

Water Analysis

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This biennial review of methods of water analysis, the fifteenth in a series that began in 1949, covers the literature from October 1970 through September 1972. It follows the plan of the previous reviews, the most recent of which appeared in *Analytical Chemistry* for April 1971 (12).

The Water Pollution Control Federation publishes, annually, a review of the literature on water pollution control which includes a section on analytical methods. Their 1970 review by Williams (39), Doughty, (9), and Carlton, Walters, and Whittle (8) includes 90 references and covers such topics as COD, cations, anions, spectrophotometric, noninstrumental, automated, monitoring, and radiochemical techniques. The 1971 review by Doughty (10), Carlton, Smith, and Walters (7), and Skrinde (29) includes 114 references and covers a section on organics in addition to the above topics.

Uematsu (33) reviewed analytical methods for determining various pollutants in water. Methods of water pollution measurement with special emphasis on instrumental and noninstrumental techniques, and the present state and future aspects of the measurement of water pollutants are discussed by Umezaki (34, 35). Improvements since 1962 in methods for determining water quality are reviewed by Liebmann (20); twenty-eight references are included. A review with 59 references on the determination of trace elements in water is given by Han (14).

Skougstad (28) outlined methods for temperature, specific conductance, density, color, turbidity, taste and odor, dissolved and suspended solids, pH, alkalinity, acidity, hardness, calcium, magnesium, sodium, potassium, chloride, sulfate, nitrate, nitrite, fluoride, phosphate, boron, silica, oxygen, manganese, iron, aluminum, rare alkali and alkaline-earth metals, transition metals, arsenic, selenium, bromide, iodide, sulfide, ammonia, BOD, COD, organic carbon, pesticides, detergents, oil and grease, bacteriological examination, and suspended sediment.

Andersen and Foeyn (2) prepared a review with 42 references on apparatus and methods used in determining sea water salinity, temperature, density, dissolved oxygen, sulfide, phosphate, silica, nitrate, nitrite, and ammonia.

Analytical methods used to determine the chemical composition of natural waters encountered during prospecting of uraniferous deposits, or in the course of mining operations, are described by Berthollet and Cavalier (5). Because of the diversity of the samples and different items of information requested, methods were selected and adopted to answer the demands peculiar to mineral research. Moore (23) reviewed and described the principles and limitations of some of the more important techniques used in water analysis. In brines, where the total solid content may approach 35%, gravimetric and volumetric techniques are recommended when high precision and accuracy are required. For minor and trace elements, and for major constituents when precision is not required, physicochemical methods are preferred.

Spencer and Brewer (30) prepared a detailed review with 331 references on inorganic analytical methods used

in oceanography. Included are determinations for total salinity, total chlorinity, major cations and anions, silicate, nitrate, phosphate, nitrite, sulfide, dissolved gases, and radionuclides. The determination of trace elements by absorption spectroscopy, atomic absorption spectroscopy, flame photometry, emission spectrography, fluorimetry, radioactivation methods, and isotope dilution is also discussed.

Concentration and separation techniques used in the analysis of water and water-pollution samples are reviewed by Andelman and Caruso (1). Techniques include precipitation, liquid-liquid extraction, freeze concentration, adsorptive bubble separations, chromatography, ion exchange, membrane techniques, carbon adsorption, distillation, evaporation, and sublimation. Three hundred and forty-one references are given.

Naumczyk (24) reported on the application of catalytic methods to analysis of water. Examples are given for the determination of both cations and anions.

A review with 30 references on the usefulness of Po-Be, Pu-Be, Am-Be, and ^{252}Cf neutron sources in the activation analysis and monitoring of pollutants in natural environments was prepared by Leddicotte (19). Buchanan and Bialy (6) reviewed recent developments in neutron activation analysis of water. Included are sources of neutrons, pre-irradiation physical and chemical treatment of samples, neutron capture γ -ray analysis, and selected applications. They also mentioned that, despite high cost and some other disadvantages, reactor neutron activation analysis is a highly selective and sensitive method for determining many trace elements in water. A review is given by Hoegdahl (15) on the application of activation analysis in oceanography.

Riseman (25) reviewed the types and uses of ion-selective electrodes in natural and waste waters. A discussion on theory, interferences, concentration measurement, and precision is included. The properties of ion-selective electrodes and the application to sea water and other natural waters are reviewed by Warner (37). Sixty-one references are given. Vigneron *et al.* (36) surveyed the applications of electrochemical analytical methods to water analysis, and they indicated that the electrochemical methods are simpler and less time consuming than the classical methods. A review with 27 references is given by Yoshimori (40) on the amperometric, coulometric, potentiometric, and conductometric methods for the continuous analysis of water. Advantages and disadvantages of these methods are discussed.

