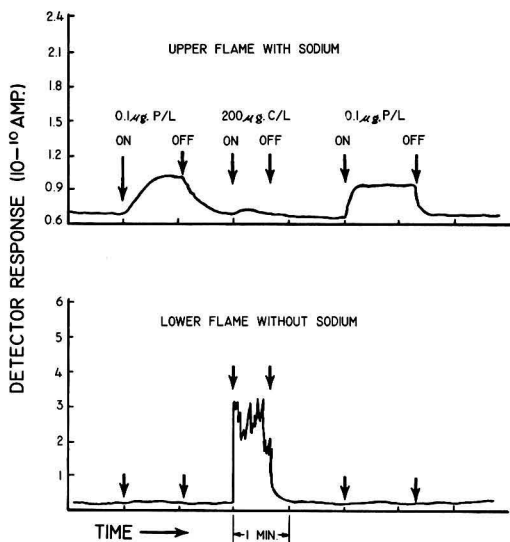


Figure 5. Dual recorder trace of stacked detector response to phosphorus and nonphosphorus compounds

between phosphorus and nonphosphorus compounds. The coil, electrically connected to the bottom burner, was negatively charged with respect to the collector as recommended for operation of flame ionization detectors (Sternberg, Gallaway, *et al.*, 1962).

The polarizing potential on the upper burner electrodes was varied from 150 to 400 volts without affecting the response of the detector. Complete ion collection was apparently achieved at 150 volts. The voltage on the lower burner electrodes was not varied, since this would not affect the phosphorus response in the upper burner. Polarizing voltages of 300 volts on both stages were used thereafter.

The hydrogen flow rates through both burners were separately controlled. Figure 2 shows that the detector response to organophosphorus compound increased as the flow rate through the bottom burner was increased to about 85 ml. per minute at constant air flow. At higher flow rates, no further improvement was obtained, and the flame became too noisy for satisfactory operation. Figure 3 shows that a much larger increase in signal was achieved as the hydrogen flow through the top burner was increased to 80 ml. per minute at constant air flow. At still higher flow rates, a noisy flame was noted. The response (Figure 4) is very much dependent on air sample flow and is greatest at about 300 ml. per minute. Variations in flame temperature appear to be primarily responsible for the great dependence of response on fuel and air flow rates. Flame temperatures determine the temperature of the coated coil and, therefore, signal and noise or sensitivity. Air flow further influences the response by its effect on the amount of sample delivered to the burner.

Response varied linearly with organophosphonate concentration over a range of 0.1 to 7 $\mu\text{g.}$ per liter of air. The minimum detectable concentration was determined to be 0.1 $\mu\text{g.}$ per liter of air on the basis of a signal to noise ratio of 2 to 1. This amount was detected in about 10 seconds. Concentrations of 0.4 $\mu\text{g.}$ per liter or greater were detected within 2 seconds.

Selectivity of the two-stage detector was investigated with a variety of hydrocarbons and oxygenated organic compounds. A typical result (Figure 5) shows a much smaller signal in the upper flame from a nonphosphorus compound containing 200 $\mu\text{g.}$ of carbon per liter of air, than from 0.1 $\mu\text{g.}$ of phosphonate per liter of air. In the bottom flame, however, only the nonphosphorus compound is detected, giving a very large signal.

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Influence of Hydrogen Fluoride Fumigation on Acid-Soluble Phosphorus Compounds in Bean Seedlings

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■ Bean seedlings were grown for four days following emergence in an atmosphere containing HF at 14 μg . of F per cu. meter. Their acid-soluble phosphorus composition was compared with that of seedlings grown in a decontaminated atmosphere. The seedlings in the HF treatment accumulated 275 p.p.m. of F but showed no symptoms of injury. The phosphorus compounds were extracted with 0.6*N* trichloroacetic acid and separated on an anion exchange resin column by elution with a variable gradient of formic acid and ammonium formate. There were no apparent differences between the atmospheric treatments with respect to the approximately 20 phosphorus compounds in the plant extracts. These results suggest that under some conditions plants can tolerate appreciable fluoride without significant inhibition of enzymes that catalyze reactions of phosphorus compounds.

Several enzymes that catalyze reactions of phosphorus compounds in plants are inhibited in vitro by fluoride or fluorophosphate. Included are enolase (Miller, 1958; Warburg and Christian, 1942), phosphoglucomutase (Chung and Nickerson, 1954; Yang and Miller, 1963), phosphatases (Bailey and Webb, 1944; Wildman and Bonner, 1947), and phosphorylases (Rapp and Sliwinski, 1956). However, attempts to detect these inhibitions in intact plants or to show that effects of fluoride on plants are related to inhibition of specific enzymes have been inconclusive.

Observed responses of plant respiration to fluoride have been ascribed to inhibition of enolase activity, but it is difficult to reconcile stimulation of respiration, which has been reported by several investigators (Applegate and Adams, 1960; McNulty and Newman, 1957; Weinstein, 1961), with enolase inhibition. Miller (1957) reported that spraying with pyruvate reduced the inhibitory effect of HF on growth of *Chenopodium murale* L., presumably because of compensation for inhibition of enolase. Enolase activity of leaf extracts was also reduced by the fluoride treatments. McCune, Weinstein, *et al.* (1964) reported increased enolase activity in leaf extracts of bean plants exposed to HF.

McNulty and Lords (1960) reported that increased oxygen consumption of *Chlorella* that was treated with NaF was associated with an increase in the total phosphorylated nu-

cleotides. Weinstein (1961) found indications of effects of fluoride on the RNA-phosphorus and DNA-phosphorus levels in tomato and bean plants.

Appreciable inhibition within the plant of any reaction in which a phosphorus compound is either a substrate or a product should affect the concentration of that compound and perhaps other compounds in the same sequence of metabolic reactions. In this study, the influence of HF fumigation on the concentrations of acid-soluble phosphorus compounds in bean seedlings was investigated.

Procedure

Tendergreen bean seedlings (*Phaseolus vulgaris* L.) were grown in a phytotron designed for air pollution research (Adams, 1961). The air entering the phytotron was cleaned to remove contaminants. Hydrogen fluoride was continuously injected by the method of Hill, Transtrum, *et al.* (1958) into the air stream entering one growth chamber. Control plants were grown in a comparable chamber in the decontaminated atmosphere. The light intensity was 1500 foot candles, and the day length was 13 hours. The temperature averaged 29° C. during the day and 13° C. at night.

The atmosphere of the HF chamber was sampled continuously through a fritted glass dispersion tower containing 0.01*N* NaOH. The solution was changed twice daily and analyzed for fluoride by thorium nitrate titration (Adams and Koppe, 1956). The average fluoride content of the control atmosphere was determined from a single integrated sample collected over the entire treatment period with a glass fiber filter (Pack, Hill, *et al.*, 1963).

Tendergreen bean seeds of uniform weight were planted in quartz sand in 1-quart polyethylene pots. Eight seeds were planted in each pot, and six pots were grown in each atmospheric treatment. The pots were automatically subirrigated three times daily with a complete nutrient solution.

