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## Water Analysis

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THIS FOURTEENTH REVIEW of the literature of analytical chemistry applied to water analysis covers a period from October 1968 through September 1970. The present review follows the plan of the previous reviews, the last of which appeared in ANALYTICAL CHEMISTRY for April 1969 (8).

A review of the literature on water pollution control, which includes a section on analytical methods, is published annually by a Research Committee of the Water Pollution Control Federation. The 1968 review of the literature (21) includes 232 references to analytical methods for determining organic and inorganic substances. The inorganic section is further divided by the specific instrumental technique used. A section on analytical methods using noninstrumental techniques is also included. Their 1969 review of the literature (22) follows the same pattern and includes 249 references.

A review of analytical procedures used in oceanography is given by Nehring, Rohde, and Berge (18). Included are procedures which involve the use of neutron activation, isotope dilution, polarography, flame photometry, paper- and gas-chromatography, automatic analyzer system, probe measurements, and multifrequency transmission.

Newton and Atkins (19) made a survey of methods used for sea water analysis. Included are colorimetric, gravimetric, titrimetric, and instrumental methods of analysis. Dyrssen (5) reviewed the need for sea-water analysis and discussed sampling, application of gravimetric and titrimetric methods, in situ sensors, atomic absorption, and activation analysis.

Fischer (7) reviewed briefly the analysis of sea-water samples. He stressed the importance of automated analysis not only for the sake of convenience but, more importantly, because it permits on-the-spot decisions to be made, and because it minimizes the danger of significant error due to changes occurring during storage of samples for subsequent analysis.

A review with 31 references is given by Bertru (1) of the principal techniques used in the determination of nitrate, nitrite, ammonia, sulfate, sulfide, phosphorus, calcium, magnesium, aluminum, nickel, manganese, cobalt, copper, iron, molybdenum, and zinc in fresh waters. The reagents used and the operational methods are given.

Brown, Skougstad, and Fishman (2) have written a manual which contains methods used by the U.S. Geological Survey to collect, preserve, and analyze water samples for their content of dissolved minerals and gases. Among the topics discussed are selection of sampling sites, frequency of sampling, sampling equipment, sample preservation, laboratory equipment and instrumental techniques, accuracy and precision of analysis, and reporting of results. Seventy-six analytical procedures are given for determining 55 water properties. The methods of analysis are applicable to a wide range of waters from those having trace concentrations of dissolved minerals or gases to those having high concentrations, such as found in sea water and brines. Each procedure specifies the type of water or range in concentration to which the method is principally applicable.

A series of simple analytical methods

for the determination of dissolved oxygen, B.O.D., ammonia, nitrite, nitrate, chloride, total hardness, and alkalinity and carbonate hardness are described by Klee (14).

Grasshoff (10) described chemical methods for the analysis of sea-water samples. Principles and procedures are given for the determination of salinity, chloride, dissolved oxygen, hydrogen-ion activity, alkalinity, phosphate, nitrate, sulfate, sodium, potassium, calcium, and magnesium.

Several types of apparatus for the continuous and direct determination of temperature, conductance, pH, redox potential, hardness, dissolved oxygen, turbidity, and radioactivity in water are described by Maschelein (15). Two types of measuring devices are described: One is formed by a totally immersed tubular sampling thief; the other by a special cell fitted with proper electrodes or detectors. Suggestions are given for the proper use and maintenance of the apparatus.

Galetti and Snowden (9) discussed new instruments useful in the analysis of water. Use of specific-ion electrodes and determination of water purity by electrolytic conductance are included.

A critical view of spectroscopic methods for water-quality control is discussed by Uman (20). The statistician's influence on the spectroscopist from the standpoint of improving the accuracy of his analyses is criticized. The problems of sample selection, water-quality criteria, analytical methods, and accuracy of results are discussed from the viewpoint of whether or not sufficient information is known about standards, controlling parameters, and meth-

ods for meaningful results. The shortcomings of the statistical approach are highlighted.

Johnson and Kelly (13) discussed the use of spectroscopy and chromatography to determine the level of constituents such as nitrogen, phosphorus, minerals, organic content, and pesticides in water. The use of automation to determine several parameters is also presented.

Iddings (12) stated that nuclear methods are well suited to the field of pollution control because of sensitivity, specificity, and relative freedom from interferences. The author discussed the advantages and disadvantages, early history, detection devices, scintillation counters, nuclear techniques, and future prospects.

The determination of metals in water and waste waters by atomic absorption spectrophotometry is discussed by Burd (3). History, theory, instrumentation, and practical applications are included in his presentation. A review is given by Burrell (4) on the applications of atomic absorption in the fields of marine chemistry and geochemistry. Particular emphasis is given to the instrumentation and techniques required to trace transition metals in marine waters and sediment.

Mattson and Mark (16) reported that colorimetric and spectrophotometric methods are useful in both qualitative and quantitative analyses in aqueous systems only when the sample is a homogeneous, transparent solution. With internal reflectance spectroscopy, optically opaque samples, such as marine sediments, water filtrates, and aqueous suspensions, can be analyzed directly. Both visible colorimetric and infrared spectrophotometric methods are described, along with some representative data and the experimental apparatus, pointing out the potentialities of the technique.

McFarren, Parker, and Lishka (17) discussed an evaluation of atomic absorption methods for the determination of zinc, chromium, copper, magnesium, manganese, silver, lead, cadmium, and iron. Fifty-nine laboratories participated in the study. Each participant had the choice of direct aspiration of the samples or chelation with ammonium 1-pyrrolidinecarbodithioate and extraction into methyl isobutyl ketone prior to aspiration. Acceptable results were obtained for lead only by chelation and extraction. Neither procedure gave satisfactory results for cadmium. The other elements gave acceptable results by direct aspiration. Greenberg, Moskowitz, and Tamplin (11) found that analyses of reference samples for calcium, magnesium, sodium, potassium, chloride, sulfate, and fluoride by 92 different laboratories gave different and sometimes unacceptable results, although all used standard methods. Statistical

evaluation indicated that 38 laboratories had perfect results, 52 had some unacceptable results, and 2 had no acceptable results. Finney and Nicolson (6) discussed the criteria used for evaluation of analytical methods in water analysis. The criteria are reproducibility, accuracy, sensitivity, limit of detection, specificity, and convenience. Examples are given.

#### ALKALI METALS

Ammer (2A) determined sodium and potassium in boiler water potentiometrically with glass electrodes. Two types of electrodes are described: One electrode responds similarly to sodium and potassium; the second responds to sodium, potassium, and hydrogen ion. The latter electrode can be made specific to sodium by adding other reagents to eliminate potassium and hydrogen ion interference. Potassium is then determined by difference. Detailed descriptions of the apparatus and methods are given. To measure the sodium-ion concentration in a continuous flow of water, Lower and Eckfeldt (5A) used a potentiometric piece of apparatus with a sodium-responsive glass electrode. Results compared favorably with those found by flame photometry. Sodium concentrations down to 0.1 to 1.0 ppb could be determined. Webber and Wilson (12A) determined sodium in high-purity water with sodium-responsive glass electrodes. At concentrations of 2 and 26  $\mu\text{g}$  of sodium per liter, the standard deviation varied from 0.4 to 1.2. Octadecylamine seriously affected the response of the electrodes, but other impurities likely to be present in power-station waters caused no significant effects. In connection with the above investigation of the performance of sodium-responsive glass electrodes, an independent method was required for determining the sodium content of water in the 0- to 50- $\mu\text{g}$  sodium-per-liter range. Webber and Wilson (13A) used a flame-photometric technique for this purpose. Special attention was given to the problem of accurately calibrating the flame photometer and a new "dry flame" technique was derived. Details of this technique and its performance are given. Selivanova and Ukhin (10A) used flame photometry to determine sodium in waters of thermal power plants. For the determination of traces of sodium by flame photometry in the water-stream cycle of thermoelectric units, Agostinelli and Bartoletti (1A) modified a Technicon AutoAnalyzer. The lithium internal standard was eliminated, thus making the instrument direct-reading. The sample intake rate was increased and, in turn, the signal increased. A potentiometer was used for scale expansion. As little as 3 ppb of sodium could be detected.

A method was suggested by Kolosova,

Bogacheva, and Barsanova (4A) for the separation of sodium from sea water by ion exchange, and its determination by the isotope dilution method.

Talreja, Parekh, and Oza (11A) developed a method using the natural radioactivity of potassium-40 for the routine determination of potassium in sea-water concentrates by measuring the  $\beta$ -activity. The method was useful above 0.5% potassium with  $\pm 2\%$  error and had the advantage that the samples were not destroyed. Moisture had no effect.

Gertner and Grdinic (3A) reported that ultramicro amounts of potassium can be determined after it is separated with the aid of  $\text{Na}_3\text{Ag}[\text{Co}(\text{NO}_2)_6]$  on the ring furnace. The reaction product is dissolved (with addition of thiocyanate) in hydrochloric acid, and the cobalt is titrated with EDTA. The method can be applied to determining the potassium-40 activity in water samples.

A fluorescence method for the determination of lithium in natural brines is reported by Markman and Strel'tsova (6A). Lithium forms a complex with 8-quinolinol in the ratio 5:1 8-quinolinol:lithium. The complex is extracted into chloroform, and lithium is determined by the measurement of the fluorescence intensity in the chloroform layer. Sodium and potassium in 20,000-fold excess, calcium and magnesium in 10,000-fold excess, and iron in 10-fold excess do not interfere. Sensitivity of the method is 0.01  $\mu\text{g}$  of lithium per ml.

Riva (8A) described a titrimetric method for determining lithium in sea water. Lithium is isolated from magnesium, sodium, and potassium by a series of precipitations. The lithium is then reacted with a ferric chloride-potassium periodate reagent and the  $\text{LiKFeIO}_6$  precipitate which is formed is dissolved in 1N hydrochloric acid. The pH is adjusted to 2-3 with solid sodium acetate, and sodium salicylate solution is then added. The solution is titrated with 0.001M EDTA until the violet color disappears.

Morozov (7A) determined lithium and rubidium in sea water directly by flame photometry at wavelengths of 671 and 780 nm, respectively. For lithium the sensitivity was 0.005  $\mu\text{g}$  per ml, and for rubidium 0.008  $\mu\text{g}$  per ml. Standard deviation was  $\pm 8\%$ . The presence of salts in sea water decreased the sensitivity of the lithium determination by approximately 30%.

A spectrochemical method for determining rubidium in sea and spring waters was reported by Schoenfeld and Held (9A). It is based upon an internal reference technique.

#### HARDNESS, ALKALINE EARTH METALS

Blake, Bryant, and Waters (2B) described a flame photometric method for determining 0 to 50 ppm of calcium

in simulated sea water in the presence of 4% (w/w) sodium. Calcium is adsorbed from solution on a column of Chelex-100 resin ( $\text{Na}^+$  form), and sodium selectively eluted with hydrochloric acid at pH 4. Adsorbed calcium is then eluted with 2*N* hydrochloric acid, and the resin washed with distilled water. The eluate and washings are diluted to volume and calcium is determined by flame photometry at 622 nm. Magnesium, zinc, nickel, barium, mercury, manganese, copper, and iron, present separately in concentrations of 25 ppm or collectively in concentrations of 5 ppm each, do not interfere. Aluminum ion depressed the amount of calcium found. A modified flame photometric method for the determination of calcium in drinking water in the presence of interfering ions, sodium, potassium, silicate, phosphate, oxalic acid, and mineral acids, was reported by Sinha, Poddar, and Sinha (17*B*). Calcium at the 1-mg-per-liter level was determined within  $\pm 1\%$  error. Pulido, Moreira de Almeida, and Gouveia de Almeida (15*B*) determined calcium in natural waters by flame photometry and atomic absorption. A 1% lanthanum chloride solution was added to eliminate anion interferences. Results agreed well with those obtained by EDTA titration.

Pasovskaya (12*B*) described a conductometric titration method for determining calcium in natural waters. The method is based on the formation of a soluble complex of calcium with nitrilotriacetate. Magnezon II is used to mask magnesium while interfering copper, aluminum, and iron ions are masked by unithiol. The accuracy was better than 0.2%. Pasovskaya (13*B*, 14*B*) also described conductometric methods for determining hardness and magnesium with nitrilotriacetate. Details of the procedures are given. Turbid and colored waters can be determined by these methods.

Eek Vancells and Casassas (5*B*) described a polarographic method for determining 0.25 to 5.0  $\mu\text{g}$  of magnesium per liter in calcium-rich waters. The method is based on the second reduction wave ( $E_{1/2} = -0.828\text{V}$ ) which appears in solutions of *o,o'*-dihydroxyazobenzene in the presence of magnesium at pH 10.0. The relative standard deviation is less than 3%. Calcium does not interfere in ratios of Ca:Mg less than 100. Cations such as iron, which form precipitates in alkaline solution, are removed by centrifugation prior to determining magnesium. A method for the determination of calcium and magnesium in water based on the decrease in the height of the anodic polarographic waves of EDTA and EGTA was proposed by Fleet, Soe-Win, and West (6*B*). The sum of calcium and magnesium is determined from the height

of the anodic wave of EDTA, and subsequently calcium is determined on a separate aliquot from the decrease in the anodic EGTA wave. Strong mercury-complex-forming agents, such as cyanide, as well as copper, zinc, cobalt, and cadmium, interfere.

Whitfield and Leyendekkers (20*B*) and Whitfield, Leyendekkers, and Kerr (21*B*) discussed the use of liquid ion-exchange electrodes as end-point detectors in complexometric titrations for determining calcium and magnesium. A calcium-selective electrode was used to monitor EGTA and DCTA titrations of aqueous mixtures of calcium, magnesium, and sodium salts. The solution concentrations were selected to span the range of natural waters, and the results were analyzed statistically. The pattern of titration curves observed with changing solution composition agreed qualitatively with that predicted theoretically, but the overall potential drop was usually lower than predicted. The technique was suitable for the determination of calcium and magnesium in sea water with an estimated accuracy of 0.5%. Van't Riet and Wynn (18*B*) determined calcium and magnesium in water by direct potentiometric titration. Calcium was titrated with a solution of the tetra-sodium salt of EGTA at pH 8. A recording pH meter indicated a sharp upward break at the equivalence point. Magnesium interference was eliminated by the addition of citrate. The titration was then continued with tetra-sodium EDTA until there was another sharp break which was the equivalence point for magnesium. The detection limit for both calcium and magnesium was 5  $\mu\text{g}$  per liter. Levchenko *et al.* (11*B*) described the preparation of liquid ion-exchange electrodes and their use for determining the activity of calcium and magnesium ions in natural water and soil solutions. The results found with the calcium electrodes agreed with theoretical values, showing their usefulness. Magnesium electrodes, however, were found to be useful only when magnesium predominates over calcium.

A successive chelatometric titration procedure for the determination of calcium and magnesium in various types of water was described by Ito and Ueno (9*B*). Calcium is titrated with di-Na-EDTA at pH 13 using Hydroxy Naphthol Blue as indicator. The pH is then reduced to 10, and magnesium is titrated with the same reagent. Iron and aluminum interferences are removed by the addition of triethanolamine or potassium cyanide.

The biamperometric titration of magnesium in alkaline solutions with EDTA at pH 9.6 and with an applied potential of 1.4 volts was studied by Vorlicek, Fara, and Vydra (19*B*) at pH 9.6 and with an applied potential of 1.4 volts.

Five  $\mu\text{g}$  to 5 mg in 40 to 100 ml of water were determined with only a 1% relative error. Calcium did not interfere. Copper and nickel were masked with potassium cyanide. Water hardness can be determined similarly.

Fritz, Sickafoose, and Schmitt (7*B*) employed visual and spectrophotometric titration techniques for determining water hardness. The titration was carried out at pH 10 with EDTA using a mixed indicator containing Arsenazo I and tris-(hydroxymethyl)-aminomethane (THAM). Barium can also be determined spectrophotometrically at a wavelength of 555 nm. Gelo (8*B*) compared a complexometric method using di-Na versenate and the Clark method for determining water hardness. Total hardness was determined with Eriochrome Black T indicator. The complexometric method was recommended. Calcium can be determined alone with "Calces" indicator.

Shtern and Nikol'skaya (16*B*) proposed a colorimetric method for determining 0.2 to 5.0  $\mu\text{g}$ -equivalent of hardness per liter in feed water. The method is based on the color formed between magnesium and Chrome Dark Blue indicator. The calcium present in the sample is changed into an equivalent amount of magnesium by addition of a solution of magnesium trilonate so that calcium trilonate and magnesium are formed. Sodium thiosulfate is used to eliminate copper interference.

Kanie (10*B*) reported that silica interferes in the titrimetric EDTA hardness determination. Low values were observed. The interference was affected by pH, time of setting, temperature, and the kind of cation contained.

Bozhevol'nov and Solov'ev (3*B*) used a phosphorescence technique to determine beryllium in water. Beryllium complexes with dibenzoylmethane in water solution at pH 5 to 6 and at a temperature of 70 °C. Upon cooling, the complex is extracted with carbon tetrachloride. The carbon tetrachloride solution is immersed into liquid nitrogen, and at liquid nitrogen temperature gives intensive phosphorescence with sharp maxima at 484 and 516 nm when irradiated with ultraviolet light of a mercury source.

An isotopic dilution spectral method was used by Aleksandrak and Stepanov (1*B*) for determining strontium in river and sea water.

Carr (4*B*) reported that salinity differences between samples and standards can be a source of error in the determination of strontium in sea water by atomic absorption. Standards must match the sample solutions in salinity.

#### ALUMINUM, IRON, MANGANESE, CHROMIUM, OSMIUM, AND RHENIUM

Vezzi (32*C*) considered a number of methods for determining aluminum in

potable water, and his choice was the aluminum colorimetric method. When aluminum is determined by this procedure, a correction must be made if fluoride is present. Hegi and Juel (17C) outlined a photometric method for the determination of aluminum in water with aluminon. Chromium interferes because it also forms a red lake with aluminon, but this interference is eliminated by oxidation of chromium(III) to chromium(VI) with permanganate and by passage through Dowex 1-X10 anion-exchange resin. Aluminum is then determined.

A modified Zirconium-Eriochrome Cyanine R method for the estimation of aluminum in the presence of fluoride was described by Bhakuni, Saroja, and Bulusu (3C). Fluoride concentrations greater than 1 mg per liter are first removed by treating the water sample with Deacidite FF, a strong basic anion-exchange resin. Aluminum is determined on the filtrate.

Titkova (31C) used Chrome Azurol S for the determination of aluminum in natural waters. Organic substances are removed by boiling the sample with ammonium persulfate. The sample is then adjusted to pH 2-3, and 0.1N hydrochloric acid, 10% hydroxylamine-hydrochloride, 0.1% Chrome Azurol S, and 2N sodium acetate solutions are added. The solution is brought to volume with water, and, after 15 minutes, the absorbance is measured at 547 nm. Sodium thiosulfate is used to eliminate copper interference. As little as 0.01 mg aluminum can be determined.

To determine trace amounts of aluminum in sea water, Nishikawa *et al.* (21C) used a fluorometric method. Lumogallion solution is added to a sample of sea water containing 0.003 to 2  $\mu\text{g}$  of aluminum. The pH is then adjusted to 5.0, and the solution heated at 80 °C for 20 minutes. After cooling, the intensity of fluorescence of the resulting solution is measured. Iron interferes, and must be present in the standards equivalent to that found in the samples. Shigematsu *et al.* (28C) found that lumogallion was an excellent reagent for the fluorometric determination of aluminum in sea water. Up to 30  $\mu\text{g}$  of fluoride and 1000  $\mu\text{g}$  of phosphate can be tolerated. Iron(III) interferes, but can be masked with *o*-phenanthroline. Interference from other ions is negligible. The method can be applied to samples containing more than 0.05  $\mu\text{g}$  of aluminum per liter.

Spencer and Sachs (29C) studied the potential interferences in the determination of particulate aluminum in sea water by atomic absorption. Interference effects of calcium, iron, sodium, and sulfate, either in individual or mutual effect, had no significant effects on the results. A nitrous oxide-acetylene flame was used.

A direct photometric method for the determination of iron in drinking water in the range of 0.01 to 2.0 mg per liter is described by Schoeller (26C). Iron reacts with thioglycolic acid to form a red-colored complex. The stable colored complex is formed in ammoniacal solution (pH 8.0-9.0).

Akaiwa, Kawamoto, and Hara (1C) reported that pyridine has a synergetic effect on the extraction of iron(II) with thenoyltrifluoro-acetone (TTA). They stated that the spectrophotometric determination of iron (7-100  $\mu\text{g}$ ) in water based on this reaction is rapid, highly reproducible, and reasonably selective. The optimal pH range is wide and the sensitivity is 0.012  $\mu\text{g}$  per  $\text{cm}^2$  at 580 nm.

Finkelsteinaite and Visockaite (13C) developed a photometric method for the determination of iron in water, employing Trilon B at pH 2.25 to 5.35. A study was made of the possible interferences from other constituents and a table of interferences is included.

Frumina, Goryunova, and Mustafin (14C) studied the reaction of ethyl bis-[(1-sodio-1H-tetrazol-5-yl)azo]acetate with ferrous and ferric iron and developed a photometric method for the determination of total iron in water. The absorbance of the iron complex is measured at 520 nm. The optimum pH for the analysis is 2 to 3. Beer's law is obeyed from 0 to 2.4  $\mu\text{g}$  per ml. Aluminum, calcium, magnesium, cadmium, lead, manganese, and zinc did not interfere. Nickel and cobalt interfered. Copper interference was masked with Complexon III. To determine aluminum in water by the hydroxyquinoline method, iron can be masked with the above complexing reagent.

A number of existing photometric methods and an emission method for determining iron in water were evaluated and tested by Eichelsdoerfer and Rosopulo (12C). A procedure is also given to bring into solution colloidal and other forms of insoluble iron prior to its determination. Detection limits are given for each procedure. The same authors (11C) also described a photometric method using bathophenanthroline for the determination of very low concentrations of iron (5 to 20  $\mu\text{g}$  per 100 ml) in waters containing organic matter. The iron complex is extracted into chloroform and the absorbance of the extract measured at 533 nm. McMahon (19C) proposed an acid-free solvent extraction procedure, using bathophenanthroline, for measuring dissolved ferrous iron in filtered lake water. Omission of hydrochloric acid eliminates the effect of acid and light on the determination. The effects of membrane filtration and ferric-bathophenanthroline interferences on absorbance at 533 nm are discussed.