General methods for determining substances at the nanogram-per-liter level in water and effluents by gas chromatography are reviewed by Weil (38). Emphasis is placed on mineral oil products and pesticides.

Keith *et al.* (16) reported on the use of nuclear magnetic resonance spectroscopy and mass spectrometry in water pollution analysis. These have enabled the characterization and specific identification of chemicals causing pollution problems in cases where this was not previously possible or practical. Examples on the actual use of these instruments are presented.

The theory of atomic absorption and procedures for determining metals are reviewed by McFarren (21). Special emphasis is placed on the use of air-acetylene, nitrous oxide-acetylene, and argon-hydrogen flames. Diagrams of apparatus and brief outlines of procedures for determining arsenic and mercury are given.

A review on the comparison of air and water pollution instrumentation is given by Rittmiller, Zomkowski, and Wadehra (26). Both sampling and analytical devices are discussed.

Suffet, Radziul, and Goff (31) reviewed the theory and methodology of electrolytic sensors presently utilized for continuous water-quality measurement. For the various sensors, sensitivity limits, problems encountered in use, and possible remedies to these problems are given. Sunahara *et al.* (32) described and illustrated instruments and instrumental modifications used in the continuous monitoring of water-quality parameters. Included are turbidimetric, fluorimetric, and coulometric methods. A review with 20 references is given by Drake (11) on pollutants found in rivers, water works, and sewage works, the acceptable levels of each, and of commercially available monitoring instruments and their detection limits.

Sampling of natural waters and sampling techniques are described in separate reviews by Saijo (27), King (17), and Grasshoff (13). Some of the topics discussed are filtration, preservation, sample size, frequency, choice of bottles, and sampling equipment.

Kroner and Ballinger (18) discussed the determination of both physical and chemical constituents in potable and sanitary water. The necessity of proper sampling and problems encountered are noted. Several sampling devices are shown. Methods for the determination of major and minor constituents are reviewed. Flame photometric, atomic absorption spectroscopic, and emission spectroscopic methods for water analysis are discussed. Ballinger (4) also reported that the requirement for accurate, precise, and rapid analytical procedures requires the use of a variety of instruments. In another publication, Ballinger (3) discussed the effect of recent developments in laboratory instrumentation on the analysis of water. The newer instruments have incorporated automatic sample handling, sequential analysis, and improved data presentation. The availability of these new instruments helps meet the need for expanded capacity and for more intensive investigation of the nature and fate of many specific water pollutants. Automatic monitoring of water quality is also discussed, and capabilities need to be expanded to include a larger variety of measurements.

Results presented by McFarren and Lishka (22) indicate that a very satisfactory way to evaluate water analysis instruments is by means of a collaborative study involving their use. At least six laboratories are recommended for each instrument being evaluated.

ALKALI METALS

Eckfeldt and Proctor (34) monitored low-level sodium ion concentrations (0.1 to 1.0 ppb) in process water with a glass electrode. Laboratory and field studies indicated that sodium, even at this low level, can be successfully monitored over long periods of time if attention is given to several conventionally overlooked factors. A strong base, such as dimethyl amine or di-isopropyl amine, is preferred to reach a pH of 11.0 to 11.6 which is needed for adequate suppression of hydrogen ion and to produce a cation which exerts minimal electrode interference. Other troublesome interferences and ways to avoid them are discussed, including potassium and silver ion interference from the ref-

erence electrode solution. A sodium monitoring apparatus which uses a sodium-responsive glass electrode is described by Diggins, Parker, and Webber (2A). Concentrations of sodium greater than 0.0001 ppm and up to 10 ppm in high-purity water can be determined continuously. Film-forming amines, such as octadecylamine, must be absent. Higher sodium concentrations can be determined with an error of $\pm 10\%$. Instrument precision, response time, temperature effects, maintenance requirements, and manual and automatic standardization are discussed. Galetti and Spear (5A) also described an analyzer for the monitoring of sodium in power plant demineralizer effluents. The analyzer has a range from 0.1 ppb to 10,000 ppm with a reproducibility of $\pm 2\%$. Kim and Kim (6A) determined sodium with an error of 20% in sea water with a sodium-sensitive glass electrode. The electrode potential is stable between pH 4.5 and 13. The effect of the presence of potassium, calcium, and magnesium is discussed.