The seedlings began to emerge 9 days after planting, and by the eleventh day over 90% had completely emerged. The plants were harvested 13 days after seeding. They were harvested 4 hours after the beginning of the light period to avoid changes in concentrations of phosphorus compounds that might occur during transition from dark to light. Any plants that were obviously irregular because of late emergence, lack of cotyledons, or distortion were discarded. Two samples of 10 to 12

grams fresh weight (about 10 seedlings) were collected at random from each treatment by severing the stems at the cotyledonary node. The cotyledons were not included. Therefore, the samples consisted of epicotyls, partially expanded primary leaves, and apical meristems. The samples were immediately weighed and frozen in 100 ml. of 0.6*N* trichloroacetic acid in diethyl ether at -80°C . A third sample from each treatment was dried in an oven at 70°C . for determination of its moisture content. It was then analyzed for fluoride by slurring with CaO, ashing, fusing with NaOH, isolating the fluoride by perchloric acid distillation, and titrating with thorium nitrate (Remmert, Parks, *et al.*, 1953; Willard and Winter, 1933).

The acid-soluble phosphorus compounds were extracted and separated essentially as described by Wilson and Harris (1966). Enzymes were denatured by homogenizing the samples in the trichloroacetic acid-ether solution with the homogenizer cup immersed in a Dry Ice-acetone bath. After centrifugation, the ether solution was extracted with water to remove any dissolved phosphate, and the residue was homogenized three times with 50 ml. of 0.6*N* aqueous trichloroacetic acid at 0° to 4°C . The trichloroacetic acid was removed from the combined extracts by washing five times with two volumes of cold diethyl ether in a separatory funnel.

The phosphorus compounds were adsorbed on a column of anion exchange resin and eluted with a variable gradient of formic acid and ammonium formate, with collection of 450 10-ml. fractions. Phosphate was determined by a modification of the Fiske-SubbaRow method (Bartlett, 1959b). The phosphorus compounds suspected of being hexose-P, P-glycerate, and adenosine triphosphate were each purified on a second ion exchange column. Hexose-P and P-glycerate were eluted with a gradient of formic acid, and adenosine triphosphate was eluted with the gradient of formic acid and ammonium formate. Hexose was determined by the anthrone method and fructose by the cysteine-carbazole method described by Bartlett (1959a). Formic acid was removed before measuring P-glycerate by the 4,5-dihydroxy-2,7-naphthalenedisulfonic acid (chromotropic acid) reaction (Bartlett, 1959c). Formic acid and ammonium formate were removed under vacuum with the aid of a heat lamp before measuring the ultraviolet absorption spectra of adenosine triphosphate at pH 2 and 11.

Results

The average fluoride content of the atmosphere, from the time the bean seedlings began to emerge until they were harvested (a period of 4 days), was 14 μg . per cu. meter in the fumigated chamber and less than 0.01 μg . per cu. meter in the control chamber. The seedlings grown in the HF treatment contained 275 p.p.m. fluoride on a dry weight basis. No fluoride could be detected in the control plants. The plants in the HF treatment showed no symptoms of fluoride injury.

Figure 1 shows the elution chromatograms of acid-soluble phosphorus in bean seedlings grown in the two atmospheric treatments. Perhaps the most significant thing about these curves is their close similarity with respect to all of the approximately 20 detectable phosphate compounds.

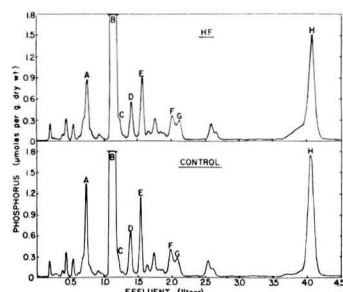


Figure 1. Separation of phosphorus compounds in acid extracts of bean seedlings grown in HF and control atmospheres

When the pooled fractions of peak *A* were eluted from a second ion exchange column with a 2-liter linear gradient of 0 to 4*N* formic acid, a small phosphate peak appeared just ahead of the main peak. Fractions in the main peak contained equivalent amounts of hexose and phosphate. Fructose-P was present in the trailing fractions and accounted for about 25% of the hexose-P. The small phosphate peak, which was about 15% as large as the main one, probably corresponds to the shoulder on the leading edge of peak *A* in the original chromatograms. The fractions in this peak exhibited ultraviolet (260 $\text{m}\mu$) absorption proportional to the phosphate content, indicating that it may be a nucleotide, but it was not further characterized. No differences were found in the composition of peak *A* for the different atmospheric treatments.

Comparison of phosphate analyses before and after acid hydrolysis indicated that peak *B* consisted entirely of inorganic phosphate except at the trailing edge where a shoulder (*C*) is evident. Some columns gave a clearly distinguishable phosphate peak at *C*.

Phosphoglycerate accounted for all of the phosphate in peak *E*. When the pooled fractions of this peak were eluted from a second column with a 2-liter linear gradient of 0 to 10*N* formic acid, the phosphate appeared in the same fractions as when authentic 3-phosphoglycerate was eluted with the same gradient. The unknown and authentic P-glycerate also gave identical absorption spectra in the chromatropic acid reaction.

The major component of peak *F* was identified as adenosine triphosphate by its ultraviolet absorption spectra at pH 2 and 11. Higher estimates of adenosine triphosphate from phosphate assay than from ultraviolet absorption suggested that another phosphate ester was also present in peak *F*.

The other peaks in Figure 1 were not identified in this study. In previous work with the same elution gradient (Wilson and Harris, 1966), known compounds have been eluted at positions corresponding to some of the other major peaks as follows: peak *C*, adenosine diphosphate; peak *D*, uridine diphosphate glucose; and peak *H*, inositol hexaphosphate.

The total phosphorus in each of the major peaks of Figure 1, obtained by subtracting the base line from the summation of the phosphorus in the individual fractions, is shown in Table I. The absence of effects of the atmospheric treatments is more obvious from these data than from the chromatograms because some differences in height of peaks are offset by inverse differences in width.

Discussion

The results of this investigation indicate that the HF treatment did not significantly inhibit any of the enzymatic reactions involving acid-soluble phosphorus compounds in the bean seedlings. Slight inhibitions may not have been evident in the measurements made, and shifts to alternate pathways may have compensated for inhibitions of some enzymes. However, it hardly seems conceivable that there was appreciable interference with phosphorus metabolism without obvious effects on the concentrations of some of the phosphorylated intermediates.

The lack of a difference in phosphoglycerate is particularly interesting in view of the common acceptance of enolase inhibition as a probable effect of HF on plants. Inhibition of enolase would be expected to result in accumulation of phosphoglycerate if there was no alternate pathway of phosphoglycerate metabolism, but no such accumulation was evident here.

Glucose-1-phosphate and glucose-6-phosphate were not separated. Therefore, no direct evaluation of phosphoglucotase inhibition is provided by the data. The total hexose phosphate and the peak believed to be uridine diphosphate glucose, both of which might be influenced by phosphoglucotase inhibition, showed a little more indication of effects of the atmospheric treatments than most of the other peaks. However, the differences did not approach statistical significance.

