Analytical chemistry for environmental sciences. A question of confidence

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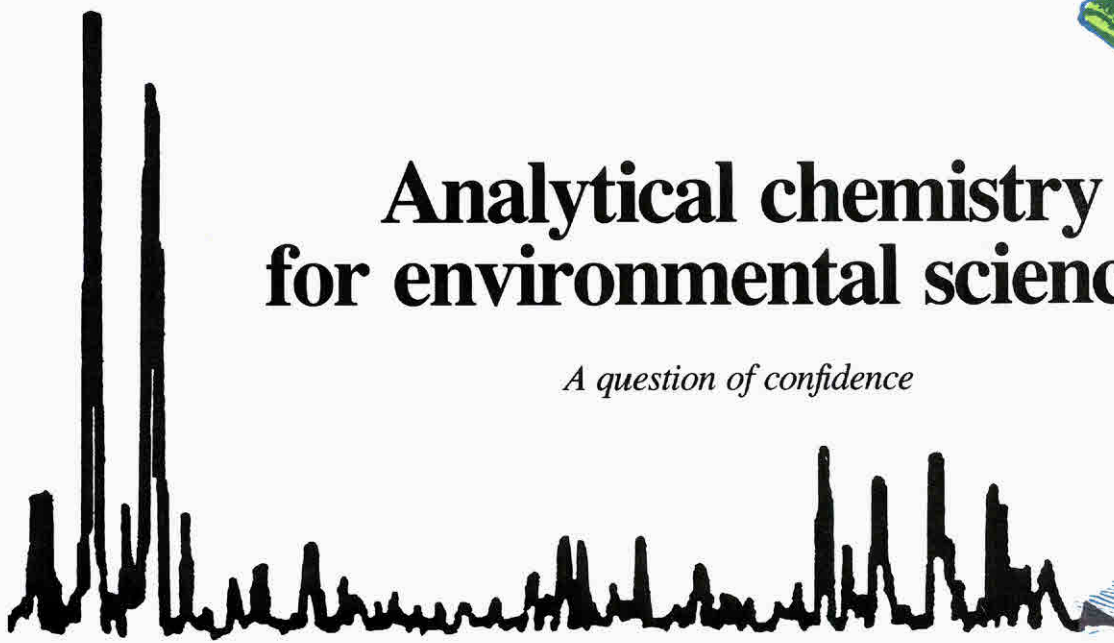
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University of Georgia
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Analytical chemistry for environmental sciences

A question of confidence

Christopher F. D'Elia
University of Maryland
Chesapeake Biological Laboratory
Solomons, MD 20688

James G. Sanders
The Academy of Natural Sciences
Benedict Estuarine Research
Laboratory
Benedict, MD 20612

Douglas G. Capone
University of Maryland
Chesapeake Biological Laboratory
Solomons, MD 20688

Federal and state agencies rely heavily on monitoring data to assess the status of and trends in the quality of the nation's natural waters. Moreover, public officials at all levels are pressing to increase monitoring efforts to establish long-term trends for chemicals that affect the environment. Unfortunately, a largely unappreciated fact is that data obtained in many monitoring studies are typically so compromised in one or more respects that they are of little value.

This situation occurs despite the expenditure of enormous sums of money on environmental monitoring programs and despite the development of comprehensive quality assurance/quality control (QA/QC) plans. Unfortunately, factors contributing to compromised analytical data (see box) are dealt with only partially or inconsistently in these plans.

Often the causes of compromised data stem from a failure to select an appropriate analytical protocol. For ex-

ample, many agencies—motivated largely by the desire to retain historical continuity—mandate or employ inadequate, outmoded, or inappropriate analytical methods. In other cases, agencies feel constrained in their choice of method by regulation or by practical limitations of available analytical, financial, or human resources.

Other problems are more subtle. Statistical errors such as censoring data near the detection limit are routinely made (1). However, in our opinion, *the greatest problem is that monitoring often becomes a goal in itself.* Moreover, differing and incompatible requirements often exist among different users of monitoring data.