Romanov and Ereemeeva (9A) discussed the accuracy of spectral methods for the determination of potassium in sea water. The variation coefficient with propane-butane-air flame photometry on an FPF-58 photometer was 14%; with an SF-4A monochromator, 4.8%; by emission spectral analysis with a spark source, ac arc, and ISP-51 spectrograph (internal standard lithium), 6%; by an emission method with photoelectric recording on a DFS-13 spectrograph by blowing the aerosol between ac arc copper electrodes (internal standard indium), 2%.

Shigematsu, Suzuki, and Tabushi (10A) determined lithium in sea water by atomic absorption. Lithium is first extracted with isoamyl alcohol.

An extraction-photometric method for the determination of lithium between 0.15 and 20.80 mg per liter in lake water is described by Morgen and Vlasov (7A). The method is based on the use of nitroanthranilazo in dimethyl formamide. Interfering elements are eliminated by extracting lithium with acetone from the dry residues. The method is accurate to 1.87% at the 95% confidence level.

A method is presented by Folsom, Hansen, and Robertson (4A) for determining cesium in sea water which involves treating two 1-liter samples together, with 0.500 μg cesium-133 added to one. The cesium is then precipitated with ammonium 12-molybdophosphate and extracted into tributyl phosphate and burned in a cup in a recording differential emission photometer. They also mentioned that prolonged storage of samples in polyethylene bottles had little effect, less than 3%, on the cesium content.

Simionescu (11A) preferred flame photometry for the determination of sodium and potassium in surface and waste waters. Conditions, which must be followed, include tracing of standard curves, using standards which contain radiation buffers, and determining either ion in the range of 0 to 100 mg per liter.

Anosova (1A) determined sodium, potassium, and lithium in high saline waters and brines flame photometrically, using interference light filters. Rectilinear calibration graphs are obtained, and the error of the determination is 1% or less. For elimination of the calcium effect on the determination of lithium and potassium, prior precipitation of calcium as oxalate or use of a compensation method is recommended. For elimination of the silica effect on lithium, aluminum nitrate is added to the sample. Prudnikov, Shapkina, and Semov (8A) used flame photometry to determine trace amounts of lithium, rubidium, cesium, and strontium in water. By the use of a sensitive acetylene flame photometer equipped with a diffraction monochromator having a 0.1-mm slit, lithium is detected at 10^{-5} mg per liter, rubidium and cesium at 5×10^{-5} mg per

liter, and strontium at 10^{-3} mg per liter. The precision is 3 to 5% at concentrations of 0.01 to 0.1 mg per liter, decreasing at lower concentrations.

HARDNESS, ALKALINE EARTH METALS

Morgen, Vlasov, and Filippova (19B) determined calcium in natural waters colorimetrically with Arsenazo I. Potassium hydroxide and Arsenazo I are added, and the color is measured at 580 nm. Accuracy is similar to that obtained by the Trilon B method, but the method is more rapid than the latter. No interference was observed in the presence of the major anions and sodium and potassium at any concentration. Ferrous and ferric iron and magnesium present in concentrations 12, 12, and 5 times that of the calcium did not interfere. The reproducibilities for samples containing less than 1000 μg per liter was 1.9 to 9.7% and 0.86 to 5.6% for samples containing between 1000 and 9000 μg per liter. Sarno (24B) described a glyoxal bis(2-hydroxyanil)-colorimetric method to determine calcium in water. At pH 12.6, the calcium glyoxal bis(2-hydroxyanil) complex is extracted with isoamyl alcohol stabilized by a mixture of ethanol and butanol. The absorbance of the organic phase is measured at 525 nm. The method is useful between 10 and 700 μg per liter. Glyoxal bis(*o*-hydroxyanil) also has been used by Johri, Handa, and Mehra (11B) for a very selective determination of calcium in mineral waters by ring colorimetry. Calcium gives a sharp red insoluble ring. The range of the determination is 0.02 to 1 μg of calcium in the total ring. Other ions which are generally present in mineral water either do not interfere or are washed out during the fixing of calcium.

Bosch Serrat, Arroyo Lopez, and Suazo Gallego (3B) described a spectrophotometric method for determining magnesium in water in the presence of diverse ions, such as calcium. Other ions at levels 100- to 1000-fold in excess of magnesium do not cause interferences. Beer's law is followed for 2 to 16 μg of magnesium per ml at 480 to 590 nm. The method is based on the color formed by adsorption of Azo Rubine S on magnesium hydroxide in alkaline media.