Although the bean seedlings showed no visible evidence of injury, the HF treatments were fairly high in comparison with concentrations of phytotoxic fluorides found in the ambient atmosphere, and considerable fluoride was accumulated by the seedlings during their exposure to it. Effects on respiration and biochemical composition have been reported for beans and other plants under less severe fluoride treatments (Applegate and Adams, 1960; McCune, Weinstein, *et al.*, 1964; Weinstein, 1961). The absence of effects in this study may reflect the influence of stage of plant growth or environmental conditions different from those where effects were reported.

The evidence that fluorine compounds inhibit enzymes that catalyze transformations of organic phosphorus compounds is well substantiated. However, demonstration of an enzyme inhibition *in vitro* may give little indication of its significance

in the intact plant. Different plant species show wide differences in resistance to fluoride, and even in sensitive species, much of the fluoride absorbed evidently fails to reach the metabolic sites in an active form. Failure of the HF treatment to produce significant differences in the acid-soluble phosphorus compounds in the bean seedlings is an example of accumulation of appreciable fluoride by plants without apparent effect. When fluoride injures plants, effects on phosphorus metabolism may be involved, but the mere presence of elevated fluoride levels in plant tissue may not signify either fluoride injury to the plant or effects on its fluoride-sensitive systems.

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Table I. Total Phosphorus in Major Acid-Soluble Phosphorus Peaks

Peak(s)	Phosphorus, $\mu\text{moles}/\text{Gram Dry Wt.}$	
	Control	HF
A (Hexose-P)	6.88 \pm 0.63	6.23 \pm 0.03
B (P _i) & C	188 \pm 10	216 \pm 0.5
D	3.11 \pm 0.13	2.77 \pm 0.11
E (P-glycerate)	4.00 \pm 0.20	4.28 \pm 0.29
F (ATP) & G	4.52 \pm 0.10	4.43 \pm 0.17
H	19.8 \pm 1.0	19.0 \pm 1.3

Further Observations on the Ferrous Ammonium Thiocyanate Reagent for Ozone

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■ Ferrous ammonium thiocyanate reagent was used for analysis of small amounts of ozone. The molar absorptivity was approximately constant at ozone levels above 2 p.p.m. (v./v.), but increased very rapidly at levels below 0.5 p.p.m. The maximum absorptivity is approximately 30,000 at ozone concentrations below 0.1 p.p.m. Bubbler frit size and collection rate were also important.

The authors have been concerned with the nature of oxidants produced photochemically in model systems. Recent publications from this laboratory have shown that with the judicious choice of methods, ozone, peracids, alkyl hydroperoxides, hydrogen peroxide, and PAN-type compounds can be determined quantitatively (Cohen, Purcell, *et al.*, 1967; Purcell and Cohen, 1967; Altshuller, Cohen, *et al.*, 1966).

During some recent work on the photooxidation of formaldehyde-NO_x mixtures, the authors have been unable to make meaningful quantitative measurements of the amount of oxidant produced. This observation has led to a reinvestigation of the ferrous ammonium thiocyanate colorimetric method for ozone.

Experimental

The reagents and chemicals, as well as the procedure employed in this work, were described in a previous paper (Cohen, Purcell, *et al.*, 1967). The data shown in Figure 1 were obtained by bubbling dilute ozone concentrations through 10 ml. of ferrous ammonium sulfate solution (0.1 gram of ferrous ammonium sulfate and 1.0 ml. of 6*N* sulfuric acid diluted to 200 ml. of water). The color development step was effected with a 2-ml. aliquot of a 50% (weight by volume) aqueous solution of ammonium thiocyanate. The absorbance was read at 480 m μ .

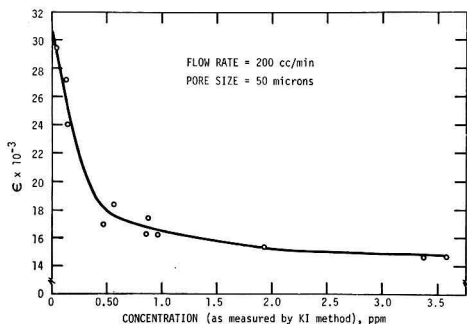


Figure 1. Molar absorptivity over a range of ozone concentrations as measured by the ferrous ammonium method

Results and Discussion

Further work with the method has shown that molar absorptivity, ϵ_m , is a function of ozone concentration. Figure 1 shows the molar absorptivity over a range of ozone concentrations as measured by the ferrous ammonium thiocyanate method. The molar absorptivity is approximately constant at ozone levels above 2 p.p.m. (v./v.), but it increases very rapidly at levels below 0.5 p.p.m. The maximum absorptivity is approximately 30,000. Why the method does not follow Beer's law with an oxidant such as ozone whereas it does so with *n*-butyl hydroperoxide, *tert*-butyl hydroperoxide, and hydrogen peroxide is unknown at present.

The method exhibits characteristics unlike those observed with the neutral KI method with which low ozone concentrations give low absorptivities (Saltzman and Gilbert, 1959).

The effect of bubbler frit size was also investigated. At an ozone concentration of 0.1 p.p.m., the molar absorptivities were 8000 and 24,000 at maximum pore diameters of 290 and 50 microns, respectively. [Pore diameter (microns) was measured by use of the equation $\mu = 30 s/p$, where s is the surface tension of water (dynes per centimeter) and p is the pressure in millimeters of Hg. This equation is derivable by equating the forces responsible for the internal pressure with those of the surface tension of water operating on a hemisphere.] These observations are again contrary to those associated with the neutral KI method, which gives better results with a large pore diameter.

Although the molar absorptivity increases with decreasing ozone concentration in the region usually encountered in polluted air, the ferrous ammonium thiocyanate method probably can be useful. For example, the method can be employed in the field and the color developed many days after collection of a sample. One laboratory has developed the color after 14 days (Clements and Morgan, 1967) and obtained reasonable values for the oxidant concentrations. No other known colorimetric method is as sensitive and still as stable. Therefore, if highly accurate ozone measurements are desired, a calibration curve should be made for use of the method at the lower levels of ozone. Frit size and collection rate should also be considered with such a calibration.

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Editorial Policy

ENVIRONMENTAL SCIENCE AND TECHNOLOGY places special emphasis on reporting original chemical research, engineering developments, and technico-economic studies in fields of science directly related to man's environment.

Contributed articles should be directed to all scientists and engineers concerned with fundamental and applied aspects of water, air, and waste chemistry and other relevant fields. These may include, but are not to be restricted to engineering, biology, ecology, economics, soil sciences, atmospheric sciences, geological sciences, marine sciences, and medical sciences. Contributions may emphasize improved understanding of water, air, and wastes from the viewpoint of their roles in pollution and its control and may consider their relationship to industrial and social progress.

Because a meaningful approach to the management of environmental quality involves more than scientific understanding, ES&T will devote serious attention to engineering, economic, legal, and other influences to give its readers an integrated view of this complex system.

The research pages of the journal are devoted to the publication of contributed and critically reviewed papers concerned with the fields of water, air, and waste chemistry, in particular, and with other scientific and technical fields, including the social sciences, which are relevant to the understanding and management of the quality of the air, water, and land environments. The journal attempts to serve the research interests of all scientists and engineers whose investigations and work fall within the broad field of environmental science and technology.

Contributed research papers will, in general, describe results of original research. Review articles will be considered when they serve to provide new research approaches or stimulate further worthwhile research in a significant area.