Common analytical problems associated with monitoring programs for natural waters

Contamination
Difficult and tedious analysis
Improper sample preparation and preservation
Improper sampling
Improper standardization
Incorrect reporting of analytical statistics
No satisfactory method available for chosen analyte
Poor technique
Right method, wrong sensitivity range
Sample matrix problems
Uncalibrated or inadequate instrumentation
Wrong chemical form determined
Wrong method

This paper draws heavily from our experiences with an extensive monitoring program for the Chesapeake Bay that provides a wide range of examples to illustrate problems inevitably encountered in other monitoring programs. Our purpose is not to criticize or embarrass the authorities who administer this program, for they recognize that problems exist and have in many cases acted to solve them. Our purpose instead is to draw attention to deficiencies in the conceptual basis of monitoring nationally and to scrutinize and improve standard methods used. We discuss some of the generic causes of inadequate quantitative determination for a broad range of environmental analytes and show how, in several cases, substantial improvements in data quality resulted from modest changes in analytical protocol. We understand that simple solutions do not always exist, but emphasize that improvement should nevertheless be the ultimate goal.

Chesapeake Bay monitoring

Nutrient enrichment, toxicant pollution, and related problems are of major concern in Chesapeake Bay. In 1984, a cooperative program between the federal government and Maryland, Virginia, Pennsylvania, and the District of Columbia established an extensive and ambitious monitoring network throughout the bay and its tributaries.

The monitoring effort is complicated by the spatial and temporal variability of the bay and the number of institutions involved in the study. The mainstem Chesapeake is long (over 180 mi.) and is fed by seven major tributaries.
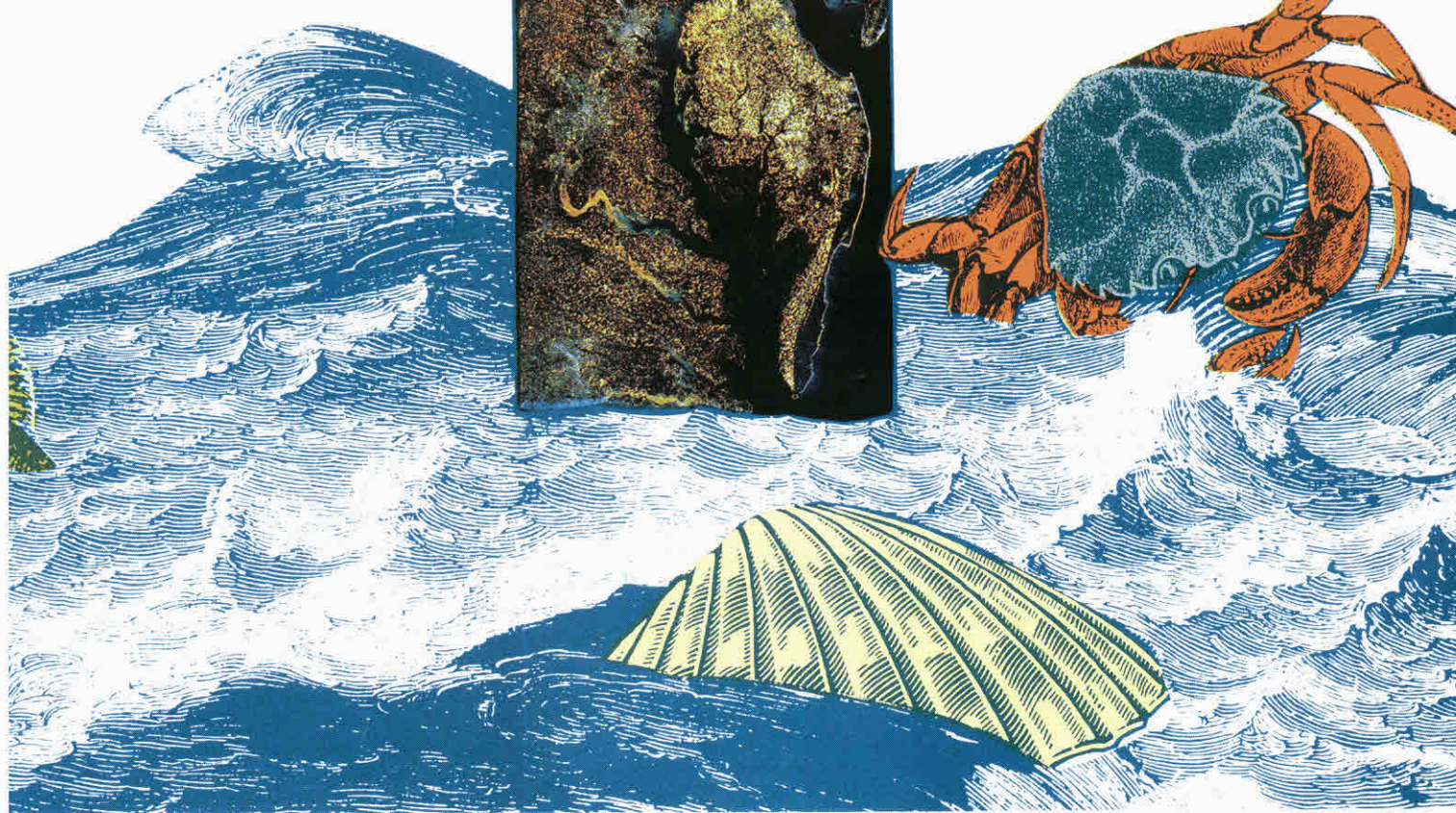
The water column alternates between stratified and unstratified conditions, and distinct seasonal variations occur in most water quality analytes. Salinity varies throughout the estuary, and extensive anoxia occurs in summer months. State and federal agencies as well as university research laboratories perform analyses.