Impedovo, Traini, and Papoff (9B) determined magnesium and calcium in water by an automated spectrophotometric method. The apparatus is described. At 571 nm with an excess of Eriochrome Black T, the variation in absorbance is proportional to the magnesium concentration regardless of the calcium concentration. By addition of MgEDTA, to displace the calcium from its calcium-Eriochrome Black T complex, calcium may be determined at the same wavelength. The standard deviation for 3 to 25 ppm magnesium and 5 to 40 ppm calcium was about 4%.

Nakagawa, Nanbu, and Ohyagi (22B) used atomic absorption with an air-acetylene flame to determine calcium and magnesium in water. They reported that large amounts of silica, aluminum, titanium, and vanadium interfered.

A photometric method for determining the total hardness of waters containing less than 0.01 meq per kg is reported by Hissink (8B). The method is based on the formation of a red complex between Eriochrome Black T and calcium and magnesium at a pH between 10 and 11. Anisimova (2B) determined calcium plus magnesium in water based on complexes formed with Chromazurol S. At pH 8.0 to 12.0, the absorbance maximum of the complexes is at 582 nm. Calcium between 2×10^{-6} and $1 \times 10^{-4}M$ and magnesium between 1×10^{-8} and $5 \times 10^{-5}M$ can be determined in mixtures.

Martin and Poudou (15B) compared the sensitivity of a divalent calcium-magnesium electrode with the complex-

ometric method for determining water hardness. Results obtained were in good agreement. Mascini (16B) reported that the copper-selective solid-state electrode is better than the calcium-selective liquid-exchanger electrode for the potentiometric titration of calcium and magnesium in water. With the solid-state electrode, well-defined potentiometric titration breaks are achieved. The standard deviation of the determination is about 0.1 to 0.5%. Moody, Oke, and Thomas (18B) made a long-life, rapid-response, calcium-sensitive electrode that nearly follows Nernstian behavior between 0 and 48 °C by incorporating a liquid ion exchanger into a poly(vinyl chloride) matrix. A description of the electrode is given. Selectivities were obtained by several methods for zinc, magnesium, barium, sodium, potassium, and hydrogen ions. The electrode was suitable for monitoring the calcium content of water supplies.

A computer-processed, high-precision photometric titration procedure for the determination of the total alkaline-earth metal concentration in sea water is described by Jagner and Aren (10B). Titrations are carried out at pH 10.5 with EDTA as titrant and Eriochrome Black T as indicator. The method yields a value of 63.32 mmole per kg of 35‰ salinity for the total alkaline-earth metal concentration in Standard Sea Water with a precision of approximately 0.01%. Details of the procedure are given.

A chelatometric titration for the determination of calcium and magnesium in natural waters, in the presence of large amounts of ferric iron, is described by Kosak and Balczko (12B). Calcium is determined by direct titration with EDTA (at pH 12.0 to 12.3, adjusted with potassium hydroxide solution) using calcon-carboxylic acid as indicator. Iron is masked with triethanolamine. To determine magnesium in the same sample, the indicator is destroyed with bromine, the pH adjusted to 10.0 with ammonia-ammonium chloride buffer, and the sample titrated with EDTA using methylthymol blue as indicator. If the iron is greater than 50 mg per liter, the sample is back-titrated with calcium chloride solution at pH 12 to 12.5 with thymolphthalexone as indicator.

Nagasaki and Toei (21B) determined calcium and magnesium in surface and ground waters by chelatometric titration. Calcium and magnesium are titrated with EDTA using 1-dicarboxymethyl-aminomethyl-2-hydroxy-3-naphthoic acid (DHNA) as a fluorescence indicator. Under irradiation at 366 nm, the fluorescence at the end point changes from sky blue to yellow-green and from green-blue to yellow-green for magnesium, and calcium, respectively.

The determination of the hardness of highly colored natural waters by titration with Trilon B, using the indicator ET-OO in a mixture with sodium chloride (1:100) without the removal of the organic substances, was performed by Lavrova, Nemtseva, and Semenov (14B).

Fritz and Kaminski (5B) demonstrated the use of Amberlyst A-21, a weak-base, anion-exchange resin, to separate ferrous iron and copper from water prior to a hardness titration. Traces of iron, copper, and several other metal ions may interfere with the titrations by combining with the indicator and reducing the sharpness of the end point, or by masking it completely so no color change occurs. Cyanide masks these metals if their concentration is not too great, but its use is hazardous.