Communications as well as full length articles will be published in the journal. Communications may be letters describing preliminary results of immediate interest, or they may be significant comments on the work of others. The author whose work is being discussed ordinarily will be allowed to reply.

Author's Guide

This manuscript preparation guide is published to aid authors in writing, and editors and reviewers in expediting the review and publication of manuscripts in ENVIRONMENTAL SCIENCE AND TECHNOLOGY. For a detailed discussion, with examples, of the major aspects of manuscript preparation please refer to "Handbook for Authors of Papers in the Journals of the American Chemical Society."

Abstracts

An abstract which will appear at the beginning of each paper must accompany each manuscript. Authors' abstracts are frequently used directly for *Chemical Abstracts*. Use between 100 and 150 words to give purpose, methods or procedures, significant new results, and conclusions. Write for literature searchers as well as journal readers.

Title

Use specific and informative titles. They should be as brief as possible, consistent with the need for defining the subject of the paper. If trade names are used, give generic names in parentheses. Key words in the title assist in effective literature retrieval.

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List the first name, middle initials, and last name of each author. Omit professional and official titles. Underline the name of the senior author, to whom all correspondence will be sent. Give the complete mailing address where work was

performed. If present address of author is different, include the new information in a footnote.

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Avoid tables and graphs which duplicate information. In general, graphs are preferable to tables, if precise data are not required. Lengthy data tables are usually not necessary. However, certain types of articles—for example, those where correlation methods are compared—do require more than the usual quantity of data. Furnish tables with appropriate titles, and number them consecutively.

Manuscript Requirements

Three complete copies of the manuscript are required. They should be typed double- or triple-spaced on 8½- by 11-inch paper, with text, tables, and illustrations of a size that can be mailed to reviewers under one cover. Duplicated copies will be accepted only if very clear.

If pertinent references are unpublished, furnish copies of the work or sufficient information to enable reviewers to evaluate the manuscript.

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Prepare tables in consistent form, furnish each with an appropriate title, and number consecutively using Roman numerals in the order of reference in the text. Double space.

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If nomenclature is specialized, include a "Nomenclature" section at the end of paper, giving definitions and dimensions for all terms. Write out names of Greek letters and special symbols in margin of manuscript at point of first use. Complete definition includes dimension of each term. If subscripts and superscripts are necessary, place them accurately. Avoid trivial names. Trade names should be capitalized and defined at point of first use. Identify typed letters and numbers which could be misinterpreted, for example, one and the letter "i," zero and the letter "O."

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Chemical formulas should correspond to the style of *Chemical Abstracts*. Chemical equations should be balanced and numbered consecutively along with mathematical equations. The mathematical portions of the paper should be as brief as possible, particularly where standard derivations and techniques are commonly available in standard works.

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Authors are requested to call special attention—both in their manuscripts and in their correspondence with the editors—to safety considerations such as explosive tendencies, precautionary handling procedures, and toxicity.

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For a periodical reference to be considered complete, it must contain the author's(s') complete name(s), journal source, volume number, issue number (if any), the first and last page numbers of the article, and the year of issue. State if a reference is in a language other than English.

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Burnet, F. M., Stanley, W. M., Eds., "The Viruses," Vol. 1, pp. 609-15, Academic Press, New York, 1959.
Chen, D. H. T., M.S. thesis, University of Rhode Island, Kingston, R.I., 1961.
Handbook of Colorimetric Chemical Analytical Methods, 5th ed., pp. 13-15, Tintometer Ltd., Salisbury, England (1959).
Luthy, R. V. (to California Research Corp.), U.S. Patent 2,855,444 (Oct. 7, 1958).
Pollard, N. D., FMC Corp., Baltimore, Md., private communication, 1962.
Saltzman, B. E., *Anal. Chem.* **26**, 1949-55 (1954).
Schmauch, L. J., Grubb, H. M., *Anal. Chem.* **26**, 308-11 (1954).
U. S. Department of Health, Education, and Welfare, Public Health Service, Cincinnati, Ohio, "Selected Methods for Measurement of Air Pollutants," 1965.

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Water environment theories outweigh facts

Equilibrium Concepts in Natural Water Systems. *Advances in Chemistry* No. 67. Edited by Robert F. Gould. A symposium. viii + 344 pages. American Chemical Society, Washington, D. C. 20036. 1967. \$8.50, hard cover. *John D. Hem is a research chemist with Geological Survey, U. S. Department of the Interior, Menlo Park, Calif. 94025*

By John D. Hem

This volume contains 16 papers presented at the March 1966 meeting of the American Chemical Society in Pittsburgh, Pa. The symposium at which the papers were presented was sponsored by the Division of Water, Air, and Waste Chemistry and chaired by Prof. Werner Stumm.

To quote from Prof. Stumm's preface, "The symposium was organized to demonstrate that elementary physical chemistry can be used to isolate and identify some of the pertinent variables that determine the composition of natural water systems; to bring together analytical chemists, geologists, oceanographers, limnologists, and sanitary engineers who need to collaborate for a better understanding of our environment; and to stimulate research in aquatic chemistry and water analysis and to guide research workers in limnology, oceanography, and geochemistry."

The broad scope of the papers suggests that this book should contain information useful not only to practitioners in the field of environmental science, but to many others who might be interested in various aspects of aqueous heterogeneous systems. In general, the papers themselves fulfill this promise. Some are general surveys of topics related to the central theme, a few present new research

results, and some are almost entirely theoretical. However, all are thought-provoking and should stimulate the further research which is so obviously needed.

Any symposium volume presents difficulties in organization and tends to give a fragmented view of its topic. There are fewer problems of this sort here than might have been expected, however. In those papers which deal directly with equilibria in natural water, many cautioning statements point out lack of knowledge of one aspect or another. Some of the authors are much less cautious than others about making broad postulations from available data.

Equilibrium model

The development of an equilibrium model for natural water systems generally must now be based on permissible evidence. That is, the conditions broadly observed in the systems do not contradict the equilibrium hypothesis. A much more rigorous set of observations is generally needed to give the proof of equilibrium conditions that would generally be demanded in other branches of solution chemistry.

The general theme of a need for more and properly designed research is present throughout. Although the more elaborate models proposed in some of the papers are thought-provoking, the next symposium on this subject will, hopefully, provide more of the facts which are now missing, facts that must be learned before the more elaborate geochemical models can be fully accepted.

One of the strong points of this volume is that it has papers on several areas of active research that are somewhat peripheral to the subject of nat-

ural water chemistry. Thereby, the book brings out clearly the fact that natural water chemistry cannot be compartmented, but relates closely to many other fields.

Some of the individual papers deserve special comment. The review by Parks of certain aspects of aqueous surface chemistry of solid minerals is clearly and simply written, and provides a number of ideas which chemists interested in natural water may find useful. Stöber, in describing his studies on silica solubility, comments on some phenomena of structural modification at surfaces which may be very useful in understanding the occurrence of metastable solids and the apparent irreversible nature of solution processes for some minerals.

Analytical methods

The paper by Hume on analytical methods and data contains some excellent observations on the problems of sampling and a realistic appraisal of accuracy and precision of analytical results for both major and minor constituents of natural water. This paper should be required reading for everyone who makes or uses analyses of natural water. Most of us are not fully aware of the limitations pointed out by Prof. Hume—or tend to forget about them.