Gorbenko and Enal'eva (15C) described a technique for determining trace amounts of iron in distilled and other types of water. With bathophenanthroline, the complex is extracted with nitrobenzene and the color compared with a scale of standards similarly extracted. The error of the determination is 25%. Cerrai and Ghersini (7C) determined iron in boiler-water samples with bathophenanthroline after preliminary extraction of ferric iron from a chloride media with bis(2-ethylhexyl) phosphate. The absorbance is measured at 533 nm and Beer's law is followed up to 30  $\mu\text{g}$  total iron. Interference due to copper, and serious interference due to V(III), Ga, Y, and the heaviest rare earths, was observed. With special precautions to avoid iron contamination, 0.7  $\mu\text{g}$  of iron per liter could be determined with a standard deviation of  $\pm 0.11$ . Burton and Head (6C) investigated certain aspects of the analysis of particulate and soluble iron in estuarine waters. Determination of total iron by evaporation of the sample to near dryness before spectrophotometric measurement, using bathophenanthroline, showed an average coefficient of variation of about 8%. They found, however, that blanks obtained were erratic and often high, leading to a poor precision for the procedure. They also examined the use of thioglycolic acid to render the iron reactive. This method gave reproducible blank values and an average coefficient of about 9%. The authors also recommended that filtered samples, to be used for soluble iron analysis, be acidified with hydrochloric acid to about pH 2 if they must be stored.

Jones and Eddy (18C) reported a sensitive atomic absorption method for determining 0.4 to 200 ppb of iron in water. Iron is complexed with 8-hydroxyquinoline and extracted from a system buffered at pH 7 into MIBK. The extract is aspirated in a total-consumption burner, using a hydrogen-air flame.

Tackett and Brocius (30C) described an X-ray fluorescence method for the determination of iron in polluted streams. No chemical pretreatment is necessary other than the addition of chromium as an internal standard. Samples are spotted directly onto chromatographic paper disks and analyzed. Best results are obtained in the concentration range of 5 to 400 ppm.

Osipova, Yakimets, and Kazantsev (22C) described a method to separate iron during the analysis of natural waters. At pH 1.5 to 2.0, phosphorylated cellulose actively adsorbs ferric iron. Iron can be eluted with oxalic acid and the column regenerated with sulfuric acid. Copper, zinc, and manganese are also adsorbed and can be

removed from the absorbant with ammonium nitrate.

To determine iron in hot-springs water, Nishiido (20C) first complexed the iron with pyrophosphate in an ammoniacal medium and then separated the complex by cation exchange on Dowex 50-X8. A 100% recovery was achieved.

Delfino and Lee (10C) evaluated the persulfate oxidation, formaldoxime, and malachite green colorimetric procedures for the determination of manganese in lake water. For membrane-filtered samples, the persulfate and formaldoxime procedures gave similar results. The formaldoxime method gave higher results on unfiltered samples. The malachite green method did not give consistently useful data unless a lengthy modification was employed. The persulfate procedure was preferred. Rab (23C) described a photometric persulfate method for determining manganese in water. For manganese concentrations up to 5  $\mu\text{g}$  per ml, the mean error was  $\pm 0.03 \mu\text{g}$ .

A spectrophotometric method using 4-(2-pyridylazo)resorcinol for the determination of manganese in potable and industrial water was reported by Yotsuyanagi *et al.* (33C). At a pH of 9.7 to 10.7, 4-(2-pyridylazo)resorcinol reacts with manganese to form a water-soluble red complex. The absorbance of the complex is measured at 500 nm. In the determination of 11  $\mu\text{g}$  of manganese per 50 ml, 20 ppm of magnesium, 2 ppm of zinc, and 1 ppm each of iron, cobalt, nickel, and chromium gave a 5% relative error.

Compton, Blaisdell, and Dorosh (9C) described a Technicon automated-formaloxime method for the determination of 0.00 to 0.25 ppm manganese in water. The standard deviation was 0.002 mg per liter. An automatic spectrophotometric reaction-rate method for the determination of 1.5 to 15 ppb of manganese(II) was described by Hadjiioannou and Kephala (16C). The method is based on the potassium periodate-diethylaniline reaction which is catalyzed by manganese. The time required for the reaction to produce a small fixed amount of colored product is measured automatically and is related directly to the manganese concentration. The effect of various ions on the determination was studied. Relative error was approximately 2% and the coefficient of variation 1%.

Chau, Sim, and Wong (8C) described an atomic absorption chelation-extraction procedure for determining traces of chromium in sea water. Chromium is complexed with acetylacetone, extracted into MIBK, and aspirated into a fuel-rich acetylene-air flame. The precision for approximately 1.6  $\mu\text{g}$  of chromium per liter was  $\pm 0.06 \mu\text{g}$  per liter. Burelli and Ecrement (5C)

also used atomic absorption to determine chromium in water.

Bilikova and Bilik (4C) modified the diphenylcarbazide method for chromium. With the modification,  $\mu\text{g}$  amounts of chromium in water are determined in the presence of higher amounts of calcium, strontium, and barium. Trichloroacetic acid is used in place of sulfuric acid to form an acid medium, and a stable colored complex is formed.

A continuous monitor for measuring hexavalent chromium was described by Alton (2C). Basically, the unit is commercially available equipment which uses a colorimetric technique for continuous determination of chromium in river water. By modifying the water-filtration system and by making other changes, as little as 2 to 3 ppb could be measured.

Scadden (24C) developed a cyclohexanone extraction procedure to isolate rhenium from sea water. Rhenium was then determined by neutron activation analysis. Sharma and Parekh (27C) described an indirect titrimetric method for determining osmium in sea water.

Schilt and Taylor (25C) described a new spectrophotometric simultaneous determination for iron and copper in water. Iron(II) and copper(I) are complexed with 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine (PDT), extracted into isoamyl alcohol, and the absorbance of the extract measured at 488 nm. The extract is then treated with sodium cyanide to convert the copper into a cyanide complex, and the absorbance of the extract is again measured at 488 and 555 nm. Calibration curves or empirical equations are then prepared. The loss in absorbance at 488 nm is directly proportional to the copper concentration, and the final absorbance at 555 nm is proportional to the iron concentration. As little as 1 ppb of iron and (or) 4 ppb of copper can be detected.

#### COPPER, ZINC, LEAD, CADMIUM, NICKEL, COBALT, GERMANIUM, AND INDIUM

Podchainova and Patsuk (27D) prepared a review, with 59 references, of numerous techniques for determining copper in various materials including natural waters. For natural waters, they recommended preliminary concentration by extraction of copper with diethyldithiocarbamate or 8-hydroxyquinolate.

Von Oppel and Weingaertner (33D) described a spectrophotometric method for determining copper in boiler feed water and turbine condensates. Copper forms an intense red complex with 4-pyridyl-2-azoresorcinol at a pH between 3.5 and 10.9. The absorbance of the water-soluble complex is measured

at 530 nm. A citric acid buffer solution completely eliminates the interference of ferric ions. Zinc also forms a complex, but it does not absorb at the same wavelength. Abraham, Winpe, and Ryan (2D) determined 0 to 10 ppb of copper spectrophotometrically in sea water with quinoline-2-aldehyde 2-quinolyldiazine (QAQH). Copper reacts with QAQH to form a colored complex which is extracted into benzene. The absorbance of the extract is then measured at 555 nm. The recovery for six replicate determinations at the 2.0-ppb level from artificial sea water was  $2.0 \pm 0.1$  ppb. Golubovic and Jovanovic (10D) determined microgram amounts of copper in mineral waters with oxaldehydehydrazide and acetic acid. The absorbance of the solution is measured at 542 nm. They stated that the method is very fast and sensitive.

Abdullaev *et al.* (1D) determined copper in natural water by neutron activation analysis, using the  $^{64}\text{Cu}$  ( $n, \gamma$ )  $^{64}\text{Cu}$  reaction. Sodium-24 severely interfered, and a radiochemical separation was employed. Copper was first precipitated as cupric sulfide, and then converted to copper thiocyanide. Comparison with results obtained by chemical analysis showed a relative deviation of less than 7.2%.

Mizuno, Hasegawa, and Takagi (22D) described a polarographic method for the determination of 1 to 20 ppb of copper in boiler water. Copper is concentrated on Dowex A-1 (hydrogen-type) ion-exchange resin, eluted with a potassium chloride-nitric acid solution, and determined by high-frequency polarography at a potential of 0.15 V (vs. mercury pool). No interference occurs from impurities likely to be present in boiler water. Baletskaya *et al.* (3D) determined copper concentrations greater than  $4 \times 10^{-7}\%$  in demineralized water by anodic stripping voltammetry.

To determine copper in hot-springs waters, Nishiido (24D) first complexed copper with pyrophosphate in an ammoniacal solution and then separated the copper by cation exchange on Dowex 50-X8. A 99% recovery was achieved.

Marvin, Proctor, and Neal (19D) studied the effects of filtration on the determination of copper in fresh water and sea water. Five types of filters were tested. Each filter tested reduced the reliability of the copper determination.

Sadilkova (29D) described a spectrophotometric method for determining zinc in water. Zinc and other metals are first extracted with diethyldithiocarbamate into chloroform at about a pH of 9. Zinc, lead, and cadmium are then reextracted with 0.16M hydrochloric acid. The cadmium is masked with iodide, and the zinc determined

spectrophotometrically with zincon at 620 nm. There is no reaction between the lead and zincon. Iron is masked with citrate before the initial extraction. The mean relative error for extraction of 2.5 to 20  $\mu\text{g}$  of zinc from 200 ml of water was 2.6%. Kish and Zimomrya (16D) reported that 4-(6-methoxy-3-methylbenzothiazolumimazo)-*N*-methyl-diphenylamine chloride forms an ion association with  $\text{Zn}(\text{SCN})_4^{2-}$ . This species is easily extracted by a mixture of benzene and butylphosphate (28:1). The absorbance of the extract is measured at 640 nm. Beer's law is obeyed for 0.1 to 3  $\mu\text{g}$  of zinc per ml. For water with 3 to 13  $\mu\text{g}$  of zinc in 900 ml, the relative error was 3%.

A direct atomic absorption method, using the sample-boat technique, for the determination of total zinc in sea water is described by Burrell and Wood (5D). The detection limit in a sea-water matrix is  $2 \times 10^{-4}$   $\mu\text{g}$  of zinc, and 0.25-ml samples can be analyzed without any preanalysis preparation. Precision for zinc standards, prepared in double-distilled water and in sea water, was  $\pm 0.04$  and 0.15  $\mu\text{g}$  of zinc per liter, respectively. Contamination problems were shown to be severe for zinc, and initial attempts to evaluate the various chemical and physical forms present in natural-water samples have had limited success.

Goncharova *et al.* (11D) determined zinc in water by emission spectrography using a specially shaped electrode.

Pavlik and Jelinkova (26D) reported a method for determining 0.001 to 0.1 mg of lead per liter in water. A 1-liter water sample is passed through Dowex 50W ion-exchange resin in the sodium form. Lead is eluted with calcium chloride and determined indirectly with dithizone. Lead dithizonate extract is decomposed with dilute hydrochloric acid and the green dithizone color measured. A square-wave polarographic technique was used by Buchanan, Schroeder, and Novosel (4D) to determine lead in potable water. As little as  $2 \pm 0.32$  ppb of lead was determined without preconcentration of the sample. In the absence of interfering organic material, the samples were analyzed directly after the addition of a combination sodium perchlorate-sodium fluoride electrolyte and adjustment of pH to 3. Samples containing large amounts of organic material were first treated with concentrated nitric and perchloric acids and then heated to fumes of perchloric acid.

Kentner, Armitage, and Zeitlin (14D) described a rapid dimethylglyoxime method for the determination of 0.5 to 40  $\mu\text{g}$  of nickel per liter in sea water and other natural waters. The complete analysis of a 750-ml sample of water requires less than 45 minutes; and the limit of detection for this

volume of sample is approximately 0.35  $\mu\text{g}$  per liter. The sample is mixed with citrate and dimethylglyoxime, the pH adjusted to between 9 to 10 with ammonium hydroxide, and the nickel extracted with chloroform. The nickel is then reextracted into acid, treated with bromine water, the pH adjusted to 10.4, more dimethylglyoxime added, and the absorbance of the solution measured at 442 nm. There is no salt effect; therefore, the reagent blank and standards are prepared in distilled water. A direct dimethylglyoxime method for determining nickel in surface and waste waters is described by Gafitanu and Marculescu (8D). The method was applied for concentrations of nickel between 0.2 and 1.5 mg per liter. Organic matter is eliminated by digesting the sample. Iron and copper are separated with cupferron, although iron concentrations below 1 mg per liter do not interfere.

Nevoral and Okac (23D) developed a spectrophotometric method for the determination of trace amounts of nickel in mineral waters. Nickel is isolated both from concentrated and dilute solutions of alkali metals and alkaline earths on Dowex A-1 ( $\text{NH}_4^+$  form) chelating cation-exchange resin. Nickel, cobaltous, ferric, and uranyl ions are eluted and then absorbed onto anion exchange Dowex 1-X10 ( $\text{Cl}^-$  form). Nickel is then eluted with hydrochloric acid. The eluate is then evaporated to dryness, the residue dissolved in water, and nickel reacted with 4-(2-pyridylazo)resorcinol. The absorbance of the solution is measured at 496 nm.

Jones and Eddy (13D) reported a sensitive atomic absorption method for determining 2 to 200 ppb of nickel in water. Nickel is complexed with 8-hydroxyquinoline and extracted from a system buffered at pH 7 into MIBK. The extract is aspirated in a total consumption burner using a hydrogen-air flame.

Kentner and Zeitlin (15D) determined cobalt in sea water by solvent extraction with 1-nitroso-2-naphthol followed by spectrophotometric determination of the 1-nitroso-2-naphthol chelate. The advantages of the method are: elimination of preconcentration (750- or 1,500-ml sample aliquot used), quantitative extraction at the pH of sea water, absence of salt effect, and rapidity. Beer's law is obeyed for 0.2 to 1.3  $\mu\text{g}$  of cobalt per liter. The standard deviations at 1.0 and 3.0  $\mu\text{g}$  per liter are 0.004 and 0.005. Gafitanu and Marculescu (9D) described a nitroso R-salt colorimetric method for determining cobalt in surface and waste waters. A discussion is given on means of removing interfering constituents. Krishnamoorthy, Sastry, and Sarma (17D) used colorimetric and activation analysis

methods for estimating cobalt in sea water. Cobalt was concentrated from 3 liters of sea water by coprecipitation with magnesium hydroxide. Using  $^{58}\text{CoCl}_2$  as a tracer, 96 to 98% of the cobalt was removed.

Dong (6D) described a spectrochemical method for determining microgram quantities of germanium in natural water containing high concentrations of heavy metals. In order to avoid undesirable dilution effects, germanium is isolated from other major and minor elements by extraction of germanium as a chloride complex, first with carbon tetrachloride and then with an ammonium oxalate-oxalic acid solution. Germanium in the resulting extract is determined by a spectrochemical method involving precipitation with 8-quinolinol, tannic acid, and thionalide, charring the precipitate at 450  $^\circ\text{C}$ , and analyzing the oxide. The average relative error and relative standard deviation for 10.0  $\mu\text{g}$  of germanium were  $\pm 9$  and 8%, respectively.

Matthews and Riley (20D) described a neutron activation procedure for the determination of indium in sea water. Indium is concentrated from one or more liters of sea water on Dowex A-1 ion-exchange resin and eluted with hydrochloric acid. It was found that the hydrochloric acid eluate also contained milligram amounts of sodium, potassium, magnesium, and calcium, and made it impossible to evaporate the eluate to a small enough volume to be transferred to an irradiation ampoule. Indium was then separated as a chloro-anion from the alkaline earths and alkali metals on Dowex AG<sub>2</sub>-X8 anion exchanger, eluted with hydrochloric acid, and finally determined by neutron activation. The precision of the method at the 1.20-ng-of-indium-per-liter level was  $\pm 0.06$  ng per liter.

Robinson, Barnekow, and Lott (28D) used atomic absorption to determine cadmium and copper in plating wastes and river waters down to concentrations of 0.001 mg per liter. The procedure is based on the extraction of the 2-mercaptobenzothiazole complexes of cadmium and copper into *n*-butyl acetate, which is then aspirated directly into an air-acetylene flame. Schaller, Lindner, and Lehnert (30D) determined a number of heavy metals in drinking water by atomic absorption. Zinc is determined directly after dilution with water (1:1). Nickel, copper, cadmium, and lead are determined by adjusting the sample to pH 2.5, chelating with APDC, extracting with MIBK, and aspirating of the organic layer.

Methods for continuous monitoring of iron, nickel, and copper in water with an AutoAnalyzer were described by Holy (12D). Concentration ranges covered were as follows: iron 0-50, nickel 0-15, and copper 0-15 ppb.



Ladanyi (18D) reported a polarographic method for the simultaneous determination of copper, lead, and zinc in snow and water. This determination is carried out in an orthophosphoric acid medium without separation. The limit of sensitivity is 4  $\mu\text{g}$  per ml. A polarographic procedure for determining nickel and cobalt in water was described by Vinogradova, Prokhorova, and Sevast'yanova (32D). Details of the procedure are given. The application of anodic-stripping voltammetry with a hanging mercury drop electrode and a fast-sweep polarograph to determine zinc, cadmium, lead, and copper in sea water is discussed by Whitnack and Sasselli (34D). Precise measurements of concentrations of  $10^{-8}$  to  $10^{-9}M$  can be made, if variables are carefully controlled. Sinko and Dolezal (31D) also used anodic-stripping polarography for simultaneously determining copper, cadmium, lead, and zinc in natural waters.

Park (25D) determined lead and zinc in natural water by X-ray fluorescence spectrometry after continuous dithizone-benzene extraction in a pulsed column. The metal dithizonates are oxidized and reextracted into water and precipitated with hydrogen sulfide. The precipitated sulfides were used for the analysis. Lead and zinc were measured at a concentration range of 2.5 to 100 ppb with relative standard deviations of 9.0 and 10.3%, respectively.

McCormick, Graham, and Bark (21D) used inorganic thin-layer chromatography to separate zinc(II), copper(II), nickel(II), cobalt(II), manganese(II), and iron(III) from each other in potable waters. Frei and Stockton (7D) combined ring-oven and circular chromatography to determine cobalt, copper, nickel, and iron(III) in both fresh water and sea water. Detection limits were approximately 0.01  $\mu\text{g}$  for nickel, 0.008  $\mu\text{g}$  for copper and iron, and 0.005  $\mu\text{g}$  for cobalt. The accuracy was between 5 and 12%, and other ions did not interfere in up to 10-fold excess.

#### MERCURY, SILVER, AND GOLD

Hinkle and Learned (9E) developed a method for determining nanogram quantities of mercury in natural waters. Mercury is collected on silver screens immersed in samples acidified with hydrochloric acid. The collected mercury is heated in a radio-frequency induction furnace, and the evolved mercury is measured in a specially built mercury-vapor absorption detector. As little as 0.1  $\mu\text{g}$  of mercury per liter can be detected. Fishman (8E) described a technique for the determination of mercury in fresh water based on the above method. Mercury is collected from an acidified water

sample by amalgamation on a silver wire. The silver wire is then electrically heated in an absorption cell placed in the light beam of an atomic absorption spectrophotometer. The mercury vapors are drawn through the cell with a water aspirator and the absorption is recorded. The detection limit is 0.1  $\mu\text{g}$  per liter. Another flameless atomic absorption method for determining as little as 0.2 ppb of mercury in water and sediments was described by Kalb (11E). Nitric acid is added to the sample to break down the organic-metallic complexes. The mercuric or mercurous ions are then reduced to elemental mercury with stannous chloride. The elemental mercury is then vaporized with a stream of air and the mercury vapor amalgamated on silver foil. The silver foil is heated in an induction coil, and the mercury vapor is carried by a stream of air into an absorption cell of an atomic absorption spectrophotometer and measured. Igoshin and Bogusevich (10E) also described a flameless atomic absorption method. Mercury ions are decomplexed and desorbed from container walls by adding potassium permanganate and sulfuric acid to the sample, and allowing the sample to stand overnight. Stannous chloride is then used to reduce the mercuric or mercurous ions to elemental mercury. The sensitivity is 0.2  $\mu\text{g}$  per liter without concentration.

Bindschedler's green leuco base (BG) is oxidized to form the green dyestuff cation ( $\text{BG}^+$ ). This system was studied by Tsubouchi (16E) to determine mercury in waste water. Mercury(II) was converted into a bromocomplex anion and extracted into 1,2-dichloroethane with the  $\text{BG}^+$ . The system followed Beer's law over the range of  $8 \times 10^{-7}$  to  $4 \times 10^{-6}M$  of mercury.

To determine mercury, 2 to 10  $\mu\text{g}$  per liter, in natural water, Ohta, Terai, and Isokawa (14E) coprecipitated mercury(II) with cadmium sulfide from a 1-liter sample adjusted to 0.1N in hydrochloric acid. The precipitate was dissolved in aqua regia, and copper was removed by extraction with trifluoroacetylacetone in chloroform at pH 4.5 in the presence of hydroxylamine hydrochloride. The aqueous phase was then adjusted to 0.25N in sulfuric acid, mercury extracted with dithizone in chloroform, and the absorbance of the extract measured at 480 nm. Average recovery of mercury from sea water was 95% with a relative standard deviation of 4.4%. Deguchi and Sakai (7E) used the reaction of mercury(II) with Methylxlenol Blue to determine mercury in water colorimetrically. Six to 60  $\mu\text{g}$  of mercury per 50 ml can be determined. The absorbance is measured at 610 nm. The method, however, is subjected to interferences from various metallic ions which necessitates

the removal of these ions prior to determining mercury. A spectrophotometric method for determining organic and inorganic bound mercury based on the formation of mercury dithizonate after decomposition of organomercury compounds by concentrated sulfuric acid was reported by Liebmann and Hempel (13E). With sample concentration and pretreatment, as little as 0.001 ppm of mercury could be detected. The mean error was  $\pm 2.03\%$  with an average recovery of 98%. Zamyslova and Ashmarina (17E) described a colorimetric method to determine ethylmercuric chloride and diethylmercury in water. Both compounds are first oxidized with potassium permanganate in acid medium to release inorganic mercury ion. The mercury is then extracted with dithizone and compared with standards. Sensitivity of the method is 0.005 mg per liter.