Villalobos (25B) described a simple and rapid method for approximating the degree of hardness in natural water. Action of methyl orange upon sodium chloride, potassium chloride, sodium sulfate, sodium bicarbonate, calcium chloride, magnesium chloride, calcium sulfate, and mag-

nesium sulfate is given for 10,000, 1000, 500, and 25.0 ppm. Occurrence of a precipitate with dilute solutions gives an idea of the water hardness.

A survey, with 73 references, was made by Bowen (4B) on analytical methods used by various authors for the determination of stable strontium in sea water. Various procedures for the determination of strontium-90 in sea water were also compared. He used this survey for the selection of some preferred methods. The descriptions of selected procedures for strontium and strontium-90 are given.

Miyanaga (17B) used atomic absorption for the determination of strontium in thermal-spring water. The samples were made up to 0.5M with hydrochloric acid, and 2000 ppm of lanthanum added to mask interference from potassium, sodium, calcium, magnesium, sulfate, and phosphate. The concentration range in the samples was 1.8 to 6.3 ppm.

Gordon and Larson (7B) described a photon activation method for the determination of strontium in sea water. The samples are acidified during storage, filtered, and freeze-dried for a 30-minute irradiation in a bremsstrahlung flux of 30 MeV, and then γ -counted after separating $^{87}\text{Sr}^m$ by the conventional strontium carbonate precipitation method.

An extraction-fluorometric method for the determination of low concentrations of beryllium, approximately 1 μg per liter, in lake, ground, and waste waters is described by Morgen, Vlasov, and Serykh (20B). Beryllium is complexed with acetyl acetone and Trilon B, extracted with chloroform, and the fluorescence measured. They obtained an accuracy of 0.64% at the 95% confidence level.

Petrova (23B) described a sensitive spectrographic technique to determine beryllium in water. At a wavelength of 2348.6 Å, beryllium concentrations of 0.14 to 0.38 mg per liter were determined with an accuracy of 6%.

Galitsyna and Polyakov (6B) used a spectrographic method to determine barium in natural waters. Five hundred milligrams of activated carbon are added to one liter of sample, the sample is shaken for 3 minutes, and the carbon filtered off in a concentrator. The obtained carbon precipitate is dried on a filter, transferred to a crucible, calcined at 600 to 800 °C, placed into a carbon-electrode channel, and then excited in a dc arc. Barium lines at 4534 and 4900 Å and the background are photometrically analyzed. Angelieva (1B) used a quartz spectrograph with an ac arc to determine barium in drinking water. Zirconium nitrate is used as an internal standard. The method showed a sensitivity of 0.001% barium with good reproducibility. The relative error was 13 to 16%.

Kozin, Korosteleva, and Yurova (13B) spectrographically determined strontium, barium, and lithium in stratal waters of petroleum deposits. A drop of water, after being saturated with sodium chloride, is placed on a carbon electrode, covered with polystyrene, and the arc spectrum compared with that of a standard. Strontium and lithium values showed good agreement with values obtained by flame photometry.

ALUMINUM, IRON, MANGANESE, CHROMIUM, RHENIUM, CERIUM, AND TITANIUM

Fishman (5C) described an atomic absorption method for the determination of aluminum in fresh waters. The element is chelated with 8-hydroxyquinoline (oxine) at pH 8.0 and extracted with methyl isobutyl ketone (MIBK). The extract is then aspirated into a nitrous oxide-acetylene flame of the spectrophotometer. Samples containing between 10 and 300 μg per liter can be analyzed. None of

the substances commonly occurring in water, except for magnesium, interferes. Magnesium forms an insoluble chelate with oxine at pH 8.0 and tends to coprecipitate the aluminum oxinate. The magnesium oxinate forms slowly (3 to 5 minutes) and its interference can be avoided if the aluminum oxinate is extracted with MIBK immediately after the sample is buffered to pH 8. A previously published method for the determination of trace-metal ions by atomic absorption was modified by Hus and Pipes (9C) for the determination of aluminum in water. Benzene is used as the organic solvent for extraction instead of ethyl propionate. The lower solubility of benzene in water allows the use of a higher extraction ratio. Diphenylthiocarbazone, 8-quinolinol, and acetylacetone are the chelating reagents. The extract is aspirated into a nitrous oxide-acetylene flame. The sensitivity of the determination is 0.009 mg per liter.