Some readers who are concerned only with fresh-water systems may find some of the individual papers are too specifically slanted toward chemical oceanography to be useful to them directly. However, almost all the papers do have useful concepts for both the oceanographers and those dealing with fresh water. Those not already familiar with Sillén's sea water model will find this an interesting concept. The decrease in degrees of freedom

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which results from increasing the number of stable solid species in a system is a concept of broad usefulness in studies of natural water chemistry.

Some whose interests are in the field of biochemistry may feel the symposium papers do not sufficiently emphasize this aspect of natural water chemistry. The subject is treated in several of the papers, however.

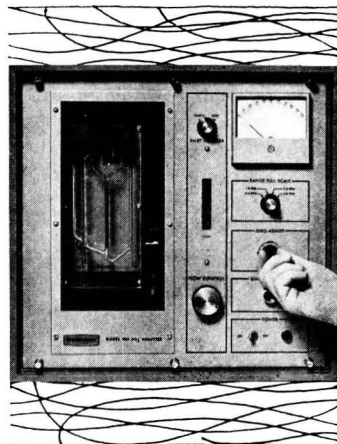
The longest of the symposium papers is one which describes current theories and research on the structure of liquid water. It has a very extensive bibliography and covers its subject thoroughly. But the inescapable conclusion is that little is really known about the structure of water. Further, the relationship of the topic to equilibrium chemistry of solutes is not adequately demonstrated. This topic could have been adequately covered for the purposes of this symposium in a much briefer paper.

Redox potentials

Several modes of thought regarding redox potentials are displayed in this volume. Two of the five papers that use the concept of redox potentials retain the standard terminology based on the law of mass action and the Nernst equation (with potentials expressed in volts), which is familiar to geochemists and others inside and outside of the water chemistry field who have utilized Eh-pH diagrams. The other three papers, two of which are by Sillén, contain a different terminology, using a function called "pE" in place of Eh. This quantity is defined by Sillén as the negative logarithm of the activity of electrons and its use in this as in other papers by Sillén, which have been published in recent years, is justified primarily on the simplified arithmetic which results when making calculations involving equilibrium constants.

My opinion is that standard terminology ought to be used in water chemistry until such time as the use of pE is more widely accepted in all the various fields of chemistry in which the Nernst equation is applied. Whether the potentials that can be measured in aqueous systems, using platinum and calomel electrodes, are directly comparable to the potential calculated from the Nernst equation,

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is a matter that is unrelated to the basic ideas involved in this proposed departure from standard nomenclature.

This book is a very useful source both of facts and of theoretical concepts to be tested by experiment. The presentations ought to be readily comprehensible to all researchers in aqueous environmental chemistry. As in many other developing fields, however, the theoretical concepts tend to outnumber the available facts, and users of this book should be careful to distinguish between them.

The Automobile and Air Pollution: A Program for Progress. U.S. Department of Commerce. iv + 51 pages. U.S. Government Printing Office, Washington, D.C. 20402. 1967. 60 cents, paper.

The Origin and Development of the Waterways Policy of the United States. William J. and Robert W. Hull. vii + 79 pages. National Waterways Conference, Inc., Washington, D.C. 1967. \$1.50, hard cover; \$1.00, paper.

Radioassay Procedures for Environmental Samples. Public Health Service Publication No. 999-RH-27. xxi + 492 pages. Public Inquiries Branch, Public Health Service, U.S. Department of Health, Education, and Welfare, Washington, D.C. 20201. 1967. No charge.

Pollution and Marine Ecology. Edited by Theodore A. Olson and Frederick J. Burgess. Conference proceedings. xvi + 364 pages. Interscience Publishers, New York, N.Y. 10016. 1967. \$12, hard cover.

Theory of Fog Condensation. A. G. Amelin. xi + 236 pages. Daniel Davey & Co., Inc., New York, N.Y. 10010. 1967. \$14, hard cover.

The Analytical Toxicology of Industrial Inorganic Poisons. Morris B. Jacobs. xxv + 943 pages. Interscience Publishers, New York, N.Y. 10016. 1967. \$25, hard cover.

Insecticides: Action and Metabolism. R. D. O'Brien. xi + 332 pages. Academic Press, New York, N. Y. 10003. 1967. \$14, hard cover.

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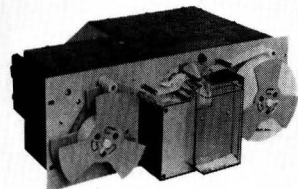
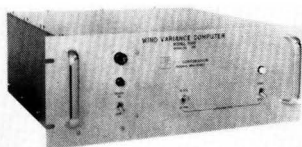
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Volume 1, Number 12, December 1967 1019

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Wind Variance Computer

Model 7000 wind variance computer records wind speed, direction, and variance data while eliminating the need for manual data extraction from strip chart records. The instrument converts standard analog input signals into digital form for direct printout on a companion 8-channel tape punch unit. NUS Corp. 62

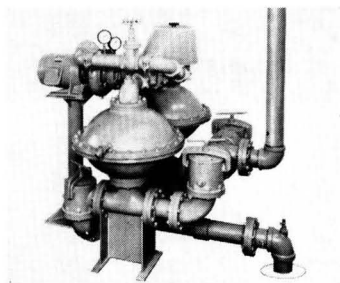
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Model 6 battery-operated portable ($4\frac{1}{2}$ pounds) pH meter features solid state circuitry, low power consumption, 0.05 pH repeatability, and a taut-band meter. Operating instructions are permanently printed on the durable, cor-

rosion-resistant plastic case of the device, which is available with an automatic temperature compensator and recorder output. Corning Scientific Instruments. 63

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Dieselex CC-2 is a combustion catalyst and smoke suppressant for diesel fuels in diesel and jet engines and for domestic furnace oil to be used for home burning and small industrial operations with residual fuel. SSI-3 is an inhibitor for sulfur trioxide and ash modifier for vanadium and sulfate deposits within boilers. MC-7 is a combustion catalyst for residual fuel in utilities, refiners, and large-size marine boilers. Coaltrol is a powdered SO_3 inhibitor and ash modifier for use in coal-fired furnaces. Apollo Chemical Corp. 64

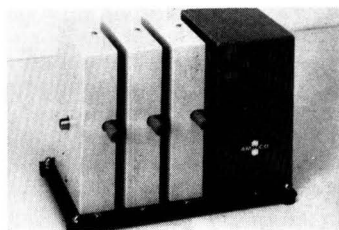


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Synthetic Organic Sequestrant

Dearborn 870 is a liquid cooling water treatment formulated to sequester iron, copper, and aluminum ions. It is compatible with other chemical treatments. The material, which has a low toxicity, requires only a low dosage and, thus, creates no pollution problem, Grace says. The sequestrant can be fed directly from the 30- or 55-gallon drums in which it comes, or through a proportioning pump-tank combination. W.R. Grace & Co. 66



Modular Fluid-Metering Pump

Panel-mounted micrometer screws on this modular fluid-metering pump provide simultaneous individual flow-rate adjustments (from 0.01 ml. to 5 ml. per min.) for up to six fluids. Long-term stability is within 0.5% of setting. American Instrument Co., Inc. 67