Chao, Jenne, and Heppting (5E) studied adsorption of traces of silver from natural water samples on sample containers as a function of pH. Maintenance of water samples at pH 1 with hydrochloric or nitric acid limited the silver adsorption on polyethylene containers to 1 to 2% for 60 days. When sample solutions contained in glass bottles were acidified with hydrochloric acid to pH 1, 2, or 3, container adsorption of silver was less than 2% for 30 days. Chao, Fishman, and Ball (4E) developed an atomic absorption method for the determination of 0.1 to 1  $\mu\text{g}$  of silver per liter in water. At pH 1, AG1-X8 anion-exchange resin quantitatively removes silver from one or more liters of water. The silver is eluted from the resin with an acetone-nitric acid-water mixture; the acetone is evaporated at 50° to 55°C, and the solution is partially neutralized with concentrated ammonia. The silver is then chelated with APDC, the silver complex extracted with MIBK, and the silver measured by atomic absorption. Silver in the 1- to 10- $\mu\text{g}$ -per-liter range can be determined by extraction without preconcentrations on an ion-exchange resin.

Salman (15E) developed a technique for determining as little as 5 parts per trillion of silver in water with an error in reproducibility within 5%. Silver is extracted from water by a solvent-extraction technique by using an organic-complexing agent. The silver in the extract is then determined by atomic absorption, using a micro-sampling boat accessory.

Chao, Jenne, and Heppting (6E) reported that adsorption of gold from natural-water samples on container walls can be prevented for more than 21 days by any of the following methods: acidification to pH 1 with hydrochloric acid and addition of 5 to 50 mg of bro-

mine per liter; acidification to 1N with hydrochloric acid without bromine addition; acidification to 2 to 3N with nitric acid. To determine gold in waters in the nanogram range, Chao (3E) first acidified 10 or more liters of water to pH 1 with hydrochloric acid and then added 50 mg of bromine per liter. The sample was then passed through AG1-X8 anion-exchange resin (chloride form) and gold was eluted from the resin with an acetone-nitric acid-water mixture. Bromine water was added to the eluate, and the gold was extracted into MIBK and determined by atomic absorption. Zlatkis, Bruening, and Bayer (18E) described a chelation-atomic-absorption method to determine part-per-billion concentration of gold in water. The method involved a concentration step which was effected by chelation on a chromatographic column containing a support coated with a polyschiff base. The latter was prepared by the condensation of 1,4-diamino-2,5-dimercap-tobenzene with glyoxal. The gold present in this chelate form was extracted with MIBK and determined by atomic absorption.

Asamov *et al.* (2E) determined gold in water using ion-exchange and neutron activation. Gold is adsorbed on AV 17 anion exchanger in the  $\text{ClO}_4^-$  form. The resin is removed from the column, placed on a filter to dry, packed into aluminum foil, and the gold determined by activation analysis.

Abdullaev, Zakhidov, and Nishanov (1E) determined tungsten and gold in mine waters by neutron activation. Details of the procedure are given.

A spectrophotometric method for the determination of mercury, thallium, and gold in water and other media is described by Kothny (12E). Iodide forms complexes with mercury and gold, whereas bromide forms complexes with thallium and gold. Crystal violet produces a toluene-extractable compound with these complexes in an acid medium. Elimination of interferences from each other is presented. As little as 0.1  $\mu\text{g}$  of each element can be determined.

#### VANADIUM, ZIRCONIUM, TUNGSTEN, MOLYBDENUM, SCANDIUM, URANIUM, AND RARE EARTHS

Chau and Lum-Shue-Chan (9F) developed an extraction-atomic absorption method for the determination of vanadium with a sensitivity of 0.3 ppb. Vanadium(IV and V) and 90% of vanadium(III) were chelated with dichlorooxine and extracted into MIBK. Radiometric recoveries of 93 and 92% were obtained for extraction of vanadium from 500 and 1000 ml of lake water, respectively.

A method for determining vanadium in water based on its catalytic action

on the reaction of bromate with 5-amino-*N*-phenylanthranilic acid was studied by Lazarev and Lazareva (16F). Details of the procedure are given.

Linstedt and Kruger (17F, 18F, 19F) developed a neutron activation procedure for determining vanadium in water. The limit of detection for 1-liter samples was 0.1  $\mu\text{g}$  per liter. There is a preirradiation concentration of the samples. The samples are passed through Dowex 50W-X8 cation-exchange resin (hydrogen form) which quantitatively retains the vanadium. The vanadium is then eluted with 300 ml of 2N  $\text{HNO}_3$  and reduced to 3 to 4 ml by evaporation. The evaporated samples are quantitatively transferred to vials for activation.

Sastry, Krishnamoorthy, and Sarma (24F) described a method for determining zirconium in sea water and sediments. A sample is evaporated to dryness and leached with EDTA. The EDTA solution is evaporated, heated at 500  $^\circ\text{C}$ , and the residue taken up with 10N hydrochloric acid and oxidized with  $\text{KBrO}_3$ . The solution is then centrifuged and passed through Dowex-1 anion-exchange resin. The zirconium is eluted with hydrochloric acid, converted to the nitrate, and coprecipitated with  $\text{Ce}(\text{IO}_3)_4$ . After decomposition with hydrochloric acid, Alizarin Red S is added and the absorbance of the zirconium lake measured at 560 nm. Recovery is approximately 25 to 30%.

A neutron activation method for determining tungsten in water was developed by Abdullaev *et al.* (2F, 3F). A description is given of sample preparation and of irradiation, extraction, and precipitation procedures. Tungsten-187 activity is measured from the  $\gamma$ -peak, 0.072 MeV., with the aid of a 100-channel  $\gamma$ -spectrometer.

Kawabuchi and Kuroda (13F) described a combined ion-exchange spectrophotometric method for the determination of molybdenum and tungsten in sea water. The two elements are absorbed on Dowex 1-X8 (thiocyanate form). Molybdenum can also be absorbed in Dowex 1-X8 (chloride form). With either procedure, both elements are easily eluted. Tungsten is separated from molybdenum by anion exchange on DEAE (chloride form). They are then determined spectrophotometrically with dithiol.

To determine molybdenum in lake waters, Chau and Lum-Shue-Chan (8F) complexed molybdenum with oxine, extracted the complex with MIBK, and determined the molybdenum in the extract by atomic absorption with a nitrous oxide flame. The sensitivity of the method was 1 ppb. The coefficient of variation for spiked lake water and natural water containing 20  $\mu\text{g}$  and 5.56  $\mu\text{g}$  of molyb-

denum per liter was found to be 1.8 and 4.0%, respectively.

Armstrong and Goldman (5F) described a spectrophotometric method capable of determining as little as 0.2 ppb of molybdenum in water. Dissolved molybdenum is coprecipitated with hydrated manganese dioxide, and molybdenum is determined with dithiol. Oxidation with perchloric acid releases molybdenum from organic material.

Kim and Zeitlin (14F) studied the behavior of ferric hydroxide to clarify its role as a collector of molybdenum in sea water. They found that at pH 4 molybdenum is quantitatively removed. Calcium and magnesium salts will also coprecipitate molybdenum at pH 10; but the recovery is less and the linearity of the working curve using the thiocyanate procedure is less pronounced than at pH 4. With these disadvantages, together with the bulky nature of the calcium and magnesium precipitate, the authors preferred the ferric hydroxide method of collecting the molybdenum.

Chau and Wong (10F) described an atomic absorption method for the determination of submicrogram concentrations of scandium in sea water. Scandium is coprecipitated with ferric hydroxide at pH 8 to 9, separated from the iron by ion exchange, and extracted into a solution of oxine in butyl alcohol. The extract is aspirated into a nitrous oxide-acetylene flame. Recoveries of 99 to 100% were obtained.

Yamabe and Takai (27F) gave a general explanation on the separation of uranium from sea water by ion exchange, adsorption precipitation, and foam separation methods. Possible difficulties of the separation were also discussed. Ogata (21F) and Ogata and Inoue (22F) studied the coprecipitation of uranium in sea water by ferric, aluminum, and titanium hydroxides. The latter was the most effective, giving greater than 85% yield. Uranium was then extracted with carbonate and determined fluorometrically. Smith and Lynch (25F) described a method for determining uranium in stream sediment and water. The samples are fused with a carbonate-fluoride flux, exposed to ultraviolet radiation, and uranium determined from the fluorescence produced. Quenching from other elements was minimized by using small samples, or by the addition of known amounts of uranium.

A method, applicable in the field, for the determination of uranium at the 1-ppm level in natural water was reported by Heunisch (12F). Uranyl ion is extracted from aqueous solution into butyl phosphate in isooctane. The organic phase is then mixed with dibenzoylmethane color reagent, and the absorbance of the solution is either measured at 410 nm, or compared



visually with a standard solution. Uray and Stoicovici (26F) determined uranium in water by measuring  $\alpha$ -particle activity. Uranyl ion is extracted with tributyl phosphate into a light gasoline fraction. After treatment of the organic phase to obtain a constant volume, a portion of the sample which still contains traces of water is mixed with 20% ethanol solution and placed in a liquid scintillation counter. Uranium was detected down to 5.35  $\mu\text{g}$  per liter. The efficiency of the method was  $88 \pm 3\%$  for a concentration of 50  $\mu\text{g}$  of uranium per liter.

Edwards (11F) used the uranium-234/uranium-238 ratio to determine the amount of uranium present in natural waters. The uranium was coprecipitated with aluminum phosphate, extracted into ethyl acetate, further purified by ion exchange, and finally electroplated on a titanium disk for  $\alpha$ -counting. Overall recovery of about 90% and a counting efficiency of 25% allowed analyses of samples containing as little as 0.10  $\mu\text{g}$  of uranium per liter.

Bertine, Chan, and Turekian (6F) determined uranium in natural waters by induced fission track analysis. It was done by utilizing a discharge counter instead of by visual counting. Comparisons with samples whose uranium concentrations were determined independently indicated that the method was accurate.

Bourseau, Fabre, and Zini (7F) reported that the measurement of internal conversion X-rays of uranium could be used for determining the uranium content of natural and industrial solutions.

Abdulleav, Kim, and Faizullaev (4F) reported an activation analysis method for determining lanthanum in natural water. Sensitivity was  $1 \times 10^{-9}$  g lanthanum with an error of approximately 10 to 20%. Sodium, calcium, magnesium, and chloride did not interfere. A neutron activation method for the determination of dysprosium in natural waters was described by Abdullaev, Faizullaev, and Kim (1F). Sensitivity was  $2 \times 10^{-11}$  g per ml. Oda (20F) used activation analysis for determining several rare-earth elements in hot-spring water.

A spectroscopic method for determining yttrium in ocean water was described by Romanov and Eremeeva (23F). The mean arithmetical error of the determination was  $\pm 7.7\%$ .

Krishnamoorthy, Sastry, and Sarma (15F) described a colorimetric method to determine cerium in marine environments. Details of the procedure are given. Sensitivity was 0.033  $\mu\text{g}$  of cerium per  $\text{cm}^2$ .

#### BORON, SELENIUM, ARSENIC, AND ANTIMONY

Bunton and Tait (2G) reported that calcium and magnesium salts interfere

appreciably with the determination of boron by the curcumin method. Hardness levels above 100 mg per liter caused a turbidity in the final solution. Introduction of an initial ion-exchange step, Zeo-Karb 225, eliminated the problem. The standard curcumin procedure was used.

To determine microgram amounts of boron in natural waters, Malyuga (7G, 8G) evaporated a sample to dryness, dissolved the residue with sulfuric acid, added solutions of sodium chloride and hydrazine, and heated the solutions until the evolution of gases ceased. Carmine solution was then added and the solution was allowed to stand overnight. The absorbance of the clear solution was measured at 610 nm. The sensitivity was 0.025  $\mu\text{g}$  of boron per ml. Ramanauskas and Suliuniene (10G) determined boron in water colorimetrically with quinolineserine in sulfuric acid. Nitrate concentrations greater than 2.9  $\mu\text{g}$  per  $\text{cm}^3$  interfered and were decomposed with hydrazine or 5% sodium chloride solution.

Lionel (6G) described an automated method on the AutoAnalyzer for the determination of boron in river waters. The method is based on the reaction of carminic acid with boron in concentrated sulfuric acid. Phenol is added to eliminate nitrate interference. The detection limit is 0.02 mg of boron per liter, and the standard deviation at 3.0 mg per liter is  $\pm 0.03$ . Hulthe, Uppstrom, and Ostling (5G) used an open system of automatic analysis for the determination of boron in sea water with curcumin. The equipment used is a module system called Autolab, which is fully described. Samples containing concentrations of boron between 0.1 and 6 mg per liter were investigated. The standard per cent deviation at mid-range was 1.5%.

To determine boron in sea water, Belyaev and Ovsyanyi (1G) used both cation- and anion-exchange resins to eliminate interferences. Boron is then determined by titration with sodium hydroxide in the presence of mannitol. Cation and anion exchange were also used by Gorene, Marsel, and Tramsek (4G) prior to determining boron by mass spectrographic techniques.

Burriel Marti and Alvarez Herrero (3G) studied the effect of a number of compounds on the spectrochemical determination of boron in water. Boron lines 2496.80 Å and 2497.72 Å were found to be highly enhanced by  $\text{CoO}$ ,  $\text{CuO}$ ,  $\text{Li}_2\text{SO}_4$ ,  $\text{NaF}$ , and  $\text{BeO}$ . A procedure for determining boron is then described.

Sherratt and Conchie (11G) reported a new method for the determination of selenium in effluents and waters. The method depends upon ion exchange to isolate the selenium from interfering substances and subsequent measurement of selenium as the colloidal metal.

Ascorbic acid or hydrazine sulfate is used as the reducing agent. Tellurium and other more common metals do not interfere.

Whitnack and Brophy (12G) described a single-sweep polarographic method for the determination of arsenic (III) in drinking waters. As little as 5 ppb arsenic can be determined. The relative error of the method is 5 to 10% of the actual concentration. They reported that this method gave more reliable results in drinking water than the Gutzeit method currently used in the analysis of arsenic at low levels.

Popescu and Papagheorghe (9G) proposed a new spectrophotometric method for the determination of antimony in waste water. The method utilizes Rhodamine B as the reactive agent. Gold, gallium, thallium, and tungsten interfere in the reaction. Sulfate concentrations greater than 200 mg per liter must be removed. The method is sensitive to 0.05 mg of antimony per liter.

#### CHLORIDE, BROMIDE, AND IODIDE

Yamamoto *et al.* (18H) described an indirect spectrophotometric method for the determination of  $0.8 \times 10^{-5}$  to  $5.6 \times 10^{-5} M$  of chloride in fresh waters. The method is based on the reaction of chloride with mercuric thiocyanate which releases thiocyanate ion proportional to the amount of chloride present. The thiocyanate ion is then extracted into nitrophenol with tris(1,10-phenanthroline)iron(II) chelate. The red color of the organic phase is measured at 516 nm. Bromide, iodide, cyanide, sulfide, and thiocyanate interfere.

Dojlido and Bierwagen (5H) determined chloride concentrations between 1 and 100 mg per liter in water with a Technicon AutoAnalyzer. Kahn (9H) used an AutoAnalyzer with two mixing manifolds to determine chloride in the concentration range of 30 to 20,000 mg per liter. The coefficient of variation between determinations compared favorably with those obtained by standard methods.

Fishman and Feist (7H) described an automated potentiometric method using a Fisher Titralyzer for determining chloride in water. Chloride is titrated with standard silver nitrate solution and the end point detected potentiometrically, using chloride ion-selective and reference electrodes. Tests indicated that the method determines chloride rapidly, eliminates color and sediment interferences, and is as accurate as other automated techniques.

Tamarchenko and Toropova (16H) described a photometric method for the determination of bromide in natural water based on the oxidation of bromide to bromate and the decolorization of

bromine formed during the interaction of bromate with excess bromide in an acid solution. The sensitivity of the determination is  $10^{-7}M$  bromide. Iodide does not interfere; however, molybdate ions accelerate the reaction in the absence of iodide. Moldal and Zyka (13H) determined traces of bromide in water spectrophotometrically with rosaniline. Beer's law is followed for 0.5 to 22  $\mu\text{g}$  of bromide. Tolerance limits for a number of other constituents found in water are given.

A completely automated procedure for determining bromide in water was described by Archimbaud and Bertrand (1H). Bromide was first oxidized to bromine with chloramine T, and the bromine reacted with phenolsulfonephthalein at pH 4.6 to give the purple tetrabromosulfonephthalein. The absorbance of the solution was measured at 589 nm. At this wavelength, chloride and fluoride interferences were eliminated. Beer's law was followed for 20 to 200  $\mu\text{g}$  of bromide per liter.

Leddicotte and Navarrete Tejero (11H) used activation analysis to determine bromide in drinking water. The samples were passed over a known weight of Amberlite IR 100, and a portion of the resin was subjected to neutron irradiation for 1 minute in a nuclear reactor operating at maximum capacity of 5 Mw and with a flux of  $10^{14}$  neutrons per  $\text{cm}^2$  sec. The sensitivity of the method was within  $10^{-10}$  g bromide for a flux of  $10^{12}$  neutrons per  $\text{cm}^2$  sec. Eckhoff, Hill, and Kimel (6H) reviewed the theory and development of neutron activation analysis. Methods for determining a number of elements in various media are given, including a method to determine bromide in sea water.

Cuttitta and Rose (4H) used a new approach to solve matrix problems in X-ray fluorescence analysis of trace elements and applied it to the determination of bromide in saline waters. In this proposed technique, the slope of a standard curve prepared from pure solutions is compared with that obtained on spiked samples. The ratio of the two slopes permits the calculation of an adjusted background which does not significantly differ from that of an absorbent impregnated with the sample matrix free of the element sought.

Matthews and Riley (12H) described a procedure for the determination of iodide and iodate in sea water. Iodide is coprecipitated with silver chloride by silver ions slowly liberated into the water by the dissolution of silver citrate. The iodide is then oxidized to iodate with bromine under the influence of ultrasonic agitation. Any hypobromite is destroyed and iodine is determined either by photometric titration or by spectrophotometry. Iodate is not coprecipitated with silver chloride, but

can be determined after reduction with hydrazine sulfate. Recoveries of iodide and iodate are excellent.

Ballezo (2H) reported that iodide in mineral water could be determined photometrically on the basis of the iodide-catalyzed oxidative stabilization of blue Tetrabase. The blue oxidation product could also be determined by titration with sodium thiosulfate to the disappearance of the color. The detection limit was  $10^{-15}$  g iodide.

Shveikina (15H) used both a kinetic method and a colorimetric method to determine 0.1 to 0.5  $\mu\text{g}$  of iodide in water. The kinetic method is based on the loss of color in the oxidation of ferric thiocyanate by sodium nitrite in the presence of iodide. The colorimetric method involves toluene extraction of the complex of Brilliant Green with  $\text{I}_2\text{Cl}^-$ .

Revel (14H) determined iodide in sea water with an AutoAnalyzer. The method is based on the reduction of  $\text{Ce}^{4+}$  to  $\text{Ce}^{3+}$  by  $\text{As}^{3+}$  which is catalyzed by iodide. Ten per cent sodium chloride is added to suppress the effects of mercury and silver. The temperature is controlled at  $57^\circ \pm 0.2^\circ \text{C}$ . Keller, Doenecke, and Leppla (10H) used the same reaction and an AutoAnalyzer to determine 0.5 to 10  $\mu\text{g}$  of iodide per liter in drinking water. The instrument will analyze 12 samples per hour with a reproducibility of  $\pm 0.1$   $\mu\text{g}$  iodide per liter.

Havas *et al.* (8H) described the use of a Pungor-type iodide-selective rubber membrane electrode for the determination of iodide in mineral water. Either a silver-silver chloride or a calomel electrode is suitable as the reference electrode. Iodide is determined directly by measuring the emf of the cell and reading the activity from a calibration curve plotted with standard solutions. An indirect titrimetric method and a direct cyanide method are also given.

An amperometric titration method for the determination of iodide in water was reported by Barikov, Songina, and Klimko (3H). Iodide was titrated with silver nitrate in the presence of excess sodium chloride, using a graphite indicator electrode.

Wilkniss (17H) used photon activation analysis to determine chloride, bromide, fluoride, and iodide from a single sample of sea water.

#### FLUORIDE

Crosby, Dennis, and Stevens (3J) evaluated the Alizarin Red S, Eriochrome Cyanine R, SPADNS, and alizarin complexone spectrophotometric methods, and the ion-selective electrode method for the determination of fluoride in potable waters and other aqueous solutions. They recommended the ion-selective electrode method because of

the speed of analysis, accuracy, and convenience. The electrode method also gave the highest recoveries (100%) of 1 ppm of fluoride added to river-water samples. Patterson, Bunton, and Crosby (14J) also evaluated visual and specific-ion electrode methods for the determination of fluoride in potable waters. They said that the Palin and electrode methods are both suitable for on-site testing.

Weiss (17J) employed a lanthanum-fluoride membrane electrode for the determination of fluoride in mineral waters. The addition of 1M ammonium nitrate kept the ionic strength at 0.1M and the pH at 5.6. Temperature can be varied from  $0^\circ$  to  $80^\circ \text{C}$ , but must be kept constant for a set of samples because the electrode potential changes 8 mV per change of  $10^\circ \text{C}$ . Sodium citrate and EDTA are added to eliminate interferences from iron and aluminum. Harwood (6J) also evaluated a commercially available fluoride electrode for the routine determination of fluoride in water. Aluminum interfered seriously. He recommended an improved ionic buffer containing CDTA to control the interference because both citrate and EDTA had limited applicability. Edmond (5J) studied the application of the fluoride-selective electrode to the determination of fluoride in waters and other materials. Results compared favorably with other methods. Warner (16J) used the lanthanum fluoride electrode to determine fluoride in sea water. Details of the procedure are given.