A weak conceptual framework greatly compromised the early stages of the monitoring. No explicit statement of goals was developed, although potential sampling frequencies and stations were hotly debated in assorted committees. The choice of analytical methods was at first given little consideration. Managers were not fully apprised of the limitations of different methods and the implications thereof. Intended uses of the data, which implicitly included trend analysis, calibration and verification of mathematical models, nutrient budgeting, development of water quality standards for receiving waters, and identification of noncompliance discharges were not specified; complementing process-oriented scientific studies were not explicitly defined.

Because of the conceptual confusion under which this program developed, the choice of methods initially fell to the established standard protocols of the lead agency involved, EPA (2). However, because EPA is primarily a regulatory and not a research agency, its methods are oriented toward legal pollutant thresholds at relatively high concentrations. In fact, state-of-the-art analytical procedures are not typically required for "end-of-pipe" compliance

monitoring of water quality.

In contrast, the best available methods are often the first choice in receiving water- or research-oriented studies, particularly when low environmental concentrations or unusual sample matrices are expected, as is the case for estuaries. Thus, although most EPA methods are suitable for policing legally mandated levels for effluent discharges, their sensitivity, precision, accuracy, and susceptibility to matrix effects may preclude their suitability for estuarine samples.

Sections 106 and 308 of the Clean Water Act do differentiate between methodologies for legal and research purposes. Methods meeting Section 308 requirements pertain to legal compliance aspects of the National Pollutant Discharge Elimination System (NPDES) effluent monitoring, whereas Section 106 methods, which are applicable to pollution research, are not as stringently constrained.

Although the Chesapeake Bay Monitoring Program falls under the latter (Section 106) category, many laboratories that perform both Section 308 and 106 analyses prefer to analyze both types of samples using the Section 308 mandated methods. In practice, they prefer to analyze all samples, regardless of source, using the fewest possible procedures or costly instruments. Accordingly, many laboratories are unwilling to vary from standard Section 308 methods.

Problems: Nutrient analyses

In 1985, after meeting some resistance, several academic laboratories

sought to supplant EPA-mandated procedures with those more typically used in the oceanographic research community. The EPA Chesapeake Bay Liaison Office agreed to support a comparison of methods prior to permitting a change to "nonstandard methods." That study, reported and discussed elsewhere (3, 4, 5), showed that data quality was improved by adapting methods used by the oceanographic community. We draw from that study to illustrate problems with standard EPA techniques.

Right method, wrong sensitivity range. The limits of detection for EPA methods are often above natural concentrations encountered for many parameters, whereas oceanographic methods offer order-of-magnitude improvements in detection limits and provide useful data at natural concentrations (Table 1).
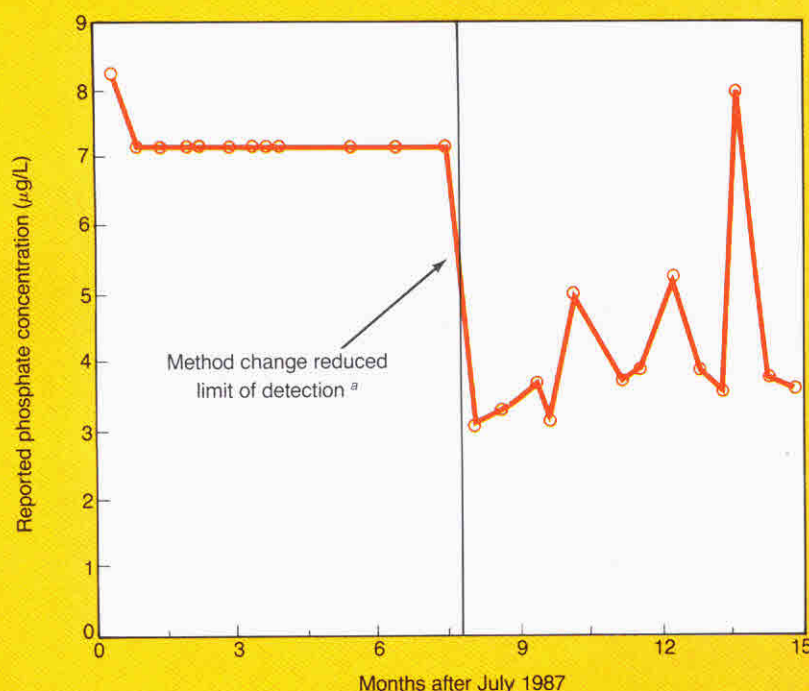
Yet in many cases, a common basis exists for the EPA protocols and their more sensitive and precise oceanographic counterparts. For example, trivial changes—such as increasing the path length of colorimeters in autoanalyzers—may greatly improve detection limits, as was the case for phosphate determination in surface samples from the bay (Figure 1).