Conductivity Bridge

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Manometer-Servo

A field instrument for use in stream gaging and water-level applications, the Stevens manometer-servo has a remote sensor to reduce flood damage possibilities, a self-purging orifice to prevent clogging, and a carriage track extending the full length of the manometer to allow considerable carriage travel and permit wide ranges of water level sensing. Leupold & Stevens Instruments, Inc. **69**

Self-Cleaning Dust Filters

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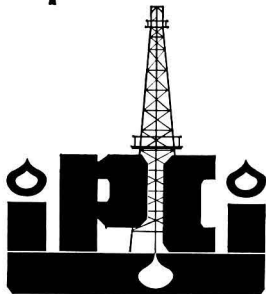
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NEW LITERATURE DIGEST

Spring-loaded thermocouples. A catalog fact sheet describing a spring-loaded thermocouple with locking cap that is adjustable over the entire length of the probe, is available. The bulletin provides complete specifications for quick-disconnect plugs, a 6-inch lead termination, and two types of mounting adaptors for the thermocouples. Thermo Electric Co., Inc. **85**

Water and wastewater analysis. Catalog 10, a 108-page equipment guide called "Water and Wastewater Analysis Procedures Catalog," describes 50 different testing procedures for alkalinity, dissolved oxygen, turbidity, coliform bacteria, hardness, chemical compounds, and many other items of importance in treating water and waste water. Where applicable, a brief description of the appropriate portable test kit or automatic analyzer is shown following the procedure. The catalog also contains listings of apparatus, glassware, solutions, and formulations required for water and waste testing. Hach Chemical Co. **86**

Centrifugal filter. A plastic, portable centrifugal filter system (capacity 50-1200 g.p.h.) is described in bulletin 802-1C. The LAI series transparent Lucite filter chamber is recommended for use at temperatures as high as 140° F.; and the AAI series epoxy filter chamber is recommended for applications to 250° F. Sethco Manufacturing Corp. **87**

Platinized honeycomb catalyst. "Platinum Catalysts and Systems for Pollution Control" is a brochure explaining the custom-installed THT catalyst air-pollution control system based on a special process of bonding platinum to a honeycomb support. In some processes such as NOX abatement for nitric acid plants, the company recommends recovery of the resultant heat energy for use as a source of power or steam. Matthey Bishop, Inc. **88**

Dust control and pneumatic conveyance equipment. Bulletin A-9167 is an illustrated 6-page folder that describes how Duclone dust collectors and Fluo/veyor pneumatic conveying units operate. Typical installations and auxiliary equipment are pictured. Bulletin W-8564 describes a Venturi scrubber (type VO) for dust and fume collection in the submicron range. Ducon Co., Inc. **89**

Direct flame fume incineration. A 12-page brochure describes the Cor-Pak fume incineration systems, covering such topics as fuel consumption, space and installation requirements, operation and maintenance, and change in process effluent. Air Pre-heater Co., Inc. **90**

Water and waste treatment equipment. Catalog D662 is a 6-page folder illustrating aero-hydraulic guns, digester mixing systems, sludge heaters and heat exchangers, walking-beam flocculation units, rotary distributors, and clarifiers. Ralph B. Carter Co. **91**

Waste treatment systems. An 8-page folder explains development of two sophisticated waste treatment pilot plants now in operation. The folder also announces the company's air, water, and waste systems group, which is a consolidation of existing air pollution and water and waste treatment systems. Infilco Fuller Co. **92**

Piston actuators. A 6-page folder illustrates pneumatic and hydraulic piston actuators for valve automation, lists features of these devices, and names accessories available. Grinnell Co., Inc. **93**

Water treatment tanks. Bulletin A-64 describes a line of polyethylene cylindrical tanks for water softening equipment and other water treatment applications. The corrosion-resistant tanks

are available in six stock sizes from 80-450 gallons capacity, or can be custom-molded in special sizes. Nalge Co. **94**

Chemical feed systems. A 4-page brochure shows a complete line of ready-to-install packaged chemical feeding systems for water treating and chemical processing applications. Also illustrated in the two-color brochure are three basic types of chemical feed systems (steel, polyethylene, and stainless steel tanks) in capacities of 50-500 gallons, the proportioning pumps used in these systems, tank dimensions, and specification charts. Neptune Chemical Pump Co. **95**

Industrial filters. Bulletin E25B lists cartridge filters for process filtration, optical clarity, cold sterilization, and removal of such things as oil and water droplets, oil and water vapor, smoke, odor, taste, and color. Pall Trinity Micro Corp. **96**

Industrial odor control. Bulletin M-81 describes Cairox potassium permanganate techniques used in oxidizing odoriferous and toxic gases responsible for air pollution. Among the harmful and irritating gases covered are hydrogen sulfide, sulfur dioxide, and the mercaptans. According to the company, the techniques described can be used to convert heavy, nauseating odors into mild, pleasant scents. Carus Chemical Co., Inc. **97**

Glass equipment supplement. A supplement to SGA catalog 66, "What's New for the Laboratory," comprises 16 pages of laboratory equipment, including glass apparatus. Ion meters, balances, ultrasonic cleaners, pumps, microscopes, stoppers, incubators, and many other items are illustrated. Scientific Glass Apparatus Co., Inc. **98**

Three New Advisors Named to ES&T



Morgan

Dr. Morgan has appointed three new members to the ES&T Advisory Board, to replace Dr. William L. Faith, Dr. T. E. Larson, and Dr. Werner Stumm, whose terms expire in January 1968.

The new members, like the present advisors, are drawn from the ranks of those active in the fields of environmental science and technology. They represent a wide variety of disciplines and interests, and lend the benefit of their expertise and experience to the continuing effort of the new publication to serve its readers in the most competent fashion.

Meeting formally once or twice a year and keeping in touch with the editor and his staff by mail and phone, the advisory board serves to advise the editor on content and subject matter and to assist, when requested, in solving problems that may arise. Members of the board are normally appointed to a three-year term.

Appointed to serve as advisors until January 1971 are Dr. Richard D. Cadle, Dr. Donald J. O'Connor, and Dr. Walter J. Weber, Jr.

Editor appoints Cadle, O'Connor, and Weber to Advisory Board

Dr. James J. Morgan, editor, is associate professor of environmental health engineering at California Institute of Technology, which he joined in 1965. He edits ES&T while continuing his duties as a faculty member.

Dr. Morgan received his B.C.E. (1954) from Manhattan College, M.S.E. (1956) from the University of Michigan, and A.M. (1962) and Ph.D. (1964) from Harvard University. From 1956-60, he was an instructor in sanitary engineering at the University of Illinois. In 1960-61, he held a Danforth Foundation Teacher Study Grant at Harvard, and in 1961-63 continued his studies under a U.S. Public Health Service predoctoral fellowship.

Dr. Morgan was appointed associate professor of water chemistry and research associate professor of civil engineering at the University of Florida in 1963. In 1964, Dr. Morgan served as a member of the National Institutes of Health's Environmental Sciences and Engineering Study Section. The recipient of a variety of professional awards, he is a member of ACS, AWWA, American Society for Limnology and Oceanography, ASCE, AAAS, Sigma Xi, and Chi Epsilon.