Collis and Diggins (2J) described an automated fluoride monitor system with a fluoride-responsive electrode for on-line analysis of fluoridated water supplies. Details of the method and illustrations of the system are presented.

Macejunas (12J) compared the zirconium chelates of xylenol orange, methylthymol, methylxylenol blue, fluorexon, and phenolphthalexon as analytical reagents for determining fluoride in potable water. Zirconium-xylenol orange gave the best results and a spectrophotometric method was developed for the determination of less than 0.8 mg of fluoride per liter. Harwood and Huyser (7J) described an automated colorimetric method using zirconium-xylenol orange for the determination of fluoride in water. A linear calibration curve was obtained from 0 to 2 mg of fluoride per liter.

To determine fluoride in water spectrophotometrically with lanthanum-alizarin-complexon, Kempf (10J) first removed interfering cations by passing the sample through a strongly acidic cation-exchange resin in the hydrogen-ion form. The absorbance of the solution, after adding the color-forming reagent, is measured at 620 nm. Lambert-Beer law is followed for

0.02 to 0.5 mg of fluoride per liter. Sulfate and chloride interfere, but curves are given to make corrections. Quentin and Rosopulo (15J) used a preliminary steam-acid distillation procedure to separate fluoride from interferences prior to its determination with lanthanum-alizarin complexon.

Marczenko and Choluj-Lenarczyk (13J) determined fluoride in tap water by the zirconium-Eriochrome Cyanine method. Fluoride is first distilled from a sample mixed with perchloric acid into water made alkaline with ammonium hydroxide.

Dozanska and Sikorowska (4J) compared three colorimetric methods for determining fluoride in water and found that the alizarin-zirconium method was the most precise and responsive after removal of interfering substances.

Wierzbicki and Pawlita (18J) determined fluoride in chlorinated water with Alizarin S and zirconyl chloride. The color is compared, visually, with standards.

A method is described by Zamanov and Rafikova (20J) for determining fluoride ion in natural waters. The method is based on preliminary precipitation of sulfate with benzidine hydrochloride, followed by titration of fluoride with thorium nitrate in the presence of Alizarin S indicator. A procedure is described by Light, Mannion, and Fletcher (11J) for the titration of fluoride with thorium(IV) as titrant at the 1-mg-per-liter level in potable water. The procedure employs an ion-exchange step for concentration of fluoride and removal of interfering ions. Precision and relative error of the method are both 1%.

To determine fluorides in natural and waste waters, Kazakova and Zolotavin (9J) added a solution of alizarin-complexon and ceric nitrate in acetone to a sample of water. After waiting 20 minutes, the absorbance of the solution is measured at 617 nm.

Bond and O'Donnell (1J) developed a highly specific polarographic method for precise determination of fluoride in the concentration range of 0.05 to 20 ppm, based on the shift of the uranium(V)-uranium(III) half-wave potential. The method is applicable to the analysis of potable waters because no prior separation or concentration of fluoride is required.

A photon activation analysis method for the determination of fluoride in sea water and the problems encountered, such as interfering reactions, sample preparations, etc., are discussed by Wilkniss and Linnenboom (19J).

A method for the analysis of fluoride in waste waters, river waters, and effluents by micro diffusion is described by Hey and Jenkins (8J).

#### SULFATE AND SULFIDE

Macchi, Cescon, and Mameli-D'Errico (12K) determined sulfate in sea water titrimetrically. A sample, diluted with distilled water, was passed through a cation-exchange resin to remove interferences. Sulfate was then titrated with barium chloride using thorin as the indicator. Chloride interference was eliminated by standardizing the titrant solution with a solution of sulfuric acid containing hydrochloric acid at a concentration corresponding to the chlorinity of standard sea water. The coefficient of variation calculated from 25 replicate analyses of the same sample was 0.13%, and the accuracy estimated from 16 analyses of 5 artificial sea waters was of the same order. Costache (5K) used ion exchange followed by titration with barium chloride to determine sulfate in water. After passing the sample through Dowex-50 ion-exchange resin, a portion of the sample is treated with alcohol, chloroacetic acid buffer (pH 2.2), barium sulfate suspension, and sodium alizarin sulfonate indicator. The solution is titrated with the standard barium chloride until a pink color appears. Sulfate concentrations between 5 and 10 g per ml can be determined with an accuracy of 0.1%. Savvin *et al.* (21K) developed a titrimetric method for the determination of sulfate in natural waters and atmospheric precipitation. A sample of water is adjusted to pH 4.9 and passed through KU-2 cation-exchange resin. The eluant is adjusted to pH 4; orthanilic K indicator and an equal amount of ethanol or acetone are added, and sulfate is titrated with barium chloride. The color change is from red-violet to green-yellow. Basargin and Nogina (1K) found that carboxyarsenazo at pH 5.6 and nitrochromeazo at pH 2 were very sensitive indicators for the determination of sulfate in natural waters using barium nitrate as the titrant. Cations interfere and must be removed by ion-exchange prior to the determination. The method compares favorably with the gravimetric method, and the relative error does not exceed 2%. Gregorowicz, Kowalski, and Olejniczenko (9K) used potassium permanganate as a redox indicator for determining sulfate titrimetrically. A sample mixed with acetone and the indicator is titrated with barium chloride.

Dollman (6K) reported that sulfate in natural water or other aqueous media could be accurately determined as sulfuric acid after passage of a sample through a cation-exchange resin (hydrogen-ion form). The method involves the evaporation of the column effluent under conditions which drive off all ordinary acids while retaining the sulfuric acid. Standard base is then used to titrate the sulfuric acid. Phos-

phate can also be determined by this procedure.

Szabo and Inczedy (23K) described a high-frequency titration method to determine sulfate in natural water after preliminary removal of cations on Varion KS cation-exchange resin. Barium acetate is used as the titrant. Barium sulfate is added to facilitate rapid separation of the precipitate. Murakami and Hayakawa (15K) determined sulfate in sea water by short-circuit amperometric titration with barium chloride at pH 5.3 to 7.3 with a relative error of 0.6%. Potassium chromate is used as indicator.

Mayer, Abel, and Kortus (13K) described an indirect polarographic micro-determination of sulfate in drinking and waste water. Sulfate is precipitated with barium chromate; and after removal of the barium sulfate precipitate, the chromium is determined polarographically.

Sarudi and Siska (20K) proposed a new method to determine sulfate gravimetrically. Interfering metals are removed by cation exchange prior to precipitation of sulfate with barium chloride. The precipitate formed by this method is free of contaminants. The relative standard deviation is 0.71%.

An aqueous solution of 2-aminoperimidine hydrochloride as a new reagent for the turbidimetric determination of 0.1 to 5 ppm of sulfate in water was proposed by Stephen (22K). As little as 0.05 ppm can be detected by precipitation of the corresponding amine sulfate. No colloidal stabilizers were needed. At the 0.5-ppm level, the relative standard deviation was 4.5%.

Petrova, Khakimkhodzhaev, and Savvin (18K) suggested 2,7-bisazo derivative of chromotropic acid as a colorimetric reagent for determining sulfate in tap water. The sample is passed over KU2 ion-exchange resin in the hydrogen form to remove interfering cations; then sulfate is determined colorimetrically in the presence of the barium - chromotropic - acid - derivative complex.

A direct spectrophotometric determination of sulfate in natural waters was described by Goguel (7K). The method allows rapid determination of sulfate in the range of 10 to 500 mg per liter by using the absorption of the  $\text{FeSO}_4^+$  complex at optional wavelengths between 325 and 360 nm. The method is accurate for samples with negligible concentrations of phosphate, fluoride, and ferric iron.

Sulfate suppresses the color development of the thorium-morin complex through the formation of thorium-sulfate complexes. Nasu (16K) used this reaction to determine 0.2 to 0.5 ppm of sulfate in river water. The absorbance of the complex is measured at 410 nm.

Fluoride, phosphate, sulfite, thiosulfate, barium, iron, and aluminum interfere. Morgen, Vlasov, and Tyutin (14K) used the thorium-morin complex to determine sulfate fluorometrically in weakly mineralized natural waters and atmospheric precipitation. A number of metals interfere, but they can be removed by cation exchange. Nasu, Kitagawa, and Mori (17K) described a photometric method (less than 100  $\mu\text{g}$  of sulfate) and a fluorometric method (less than 20  $\mu\text{g}$  of sulfate) to determine sulfate in natural waters. The first method is based on the suppression of the color development of the thorium-flavonol complex by sulfate, and the fluorometric determination is based on the quenching of the fluorescence of the complex by sulfate.

Little *et al.* (11K) determined sulfate in surface waters indirectly by atomic absorption. Sulfate is precipitated as lead sulfate in 40% ethanol, and the residual lead in solution is determined by atomic absorption. As little as 1 ppm of sulfate can be determined. Borden and McCormick (2K) also used atomic absorption to determine sulfate in water. Sulfate is precipitated with barium, and the remaining barium is measured by atomic absorption. Sensitivity was 1.5 ppm sulfate per 1% absorption. The method was used without dilutions for samples containing up to 150 ppm sulfate. Pleskach and Chirkova (19K) used flame photometry to determine sulfate in water by measuring the excess barium after precipitation of sulfate as barium sulfate. Samples containing 600 to 800 mg of sulfate per liter were mixed with a solution of barium chloride containing 15,000 mg per liter.

A microbiological assay method for determining sulfate in rain water by means of cultivation of *Escherichia coli* is described by Yamamoto and Oba (24K). The calibration curve ranged from 0.5 to 20 ppm of sulfate.

Lambert and Manzo (10K) reported that sulfide can be determined in water by using a column of crystal violet-tetraiodomercurate(II) ion-association reagents. Details are given on preparation of the reagent. A sample is passed through the column and the absorbance of a portion of the eluted ion is measured at 590 nm. Beer's law is obeyed for 0 to 6.0 ppm of sulfide. Cyanide, strong oxidants, and reductants interfere. Cline (4K) described a spectrophotometric method for the determination of hydrogen sulfide in natural waters. A mixed diamine reagent is used to determine sulfides in the concentration range of 1 to 1,000  $\mu\text{g}$  per liter.

Grasshoff and Chan (8K) adopted the methylene blue method to the AutoAnalyzer for the determination of 0 to 500  $\mu\text{g}$  atoms of hydrogen sulfide per

liter. The sulfide samples are stabilized with zinc chloride gelatin solution prior to determination. Casapieri, Scott, and Simpson (3K) also determined sulfide automatically with an AutoAnalyzer. The method is based on the measurement of the violet color that forms from the reaction of sodium nitroprusside and sulfide in a pH 12 buffered solution. Sulfide is distilled from the water as hydrogen sulfide and swept into the buffered solution.

#### PHOSPHORUS AND SILICA

Lee (17L) prepared a review on sources of phosphorus in water bodies, methods of sampling, preservation, and choice of analytical methods. Olsen (24L) reviewed methods for the determination of orthophosphate in water. Included are 137 references. The use of ascorbic acid is discussed, as is solvent extraction.

Piton and Voituriez (25L) reported that the total phosphorus in sea water is made up of inorganic phosphorus in solution, organic phosphorus in solution, and phosphorus in the suspended matter. They used known methods to determine each form. Piton and Voituriez (26L) also compared the stannous chloride and ascorbic acid methods for determining phosphate, and reported that the ascorbic acid method gave better precision and much greater stability of color.

A number of investigators, Link (18L), Harwood, Van Steenderen, and Kuehn (14L), and Drews (7L), described modifications of the orthophosphate method of Murphy and Riley (23L), in which the phosphorus-molybdenum complex is reduced with ascorbic acid in the presence of antimony. Isaeva (16L) used a similar procedure but extracted the colored phosphorus complex with 1:1 iso-butyl alcohol:benzene. The absorbance of the extract was measured at 690 nm. As little as 0.1  $\mu\text{g}$  of phosphorus per liter could be determined.

Fossato (9L) determined low levels of reactive phosphorus in sea water with a modification of the Strickland and Parsons method. The organic phase is centrifuged prior to measurement, which eliminates haziness observed with seawater samples.

Soier and Semenov (28L) studied the determination of organic phosphorus in fresh water by ultraviolet irradiation. The observed results were found to agree well with those obtained by the ignition method.

Blaisdell, Compton, and Nair (4L) described an automated method to determine the various forms of phosphate as orthophosphate. The aminonaphtholsulfonic method was used and the concentration of the standards ranged from 0 to 6 mg per liter. Acid

hydrolysis of the samples was done manually before placing them in the AutoAnalyzer. Campbell and Thomas (6L) found that values obtained for dissolved phosphorus in the low microgram-per-liter range in lake and stream waters, in some cases, were higher than total phosphorus values. They showed that the error was due to silica which also reacts with ammonium molybdate. They also mentioned that silica is dehydrated during perchloric acid digestion for total phosphorus and, thus, does not interfere. A method is presented to permit an automated simultaneous determination of dissolved phosphorus and the silica interference so that a correction can be made. Heinke and Behmann (15L) determined condensed phosphates in water with ion-exchange chromatographic analysis. Color was developed by an automated stannous chloride procedure.

Addison and Ackman (1L) determined elemental phosphorus in water by gas-liquid chromatography, on either a 3% OV-1 Chromosorb W column at 100° or on a 3% SE-30/Chromosorb W column at 120°, using helium as the carrier gas. The phosphorus was first extracted with benzene or isooctane. A flame photometric detector with a 526-nm filter was used for measuring the phosphorus. The detection limit was  $10^{-12}$  g of phosphorus.

An indirect flame spectrophotometric determination of phosphate in water was described by Sakai and Okura (27L). A known volume of standard lead nitrate was added to a sample containing less than 700 ppm of phosphate. The pH of the solution was adjusted to 4-5 with sulfuric acid or ammonium hydroxide, and then filtered. The decrease of luminosity of the filtrate was measured at 405.7 nm in an oxygen-hydrogen flame, and the luminosity of the sample at 410 nm was subtracted. The calibration curve of each phosphate (ortho, pyro, tetrapoly, or meta) had a characteristic slope. Sulfite, sulfate, and chromate interfered and were removed with barium salts.

Guyon and Shults (12L) described two fluorometric procedures for the determination of phosphate in water. For concentrations below 1  $\mu\text{g}$  per ml, an aluminum-morin system was used. Concentrations greater than 10  $\mu\text{g}$  per liter were determined by a tin-flavonol system. The theory of the methods and details of the procedures are given.

Hahn and Schmitt (13L) determined 10 ppb to 100 ppm of phosphate in water radiometrically. Molybdic acid and tungsten-185 were added to a sample to give a radioactive tungstomolybdophosphoric acid complex which was then extracted into 2-octanone and counted. The optimum pH for the formation and extraction of the complex was 0.9 to 1.2. Arsenic, silica, and niobium interfered

to about the same extent as in spectrophotometric methods.

Allen and Hahn (2L) used activation analysis to determine phosphate in natural waters. Molybdate and tungstate ions are added to the sample and the tungstomolybdophosphoric acid is extracted with 2,6-dimethyl-4-heptanone. An aliquot of the organic phase is activated for 1 hour at a flux of  $10^{13}$  n per  $\text{cm}^2$  per sec. The gamma spectrum from the activation is essentially tungsten-187 and is proportional to the concentration of phosphate in the sample. By using a 100- $\mu\text{l}$  aliquot for activation, accurate results are obtained at concentrations of 4 to 200 ppb of phosphorus.

Braun (5L) reviewed the advantages and disadvantages of the various methods for determining silicate in surface water. Various factors which affect the molybdenum blue procedure were studied and a procedure was developed. Metol and sodium sulfite were used to reduce the heteropolyacid to molybdenum blue. Liss and Spencer (19L) investigated several methods used for the determination of silicate in sea water.

Duce and Yamamura (8L) described a spectrophotometric method for the determination of microgram quantities of silica, as molybdenum blue. It combines the desirable features of existing spectrophotometric methods with three pretreatment procedures. The pretreatments are: (a) conversion of nonreactive silicon species into the reactive monomer with hot sodium hydroxide solution, (b) removal of cations and excess sodium hydroxide with cation-exchange resin, and (c) complexation of fluoride with boric acid in the presence of cation-exchange resin. The attainable precision at the 30- to 40- $\mu\text{g}$  silica level is 1.25% relative standard deviation.

A method is described by Morrison and Wilson (21L) for determining polymeric silicic acid in water. The sample is first digested with sodium hydroxide to convert the polymer into reactive silica which is then determined spectrophotometrically as the reduced  $\beta$ -molybdosilicic acid. At concentrations of about 0.02 ppm, the standard deviation was 0.001. Webber and Wilson (31L) described an ion-exchange procedure for determining the total silica content of high-purity water. Silicon is first concentrated on a mixture of finely ground cation and anion-exchange resins, which are then ignited and fused with sodium carbonate. The resulting melt is then determined spectrophotometrically as the reduced  $\beta$ -molybdosilicic acid. The standard deviation for 1-liter samples containing between 0 and 100  $\mu\text{g}$  of silica was approximately 3  $\mu\text{g}$  of silica. Baker and Farrant (3L) determined total silica in water spec-

triphotometrically by the molybdenum blue procedure. Silica is first converted to its reactive form by evaporation and fusion with sodium carbonate. Fukui, Mizumoto, and Fujii (10L) studied a number of procedures for converting colloidal silica into ionic silica in boiler feed water. Results of their investigation are given. Vainer and Yakimets (30L) described a method to determine total silica in thermal powerplant water. Sodium chloride solution and hydrofluoric acid are added to an aliquot of water. The solution is evaporated to dryness and the residue dissolved in water. Silica is then determined by the molybdenum blue method, using stannous chloride as the reducing agent. This treatment gives better results than that with sodium carbonate. Gryk (11L) used hydroxide to convert water-insoluble silica to its reactive form.

Sturla (29L) determined silica in pure water by extraction. Silica is reacted with ammonium molybdate in acid solution to form the molybdosilicic acid. The molybdosilicic acid is then reduced and extracted into butanol, and the absorbance of the extract measured at 750 nm.

Munao (22L) proposed the separation of silica from phosphates on Amberlite IRA-400 ion-exchange resin prior to determining silica. Mizuno (20L) determined traces of silica in water after ion-exchange concentration. A liter of sample is passed through Amberlite CG-400 anion-exchange resin. The silica is then eluted with a small volume of 1N potassium hydroxide and silica in the eluant determined by the molybdosilicic acid method. The standard deviation in the concentration range of 3 to 14 ppb varied from 0.2 to 0.5 ppb. No interference is caused by other impurities.

#### NITRATE, NITRITE, AND NITROGEN COMPOUNDS

Babkin (4M) described the use of rivanol for the colorimetric determination of nitrites and nitrates in natural waters after their reduction with zinc. The Bouger-Lambert-Beer law is obeyed for 0.5 to 10 mg of nitrite per liter and 0.7 to 15 mg of nitrate per liter. The accuracy of the determination is  $\pm 10\%$ . Iodide, fluoride, sulfite, thiosulfate, and thiocyanate interfere with the nitrite determination. A brucine photometric method for determining nitrates and nitrites in water is outlined by Fadrus and Maly (15M). Concentrations of greater than 5 mg of ferric ion per liter and 25 mg of ferrous ion per liter must be removed with an ion exchanger.

Henriksen and Selmer-Olsen (16M) reported on automatic methods for determining nitrate and nitrite in water and soil extracts. Twenty samples per hour can be analyzed with the Auto-

Analyzer by using a method modified from a standard manual procedure. The method makes use of a copperized cadmium reductor and is not subject to interference from humic acids.

Twenty-six references are included in the review presented by Thielemann (36M) on the various methods for determining nitrate and nitrite in water and waste water.

Matsunaga and Nishimura (22M) determined nitrate in sea water by a modification of Bendschneider and Robinson's method. Nitrite is determined, and the nitrate is calculated by difference. The absorbance of the solution is measured at 543 nm, and the sum of the nitrite plus nitrate in a sample is determined from the absorbance concentration curve. The determination is quantitative between 0.31 and 1.55 mg of nitrate per liter in the presence of 0 to 0.230 mg of nitrite per liter. An ultraviolet spectrophotometric determination of nitrates in the presence of nitrites in water and sewage is outlined by Sobota (34M). To determine the sum of nitrate and nitrite, add to a 10-ml sample, containing more than 1 mg nitrate and nitrite per liter, hydrochloric acid, remove organic substances with charcoal, dilute, and determine the absorbance at 205 to 210 nm. To determine nitrate alone, transform the nitrite into the corresponding azo compound, decompose this compound, and read the absorbance of the sample.

The reactions of 2-nitroso-1-naphthol-4-sulfonic acid with nitrate in acidic solutions were studied by Abercrombie and Caskey (2M). When the concentration ratio of nitrate to 2-nitroso-1-naphthol-4-sulfonic acid is one or less, 2-nitro-1-naphthol-4-sulfonic acid is the product. Under these conditions, Beer's law is followed over a nitrate (as nitrogen) concentration range of 0.3 to 13 ppm. Isaeva and Bogoyavlenskii (18M) have determined nitrates in sea water by reducing them to nitrites with amalgamated cadmium and reacting the resulting nitrites with sulfanilamide and  $\alpha$ -naphthylethylenediamine to form colored azo compounds. Approximately 96 to 97% of the nitrates in sea water are reduced by this method. Phenolsulfonic acid and *o*-tolidine were employed by Sadowski (31M) to determine nitrates in the presence of chloride. This modified method of Macejanus can determine nitrates in the range 1 to 90 mg per liter. Iron(III), manganese(IV), manganese(II), nitrites, some oxidants, turbidity, and colored waters may interfere.