The data shown in Figure 1 were taken directly from the monitoring database. The left side of the graph represents censored data using EPA techniques that were reported numerically as the limit of detection (7.5 $\mu g/L$) and exceeded actual values. The right side of the graph represents values obtained with the standard oceanographic procedure.

FIGURE 1
Effect of reporting "nondetects" on samples from a station in mid-Chesapeake Bay

Method change reduced limit of detection [a]

Months after July 1987

[a]The limit of detection for the methods used changed from 7 to 0.6 μg/L.

Incorrect reporting of analytical statistics. Figure 1 also illustrates a frequent and serious problem with much environmental data held by various agencies: data records reported as "nondetects" or "less-thans." Hence, when trend analysis is desired some arbitrary value must be substituted (4). Even though the American Society for Testing and Materials has strongly recommended against censoring nondetects (6), a recent paper has focused on the failure of the environmental community to come to grips with the problem and has discussed how information is lost by this practice (1).

This is not a strictly academic matter, for the censoring of data not only biases statistical comparisons, but also can result in erroneous verification and calibration of water quality models. Moreover, it can severely affect mass balance and budgetary calculations when the analyte in question is found at low concentrations in large water bodies.

Wrong method. For some analytes, EPA-mandated protocols are simply poor choices. For example, elemental analysis for particulate carbon (PC), nitrogen (PN), and phosphorus (PP) are not satisfactorily accomplished by EPA procedures which rely on indirect, not direct quantitation (3, 4, 5). Yet information on these variables is critical for water quality modeling purposes.

The particulate fraction includes the biological part of the ecosystem, and the temporal and spatial variations associated with this fraction could be overlooked were the analyses not made. Moreover, unlike dissolved solute phases—which mix conservatively (i.e., dilute proportionally) between different water masses—particulate phases may, through biological transformations and sedimentation, repartition nutrients nonconservatively (i.e., not by simple dilution) between one water mass and another.

Management agencies in the United States have typically calculated PC, PN, and PP indirectly "by difference" from two other determinations, that is, by subtracting values determined in sample filtrate from those determined in whole water. This indirect approach amplifies analytical error and often results in poor correlation between field replicate samples and in negative values for particulate analytes (Figure 2a).

In contrast to management agencies, oceanographic research laboratories have for decades employed direct elemental analysis of particulate fractions collected on inert filters using a variety of analytical approaches (7, 8, 9). Direct measurement of the particulate fraction vastly improves precision and lowers detection limits (Figure 2b). This too has important implications for water quality modeling of biological constituents.

No satisfactory measurement method available. For saline and estuarine waters, no satisfactory measurement method exists for either dissolved organic carbon (DOC) or nitrogen (DON), which are cornerstone measurements of many aquatic monitoring programs. Such measurements are needed to provide the direct estimates of oxidizable organic substrates available for heterotrophic, biological oxygen demands and estimates of bound nitrogen available for growth requirements that any credible nutrient modeling effort requires.

Oceanographers have realized for some time that the venerable Kjeldahl nitrogen determination, although proven reliable for many sample matrices, lacks sensitivity and does not provide reliable DON data for estuarine and marine samples (10). Accordingly, more precise, alternative techniques (7, 8, 9) have been developed based on peroxydisulfate (11) or UV (12) digestion procedures for DOC.

Recently, however, concern has arisen about the shortcomings of all existing procedures for measuring dissolved organics (13), and a new oxidation procedure involving catalytic high-temperature combustion of DOC and DON (14, 15) surprisingly suggests that estimates from peroxydisulfate- or UV-based procedures underestimate DOC and DON pools by as

much as 80% *(15)*.