Dr. Richard D. Cadle, head of the chemistry and microphysics department of the National Center for Atmospheric Research, Boulder, Colo., earned his B.A. (1936) at Western Reserve University and his Ph.D. (1940) in physical chemistry at the University of Washington (Seattle).

From 1940-47, Dr. Cadle was employed by Procter & Gamble Co. as a research chemist, and in 1947-48 he was unit head, Naval Ordnance Test Station, China Lake (Calif.). In 1948, he joined the Stanford Research In-



Cadle



O'Connor

stitute as senior physical chemist. Then in 1954 he was named chairman of SRI's atmospheric chemical physics department, a position he held until 1963. Since 1963, Dr. Cadle has been with the National Center for Atmospheric Research, first as program scientist, and now as chairman of the chemistry and microphysics department.

Since 1948 Dr. Cadle has worked almost exclusively on problems related to the chemistry and physics of the atmosphere. His research has included analyses of contaminants in the atmosphere, studies of aerosols, and investigations of the kinetics and photochemistry of chemical reactions in the atmosphere.

Dr. Cadle is the author of close to 55 articles in scientific and technical journals. He is a member of ACS and American Geophysical Union. Dr. Cadle will serve on the Advisory Board until January 1971.

Dr. Donald J. O'Connor is professor of civil engineering at Manhattan College. He earned his B.C.E. (1944) at Manhattan College, his M.C.E. (1947) at Polytechnic Institute of Brooklyn, and his Eng. Sc. D. (1956) at New York University. Dr. O'Connor served as instructor in sanitary engineering at Manhattan College (1946-47) and at Polytechnic Institute of Brooklyn (1948-50). In 1952 Dr. O'Connor returned to Manhattan



Weber

College as assistant professor. He became associate professor in 1956 and professor in 1964.

Dr. O'Connor received a Founders' Day Award in 1956 at New York University; the Rudolph Hering Award in 1958 (for "Mechanism of Reaeration in Natural Streams") and in 1966 (for "Estuarine Distribution of Non-Conservative Substances"); and Honorary Mention (1960) from the New York State Water Pollution Control Federation for "Treatment of Organic Waste in Aerated Stabilization Basin." He is the author of some 40 technical papers published in journals, and co-author (with W. W. Eckenfelder) of **Biological Waste Treatment**.

Dr. O'Connor is specially interested in mathematical analysis of water pollution in all natural bodies of water. He is a member of ACS, the American Geophysical Union, New York State Water Pollution Control Association, American Society of Engineering Education, American Association of University Professors, American Society of Limnology and Oceanography, American Association of Professors of Sanitary Engineering, Chi Epsilon, Sigma XI, and is a Professional Engineer of the State of New York. Dr. O'Connor will be on the Advisory Board until January 1971.

Dr. Walter J. Weber, Jr., associate professor of civil and water resources engineering, University of Michigan (Ann Arbor), received his



Friedlander

B.S. (1956) in engineering from Brown University, his M.S. (1959) in sanitary engineering from Rutgers University, and an M.A. (1961) in water chemistry and aquatic biology and Ph.D. (1962) in water resources engineering from Harvard. Dr. Weber was named Doctoral Marshall, Outstanding Ph.D. Recipient, Division of Engineering and Applied Physics in 1962 by Harvard. He was given the Presentation Award by the Division of Water and Waste Chemistry, ACS, the same year. He received the Distinguished Service Award, College of Engineering, University of Michigan, in 1967.

In addition to his professorial duties, Dr. Weber has published widely, and found time to chair the 1967 ACS National Symposium on Adsorption from Aqueous Solution and the 1968 Gordon Research Conference on Environmental Sciences.

Dr. Weber is a member of ACS, AIChE, AWWA, ASCE, AAAS, WPCF, American Society of Professors in Sanitary Engineering, American Geophysical Union, University Council on Water Resources, Rhode Island Society of Professional Engineers, Sigma XI, Delta Omega, and Chi Epsilon. Dr. Weber will serve on the Advisory Board until January 1971.

Dr. S. K. Friedlander is professor of chemical engineering and environmental health engineering at California Institute of Technology, a posi-



Goldberg

tion he has held since 1964. Previously (1957-64), he had taught at Johns Hopkins, chairing the biomedical engineering committee there in 1962. Dr. Friedlander received his B.S. (1949) in chemical engineering from Columbia University, his M.S. (1951) from MIT, and his Ph.D. (1954) from the University of Illinois.

Dr. Friedlander's research interests lie primarily in the fields of diffusion, interfacial transfer, and aerosol physics. He is a member of ACS, Sigma Xi, Tau Beta Pi, and Phi Lambda Upsilon. Dr. Friedlander will serve on the Advisory Board until January 1970.

Dr. Edward D. Goldberg, professor of chemistry, University of California, San Diego, received his B.S. (1942) from the University of California, Berkeley, and his Ph.D. (1949) from the University of Chicago. He joined the faculty of UCSD in 1949, and was appointed full professor in 1961; from 1965-66 he served as the first provost of the university's Revelle College.

Dr. Goldberg's major areas of scientific interest are in the geochemistry of marine waters and sediments and the application of radioactive dating techniques to problems in the major sedimentary cycle. The editor of two journals (**Earth and Planetary Science Letters** and **Journal of Marine Research**), Dr. Goldberg will serve on the Advisory Board until January 1970.



Gregor



Pitts



Altshuller



Gaudy



Lee

Dr. Harry P. Gregor, professor (since July 1967) of chemical engineering at the Columbia University school of engineering and applied science, earned his B.A. (1939) and Ph.D. (1945) at the University of Minnesota. Following a year of experience in the water-treatment industry, Dr. Gregor joined the faculty of Polytechnic Institute of Brooklyn, where he remained until his appointment at Columbia.

Dr. Gregor has specialized in colloid and electrochemistry, with particular emphasis on the study of high-polymeric materials. His interests in environmental science and technology have led him to an active role as an advisor to many private and government agencies in the field of water purification and desalination. Dr. Gregor, a member of ACS, will serve on the Advisory Board until January 1970.

Dr. James N. Pitts, Jr., is professor of chemistry, University of California, Riverside, a position to which he was appointed in 1959; Dr. Pitts served as chairman of the department of chemistry there from 1961-63.

Dr. Pitts received his B.S. (1945) and his Ph.D. (1949) in physical chemistry from UCLA. He worked as a research assistant (1942-45) for division 10, National Defense Research Committee, OSRD, and as research associate, special projects division, U.S. Army (1945-46). Then he was appointed instructor and assistant professor of chemistry at Northwestern University (1949-54). From Northwestern he went

to the University of California, Riverside, where from 1954-59 he was associate professor of chemistry. A member of ACS, APCA, Faraday Society, Sigma Xi, Alpha Chi Sigma, Phi Lambda Upsilon, Phi Beta Kappa, and the American Physical Society, Dr. Pitts will serve on the Advisory Board until January 1970.