Sarudisen (32M) described a gravimetric procedure for the determination of nitrate in drinking water. A nitron solution is added to a warm, acidified water sample. After 24 hours, the precipitate is removed by filtration, washed, dried at 105°-110°, and



weighed. The conversion factor for nitrate is 0.1652.

Nitrate in the range of 0.1 to 10  $\mu\text{g}$  of nitrogen per liter can be determined by using marine nitrate-reducing bacteria as outlined by Yoshida (39M). Bacteria and sodium acetate are added after the solution is centrifuged to remove the bacteria originally present. The concentration of nitrite is determined by a standard method after incubation and recentrifugation.

Several investigators discussed the determination of nitrate in waters and effluents using an ion-selective electrode. Bunton and Crosby (8M) carried out their measurements at constant temperature and in a buffered solution to overcome the ionic concentration effect. Other ions found in water can interfere, and the electrode is not accurate below 5 mg per liter of nitrogen. Abbott (1M) found that the common commercial nitrate-activity electrode is insensitive to variations between 0 and 100 micromoles per liter of nitrate in solutions with salinity between 0.153 and 15.32 g per kg. According to Keeney, Byrnes, and Genson (19M), the nitrate ion-selective electrode can be used for the routine determination of greater than 10 mg per liter of nitrate (as nitrogen) in well and surface waters. Interferences from diverse anions were small at commonly observed levels, and recoveries of nitrate from well and surface waters were 88 to 105% and 83 to 98%, respectively. However, the results from the electrode method were consistently lower and less precise than results by the phenoldisulfonic method.

A photometric method for the determination of nitrite in waste waters is described by Bagdasarov *et al.* (5M). Violet reaction products are formed by diazotization of 3,6-diaminoacridine sulfate. The reaction takes place at room temperature in a wide range of pH (<2.05). The relative mean error of the determination is  $\pm 1.6\%$ . Dabrowska and Hernas (11M) studied the possibility of using chlorpromazine hydrochloride for the colorimetric determination of nitrites in natural waters. This reaction produces satisfactory analytical results in the range of 0 to 100  $\mu\text{g}$  of nitrite (as nitrogen) per liter. An extraction-photometric method using Basic blue "K" was developed by Podberezskaya (29M) for the determination of nitrite in water. Calibration curves were plotted for similarly produced series of standard solutions, containing 2.5 to 80 mg of nitrite per liter.

Several authors describe the determination of ammonia by the phenol-hypochlorite method. Details of this method are given by Solorzano (35M) for natural waters. Emmet (13M, 14M) adapted this method to determine  $\mu\text{g}$  quantities of ammonia per liter. A new modification of Lubochinsky's

indophenol method for direct microanalysis of ammonia in sea water is described by Manabe (21M). By using this method, it is possible to determine 0.005 to 3.00 ppm of ammonia (as nitrogen). Inorganic electrolytes, amino acids, or urea, which were detectable in polluted sea water, do not interfere.

A method for the rapid determination of ammonia and total nitrogen in municipal waste water by microcoulometry is reported by Albert *et al.* (3M). The relative error and the relative standard deviation is generally less than or equal to  $\pm 6\%$ .

Yoshida (38M) used a marine nitrifying bacterium to determine ammonia. The concentration of nitrite formed was determined by using the Griess and Romins reagent. The lowest concentration of ammonia in sea water determinable by this method is 0.1 to 10  $\mu\text{g}$  of nitrogen per liter.

Moore and McNulty (26M, 27M) determined trace total nitrogen in water by microcoulometric titration. A flow diagram and applications of a microcoulometric titrating system are given. The sample is pyrolyzed over a granular nickel catalyst in a stream of humidified hydrogen. The bound nitrogen in the sample is converted quantitatively to ammonia and subsequently titrated electrochemically. Total nitrogen can be measured down to the sub-ppm range.

Trifonova (37M) reported on the use of diffusional-isothermal ammonia distillation for determining organic nitrogen in natural waters. Control determinations with 1 to 50  $\mu\text{g}$  of nitrogen in the sample gave good results with 0.15 to 10% error.

The Kjeldahl digestion method, followed by distillation and colorimetric determination using Nessler reagent, was critically examined as a procedure for the microdetermination of organic nitrogen in lake water by Schmid (33M).

Povoledo (30M) discussed the determination of organic carbon and nitrogen by wet-dry combustion with gas chromatographic detection. Several existing techniques were adapted for these determinations in natural waters.

McDaniel, Hemphill, and Donaldson (23M) used an AutoAnalyzer to determine total Kjeldahl nitrogen in estuarine water. The samples were analyzed for nitrogen with an 85% recovery and a lower detection limit of 0.02 mg per liter by using a mercury-catalyzed digestion. Chapman, Cooke, and Whitehead (10M) described automated determinations for ammonia, nitrate, and nitrite.

The determination and evaluation of water-borne cyanide is discussed by Bucksteeg and Dietz (7M). A vacuum distillation method is used to determine total cyanides and decomposable cyanides. Data are tabulated for several

complex cyanides, and results are compared with two modifications of the ASTM procedure. A modified colorimetric method using picric acid for the determination of cyanides in waste water is outlined by Zika (40M). Absorbance is measured at 520 to 530 nm, and the accuracy of the method for 0.2 mg of hydrogen cyanide is  $\pm 1\%$ . A titrimetric method for the determination of cyanides in some industrial and waste waters is reported by Dolzhanskaya and Edel'man (12M). Nickel sulfate is the titrant and murexide is the indicator. A sulfide correction is made, if necessary.

Mertens (24M) described a method for the determination of free cyanide in water which prevents the loss of volatile hydrogen cyanide, and which prevents the gain of free cyanides by the decomposition of complex cyanides. A titrimetric and a photometric method are both discussed. In the former, the titrant is silver nitrate and the indicator is *p*-dimethylaminobenzylidene. In the latter, the absorption is measured at 578 nm after the addition of chloramine-T solution and barbituric acid reagent. Montgomery, Gardiner, and Gregory (25M) reported a solvent extraction method for the determination of free hydrogen cyanide in river water. Undissociated hydrogen cyanide can be determined spectrophotometrically by this method, which is designed to avoid disturbance of the equilibrium between hydrogen cyanide, cyanide ion, and complexed cyanide. The detection limit is less than 0.01 mg hydrogen cyanide per liter.

An AutoAnalyzer procedure for the determination of cyanide ions in waters and effluents has been designed by Casapieri, Scott, and Simpson (9M). Cyanide between 0.5 and 40 ppm is determined spectrophotometrically (40 determinations per hour) in natural waters, trade effluents, and complex metal-cyanide solutions. The diagram of the analytical cyanide manifold is given.

An amperometric cyanide determination in waste water is described by Berndt (6M). Cyanide is determined with a rotating gold-disk electrode at a potential of +150 mV vs. S.C.E. and at a pH of 11.

A review with 24 references was compiled by Lee (20M) on the analytical chemistry of plant nutrients. The sources of nitrogen and phosphorus in water bodies, methods of sampling, and choice of analytical methods are included.

Howe and Holley (17M) conducted a comparison study of mercury(II) chloride and sulfuric acid as preservatives for nitrogen forms in water samples. Ammonium, organic, nitrate, and nitrite nitrogen forms in surface water samples underwent biological changes which were more effectively prevented



by addition of 42 mg of mercuric chloride per liter of sample than by 0.8 ml of concentrated sulfuric acid per liter. Ostrowski, Bojanowski, and Malewicz (28M) reported on the preservation of sea-water samples for nitrate determination. The samples were filtered; and chloroform, mercuric chloride, thymol, and sodium fluoride were added to study their preservative values. It was concluded that the use of these compounds for preservation of sea water is impossible, and the best preservation method is the filtration of water through a 0.5-micron pore filter. This method assures invariability of nitrate contents for 8 days at room temperature.

#### pH AND ALKALINITY

Legler, Pietschmann, and Huhn (7N) critically reviewed the various analytical methods for determining free carbonic acid and for measuring pH of slightly buffered waters by colorimetric and electrometric means.

Culberson, Pytkowicz, and Hawley (3N) investigated the determination of sea water alkalinity by the pH method. An attempt was made to increase the accuracy of the method of Anderson and Robinson for the determination of the total alkalinity of sea water, and to provide an interpretation of the empirical term,  $f_H$ , which is related to the activity coefficient of hydrogen ions. The procedure and calculations used to determine  $f_H$  and the apparent dissociation constants of bisulfate and hydrofluoric acid are described. The alterations of alkalinity, pH, and conductance of sea water when it is collected in metallic containers were described by Park (8N). These containers are usually made of brass, and they react with sea water according to the following reaction:  $2(\text{Cu,Zn}) + \text{O}_2 + 4\text{HCO}_3^- \rightarrow 2(\text{Cu,Zn})^{2+} + 4\text{CO}_3^{2-} + 2\text{H}_2\text{O}$ . A portion of the metal ions form complexes. As a result of these reactions, the pH and alkalinity of the sea water increase, and the conductance decreases.

Kunkler (6N) has devised a field kit for determining alkalinity. The temperatures of the standard buffer solutions and the samples are maintained within 2° of the temperature of the sampling source. Field determinations of standard solutions indicate that the results deviate less than 1% from the correct values.

A high-pressure pH sensor was developed by Ben-Yaakov and Kaplan (2N) for oceanographic applications. The sensor, composed of a specially-designed glass membrane and reference electrodes, performed well at a pressure of 150 kg per cm<sup>2</sup> in the laboratory, and at a depth of 270 meters in the ocean. A reproducibility of 0.02 pH unit was obtained when calibrated with sea-water buffer.

The methods of determination of bicarbonate and hydroxide ions in natural water by the use of nomograms, and their dependence on temperature, pH, and salt concentrations, was described by Apel'tsin and Sorkin (1N).

Polozhentsev and Vinnichenko (10N) developed a rapid method for determining ammonia and sodium alkalinity in boiler water and ammonium-sodium-cationized water which is based on consecutive titrations to determine sodium and ammonia alkalinity in the same sample. After estimation of the total alkalinity by the normal way, a 10% solution of *s*-trioxane or 40% formaldehyde is added and the hydrochloric acid is titrated with 0.1N sodium hydroxide.

Pittwell (9N) determined traces of carbonate ( $10^{-4}$  to  $10^{-6}M$ ) in natural waters by a turbidimetric method after the sample is mixed with an equal volume of barium chloride solution. Trace quantities of sulfate are determined after acidification of the above sample, and the carbonate reading is then corrected. Bicarbonate does not interfere up to 1 g per liter. A method involving the extraction of dissolved carbonate species from natural water for carbon-isotope analysis has been developed by Gleason, Friedman, and Hanshaw (4N). A concentrated solution of strontium chloride in ammonium hydroxide is added directly to the water sample. The strontium carbonate precipitate is separated, dried, and analyzed for its  $\delta^{13}\text{C}$ , where  $\delta^{13}\text{C} = [({}^{13}\text{C}/{}^{12}\text{C} \text{ sample}) / ({}^{13}\text{C}/{}^{12}\text{C} \text{ standard}) - 1]1000$ . Both the water sample and the prepared solution must not be exposed to air during handling.

A method was described by Johnson and Michalski (5N) for the determination of low concentrations of inorganic carbon in lake water. The sample is acidified to pH 3.0, and the inorganic carbon is absorbed quantitatively in base which was contained in a rubber balloon suspended within the sample bottle. Inorganic carbon in the absorbent is analyzed by back-titration with acid and with a carbon analyzer; results of the two methods varied by approximately 6.5%.

#### OXYGEN DEMAND

A new automatic instrumental method for measuring total oxygen demand (T.O.D.) was described by Goldstein *et al.* (7P). The T.O.D. method is based upon the quantitative measurement of the amount of oxygen used to combust the impurities in a water sample, and it is not subject to the interferences and special techniques required of the C.O.D. and B.O.D. methods. The applicability of the T.O.D. method and instrument is demonstrated by data from three

laboratory installations and six process-waste-stream applications. The close correlation of the T.O.D. measurements with the standard C.O.D. measurement is also shown. Clifford (5P) used an automatic instrument to directly and quantitatively measure the T.O.D. The combustion chamber is platinum catalyzed and maintained at 900°. An automatic sampling and injection system is incorporated into the device.

Stenger and Van Hall (12P) compared the C.O.D. results obtained from an instrumental, vapor-phase redox method which they developed in 1967 with results obtained by a conventional method. Several different waste-water sources were used, and C.O.D. results were generally higher with the authors' method. Reasons for the higher results and the advantages of the new method are discussed.

Leithe (9P) introduced a new method for C.O.D. analysis in waste water, replacing potassium dichromate with potassium persulfate. With the exception of the amino-nitrogen being converted to nitrates, most of the organic matter is oxidized to carbon dioxide and water. Silver nitrate is used to mask chloride and to serve as a catalyst for persulfate decomposition. Results average close to 100% theoretical for a wide variety of organic compounds. A modified Jeris rapid C.O.D. test was evaluated by Wells (14P). The digestion time, time of standing after digestion, and temperature of digestion were varied to determine the effect that these have on results.

An automated system for the determination of C.O.D. was described by Hey, Green, and Harkness (8P). Measured volumes of sample and reagent are mixed, passed through a heated glass coil, and cooled. The sample is then passed through a colorimeter, and the change in absorbance is recorded on a stripchart recorder. The C.O.D. of the samples is computed by reference to peak heights given by known standards. An automatic potentiometric titration for low C.O.D. determinations was developed by Baughman, Butler, and Sanders (1P). The standard procedure is modified by using 0.03N potassium dichromate, 0.025N ferrous ammonium sulfate, and an automatic titration. The precision is  $\pm 5\%$  at C.O.D. values above 5 mg per liter but becomes erratic at lower values. It is imperative that all glassware be scrupulously clean.

The ultraviolet absorption of natural waters in the range of 210 to 300 nm was determined by Ogura (11P). The absorption values are closely related to the chemical oxygen demand of fresh water and are a good index for the pollution of sea water.

Bridie (3P) described an electrolytic respirometer which is applicable to rou-

tine 5-day B.O.D. determinations as well as to biodegradability studies. Results obtained from B.O.D. tests agree well with the values found in literature. The method is more reliable, versatile, and less laborious than the standard 5-day dilution method.

Ford (6P) described a total carbon analyzer containing an infrared spectrophotometer. A correlation was found between T.O.C., B.O.D., and C.O.D. results for waste waters. This type of correlation becomes more difficult for industrial waste waters. The degree of C.O.D./T.O.C. variability in an industrial waste effluent was used to indicate the fluctuation in the effluent composition. A new instrumental technique was discussed by Christie and Bergmann (4P) for the short-time measurement of biological and chemical oxygen demands in water pollution control.

Beck (2P) used an amperometric titration with phenylarsenite to determine the residual permanganate and permanganate demand of raw water. In the absence of chlorine, a sample of raw water is treated with permanganate, mixed, and allowed to stand. Samples are treated at 0.5-hour intervals, and the difference between the values obtained and the original concentration constitutes the permanganate demand for each time interval. A comparison between the total organic carbon content of sea water and potassium permanganate consumption was made by Szeikielda (13P). The results with the permanganate method were consistently lower, in particular for suspended matter.

A radiochemical procedure to study persulfate wet oxidation of organic matter in sea water was described by Williams (15P). Leithe (10P) mentioned that C.O.D. determinations as given by "Standard Methods" will oxidize nearly 95% of the carbon and oxygen which is present to carbon dioxide and water; simultaneously, the nitrogen compounds are converted to ammonium sulfate. In this manner, the carbon and nitrogen can be determined simultaneously.

#### OXYGEN AND OTHER GASES

Several review and comparison studies were reported for determining oxygen concentrations in water. Rotshtein and Shemyakin (39Q) compiled a review with seven references on the determination of oxygen in natural waters. Ivanitskaya (19Q) compared several colorimetric methods for determining small concentrations of oxygen in water. He concluded that the most precise in the 0- to 30-mg-per-kg range is the safranine method. For industrial control purposes at concentrations between 0 and 50 mg per kg, the safranine method is recommended, using a solution of

safranine in a mixture of ethanol and glycerol with a specific gravity greater than 1. The indigo carmine method can be used when the oxygen concentration is greater than 10 mg per kg. The methylene blue method with glucose as a reducing agent is not recommended. Colorimetric, volumetric, paramagnetic, and electrochemical methods for the determination of oxygen in water and sewage were discussed by Thiele (46Q), with special emphasis on the electrochemical method. New research on continuous recording of oxygen consumption was also reported.

Boutin *et al.* (3Q) compared an electrochemical method to the Winkler method for the determination of oxygen in sea water. Agreement between methods was good. A dissolved-oxygen comparison study between oxygen sensor and modified Winkler methods was made by Reynolds (35Q). Excellent agreement was obtained.

Thallium was employed by several investigators in dissolved oxygen determinations. Doronin, Kabanova, and Timofeev (9Q) used a thallium column to measure dissolved oxygen in the range of 10 to 200  $\mu\text{g}$  per liter at temperatures between 0° and 80°. The addition of 100  $\mu\text{g}$  of oxygen to a liter of water changes the conductance of water after passing through the column by  $5.5 \times 10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$  at 60° and  $6.5 \times 10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$  at 80°. A similar thallium-oxygen analyzer is discussed by Lueck (28Q). Included are measurement conditions and influencing factors. Advantages of oxygen monitoring using a thallium-oxygen meter, comparison of equipment, industrial installation, and operational experience are also discussed.

Rygaert *et al.* (40Q) developed a portable method for determining dissolved oxygen using a column containing  $^{204}\text{Tl}$  deposited on copper-wool. When water is passed through a column, dissolved oxygen reacts according to:  $4\text{Tl} + \text{O}_2 + 2\text{H}_2\text{O} \rightarrow 4\text{Tl}^+ + 4\text{OH}^-$ . Consequently, the oxygen concentration can be determined by measuring the  $^{204}\text{Tl}$  concentration with a  $\beta$ -detector. Only nitrate interferes. A radio-release instrument for dissolved oxygen analysis has been constructed by Gillespie and Richter (14Q). The dissolved oxygen in the sample reacts with  $^{204}\text{Tl}$  in the instrument and releases a stoichiometric quantity of  $\text{Tl}-^{204}\text{Tl}$  ion whose activity is measured on a beta counter. This instrument is portable, battery operated, and not significantly affected by most dissolved salts and some common oxidizing agents above pH 5. Oxygen concentrations above 0.2 ppm are easily measured, and the precision is  $\pm 5\%$  at 1 ppm.

Tolgyessy and Varga (47Q) investigated a dissolved oxygen method in which the oxygen dissolved in the sample

reacts with the surface layer of radioactive kryptonate of thallium and releases a stoichiometric amount of radioactive krypton.

A procedure for the determination of dissolved oxygen in the range of 0 to 50  $\mu\text{g}$  per liter has been devised by Broenkow and Cline (4Q). Oxygen concentrations are determined colorimetrically by measuring the absorption of ultraviolet light by the triiodide ion which is produced when Winkler reagents are added to the sample. Care must be taken to avoid air contamination. The absorbance is measured at 352 nm, using a spectrophotometer having either a tungsten or hydrogen lamp. A manganese(III) complex was used by Sastry, Hamm, and Pool (41Q) to spectrophotometrically determine dissolved oxygen in water. The samples were treated with manganese chloride and sodium hydroxide solutions. After the precipitate settles, solid *trans*-1,2-diaminocyclohexanetetraacetic acid is added and the pH is adjusted to a range of 2.0 to 2.3 with sulfuric acid. After filtering, the absorbance was measured at 500 nm. Calcium interferes with the determination.

Epeikina (11Q) determined the oxygen content in power-station waters by the safranine method. The safranine solution is standardized with picric acid. Alternatively, a stable solution corresponding to 0.02 mg per liter can be prepared from Alizarin Red C solution.

The measurement of dissolved oxygen in the presence of chlorine and ferrous ion interferences was discussed by Berka, Glassl, and Hofmann (2Q). The ferrous ion interference is eliminated by oxidation with excess permanganate. Interference due to chlorine is eliminated by treating the samples with manganese sulfate and potassium hydroxide solutions, and determining oxygen as in the presence of ferrous ion. The relative error was less than 5%. Mori, Ishihara, and Goto (29Q) studied the effect of metal ions on the Shibata-Miller method for the measurement of dissolved oxygen in water. When silver, mercurous, cupric, barium, and chromous ions are present, the amount of ferric sulfate solution used for the titration is larger than theoretical. The reverse is true when manganous, ferrous, and stannous ions are present. Therefore, the Shibata-Miller method cannot be used to determine dissolved oxygen when these ions are present in water.

Dissolved oxygen in aqueous systems can be determined by titrating with chromium(II) in the presence of iodide, as outlined by Woolfolk and Dinius (52Q). The stoichiometry for the titration is four moles chromium(II) per mole of dissolved oxygen. Corrections must be made if other oxidants, such as nitrates, are present. The deter-

mination of oxygen in fresh water using trivalent cerium salts was discussed by De Graff Bierbrauwer and Golterman (7Q). Cerous and manganous salts have similar oxygen-binding properties. Cerous nitrate or cerous chloride and sodium hydroxide are added to lake samples, and the resulting hydroxide precipitates are dissolved in sulfuric acid. The ceric absorbance can be measured at 320 nm, or the oxygen can be determined by titration with arsenous oxide. Ferroin indicator or a potentiometer is used to determine the end point. Dissolved organic substances interfere; but this interference can be prevented by preoxidation with ceric sulfate.

The methylene blue method was used by Devdariani and Shaml'tsel (8Q) to determine small concentrations of oxygen in water. The methylene blue reagent is reduced in alkaline media with glucose. The absorbance of the color produced by the reaction of the oxygen with the reduced methylene blue reagent is measured at 660 nm. The sensitivity is 0.5  $\mu\text{g}$  of oxygen per kg; and the error for 0 to 100  $\mu\text{g}$  of oxygen per kg is 5%.

Padrus and Maly (12Q) described a method for the determination of oxygen in water. The oxygen is fixed by adding manganese sulfate and potassium hydroxide. The precipitate is dissolved in acetic acid, and EDTA is added to form the manganese(III)-EDTA complex. This complex is titrated with standard ammonium ferrous sulfate. At the equivalence point, the violet color changes to yellow in the presence of a few drops of 1% 3,3'-dimethylnaphthidine.