Strong statistical correlations between DOC determined by catalytic combustion and the oceanographic parameter "apparent oxygen utilization" suggest the presence of previously undetected biologically reactive compounds *(15)*. Thus, the ability of current procedures to quantify this DOC pool is in serious doubt, and previous estimates based on them may be very low *(13, 14, 15)*. However, this issue is unresolved and still under intense scrutiny by oceanographers.

The debate on this topic goes beyond local or regional water quality concerns and has profound global implications. A potential underestimate of inventories of organic carbon bears directly on the accuracy of our estimates of the fate of anthropogenic CO_2 emissions.

To compound the problem, recent analyses of global and oceanic carbon cycles have indicated major inconsistencies between the predicted and actual atmospheric CO_2 increase (actual is far less than predicted), and between observed rates of primary production by bottle techniques and estimates based on export from the euphotic zone of the ocean (which were high) or inferred from seasonally intergrated trends in O_2 (which were higher yet).

Such observations about dissolved organics in marine water samples give rise to concern in other areas of the environmental sciences. Indeed, several groups have initiated rigorous comparisons of DOC and DON in different sample matrices. The discrepancies in marine DOC and DON analyses reconfirm the continuing need to reassess, improve, and calibrate routine field measurement procedures.

Problems: Metals and organics

Routine monitoring of aquatic systems for inorganic and organic toxicants has also become more prevalent in recent years. This issue is particularly topical in Chesapeake Bay, for EPA is now under intense pressure to increase monitoring efforts for toxic substances in the bay.

One issue, larger than we can adequately address here but which deserves more national debate, is the usefulness of such data in the first place. In many instances, as in water column sampling, pollutant concentrations are likely to be at or below nanomolar concentrations—well below the detection levels established by regulatory agencies. Thus, many agencies are accumulating vast quantities of nondetect data. In addition, our knowledge of what constitutes an environmental effect is very poor; the action levels that have been set may be too stringent or too lenient to protect the system in question. Therefore, even the most carefully collected and accurately analyzed samples may not allow us to detect actual pollutant impact or to predict potential degradation.

We are faced with a complex set of issues when sampling and analyzing for most trace toxicants. The large variety of elements and compounds present in a variety of matrices presents many analytical problems, some of which we illustrate below.

Contamination. The first, and until recently the overriding, issue was contamination. Analysts blithely measured metal and organic levels in aquatic systems for decades without knowing that, in many cases, they were analyzing only the analyte introduced into the sample during its handling and analysis.

During the past 20 years, the magnitude of this problem has been clearly established, and chemists have developed new methods to collect, process, and quantify samples cleanly. Years' worth of data have been discarded. Consequently, reported trace metal and trace organic levels have dropped steadily, possibly as much because of improved chemical analyses as because of improved water quality.

Perhaps the best example of such progress has been the analysis of trace metals in the open ocean. Early reports indicated widely variable, relatively high concentrations of trace metals *(16)*. By the mid-1970s, however, most investigators had realized that the variability in analyses was due to rampant contamination, difficult to avoid when taking samples from dirty metal ships.

Such realization led to a reevaluation of techniques and approaches *(17)*, resulting in analyses that were consistent with theory and with one another *(18)*. Researchers of other aquatic systems have, in time, addressed the problems of contamination as well. "Clean technique" procedures are now routine for