Dr. A. P. Altshuller is chief of the Chemical Research and Development Section, Laboratory of Engineering and Physical Sciences, Division of Air Pollution, Public Health Service, at the Robert A. Taft Sanitary Engineering Center in Cincinnati, Ohio. He received his B.S. (1948) at the University of Chicago, and his M.S. (1950) and Ph.D. (1951) from the University of Cincinnati.

From 1951-55, he was an aeronautical research scientist engaged in fuels research at what is now the Lewis Research Center of NASA. Dr. Altshuller has published numerous articles related to spectrophotometric and coulometric analysis, gas chromatography, and various aspects of atmospheric chemistry. He is the chairman of the ACS Committee on Air Pollution, a member of the ASTM Committee on Air Pollution, and a member of APCA. Dr. Altshuller will be on the Advisory Board until January 1969.

Dr. A. F. Gaudy, Jr., is professor and acting head of Oklahoma State University's school of civil engineering. He received his B.S. (1951) in

civil engineering from the University of Massachusetts, his M.S. (1953) in sanitary engineering from MIT, and his Ph.D. (1959) in sanitary engineering from the University of Illinois, where he was an assistant professor of sanitary engineering from 1959-61.

A consultant on industrial waste abatement problems that require a research approach, Dr. Gaudy is a member of ACS, ASCE, WPCF, AWWA, AAAS, APHA, Sigma Xi, and the American Society for Microbiology. His term on the Advisory Board runs until January 1969.

Dr. G. Fred Lee is professor of water chemistry and director of the water chemistry program at the University of Wisconsin (Madison). He received his B.S. (1955) from San Jose State College (Calif.), his M.S. (1957) in public health from the University of North Carolina, and his Ph.D. (1960) from Harvard. From 1960-61, he was assistant professor of water chemistry at the University of Pittsburgh (Pa.) before moving to the University of Wisconsin.

An authority in the fields of the chemistry of natural waters, water and waste water treatment, and water pollution control, Dr. Lee is a member of ACS, AWWA, AAAS, Pollution Control Federation, American Society of Limnology and Oceanography, American Geochemical Society, American Ecological Society, Sigma Xi, and Delta Omega. He will serve on the Advisory Board until January 1969.

MEETING GUIDE

January 22-26, 1968

Instrument Society of America

4th Marine Science Instrumentation Symposium

Ramada Inn, Cocoa Beach, Fla.

The symposium will emphasize marine instrumentation problems and considerations involving data recording, handling, and processing. Sessions are planned on human performance in the undersea environment, control and analysis of hyperbaric environments, instrumentation for physical oceanography, and others. In conjunction with the meeting, ISA will offer its first four-day, short course on dynamic measurements in ocean studies. The course is designed to provide nonelectrical engineers with a knowledge of basic definitions and instruments used in dynamic measurements.

January 22-26, 1968; April 1-5, 1968; April 15-19, 1968; May 27-31, 1968

Manufacturing Chemists Association

Seminars on Water Pollution Control for Chemical Wastes

University of Texas, Austin

Each seminar is designed as an intensive course on chemical waste treatment for water pollution control. These seminars will provide attendees with information on the latest techniques in the field, both as to practice and selection, planning and designing of particular treatment methods. Registration is available to industrial representatives only and should be made to the MCA. Fee for each seminar is \$150.

February 2, 1968

American Chemical Society-Middle Atlantic Regional Meeting

Colloquium on the Application of Physical Chemistry to Air Pollution Problems
Philadelphia, Pa.

The colloquium will feature a discussion of mechanism and kinetics of photochemical air pollution. Special reference will be made to East Coast smog and such topics as sulfur oxide kinetics in combustion, effect of air pollutants on polymers, carbon oxide conversion in the afterburning region of a hydrocarbon flame at atmospheric pressure, mechanistic studies of photochemical air pollution systems, and the hydrocarbon-NO_x ratio effect on individual hydrocarbon photochemical reactivity.

February 6-7, 1968

University of Illinois-Illinois Department of Public Health

10th Sanitary Engineering Conference
Illini Union, Urbana, Ill.

This conference will feature discussions on waste disposal from water and waste water treatment processes. The conference is intended for sanitary engineers and water works managers and operators. In addition to the technical presentations, the conference will feature a tour of the new sanitary engineering laboratories of the University of Illinois.

February 7-9, 1968

California State Department of Public Health

9th Conference on Methods in Air Pollution and Industrial Hygiene Studies

Huntington-Sheraton Hotel,
Pasadena, Calif.

The conference features both lecture and workshop sessions devoted to current practices as well as new and improved methods for atmospheric sampling and analysis. Topics for the lecture sessions include states of standard methods, programs in calibration methods, pesticide analysis, fluorides, gaseous pollutants, and others. Topics for the workshop sessions include range of application of detector tubes; determinations for SO₂, CO, NO_x, O₃, hydrocarbons, and relative humidity; and direct reading instruments.

February 19-22, 1968

Technical Association of the Pulp and Paper Industry

53rd Annual Meeting-7th Pulp and Paper Industry Exhibit

Hilton Hotel, New York, N.Y.

Among the 20 sessions scheduled for the meeting is a discussion on odor abatement. The 7th Pulp and Paper Industry exhibit will be held during the first three days of the meeting and will feature air and water treatment equipment, and instruments for measuring and controlling air and water pollutants.

March 3-8, 1968

Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy

19th Annual Conference

Penn-Sheraton Hotel, Pittsburgh, Pa.

The theme of the 1968 conference is analytical chemistry and spectroscopy for the future. The conference will feature symposiums on specific ion electrodes and the role of government and industry in pollution control. Exhibits from more than 200 companies will highlight the latest in analytical instrumentation.

March 27-28, 1968

IIT Research Institute-Bureau of Mines

Symposium on Mineral Waste Utilization

IIT Research Institute, Chicago, Ill.

The symposium reflects growing national concern about difficulties associated with mineral waste disposal. The meeting also reflects growing realization of benefits that can be gained from utilization of such wastes. Presentations, highlighting current efforts to develop methods for recycling and using mineral wastes, will be directed to aluminum ores, phosphate slimes, coal wastes, iron ore wastes, copper wastes, furnace slags, and others. Included will be the economic, ecological, and systems aspects of the problems associated with such disposal.

March 31-April 3, 1968

Desert Research Institute of University of Nevada

Symposium on Erosion as Related to Air and Water Pollution

Stardust Hotel, Las Vegas, Nev.

The theme of the symposium is the mechanics of erosion as it is related to air and water pollution. The symposium is sponsored by Kennecott Copper Corp., Golden Bean Oil Co., American Meteorological Society, and Desert Research Institute. Topics include physics of soil erosion by air movement, water erosion and erosion cycles, meteorological aspects of soil erosion, medical aspects of airborne particulates, and others. A planned field trip to Death Valley will afford an opportunity to examine various areas affected by both wind and water erosion.

March 31-April 4, 1968

American Institute of Chemical Engineers

1st Materials Engineering and Sciences Exposition and Conference


Sheraton Hotel, Philadelphia, Pa.

Aware of the fact that one third or more of chemical engineering graduates are actively engaged in the field of materials, AIChE has planned 13 symposiums on various materials-oriented subject areas.

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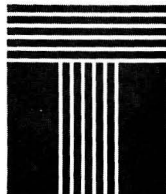
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