Schmid and Mancy (42Q) described an electrochemical method for the determination of dissolved oxygen in the presence of hydrogen sulfide. Measurements include lake and sediment samples. The silver-lead electrode is covered by a multilayer membrane consisting of a potassium hydroxide layer on lens paper and a cadmium nitrate layer on lens paper separated by an oxygen-porous membrane (polyethylene, polypropylene, Teflon). Foeyn (13Q) described an instrument which measures the oxygen content of brackish waters electrometrically when the electrodes are connected with a recording microammeter. Konnik, Mordukhovich, and Kuz'min (24Q) described an oxygen analyzer containing a gold-wire grid cathode and a zinc plate anode placed in a 0.1N sodium acetate solution. The electroreduction of oxygen shows a single wave up to -1.2 volts in the cathodic polarization curve, indicating the reduction of oxygen to water. It is possible to determine oxygen concentrations from  $10^{-6}$  to 100 volume per cent.

Parekh and Oza (32Q) determined

dissolved oxygen in sea water by a derivative polarographic method. A calibration curve was made from observed peak heights of the polarogram, taking the amount of air-saturated, distilled water as 8 mg per liter. Similar recordings were made on sea-water samples, and the amount of oxygen was read from the calibration curve.

Kuz'min and Smirnov (26Q) developed an electrochemical method for continuous measurement of dissolved oxygen in waste water. The construction of the automatic analyzer with its gas-permeable selective membrane is described. A gold-zinc electrode pair is submersed in alkaline electrolyte and hermetically encapsulated with a gas-permeable membrane. Rossi (38Q) used a galvanic cell-type electrode for the determination of dissolved oxygen in river and sewer water. Operational parameters and applications are also included.

Khranov, Pavlenko, and Nechvalenko (22Q) described the design of diffusion sensors and their supporting electronics for in situ measurement of the oxygen concentration in sea water. Principles of sensor operation and factors affecting the sensitivity and accuracy of oxygen concentration determinations are also mentioned. The methods, applications, and electronics used in continuous, in situ monitoring of dissolved oxygen in sea water are described and discussed by Khranov, Nechvalenko, and Berkutova (21Q). The sensors are useful in depths of 400 meters and measure dissolved oxygen concentrations of 0 to 12 ml per liter with an accuracy of  $\pm 0.1$  ml per liter.

An electrochemical device for measuring the oxygen content in water, waste water, and sludge was described by Kalman (20Q). A membrane is not required; and the readings are only slightly affected by salt concentrations and pH of the medium.

Lilley, Story, and Raible (27Q) investigated the chronoamperometric determination of dissolved oxygen using membrane electrodes. In the determination of dissolved oxygen in situ with Clark membrane electrodes, stirring can be eliminated and sensitivity improved by using a chronoamperometric method. This consists of applying 0.7 volt to the electrode, measuring current after 3 seconds, and reequilibrating for approximately 5 minutes.

Ropars (37Q) used gas chromatography to determine traces of oxygen in steam-boiler feed water. The water sample is ejected from a capillary where the jet entrains helium. The helium-oxygen mixture escapes from the liquid, passes through a de-entrainment column, and then flows to the chromatograph. The precision is better than  $\pm 4\%$  at 1  $\mu\text{g}$  per liter.

Smirnov, Kuz'min, and Saifi (44Q)

discussed the polarographic determination of dissolved oxygen in waste waters. The probe of the polarographic apparatus consists of two solid electrodes (gold and zinc), protected by the gas-permeable membranes. The electrolyte between electrodes is sodium acetate solution with agar-agar. Radchenko and Davidyuk (34Q) isolated dissolved oxygen in water by the guanidine method for precision mass-spectral analysis.

Reusmann (36Q) determined dissolved gases in sea water by gas chromatography. The flow scheme of the system is included. Helium acts both as a purging and a carrier gas. The carrier gas containing nitrogen, oxygen, argon, and carbon dioxide is dried over magnesium perchlorate and analyzed in a fractometer. The sequence of the fractometer is as follows: (1) a 1-meter column filled with silica gel; (2) a thermistor device; (3) a tube filled with soda lime for absorption of carbon dioxide; (4) a 1.5-meter column filled with molecular sieve 5A; (5) a thermistor detector; (6) a soap-bubble flow meter. The first detector indicates carbon dioxide and air; the second, nitrogen, oxygen, and argon. Gas chromatography was used by Hosokawa and Oshima (18Q) to determine carbon dioxide, oxygen, nitrogen, argon, and methane in natural waters.

A system for the shipboard determination of dissolved oxygen, nitrogen, and argon in natural waters has been designed by Williams (50Q). Included is an in situ sampler, a gas chromatograph with thermistor detectors, and three modes of calibration. Estimations of the composition of argon, oxygen, and nitrogen in dry air and in air-saturated water are reported.

Krajca (25Q) determined dissolved gases in the ground water from deep drill holes with low influx and large depression. A device for the sampling of gas-saturated waters from strata with low influx was developed where the composition of the gases is maintained. Such samples are indicative of possible oil and gas deposits, and permit the estimation of reserves and planning of production of springs.

Traces of hydrogen, nitrogen, and oxygen in aqueous solutions were determined by Tolk *et al.* (48Q). Helium strips the gases from water samples, and the gases are absorbed on a molecular sieve at liquid-nitrogen temperature. At room temperature, the gases are desorbed from the molecular sieve and determined by gas chromatography. Carbon monoxide, methane, and argon can be determined simultaneously.

Walker and France (49Q) described an apparatus for the continuous removal of dissolved gases (hydrogen, methane, carbon monoxide, and carbon dioxide) in water by mixing pure helium and water on a continuous-flow basis. The

stripper unit was tested by using a helium ionization chromatograph. The relative standard deviation ranged from 4 to 10%, depending on the rates of flow of the helium and water streams.

A method for onstream determination of free chlorine in water was described by Baker (1Q). Features of amperometric and potentiometric analyzers are combined in this instrument. The analyzer is specific, exhibits a sensitivity in the ppm range, and can be applied to samples ranging from fresh water and sea water to highly-contaminated brackish waters. Dozanska, Kelus, and Sikorowska (10Q) investigated the determination of chlorine in water using *p*-aminodimethylaniline oxalate. This colorimetric method makes it possible to determine free, total, and bound chlorine in the range of 0.02 to 2.0 mg of chlorine per liter. The determination can be made in the temperature range 4° to 35°. This method compares favorably with the *o*-tolidine-arsenate method in terms of selectivity, accuracy, speed, and temperature independence.

The amperometric determination of free chlorine and hypochlorite, using mercurous nitrate, has been proposed by Tarayan, Acharyan, and Darbinyan (45Q). An external potential of +0.6 volt is applied to the electrode couple (platinum and molar mercurous iodide) for the selective determination of chlorine and hypochlorous acid in the presence of chloric acid. Potassium bromide, mercuric nitrate, and sulfuric acid are added to the sample before it is titrated with mercurous nitrate. This method is suitable for analysis of natural and waste water. The highest relative error in the range of 0.009 to 3.4 mg chlorine was 1.25%.

Herculano de Carvalho (15Q) studied the determination of total carbon dioxide in natural water by precipitation with barium chloride and aqueous ammonia followed by fusion of the precipitate with borax. The method is precise if fusion is carried out at 800° to 850° for 30 minutes. This method is simpler than the one currently used in which hydrochloric acid is added to the precipitate, and the evolved carbon dioxide is collected in a suitable absorber which is then reweighed. An extraction method for the quantitative determination of total carbon dioxide in sea water has been developed by Wong (51Q). The new method, which employs a vacuum extraction and infrared analysis, has a precision of  $\pm 0.15\%$  standard deviation, and an accuracy of approximately  $\pm 0.2\%$ . A comparison of results showed that ocean water sterilized with 100 mg per liter of mercuric chloride does not change its total carbon dioxide on storage in stoppered borosilicate glass bottles at room temperature for a period up to 3 months.

Seidler (43Q) conducted a comparison study of three methods for the determination of total free carbon dioxide in water. The method of Kegel, a pH-difference technique, was compared with the sodium hydroxide titration method and the method involving calculation of the dissociation equilibrium of the carbon dioxide by a series of parallel precision tests. The Kegel method was shown to be reliable and especially suited for hard and medium-hard waters. For soft waters that are uncontaminated by heavy-metal salts, the sodium hydroxide method is more suitable. The applicability, concentration range, time, interferences, and accuracy of each method is compared by tabulation.

Myhrstad and Samdal (30Q) discussed the behavior and determination of chlorine dioxide, and outlined a series of methods for determining chlorine dioxide, hypochlorite, and chlorine. These methods are based on direct spectrophotometric determination of chlorine dioxide with Acid Chrome Violet K; iodometric-potentiometric titration at pH 7 to give chlorine + 0.80 hypochlorite, and an iodometric-potentiometric titration at pH 7 after acidifying to pH 2.5-3 to give chlorine + chlorine dioxide + hypochlorite. Chlorine dioxide and chlorite in water can be determined by a modified method using diethyl-*p*-phenylenediamine as described by Palin (31Q). The end point can be determined either titrimetrically or colorimetrically. Malonic acid is used to suppress free chlorine.

A photometric determination of low concentrations (0.01 mg ozone per liter) of ozone in water was described by Hofmann and Stern (17Q). Ozone reacts with the manganese(II) diphosphate complex to form the manganese (III) diphosphate complex which is then treated with *o*-tolidine. Ozone can be determined if it is swept out of the sample by a stream of gas and absorbed in the manganese(II) diphosphate solution in the presence of chromium(III) as a catalyst. At 0.52 mg of ozone per liter, the standard deviation is 3.1%. Hofmann and Stern (16Q) also investigated a photometric determination of ozone in water when the concentration is greater than 1 mg of ozone per liter. This determination is based on the fact that ozone reacts with manganese(II) diphosphate to form a violet-colored complex. The standard deviation was 2.42% at 4.3 mg per liter. Chlorine, chlorine dioxide, hypochlorite, chlorite, chlorate, and perchlorate do not interfere.

Kokoshkin (23Q) reported an improved method for determining hydrogen in a condensate. Experiments with the A. A. Avdeev-type chromatographic apparatus show that the hydrogen results in the range of 1 to 10  $\mu\text{g}$  per liter may be approximately 50% low

because of surface absorption. This phenomenon is fully compensated in continuous analyzers. The method is based on utilization of a continuous-contact device, where air is the gaseous carrier. Standardized activated charcoal K-7 is used as an absorbent. The analysis is made within 90 seconds with high accuracy.

Craig and Weiss (6Q) compared results obtained for argon concentrations in the ocean by two different methods. The use of the isotope dilution method in the mass spectrometric analysis, and the gas chromatographic method have produced excellent agreement, especially below 1000 meters in depth.

Quentin and Schretzenmayr (33Q) described a radon determination in mineral and curative waters using a static electrometer. They stated that the method can be improved by using an oscillating condensing electrometer. A method was devised by Chung, Yu, and Chung (5Q) to determine the concentration of radon in underground water. The radioactivity was measured with a liquid scintillation counter after extraction of radon by ethyl ether from water samples.

#### DETERGENTS

Seventy-four references are included by La Noce (6R) in his review on the principal analytical methods for the determination of anionic and nonionic surfactants in water. Official and other published methods of general applicability are listed.

Atomic absorption spectrophotometry was used by Le Bihan and Courtot-Coupez (7R) and Courtot-Coupez and Le Bihan (2R) to determine traces of anionic and cationic detergents in sea water and soft water. Anionic detergents (10 to 500  $\mu\text{g}$  per liter) were determined after complexing with *o*-phenanthroline-copper sulfate and extraction with either isobutyl acetate or MIBK, copper being determined in the solvent by atomic absorption spectrophotometry. The precision was 5% at greater than 50  $\mu\text{g}$  per liter and less than 15% at lower concentrations. Ferric, perchlorate, thiocyanate, and nitrate ions caused a small, constant interference which was compensated for by using a blank. Hydrogen sulfide should be removed by oxidation. Cationic detergents were determined by complexing with known amounts of anionic detergent and determining the residual anionic detergent by difference. The precision of this method was 5 to 20% at greater than 20  $\mu\text{g}$  per liter. Stroehl (10R, 11R) described a method for separate determination of anionic and cationic detergents in waste waters and effluents. If both anionic and cationic surfactants are present, the latter is separated by ion exchange on Zeo-

Karb 225 at a pH of 6.5 to 7.0. A determination before and after separation gives both anionic and cationic surfactant content as one surfactant cation corresponds to one anion.

The use of Azure A instead of methylene blue for the determination of anionic detergents in drinking and surface waters was discussed by Den Tonkelaar and Bergshoeff (4R). They concluded that the Azure A method is more rapid, more sensitive, and more selective for detergents in drinking and surface waters. A detailed procedure is included.

The development of a rapid analytical method for the determination of alkylbenzenesulfonate (A.B.S.) in surface waters and sewage, in the presence of biological interferences, was reported by Dessouky, Abu-Shady, and Miller (5R). A basic dye salt is reacted with A.B.S., extracted with suitable organic solvents, and measured colorimetrically. Several dye-solvent systems show promise.

A methylene blue field method for the determination of anion-active surface-active agents in water is described by Mozhaev, Lin'kov, and Kapyrina (8R). The color of the chloroform layer is compared to that of similarly prepared standards.

Belen'kii *et al.* (1R) reported on the determination of OP-7 and OP-10 (nonionic surface-active agents) in colored waste waters. In order to perform a colorimetric determination, usually with cobalt thiocyanate, it is necessary to remove the colored substances from waste water with activated charcoal. However, the nonionic surface-active agents are also removed by the charcoal. A mixture of benzene, chloroform, petroleum ether, and ethanol are used to elute these nonionic agents from the charcoal.

An automatic apparatus was described by Sawyer, Stockwell, and Tucker (9R) for the solvent extraction of traces of nonionic detergents in river and waste water. The extraction of nonionic detergent into chloroform was 93 to 98% effective.

A procedure for the determination of nonionic surfactants in water was reported by Courtot-Coupez and Le Bihan (3R). The procedure relies on the extraction of a cobaltthiocyanate adduct from an aqueous sample into a benzene phase. The amount of cobalt in the extract is determined by atomic absorption spectrophotometry and correlated linearly with the corresponding surfactant.

#### PESTICIDES AND HERBICIDES

The use of OV-17 as a stationary phase for the systematic analysis of herbicides, organochlorine and organophosphate pesticides, and related compounds was investigated by Leoni and Puccetti

(11S). At 198°, separation with OV-17 was comparable or superior to that obtained with the mixed stationary phase, QF-1 + DC-200. Separation of dieldrin/*p,p'*-DDE was not satisfactory at 198°, but was at 175°.

Pertinent references on the determination of organochlorine insecticides in soil and water samples are reviewed by Pionke and Chesters (14S). Extraction of organochlorine insecticides from soil and waters, cleanup of soil and water extracts, and analysis of purified soil and water extracts are the three principal sections of this review. In the section on cleanup, special attention is devoted to chromatographic procedures.

Dyatlovitskaya and Gladenko (4S) discussed the determination of organochlorine insecticides in water by thin-layer chromatography. DDT, hexachloran, aldrin, and dieldrin are extracted from water with benzene and separated on an aluminum oxide chromatographic plate by using *n*-hexane as a developer. The spots are developed by spraying with an ammoniacal solution of silver nitrate in acetone followed by exposure to a mercury lamp.

The determination of pesticide residues in natural water is discussed by Mestres *et al.* (18S). The residues are extracted, and the extract is purified by chromatography. An electron detector is used to assume the absence or presence of certain pesticide residues. Organophosphorus pesticides are detected enzymically and confirmed by gas chromatography. Taylor and Bogacka (16S) colorimetrically determined DDT, methoxychlor, and lindane after extraction and separation by thin-layer chromatography. Gapotchenko and Nezdoimishapka (7S) outlined a procedure for the determination of DDT and hexachlorocyclohexane in water by radial partition paper chromatography. Koppe and Rautenberg (10S) reported a simple, thin-layer chromatographic method for the determination of less volatile lipophilic chlorine compounds in water. Both readily and difficultly decomposed compounds are detected in the range of 1 to 1000 µg of chlorine per liter.

The determination of pentachlorophenol, sodium pentachlorophenate, and lauryl pentachlorophenate in river, industrial, and sewage water, as pentachlorophenol, was described by Colas (2S). It was possible to determine 0.5 ppm of pentachlorophenol with precision. No interferences were found.

Croll (8S) concluded that pyrethrins can be quantitatively recovered from water by extraction with light petroleum and that electron-capture gas chromatography is capable of accurately estimating pyrethrins in water down to a level of 0.00026 mg per liter. It is possible to estimate the total pyrethrins by measurement of the com-

ponent which gives the greatest response to the detector.

Konrad, Pioke, and Chesters (9S) discussed the extraction and determination of organochlorine and organophosphate insecticides from lake water. The concentrated benzene extract is analyzed directly by gas-liquid chromatography with an electron-capture detector for organochlorine insecticides and with a potassium chloride thermionic detector for organophosphates.

Vylegzhanina and Kalmykova (17S) used thin-layer chromatography for the determination of O,O-dimethyl 2,2-dichlorovinyl phosphate in plants, soils, and water. A detailed description of the procedure is included. DDT and chlorophosphate do not interfere.

Askew, Ruzicka, and Wheals (1S) described the determination of organophosphorus pesticide residues in river waters and effluents by gas, thin-layer, and gel chromatography. A general comprehensive scheme for chloroform extraction of organophosphorus pesticides from river water and sewage effluent for chromatographic analysis is included. Gas, thin-layer, and gel chromatographic data are presented for 40 organophosphorus pesticides.

A gas chromatographic method for the determination of low concentrations of Abate in water was described by Shafik (15S). The product resulting from the hydrolysis of Abate residues to 4,4'-thiodiphenol followed by silylation with hexamethyldisilazane is determined by gas chromatography. The average recovery was 88%. Howe and Petty (8S) reported a thin-layer chromatographic procedure for quantitatively measuring Abate concentrations in surface waters. Average recovery was approximately 75%.

El-Dib (5S) discussed the use of thin-layer chromatography to detect, separate, and identify *N*-phenyl, *N*-methylcarbamates, and related ureas in natural waters. Residues of chlorinated or organophosphorus pesticides do not interfere with the procedure.

The sampling and storage of water samples containing chlorinated hydrocarbons was investigated by Weil and Quentin (18S). Solutions containing 10 µg per liter of the following insecticides were stored in glass, aluminum, and polyethylene containers: DDT, dieldrin, heptachlor epoxide, lindane, and methoxychlor. With the exception of lindane in glass, all samples showed a decrease in insecticide content after 1 or more days. Aluminum showed somewhat higher absorption losses than glass, and polyethylene the greatest. The authors recommended that samples be taken in glass bottles with ground-glass stoppers, that the total sample be extracted with petroleum ether, and that the container be washed with petroleum ether and combined with the extract



from the sample. They recommended that pesticide determinations be made as soon as possible after sampling.

McKone and Hance (12S) described a method for estimating diuron in surface water by electron-capture gas chromatography. Overall mean recovery was 94%. An electron-capture gas chromatographic procedure for the extraction and determination of dalapon in water was developed by Frank and Demint (6S). Recoveries averaged 96%, and the minimum level of detection was 0.1 ng.

## ORGANICS

Determinations of organic materials in water are listed for five groups: acids, organic carbon, phenols, petroleum products, and miscellaneous.

To determine organic acids in ground water, Bryzgalov and Semenov (17T) passed the sample through a column with KU-2 cation exchanger and AN-22 and AV-17 anion exchangers. The organic acids were eluted from the AV-17 resin with ammonium carbonate. The solution was made alkaline with sodium hydroxide and evaporated to dryness. The precipitate was dissolved in hydrochloric acid, and, after removing carbon dioxide, the organic acids were extracted with ethyl ether. The extract was titrated in ether-butanol medium with an alcoholic solution of sodium hydroxide.

Nichikova *et al.* (72T), and Declercq, Blaton, and Peeters (24T) described gas chromatographic methods to determine low-molecular-weight aliphatic acids in water. Details of the procedures are given. Harwood and Huyser (40T) proposed an automated method for the determination of the total volatile fatty-acid content of aqueous samples.

Khomenko, Goncharova, and Stradomskaya (53T) developed a thin-layer chromatographic method to determine a number of nonvolatile organic acids in natural water. Included are formic, fumaric, glutaric, adipic, pyruvic, lactic, succinic, oxalic, aconitic, malonic, gallic, citric, malic, and tartaric acids.

A method for separate determination of organic acids and amines in ground water, based on the use of partition column and gas-liquid chromatography, was developed by Semenov *et al.* (86T).

Wershaw and Burcar (106T) described procedures for the isolation of humic and fulvic acids from natural waters and methods for characterization of the humic acids with spectrophotometry, polarography, chromatography, N.M.R., E.P.R., small-angle X-ray scattering, electron microscopy, and others. Van Beneden and Van Beneden (104T) reviewed the origin and characteristics of the humic acid compounds, and examined the different analytical

methods used for the determination of humic acids in water. In addition, they proposed a new spectrophotometric technique which is described in detail. Shapiro (89T) concentrated humic acids in natural waters by freeze concentration. The acids were concentrated by agitation of the sample at a controlled rate of freezing. A 45-gallon volume of sample was reduced to 5 to 10 gallons in 48 hours. Further concentration is also possible.

Bykova (20T) used a capillary-luminescence method in a study of naphthenic acids in water. This method provides for separation of individual compounds or groups of organic substances with subsequent quantitative evaluation.

Goncharova and Khomenko (38T) described a spectrophotometric method for determining benzoic acid in water. Benzoic acid is first extracted with diethyl ether and then reextracted with sodium hydroxide. The pH is adjusted to between 3 and 4 with hydrochloric acid, and the absorbance of the sodium benzoate solution measured at 230 nm.