FIGURE 2
Comparison of replicates by (a) standard Kjeldahl "by difference" method[a] and (b) elemental analysis method
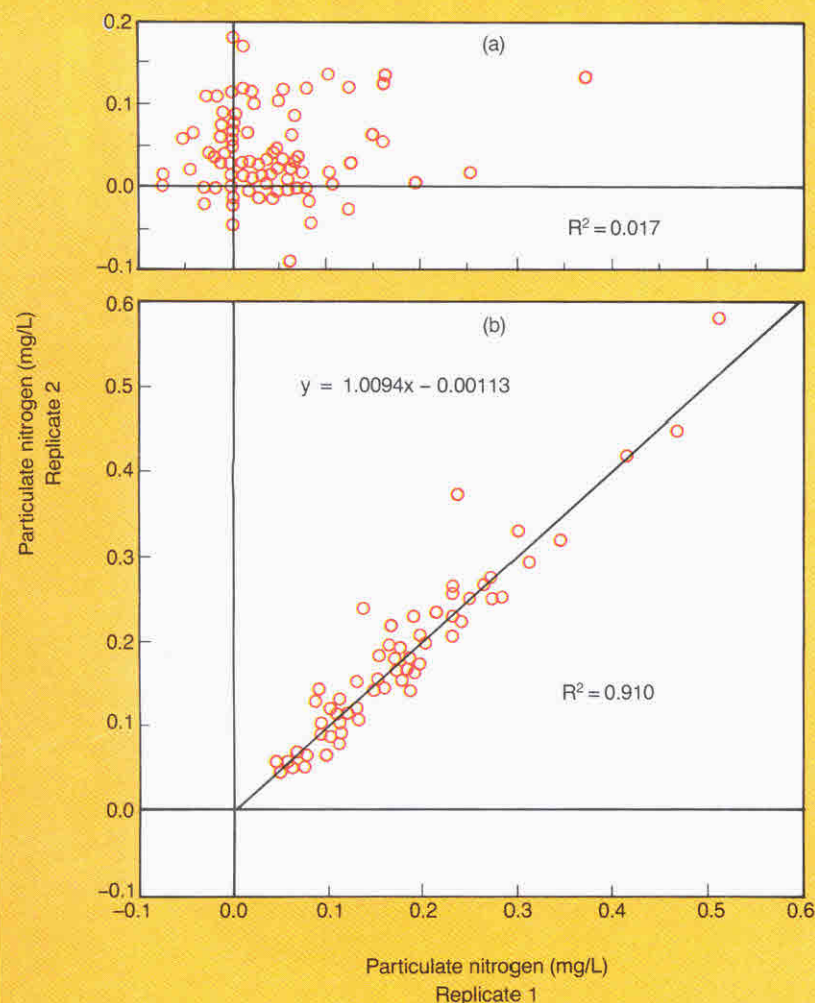
[a]Duplicates for both whole-water and filtrate samples subjected to Kjeldahl N determination were randomly paired and the difference obtained.

sample collection and processing.

Wrong chemical form analyzed.
Recent research has shown that the toxicity of many trace elements is a function of the chemical form of the element; for example, copper toxicity is related directly to the free metal ion Cu^{2+} *(19)*. In the case of copper, the free ion concentration depends on the formation of copper complexes with inorganic ions and organic complexing agents, and generally comprises less than 1% of the total dissolved copper. Unfortunately, our ability to measure this form of copper directly has lagged behind our appreciation of its relative toxicity. Although free copper can be detected by the use of a selective ion electrode, detection limits are fairly high, and determinations in most natural waters require an indirect method of analysis.

Thus, although recent water quality criteria recognize the importance of the metal form, such as the free copper ion *(20)*, criteria continue to be developed based on analysis of total dissolved metal. Consequently, the criteria developed may bear no direct relationship to the most relevant toxic form of the element and cannot adequately protect aquatic ecosystems without being unrealistically stringent.

Right method, wrong sensitivity range. As for inorganic nutrients, standard methods are often not sensitive enough to measure elements present in low concentrations. At present, the most common analytical technique for inorganic trace elements is the use of atomic absorption spectrophotometry (AA). AA is quick, versatile, and quite flexible. Whereas numerous interferences exist with specific elements, the literature is replete with methods for overcoming them. Unfortunately, this instrument, even when coupled with electrothermal techniques, is insufficiently sensitive to detect many of the elements present in natural waters (e.g., Table 2). Therefore, an analyst using these techniques will consistently be faced with environmental values below the detection limit.

When such techniques are used in a monitoring context, the results are often of little value. For example, samples taken by the U.S. Geological Survey in conjunction with their monitoring of water resources throughout the United States include a number of trace metals monitored quarterly. For the water year 1987 (October 1986–September 1987), 19 metals were measured in Chesapeake Bay tributaries. For three of these (Patuxent, Choptank, and Susquehanna), a total of 226 measurements were made. Of these measurements, 129, or 57% of the total, were reported as less-than-detec-

FIGURE 3
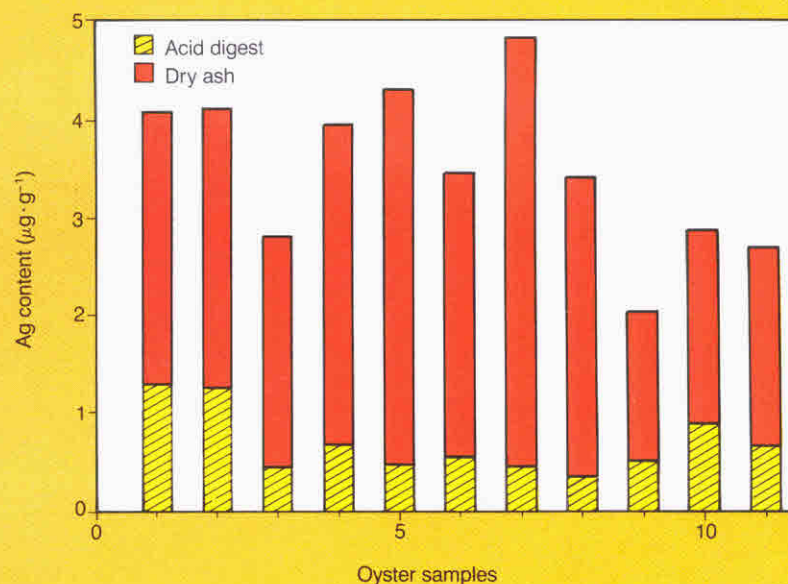**Silver content (dry weight) of oysters sampled monthly from the Patuxent River compared by analytical method**

tion *(21)*. Attempts to determine metal loadings to the Chesapeake Bay based on such data are futile.

Sample matrix problems. Problems encountered with analyzing tissues and other solids for toxic substances are somewhat different from the low-level analyses encountered in water column samples. Sensitivity of detection may not be a big problem for tissues, where biological concentration factors markedly increase the levels present naturally, or for sediments, where sorptive and redox reactions enrich concentrations of metals and hydrophobic organics. Rather, the matrix containing the pollutants is often insufficiently considered by the method employed. Matrix interferences, difficult problems for dissolved substances, are greatly amplified for solids. Familiar interfering substances may occur in higher concentrations, and totally unknown sources of interference may also be encountered because of physicochemical factors

such as redox transitions (e.g., anoxic sediments).

The potential for matrix interferences must be considered at the outset. Different elements often require different preparation techniques. A widely accepted practice in preparing tissues for metal analysis is to use a wet acid digestion to decompose and solubilize the organic matrix. In many instances, complete decomposition does not occur and the residue contains refractory compounds that can interfere greatly.

For example, a series of oyster samples were collected monthly in the estuarine portion of the Patuxent River in Maryland. A portion of the sample was subjected to a standard wet digestion with nitric acid, and the remainder to a dry ash technique that results in total sample destruction *(22)*. Analyses performed on the wet-digested samples of oysters significantly underestimated silver content, with a mean of only 18% recovery in the acid digests (Figure 3).

Difficult and tedious analysis. The analysis of polychlorinated biphenyls (PCBs) presents probably the best example of generic problems in organic contaminant analysis. Until recently, packed column gas chromatographic (GC) procedures were used to separate and quantify environmental levels of PCBs and are responsible for much of the extant information.

However, the resolution and sensitivity of such procedures are severely restricted and minimize the information gained. Of the 209 potential PCB congeners, most can now be effectively separated and authenticated with standards using glass capillary GC (23), and it is now possible to evaluate compositional spectra and to identify postdepositional transformations (e.g., 24, 25, 26). Nevertheless, few laboratories have these high-resolution techniques in regular use.

Routine comparisons illustrate the degree of divergence in capabilities among laboratories (27, 28). Variations in sample preparation methods and instrument response contribute to poor comparability. Our ability to interpret and compare trends in organic contaminants will be constrained until procedures become as comparable as is practical.

For PCBs, problems of poor precision and comparison are associated with the whole analysis train from sample collection to extraction, cleanup, and storage (29). The wide range of solubilities and partition coefficients among the PCBs and the effects of the various sample matrices account for discrepancies. Ongoing development of standard reference material in representative matrices, for various organics as well as for metals, should greatly improve future comparisons.

Discussion and recommendations

We do not pretend to have easy solutions to many of the problems discussed above, but we do believe the time is right for a serious national dialogue to begin on this topic. A substantial fraction of the limited fiscal resources for environmental research is now devoted to monitoring studies that are clearly of limited scientific or practical value. We recommend the following steps.