A detailed spectrophotometric method for the determination of malic acid in natural waters is described by Smotrakov, Stradomskaya, and Goncharova (94T). Malic acid is concentrated by a series of evaporations, oxidized with permanganate, and finally reacted with 2,4-dinitrophenylhydrazine. The absorbance of the solution is measured at 554 nm. The sensitivity is 50  $\mu$ g per liter and the average yield is 50%.

The determination of lactic acid in water, which was reported by Stradomskaya and Goncharova (97T), is based on the transformation of lactic acid in the presence of sulfuric acid to acetic acid, followed by the color reaction of acetic acid with  $\alpha$ -naphthol. Samples are first concentrated by evaporation, and the lactic acid is then extracted with butanol and reextracted with sodium hydroxide. Beer's law is followed up to 100  $\mu$ g of lactic acid. The minimum detectable amount of lactic acid is 5 micrograms.

A method of concentrating 0.001 to 0.1 mg of glycolates per liter in natural water by a 2-stage ion-exchange procedure, and their estimation by the modified Calkins colorimetric method, is described by Fogg, Eagle, and Kinson (32T). A 2-liter sample of water is used. The method is laborious, but it has an accuracy of  $\pm 0.0003$  mg per liter.

Shaova, Shelepin, and Kaplin (88T) determined the total monocarboxylic acid content of water polarographically. The analyses of the solutions were carried out in an electrolyzer equipped with a device for blowing a stream of hydrogen through the solution. A dropping-mercury electrode was used as the cathode, and the anode consisted of a pool of

mercury. Sensitivity of the method for the sum of acetic, propionic, and butyric acids was 0.01 mg per liter. Accuracy in the concentration range of  $10^{-4}$  to  $10^{-6}$  mole per liter was 10%.

Kriul'kov, Ganin, and Kaplin (59T) described an ultraviolet spectrophotometric method for determining water-soluble sulfuric acid lignin in polluted natural waters. Phenols interfere, but are removed by extraction with diethyl ether. The absorbance of the aqueous phase is measured at 275 to 280 nm, using a sample of unpolluted natural water as a blank.

Stroehl (98T) reported phenol losses of approximately 10 and 20% for normal distillation and steam distillation methods, respectively, when determining volatile phenols in the range of 0.01 to 0.5 ppm in surface and waste waters. The yield was increased to 98% for the normal distillation of 250  $\mu$ g phenol in 500 ml of water by adding 50 ml water to the sample after distillation of approximately 450 ml, so that a total volume of more than 500 ml was distilled. After distillation, the phenols were determined colorimetrically with 4-aminoantipyrine. Zannoni (110T) determined phenols in water by reaction with 4-aminoantipyrine and  $K_4Fe(CN)_6$  in an alkaline medium. The complex was extracted with chloroform, and the absorption measured at 460 nm.

Friestad, Ott, and Gunther (35T) used the coupling reaction of phenols with 3-methyl-2-benzothiazolinone hydrazone to determine phenols in water by an automated method. The system operates at a rate of 20 samples per hour, either alone or when combined with automated steam distillation of the phenols prior to the colorimetric reaction.

Melpolder *et al.* (69T) reported the development of an ultraviolet spectrophotometric method for determining ethoxylated alkylphenols in river water and sewage. The method utilizes physical separation and purification procedures to isolate the phenol fraction.

Smith and Lichtenberg (92T) described a thin-layer chromatographic method for the determination of phenols in surface water. Concentrated extracts from water grab and carbon absorption samples were chromatographed on silica gel G thin layers and developed in benzene-cyclohexane- $Et_2NH$  (5:4:1) or chloroform. Specific phenols were identified by their  $R_f$  value and the color of the spots produced with *p*-nitrobenzenediazonium tetrafluoroborate and Gibbs' reagent. For several phenols, a sensitivity of less than 1  $\mu$ g per liter was obtained. A thin-layer chromatography method was also described by Aly (2T). Phenols were separated as their antipyril or *p*-nitrophenylazo dyes and chromatographed on basic gel plates. The procedure was applied to



the separation and identification of chlorination products of phenols in water.

The techniques used for the determination of total phenol content of the River Test and its tributaries after a heavy snow and subsequent thaw are described, and the results are evaluated by Wallwork, Bentley, and Symonds (105T). A detailed description is given of the 4-aminophenazone method of analysis, the working up of carbon filters, and the working details of gas-liquid and thin-layer chromatography procedures.

Semenchenko and Kaplin (83T) carried out a gas-liquid chromatographic separation of monohydric phenols in aqueous solutions after methylation of the phenolic compounds in an alkaline medium with dimethyl sulfate. Bidlo (13T) described a combination paper-gas chromatographic procedure for the determination of phenols in water.

The automated layout designed to determine phenol in water in the presence of chloride by the C.O.D. method is given by Hertzog (43T). A fresh dextrose solution is used as a standard for comparison.

Berka and Zaveksy (12T) reported that the oxidizability of phenolic waters can be determined by refluxing the sample for 15 minutes with an excess of potassium permanganate in the presence of sodium carbonate, followed by oxidation in an acidic medium for 15 minutes at room temperature.

A method for the bromometric determination of phenol in water was proposed by Selezneva and Kogan (81T).

A brief description was given by Szekeida (99T) of the methods used in oceanography and limnology for the measurement of particulate and dissolved carbon in aqueous solutions. The review covers the literature for the past 12 years and includes titrimetric, coulometric, potentiometric, infrared, gas chromatographic, and conductometric methods. Hill (44T) reviewed the development of instruments for the determination of total organic carbon in water and effluent samples. Recently developed chemical oxygen demand, Rocketdyne organic, and total oxygen demand analyzers are described. The use of carbon analyzers for many types of water is considered, and techniques are compared. Gordon (39T) examined analytical procedures to determine accurate confidence limits for individual determination of particulate organic carbon analyses. Freimuth *et al.* (34T) reviewed the literature and tested several methods for determining organically bound carbon in water and sediment. None of the methods tested could be generally applied to all samples.

A dual-channel carbonaceous analyzer for measuring the total carbon in polluted water is described by Jones and

Dageforde (50T, 51T). The instrument measures the total carbon from both inorganic and organic sources by estimating the combustion and decomposition of the compounds in a catalyst-packed column at high temperature. Organic carbon is determined by difference. Sensitivity is less than 5 ppm carbon. Cropper and Heinekey (23T) adapted a commercially available flame-ionization detector for the determination of total carbon in industrial wastes, sewage effluent, and river water. As little as 1 ppm could be determined by direct injection of the sample in the apparatus.

To determine total carbon in water, Busch (19T) used an infrared analyzer. The sample is vaporized to form steam and the carbonaceous material oxidized to carbon dioxide, which is then measured. The carbonates and dissolved carbon dioxide are first removed by precipitation with a barium salt. A mass culture procedure is also described, by which the degree of biodegradability of carbonaceous material can be determined.

Bartz (10T) reported that a carbonaceous analyzer normally measures 0 to 100 ppm full-scale total organic carbon in water. He then described a technique to modify the instrument to improve its sensitivity to 0 to 1 ppm full scale. Details of the modifications and equipment used are given. Bakulina and Skopintsev (9T) gave details of the equipment used for the determination of trace amounts of carbon in natural water without preliminary evaporation. A sensitivity of 10 to 20 micrograms was achieved with an accuracy of  $\pm 3\%$ .

To determine between 10 and 900  $\mu\text{g}$  of particulate carbon in sea water, Szekeida (100T) passed a one-half- to 2-liter sample through a filter of glass-fiber paper. The filter is dried; it is then burned in oxygen at  $700^\circ$  in a combustion tube. The combustion products are passed through absorbents to remove interfering gases and the carbon dioxide is absorbed in 0.01N sodium hydroxide. The electrical conductance is then measured. The coefficient of variation is 4% at the level of 350  $\mu\text{g}$  of carbon per liter.

A technique for determining organic carbon directly in various mineralized waters is proposed by Kogan and Mikheeva (54T). It is based on the burning of organic carbon substances with boiling sulfuric acid in the presence of chromic acid and chromyl chloride. For amounts of carbon between 120 and 1200  $\mu\text{g}$ , in the presence of 0 to 900 mg chloride and without any pretreatment, the error was less than 10%. Samples high in carbonate must first be boiled with sulfuric acid to remove carbon dioxide.

Ohmori, Hirayama, and Iwasaki (73T), Iwasaki *et al.* (47T, 48T), and

Togashi and Hirayama (102T) determined organic carbon in water by a potassium peroxydisulfate oxidation method. Organic substances are decomposed with potassium persulfate and silver nitrate solutions in the presence of sulfuric acid at  $200^\circ$  to  $250^\circ$  for 60 minutes. The carbon dioxide produced is bubbled into potassium hydroxide, precipitated as barium carbonate, and determined titrimetrically. Perel'shtein and Kaplin (75T) also described a persulfate decomposition method to determine total carbon in natural water. In addition, they described a method to determine the oxidizability of the organic compounds.

Lyutsarev (65T) described an automated device for the coulometric estimation of organic carbon in natural water. Inorganic carbonates are first destroyed, and then the organic substances are oxidized with a mixture of silver and potassium dichromate in the presence of sulfuric acid at  $130^\circ$ . The carbon dioxide is carried over to a coulometric cell and is measured. The accuracy for a sea-water sample with a concentration of 2 mg per liter was  $\pm 5\%$ . The method does not require initial evaporation of the sample.

Comparative data for the amount of dissolved organic carbon in sea water by wet and ultraviolet oxidation methods were reported by Williams (107T). The difference between the two methods was not great.

Enrhardt (31T) described an automated AutoAnalyzer method to determine dissolved organic carbon in sea water. The organic carbon compounds are oxidized by means of ultraviolet radiation with potassium persulfate as the source of oxygen. The carbon dioxide formed is absorbed in dilute sodium hydroxide solution and the electrical conductance change of the base is proportional to the amount of dissolved organic carbon.

Otsuki and Hanya (74T) studied the Katz method of determining organic carbon in polluted water with potassium persulfate in the Thumberg tube.

Goebgen and Brockmann (37T) reviewed a number of methods for the determination of hydrocarbons, both of gasoline and gasoil origin. A method is suggested which meets the sensitivity requirements for water-quality control. The volatile hydrocarbons are determined as carbon dioxide and the heavy oils by thin-layer chromatography. Maehler and Greenberg (66T) presented a sequential scheme for the identification of petroleum products in estuarine waters which included gas chromatographic analysis of vapor space gas and solvent extraction followed by infrared spectrophotometric and paper chromatographic examinations for the nonvolatile products.

Hellmann (41T) studied the possi-

bilities and limits of infrared spectroscopy in determining mineral oils and fuels in surface water. Topics discussed were: sampling and sample-treatment, separation of nonpolar hydrocarbons from compounds of different polarity, measurement of infrared absorption spectra, determination of hydrocarbon content, and sources of error.

Imanuilov (46*T*) reviewed the use of modern spectrophotometric techniques for determining petroleum products in water. Infrared is very useful, especially if combined with chromatographic techniques to clarify the component of a contaminant. Ultraviolet, fluorescent, and luminescent methods have less accuracy.

Koppe and Muhle (56*T*) used a combination of oxidation and infrared analysis to determine the difficult oxidizable odoriferous oils in water.

An oleometer for the automatic determination of oil content in water was proposed by Yoshii, Katsuda, and Ueda (108*T*). The oil content of a sample of sea water was estimated, after emulsification, by determining the ratio of the intensities of the transmitted and reflected light.

Rychkova (78*T*) described a fluorometric method for determining petroleum products in water. A direct-reading instrument with incandescent and mercury lamp sources was used. Light filters with 50 to 70 nm band covered the 300 to 750 nm range. The sensitivity of the method was 0.5  $\mu\text{g}$  per liter, and the maximum error in the 0.5- to 10- $\mu\text{g}$ -per-liter range was 1  $\mu\text{g}$  per liter. Bauer and Driescher (11*T*) used a fluorescence analyzer for determining 1 ppm of oil in water. The fluorescence of ultrasonically prepared oil emulsions is determined at 365 nm.

Semenov, Beisova, and Obraztsova (85*T*) used a paper-chromatographic method for determining petroleum products in both natural and oilfield waters. The chromatographic paper is impregnated with a 1 to 5% solution of silicone oil in cyclohexane. A mixture of chloroform and petroleum ether is the moving phase. Values of  $R_f$  are given for the three chromatographic zones determined in analyzing samples containing crude oils, distillation residues, or oil fractions.

The qualitative and quantitative detection of mineral oil in water by organoleptic, pycnometric, thin-layer, and gas chromatography is reviewed by Lawrenz (61*T*). A method is also described for the detection of benzene in concentrations greater than 0.01 mg per liter. Jeltes (49*T*) determined mineral oil in water by the extraction of the oil from the water and subsequent gas-chromatographic analysis of the resulting extract.

Existing methods for the determination of diesel oil in water were critically

reviewed by Reissaus (76*T*), and a new method was developed. The method was based on hot extraction with *n*-hexane for three hours and on measurement of the absorbance of the organic phase at 220 nm. Techniques to remove interferences were provided.

The remainder of the methods under Organics pertains to determinations of miscellaneous organic compounds. Because they are numerous, only brief descriptions will be made. The reader should refer to the original reference or to the Chemical Abstract reference for more complete details.

To improve the limit of detection of benzene, toluene, ethylbenzene, diethylbenzene, and isopropylbenzene from 1 mg per liter to  $10^{-3}$  to  $10^{-4}$  mg per liter in water by chromatographic analysis, Krichmar and Stepanenko (58*T*) introduced an intermediate concentration step in which nitrogen gas was used to volatilize and entrain the hydrocarbons.

McAuliffe (67*T*) discussed a number of available techniques for separating dissolved hydrocarbons from aqueous solution and for analyzing them by gas chromatography. Koppe and Muhle (57*T*) used microcircular thin-layer chromatography for the detection of dissolved hydrocarbons and lipophilic substances in water.

Dudova and Diterikhs (27*T*) determined aromatic hydrocarbons in ground water by extraction with *n*-hexane followed by treating the extract with a nitration mixture and determining the nitro derivatives colorimetrically with a sensitivity of 0.2 mg per liter. Saltzman (80*T*) used ultraviolet absorption as a basis for continuous monitoring of aromatic compounds in water by differential photometry.

Borneff and Kunte (15*T*) determined several polycyclic compounds by thin-layer chromatography after extraction of the compounds from a 10-liter sample with benzene and evaporation of the solvent. Thielmann (101*T*) used thin-layer chromatography to identify a number of organic pollutants in filtered surface and ground waters.

For the determination of benzene in natural mineralized water, Kontsova (55*T*) used gas-liquid chromatography. Boldina (14*T*) determined propylbenzene and isopropylbenzene in reservoir water spectrophotometrically. The method is based on nitrating the compounds, converting the nitro compounds to color complexes, and measuring the absorption at 460 nm. The sensitivity limit is given as 0.1 mg per liter. To determine hexachloro-, nitrochloro-, dinitrochloro-, and dichlorobenzene in water titrimetrically, Nazarova and Eremenko (70*T*) first extracted these compounds with carbon tetrachloride, evaporated the solvent, and oxidized the compounds. The chloride was then

determined by titration with mercuric nitrate.

The formation of molybdenum blue during the reduction of ammonium molybdate by hydrazine was recommended by Rychkova (79*T*) as a colorimetric method for the determination of trace amounts of hydrazine. Beer's law is followed from 0.01 to 0.6 mg per liter. Aksel'rud and Berezyuk (17*T*) described a titrimetric method to determine hydrazine and ammonia when both are present in a solution.

Zaitsev and Nesterova (109*T*) determined cyclohexanone oxime and hydroxylamine together by polarography. Hydroxylamine is then destroyed with potassium ferricyanide in sodium hydroxide solution, and cyclohexanone oxime again determined polarographically. Hydroxylamine is determined by difference.

A method for the determination of 20 to 200  $\mu\text{g}$  of pyridine per liter in mineral waters was described by Spiridonova and Kogan (96*T*). The accuracy was 2 to 6%.

Dyatlovitskaya and Botvinova (28*T*) determined hexamethylenediamine in water from natural sources by chromatographic separation followed by spectrophotometric measurement. Ninhydrin is used to develop the color. Sensitivity is approximately 0.02 mg per liter.

Burkert (18*T*) determined flocculating agents, such as polyacrylamide and acrylamide-sodium acrylate copolymers, at concentrations of 0.005 to 0.1 ppm in drinking water by adding a powder solid, such as kaolin, and comparing the settling rate of the solid with that of the solid in water containing known amounts of the flocculating agent.

A simple method for the determination of urea in sea water is described by McCarthy (68*T*). It involves addition of urease and subsequent measurement of the liberated ammonia. Emmet (30*T*) described a spectrophotometric method for the determination of urea in natural waters. Urea reacts with hypochlorite and phenol to form a yellow compound which has a maximum absorption at 454 nm.

Corwin (22*T*) determined acetone, 2-butanone, and butyraldehyde in sea water by gas-liquid chromatography in a 10% Carbowax 20M/Chromosorb W (acid-washed) column at 95° with a hydrogen ionization detector and helium gas carrier.

To determine furfural in water, Kuznetsov (60*T*) used ultraviolet spectrophotometry. Beer's law is obeyed for 0.6 to 3 mg of furfural per ml.

Aoki, Okaichi, and Ochi (3*T*, 4*T*) determined as little as 0.1 mg of dichloromono-fluoromethane per liter (Freon 21) in fresh water spectrophotometrically with pyridine. The absorbance of the solution is measured at 366 nm.

To determine aliphatic alcohols in water, Sokolova and Kaplin (95T) concentrated the alcohols by distillation, converted them to alkyl nitrites, and measured the absorbances at 355.5 nm. Tests with addition of alcohols to river water have shown a 20% error for 0.3 to 0.5 mg per liter of mixed alcohols.

A spectrophotometric method for the determination of complex esters in ground water was suggested by Bryzgalo and Semenov (16T). The sensitivity was reported to be 1.5 to 2  $\mu$ g-equivalent per liter.

Dyatlovitskaya and Maktaz (29T) studied the determination of caprolactam in water by thin-layer chromatography. Caprolactam is hydrolyzed to caproic acid, and chromatographic patterns of the latter developed with ninhydrin. The compound is then extracted with methanol and measured photometrically.

Garside and Riley (36T) described a combined thin-layer chromatographic-reflection densitometric procedure for the determination of chlorophylls and carotenoid pigments in sea water. Chlorophylls a, b, and c, carotene, many of the individual xanthophylls, and certain degradation products of chlorophylls could be determined.

Dowgiallo (26T) described a procedure to clarify water samples prior to determining the carbohydrates. Samples were treated with cadmium sulfate and barium hydroxide, shaken, and finally filtered. The filtrates were then analyzed for sugars by paper chromatography after concentration, followed by desalting with ion-exchangers.

Sinel'nikov (91T) used a combination of extraction with chloroform and paper chromatography to determine bituminoids in water. The identification was done by using fluorescent light.

Holm-Hansen, Sutcliffe, and Sharp (45T) determined the deoxyribonucleic acid (DNA) content of sea water by measuring the fluorescence of the complex formed between DNA and diamino-benzoic acid dihydrochloride.

Umbreit, Nygren, and Testa (103T) determined C<sub>1</sub>-C<sub>5</sub> alkylamine hydrochlorides in water by gas chromatography.

Smith and Gouron (93T) devised a thin-layer gas-chromatographic method for the specific detection of the human fecal sterol 5 $\beta$ -cholestan-3 $\beta$ -ol in natural water.

A number of other investigators described instrumental techniques for determining a variety of organic substances.

Gas chromatographic techniques were studied by Lysyj *et al.* (64T); Lysyj, Nelson, and Newton (63T); Nelson and Lysyj (71T); Caruso and Koslow (21T); Dmitriev and Kitrosskii (25T); Baker and Malo (8T); Semenchenko

and Kaplin (82T); and Semenchenko, Kaplin, and Lur'e (84T).

The infrared absorption spectra of hydrocarbon fractions from the acid part of organic substances in natural waters was studied by Shaks (87T).

Formaro and Trasatti (33T) discussed the possibility of testing the organic content of water by plotting cathodic charging curves and by capacity measurement.

A review is given by Bagdasarov *et al.* (5T) describing the basic principles, instrumentation, and advantages and disadvantages of the photocolormetric method of determining a number of organic substances in pure water. Lur'e (62T) discussed the use of chromatography, polarography, and infrared and ultraviolet spectrophotometry methods for the identification and determination of organic substances in water.

Investigations by Hellmann (42T) revealed that plastic containers allowed the permeation of hydrocarbon material from the outside to the water sample to be tested, and from the sample to the outside.

Freeze-concentration techniques for the concentration of organic materials in water have been described by Baker (6T, 7T) and Kammerer and Lee (52T).

Siegel (90T) and Riley and Taylor (77T) reported on ion-exchange procedures for concentrating organic substances in sea water.

#### RADIOACTIVITY AND ISOTOPIC ANALYSIS

A review by Pazdernik (26U) contains 18 references for the determination of some isotopes in water. Rapid procedures for the estimation of the contents of radioactive cations in water were described by Haber and Stuerzer (13U). Various acid treatments of the radioactive cations, which were removed from neutral water (pH 5-7) by filter paper loaded with ammonium phosphomolybdate, and SA-2 filters loaded with Amberlite IR-120 in the sodium-ion form, permitted the estimation of the content of radioactive cations. Wood *et al.* (34U) reported the sequential separation and quantitative determination of <sup>90</sup>Sr, <sup>144</sup>Ce, <sup>137</sup>Cs, and the stable elements of strontium and calcium in plant, feces, milk, and water. The techniques and methods described in the procedure were developed or are standard procedures that were modified.

Picer (27U) discussed three methods for the determination of beta radioactivity in natural waters. A cation-exchange resin is placed over filter paper, and water is filtered through it to remove radioactive cations. After partial drying, radiation is measured with a Geiger-Mueller counter. This method was found more expedient than the

conventional evaporation to dryness followed by igniting of the residue at 750°, and was superior to the procedure in which water is treated with citric acid and calcium acetate prior to evaporation and drying at 110°. The detection of beta radioactivity in natural waters by another evaporation technique was also reported by Picer (28U). Berke, Gyonyor, and Viragh (4U) presented a procedure for the determination of total  $\beta$ -activity of polluted and fresh waters. The radioactive cations are precipitated by adding calcium chloride and ferric chloride solutions, and by adjusting the pH to 10.5 with sodium hydroxide. The apparatus for the detection of radiation and its calibration is described.