- Review standard EPA methods in the context of the concentrations of analytes encountered in natural waters. Clear differentiation must be made between methods used for discharge compliance and methods used for monitoring receiving waters.
- Establish a national system of regional centers of analytical expertise based on existing academic analytical capabilities to provide information concerning analytical techniques, to
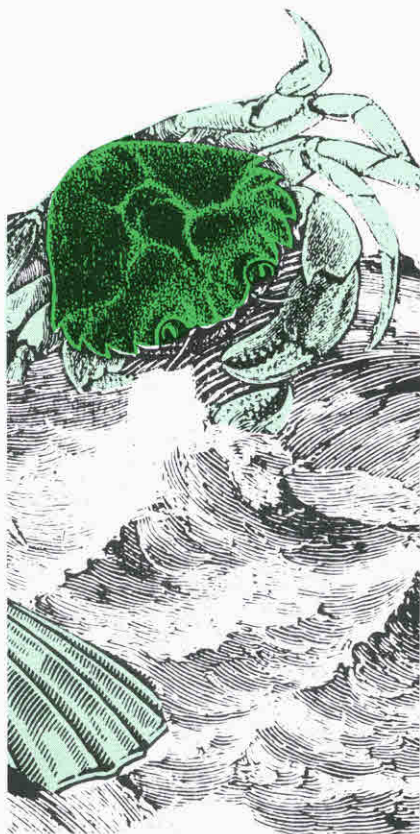
intercalibrate methods and develop reference materials, to train analysts, and to perform unusual or state-of-the-art analyses. An enormous chasm exists between most laboratories performing routine monitoring analytical work and the academic research community. This must be bridged if we are to improve environmental chemistry.

- Increase research into new methods. Research in analytical chemistry has shifted, we believe, away from this goal. We must have more scientists investigating analytical methodologies, and we must ensure that sufficient funding exists for such research.
- Abolish the practice of reporting analytical measurements as nondetects. Such information at best is essentially useless and at worst is misleading. This recommendation is not new (1, 6) but has been consistently ignored.
- Improve quality assurance/quality control plans to address specific objectives and provide the rationale for making the measurements. At present these plans constitute little more than bureaucratic enfranchisements of bad standard methods. Until methods used in a given monitoring effort are viewed in the context of explicit goals, enormous problems with data quality will persist.

These recommendations are hardly novel and involve little more than common sense. As a nation with a reawakening concern about the quality of the

environment, we must strive to ensure that the most current and best techniques are applied under the best possible conceptual framework or we will make little long-term progress in environmental management.

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Christopher F. D'Elia is a professor at Chesapeake Biological Laboratory and Director of the Maryland Sea Grant College Program. He received his Ph.D. in zoology from the University of Georgia in 1974. His interests include the nutrient dynamics of aquatic ecosystems and science-policy relationships.
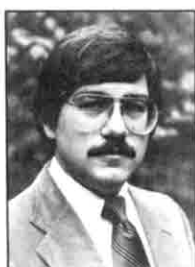
James G. Sanders (l) is Director and Associate Curator of the Academy of Natural Sciences' Benedict Estuarine Research Laboratory on the Chesapeake Bay. His research interests include trace element biogeochemistry in coastal ecosystems and organism response to chronic loading of toxic substances. He obtained his B.S. from Duke University and his Ph.D. from the University of North Carolina at Chapel Hill.

Douglas G. Capone, (r) a professor at Chesapeake Biological Laboratory, received his Ph.D. from the University of Miami in oceanography in 1978. His research deals with microbial ecology, biogeochemistry, and microbe-pollutant interactions.

Cocombustion of refuse-derived fuel and coal

A review of selected emissions

Glenn A. Norton
Ames Laboratory
Iowa State University
Ames, IA 50011

Audrey D. Levine
Iowa State University
Ames, IA 50011

It is estimated that the annual generation of municipal solid waste (MSW) in the United States will reach about 200 million tons by 1990 *(1)*. Alternatives to disposal of solid waste in landfills are needed because of increasingly stringent environmental regulations. One alternative is to process MSW into refuse-derived fuel (RDF) that can be used as a fuel supplement for combustion of coal in dedicated or retrofitted boilers for the production of electricity.

The energy content of 200 million tons of MSW is equivalent to about 75 million tons of coal or about 300 million barrels of oil and thus represents a significant fuel source *(2)*. In addition, by producing RDF from MSW, the volume of waste to be disposed of is decreased; therefore the useful life of a landfill can be extended by as much as 400% *(1)*.

The processed MSW can be fired as "fluff" RDF or densified RDF (dRDF). Fluff RDF is typically 3/4–1 in. in size and is generally prepared by processing the MSW through primary and secondary shredding, magnetic separation, and air classification *(3, 4)*. The fluff RDF can be densified to form dRDF by pelletizing, briquetting, or extruding techniques *(1, 4)*. (In this paper, "RDF" refers to both the fluff and densified forms unless otherwise noted.)

The first major experiment in which RDF was cofired with coal was performed in St. Louis at the Union Elec-