A rapid qualitative and quantitative method is described by Berger *et al.* (3U) for determining  $\gamma$ -emitting radio elements in rain water, water from hot springs, or effluents from nuclear reactors. The instrument utilizes a sodium iodide-activated scintillation counter in the energy range 50-1.5 kev.

Biro *et al.* (5U) developed an ionization chamber for the investigation of natural  $\alpha$ -emitters with a specific activity of the order of a picocurie per gram. The  $\alpha$ -spectrometry of natural waters, minerals, and biological samples is described.

A liquid scintillation procedure using 1,4-dioxane, naphthalene, PPO, and POPOP as the scintillator was evaluated in a collaborative study of the determination of tritium in water, as reported by Sodd and Scholz (30U). The minimum detectable activity, based on 99% confidence limits, 100-minute counting time, and backgrounds from 9 to 67 counts per minute, ranged from 0.2 to 0.5 picocurie per ml. The coefficient of variation ranged from 3.4 to 7.3% over a concentration range of 2.91 to 115.8 picocuries per ml. An apparatus was described by Metson (20U) for the electrolytic enrichment of tritium in rain and river water prior to liquid scintillation counting. Tritium activities were determined with  $\leq \pm 15\%$  error without the need for deuterium determinations. Osborne (24U) developed a detector for tritium in water. A cell containing a stack of plastic scintillator sheets measured concentrations of tritium in water down to 1 microcurie per liter. The response time constant is less than 1 minute. An improvement in the enrichment procedure for the determination of tritium in natural waters was discussed by Takahashi, Nishida, and Hamada (32U). A constant separation factor for tritium was obtained, and the enrichment factor for tritium was calculated with an accuracy of  $\pm 10\%$  for the volume reduction rate of  $\frac{1}{2}$ - $\frac{1}{3}$ .

A photonutron method for isotope analysis of hydrogen in natural waters

was outlined by Khristianov (18U). The H:D ratio can be obtained with 1% accuracy by measuring the photo-neutron current due to the effects of  $^{24}\text{Na}-\gamma$  radiation after  $^2\text{D}(\pi\gamma, n)\text{H}$ . A disadvantage of this method is the great sample volume (100 ml) necessary. A polarographic method was used by Calusaru (6U) to determine the isotopic composition of water. The height of the polarographic wave varies linearly with the deuterium content in the presence of cobalt. This method can be used only when the concentration of either hydrogen or deuterium is greater than a few per cent. An interferometric method for the determination of small amounts of  $\text{D}_2\text{O}$  and  $\text{H}_2\text{O}$  was discussed by Mercea (19U).

A nondestructive method was developed by Overman, Corey, and Hawkins (25U) for determining deuterium by the  $(\gamma, n)$  reaction. The method was used to determine deuterium oxide extracted from river water and to locate deuterium oxide in moisture studies. The  $\gamma$ -source is metallic  $^{208}\text{Tl}$  produced from  $^{228}\text{Th}$ . Balakhovskii and Pavelkovskii (1U) determined the deuterium content in water by the absorption of deuterated acetylene in the infrared region. Partially deuterated acetylene is formed by the reaction of calcium carbide with a mixture of  $\text{H}_2\text{O}$  and  $\text{HDO}$ . The infrared absorption maximum caused by C-D and C-H vibrations differs; therefore, it is possible to determine the amount of deuterium present from the relative intensities of C-D and C-H absorptions.

A rapid method for the estimation of radium and radon in water samples with activity levels greater than 1–2 picocuries per liter was related by Kelkar and Joshi (17U). Radium and daughter products of radon are precipitated with greater than 95% efficiency in the presence of  $\text{ZnS}(\text{Ag})$  scintillation powder giving a composite scintillator-sample combination. Conlan, Henderson, and Walton (7U) discussed the assay of picocurie concentrations of  $^{226}\text{Ra}$  with an emanation system, and its application to a study of the natural radioactivity in surface waters. The apparatus was constructed from readily available materials and equipment. This emanation system can be used for the routine assay of  $^{226}\text{Ra}$  concentrations  $\geq 0.01$  picocurie per liter following enrichment by evaporation. The first measurements of  $^{226}\text{Ra}$  in the ocean were reported by Moore (22U). Analytical methods of determining  $^{226}\text{Ra}$  and  $^{228}\text{Th}$  were also discussed.

Doshi and Sreekumaran (9U) described a simplified procedure for estimating  $^{90}\text{Sr}$ . Ferric hydroxide was used for scavenging the fission products and naturally occurring radionuclides, which interfere in the assay of  $^{90}\text{Y}$ , and

for collecting  $^{90}\text{Y}$  after equilibration with the parent  $^{90}\text{Sr}$ . A method for the determination of  $^{90}\text{Sr}$  was discussed in detail by Montag (21U). Stradomskii and Gunbin (31U) described the determination of  $^{90}\text{Sr}$  and  $^{90}\text{Y}$  in natural waters. After concentration and removal of radiochemical impurities,  $^{90}\text{Sr}$  is coprecipitated with calcium carbonate. After 12–14 days, the total  $\beta$ -activity of the calcium carbonate precipitate is measured, and the absolute radioactivity of  $^{90}\text{Sr} + ^{90}\text{Y}$  determined with the aid of standards. The design of a detector of very low energy  $\beta$ -particles is described.

A solvent extraction method for the determination of  $^{137}\text{Cs}$  was outlined by Flynn (10U). After several preliminary steps, including the addition of sodium dodecylbenzene, the extraction was done with phenol-nitro benzene mixture. The final recovery of  $^{137}\text{Cs}$  from vegetation, rainwater, and effluent samples was about 80%. The method is applicable in the presence of large quantities of interfering ions. A determination of  $^{137}\text{Cs}$  in water was described by Benes (2U). Cesium can be concentrated by coprecipitating with the heavy metal ferrocyanides ( $\text{Fe}(\text{III})$ ,  $\text{Mn}$ ,  $\text{Co}$ ,  $\text{Ni}$ ,  $\text{Zn}$ ,  $\text{Cu}(\text{II})$ , or  $\text{Pb}$ ). The use of Prussian blue is advantageous for separating mixtures containing potassium and other interfering elements in high concentrations. Derecki *et al.* (8U) determined  $^{137}\text{Cs}$  activity in water and urine samples by the ion-exchange collection of  $^{137}\text{Cs}$  on ammonium molybdophosphate mats. After the sample drains through the mat, the  $^{137}\text{Cs}$  activity on the mat is counted, correcting for the background with a blank mat. Modifications will permit the determination of approximately 1-picocurie amounts of  $^{137}\text{Cs}$  in water.

Haberer and Stuerzer (12U) reviewed the various methods of determining  $^{131}\text{I}$  in water. Two variations of the method of Fairman and Sedlet, which were considered the most promising, are described. Both are based on the ion-exchange reaction between iodide and silver chloride. The determination of radioactive iodine in rainwater and urine is discussed by Jaworowski (15U).  $^{131}\text{I}$  ( $\geq 5$  picocuries per liter) can be determined with a 97% mean recovery by isotopic exchange between inactive and radioactive iodine on silver iodide impregnated asbestos mats.

A technique has been developed by Williams and Gordon (33U) for the determination of  $^{13}\text{C}/^{12}\text{C}$  ratios in the dissolved organic matter found in the sea. The ratios are essentially invariant with depth and time and do not correlate with dissolved oxygen or total dissolved organic carbon.

The determination of cobalt-60 in water, biological material, and soil was discussed by Yurasova (35U). Co-

baltous nitrate and ferric ion are added to the sample. After precipitation of the ferric ion with ammonium hydroxide, cobalt is extracted as the thiocyanate, reextracted into water, and determined by a mercurithiocyanate method.

The activity ratio of  $^{234}\text{U}/^{238}\text{U}$  and the  $^{238}\text{U}$  content can be determined by the precise and relatively rapid method described by Nguyen and Lalou (23U). After several preliminary steps, the uranium is deposited electrolytically on a platinum electrode. The electrode is washed, dried, and heated to incandescence. The  $\alpha$ -activity of the uranium isotopes is counted with a semiconductor device.

Jaworowski (16U) reported that radioactive phosphorus, strontium, lanthanum, and cerium can be determined in urine and water, with 77 to 99.5% recoveries and a 10-picocuries-per-liter detection limit, by sorption of the nuclides on phosphate gel mats. After sorption on the mat, it is dried under vacuum for  $\beta$ -counting.

A method was outlined by Shannon, Cherry, and Orren (29U) for the determination of  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  in sea water by solvent extraction, followed by electrodeposition and  $\alpha$ -counting. The mean activities of  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  in 29 sea-water samples were  $38 \times 10^{-15}$  curie per liter and  $20 \times 10^{-10}$  curie per liter, respectively.

A procedure for the radiochemical determination of technetium-99 in environmental water samples was reported by Golchert and Sedlet (11U). In the final steps, the technetium is electrodeposited on stainless steel and determined by a beta-counter. The overall chemical recovery was 79.6%, the counting efficiency was 41%, and the relative error of a single determination ranged from 30% at 0.5-picocurie-per-liter levels to 13% for greater than 10-picocuries-per-liter activities.

Ikeda *et al.* (14U) outlined a radiochemical determination of  $^{95}\text{Zr}$ - $^{95}\text{Nb}$  in sea water. The method is based on the coprecipitation of  $^{95}\text{Zr}$ - $^{95}\text{Nb}$  with 5  $\mu\text{g}$  of zirconium carrier on lanthanum hydroxide followed by the extraction of  $^{95}\text{Zr}$ - $^{95}\text{Nb}$  with 5% tri-*n*-butyl phosphate in toluene. The yield of  $^{95}\text{Zr}$ - $^{95}\text{Nb}$  was greater than 99%.

#### MISCELLANEOUS

Charpiot (10V) investigated several techniques for the preservation of water samples for phosphate, nitrite, nitrate, silica, and boron determinations. He found that 2 ml of 8N sulfuric acid per 100 ml of sample and storage in polyethylene bottles in the dark was the best technique for phosphate. For the other constituents, 2 ml of a 2% mercuric chloride solution per 100 ml of sample and storage at 4 °C in poly-

ethylene bottles provided the best means of preservation.

Robertson (54V) studied the adsorption behavior of 11 trace elements in sea water at pH 8.0 and 1.5 in polyethylene bottles. For prolonged storage, up to 75 days, the sample should be acidified to pH 1.5 with hydrochloric acid immediately upon collection of the sample. Gavrishin (23V) also studied the adsorption of trace elements on container surfaces. He recommended the use of glass containers and acidification of the filtered sample to a pH of 2.5 to 3 after 5 days. He reported that to acidify the sample immediately may cause the acid water to leach elements from the glass bottle.

Shevchik (58V) compared and evaluated three methods of concentration of trace elements in formation water and found the following: (1) Evaporation to a dry residue was the most efficient for Cu, Be, and Bi; (2) coprecipitation with CdS gave maximum efficiency for Ag, Cr, Mo, Sn, Be, Ge, Y, Yb, and Sc; and (3) coprecipitation with  $\text{Al}(\text{OH})_3$  was the most efficient for Sb and Se. High sensitivity was obtained for Mn and W by all three methods.

Karakhanov (31V, 32V, 33V) described three methods of determining the total mineralization of waters. The methods are based on either passing the water through cation- or anion-exchange resins and titrating the filtrates for total cations with sodium hydroxide, or total anions with silver nitrate.

Ceausescu (9V) described a procedure to determine sulfate, chloride, and nitrate in the same test sample. A decationized water is titrated with sodium hydroxide to give the sum of sulfate, chloride, and nitrate. Sulfate is then precipitated with excess barium ion, and the excess barium is titrated with tetra-Na EDTA. Sulfate is determined by difference. Chloride is titrated with mercuric nitrate in the presence of an indicator. Nitrates are then determined from the difference.

Korotkov (35V) studied the use of an electrical conductance method in the determination of the total mineral content in natural waters containing calcium chloride and sodium chloride by obtaining electrical conductance *vs.* total mineral content curves at 20 °C. Teterin (63V) also described an electrical conductivity method to determine the total salinity of natural water.

Vaidya (64V) used a pocket refractometer to determine the salt concentration of sea water. The per cent refractometer reading *vs.* concentration in degrees Baumé is linear over the range 5° to 30° Baumé. Brown (7V) and Kozlovskaya and Ermakov (37V) described salinometers to determine the salinity of sea water.

Two models of potable-water testing laboratories were described by Vul'fon

and Saburova (65V). One is a titrimetric unit and the other a colorimetric unit.

Berkstresser (4V) reported that water samples should be field-filtered at the time of collection since solids may cause changes in water quality of samples during storage. Vacuum or pressure filtration with chemical-active gases may cause chemical changes in the samples, and he suggested the use of nitrogen gas to minimize chemical change.

Jakovljevic (30V) described a mathematical procedure to check the validity of a chemical water analysis. Details are given.

The errors in the methods for the determination of nitrate, fluoride, calcium, and total hardness in drinking water are discussed by Gitsova and Kalpazanov (24V) from a statistical point of view.

Several references on the use of atomic absorption for the determination of individual elements appear throughout this review. However, several publications on atomic absorption have dealt with multiple-element analysis in natural and industrial water, using direct, precipitation, and extraction procedures. Elements that have been determined include: silver, copper, iron, manganese, lead, zinc, cobalt, nickel, lithium, strontium, silica, calcium, magnesium, cadmium, chromium, barium, sodium, and potassium [Briley and Rankin (6V); Galle and Angino (22V); Grillo (27V); Sinha, Singhal, and Banerji (59V); Soman, Panday, and Joseph (61V); Riley and Topping (52V); Brewer, Spencer, and Smith (5V); and St. John (57V)].

In a review on atomic absorption, Price (50V) outlined procedures for handling water samples and many other types of materials.

Fishman and Erdmann (18V) automated an atomic absorption spectrophotometer with a sampler, proportioning pump manifold, and strip-chart recorder. Copper, lithium, manganese, potassium, sodium, iron, and zinc are determined by direct aspiration. The proportioning pump manifold is added to the system for the determination of calcium, magnesium, and strontium. It introduces, automatically, an interference suppressant to the sample prior to aspiration. The latter system can also be used to make dilutions.

Pittwell (49V) reported that molecular bands, as well as  $\text{C}_2\text{N}_2$  and some metal oxides, notably of CO and SiO, appear on many d.c. arc emission spectrograms of ashed oxide-dithizone extracts of natural water. Wavelength data are presented to help avoid the misidentification of the lines of trace elements.

Durisova and Mrna (15V) concentrated heavy metals from surface waters by electrolysis and then determined their

concentration spectrographically. Lotrian and Johannin-Gilles (39V) determined boron, strontium, and silica in sea water by emission spectroscopy by the tube electrode-spray electrode and porous-cup methods without any chemical pretreatments. They claimed that the tube electrode method was superior both in reproducibility and detection. Kozin, Korosteleva, and Yurova (36V) described a spectral method for the determination of lithium, strontium, and barium in stratal waters. For the determination of lithium, the water was first saturated with sodium chloride. The water was analyzed directly for strontium and barium.

Sokolov *et al.* (60V) used organic coprecipitators, sulfaniloxine, Rhodamine 6 Zh, and  $\text{Al}(\text{OH})_3$ , to preliminarily concentrate 27 elements, from ground and mineralized water, and then determined their concentrations spectrographically. Kuz'min, Yakimenko, and Kalinachenko (38V) described an extraction spectrographic method for the determination of Al, V, Bi, Ga, Fe, In, Co, Mn, Cu, Ni, Pb, Ti, and Zn in water. The metals were extracted as complexes with 8-hydroxyquinoline and sodium diethyldithiocarbamate at a pH of 5.5 to 6.0 with a mixture of chloroform and isoamyl alcohol (3:1). Reeben (51V) extracted trace metals into chloroform after complexing the metals with  $\text{Et}_2\text{NCS}_2\text{Na}$  and oxine at pH 4.8 and 9. The extract was then evaporated in the presence of a spectroscopic base and determined by an a.c. arc spectrograph.

Diaz-Guerra and Capdevila Perez (13V) investigated the effects of 1000 ppm calcium and sodium and 300 ppm magnesium on the spectrographic determination of a number of trace elements in natural water. With the use of Tl-In as a buffer and Ga, Tl, and Y as internal standards, the determinations were accurate to within 10%.

Eremenko and Mel'nikov (17V) studied the effect of high concentrations of manganese on the spectrographic determination of heavy metals in natural waters. An extraction procedure is described which will help to eliminate the problem.

A direct-reading spectrographic method, using a special push-up filter-paper technique to minimize sample preparation, for determination of elements in industrial waste waters is described by Steiner, Austin, and Lander (62V).

A dropping mercury polarographic method for the determination of Sb, As, Cd, Pb, Hg, Se, Te, and Th was reported by Offner and Witucki (45V). A 0.25M tartaric acid and hydrochloric acid solution was used as the supporting electrolyte. Solutions were polarographed in a standard hydrogen-type cell, following deaeration with oxygen-



free nitrogen. Cravo (12V) determined zinc and iron simultaneously in water polarographically using a support solution containing 1M ammonium sulfosalicylate and 6M ammonium hydroxide.

Park (46V) used X-ray fluorescence spectrometry to determine Cu, Pb, Zn, Ni, Co, and Fe in water. These cations are concentrated by evaporation, precipitated as their sulfides, and fixed in gelatin. Each metal was measured at a concentration range of 2.5 to 50.0 ppb with a relative standard deviation of 14.0%. Rose and Cuttitta (56V) described an X-ray fluorescence spectroscopy method which could be used for the analysis of waters. Two single-channel X-ray spectrometers were used: an air or helium unit with a platinum target X-ray tube, and a vacuum unit with dual-target (Cr and W) X-ray tube.

Funk, Bhagat, and Filby (21V) determined a number of trace elements in lakes and streams by neutron activation with minimal or no chemical preparation. Robertson, Rancitelli, and Perkins (55V) employed neutron activation and direct-counting techniques to measure the concentration of numerous trace elements in sea water. The activated samples were counted on either a multidimensional gamma-spectrometer or a large-volume Ge(Li) diode spectrometer after optimized decay periods.

Piper and Goles (48V) determined Co, Cr, Cs, Fe, Rb, Sb, Sc, Sr, and Zn in ppm or ppb in the freeze-dried salts of 100 ml of sea water by means of neutron activation.

Mark *et al.* (42V) employed ion-exchange membranes in conjunction with neutron activation and anodic stripping voltammetry to determine certain trace metals in aqueous media. The parameters which govern their applicability, limitations, and sensitivity were investigated.

Korkisch and Orlandini (34V) described a quantitative method for the isolation of more than 30 trace elements from sea water. The technique consists of 2 organic extractions and a cation-exchange step. The isolated elements can then be determined radiochemically.

Fleischer and Lovett (20V) described a particle-track etching technique to determine the uranium and boron content of water.

Riseman (53V) reviewed the application of specific-ion electrodes to the analysis of inorganic materials in water. The precautions necessary to ensure correct analysis are emphasized. Andelman (1V, 2V) discussed the principles of operation of ion-selective electrodes and reviewed their application for the determination of the concentration of specific ions in water.

A review of continuous turbidity measurements during flood and sewage con-

trol is given by Malz, Schniewind, and Krone (41V); their experiences on mechanized photometric analyses also are discussed.

Green (26V) discussed the apparatus used in water pollution control and divided it into three types: laboratory analysis, field analysis, and permanent or semipermanent field monitors. He stressed the need for improved monitoring instruments and the desirability for measuring a number of constituents not presently being monitored.

Babcock (3V) discussed the guidelines needed to maintain laboratory standards and to prevent loss of accuracy when laboratory techniques are transferred to water-plant routine.

Fishman and Pascoe (19V) have applied automated titrimetric analysis to the determination of alkalinity, chloride, and sulfate in fresh water. Alkalinity is determined potentiometrically and chloride and sulfate spectrophotometrically. Replicate analyses and comparison of results with those obtained by manual methods show that the automated methods are accurate and reproducible.

Watanuki (66V) adopted automated colorimetric methods for the determination of silica and nitrate in sea water. Silica is determined by the molybdate blue method and nitrate by the diazotization method after reduction of nitrate with copper-cadmium.

Several investigators, Williamson and Millar (67V); Hertzog (29V); Grasshoff (25V); Conetta and Adelman (11V); Catanzaro *et al.* (8V); Marten and Catanzaro (43V); and O'Brien and Olsen (44V), used Technicon Auto-Analyzer systems (field monitoring and laboratory analysis) to determine a number of constituents in fresh water, industrial water, and sea water.

Henriksen (28V) described a photo-oxidation method for the determination of total nitrogen, phosphorus, and iron in fresh water. The samples are subjected to ultraviolet irradiation with a high-pressure mercury-arc lamp. Organically-combined phosphorus and iron are converted into orthophosphate and ferric iron in the presence of acid and excess of oxygen after an irradiation time of one hour. Four hours of irradiation in the presence of excess oxygen in the pH range of 6.5 to 9 are required to convert organic nitrogen compounds and ammonia to nitrate and nitrite.

Dojlido and Metler (14V) described the construction, operating principles, and applications of a water and sewage autoanalyzer.

Malz and Krone (40V) compared two processes for the continuous conductivity measurement of river and waste water. One uses a direct contact between the electrode surface and the water. The other measuring cell is a glass tube, in which the analyzed water

flows and the electrodes are connected to the outer wall. This adjustment represents a condenser, where the loss of resistance affected by the conductance of the liquid is measured by a high-frequency oscillator and the amplitude is the measure of the conductance.

Effenberger (16V) described a sample flow cell for the continuous measurement of redox potential.

Pavelka and Rebuzzini (47V) described the microchemical detection of the most common cationic and anionic pollutants in water. The method is based on modified Feigl microanalyses.

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### Introduction

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