Nozdrunova (15C) described a colorimetric method for determining soluble aluminum in the presence of iron in various types of water. Iron is masked by adding a 2% thioglycolic acid solution. The sample is then buffered to pH 5.4 with ammonium acetate and the color developed with a 0.2% aluminon solution. To determine total aluminum, the aluminum present in organics is extracted by multiple treatments with 30% hydrogen peroxide; the solution is then evaporated to dryness, dissolved in hydrochloric acid, and determined as above.

Hiiri, Tanaka, and Sawada (8C) used solvent extraction followed by atomic absorption to determine ppb amounts of iron in ground water and tap water. A liter sample is buffered with an acetic acid-sodium acetate solution and the iron extracted with 30 ml MIBK. A linear calibration curve is obtained for 10 to 50 ppb. Results by the atomic absorption method agreed with results obtained by the colorimetric, 1,10-phenanthroline method.

Resch (18C) compared various methods for the determination of total iron in water-steam cycles of power plants and listed their advantages and drawbacks. Methods using phenanthroline give consistent results both for metallic iron and iron oxides. The thioglycolic acid method is as good as the phenanthroline method when the nondissolved iron occurs as magnetite.

Mizuno (12C) determined ferrous iron in boiler water between 1 and 30 ppb in the presence of ferric iron by the bathophenanthroline method. Ferric iron is masked with sodium pyrophosphate. The ferrous iron is then extracted with *n*-butanol at pH 4.2 to 4.7, and the absorbance measured at 533 nm. Nickel, zinc, manganese, and chromium have little effect on the determination; however, only 25 ppb of copper and 5 ppb of cobalt can be tolerated.

Fukasawa, Iwatsuki, and Asakawa (7C) described a spectrophotometric method to determine between 0.04 and 1 ppm of iron in water with sodium sulfide in an alkaline media. Ferric iron is first reduced to ferrous iron with hydroxylamine sulfate. The solution is then neutralized with sodium hydroxide, sodium sulfide is added, and the solution is brought up to a specific volume. After standing for 45 minutes at 30 °C, the absorbance of the solution is measured at 430 nm. Copper, manganese, magnesium, and phosphate, 1 ppm each, interfere.

Andreeva and Samozvon (1C) reported that total iron in ground waters in the presence of organic matter can be determined spectrophotometrically with sulfosalicylic acid. The organic material is decomposed with acid-permanganate solution.

Cherkesov and Krasnov (4C) determined iron in water photometrically at 530 nm after ferric iron is complexed with Phthalexon S at pH 1. Beer's law is followed for 0.2

to 0.8 μg of iron per ml. Most cations commonly occurring in water do not interfere.

A spectrophotometric method for the determination of iron in natural and industrial waters is described by Vajda, Jarabin, and Szarvas (21C). Ferric iron is reduced to ferrous iron with ascorbic acid and the solution buffered to pH 8.8 with an ammonium chloride-ammonium hydroxide solution. The reddish brown color developed upon addition of gallic acid is measured at 500 nm.

Obraztsov and Bocharova (16C) used a highly sensitive luminescence-kinetic method for the determination of iron in fairly pure water. Iron is determined in acetic acid medium by the method of addition using the catalytic effect of ferric iron on the oxidation reaction by hydrogen peroxide solution of a luminescent indicator, Stilbexone. The sensitivity of the method is 0.001 μg of iron per ml.

Nakagawa and Ohyagi (13C) used atomic absorption to determine manganese in river and hot-spring waters. The optimal parameters are given for the determination of 0 to 3 ppm of manganese. The presence of nickel, cobalt, silica, titanium, and copper decreases the absorbance of manganese, but this effect is eliminated by the addition of a salt of magnesium, calcium, strontium, barium, or lanthanum.

Wudzinska and Ponikowska (22C) employed a Technicon AutoAnalyzer to determine manganese in water. The method is based on the color reaction between manganese(IV) ions and formaldoxime in an alkaline medium. Measurement is made with a 50-mm cell at 480 nm. The range of the method is 0.01 to 0.3 mg per liter. No interferences are mentioned.

Angino, Hathaway, and Worman (2C) reported that the use of electron spin resonance spectroscopy to determine the presence and concentration of equilibrium and non-equilibrium species of manganese in natural waters has not been adequately investigated. The presence of manganese(II) in a stream water with pH 8.5 suggests that while the manganese(II) was in an equilibrium state with regard to the solubility product of manganese hydroxide and manganese carbonate, it was not in equilibrium relative to the oxidation-reduction conditions of the environment. Investigation of signal strength *vs.* manganese(II) concentration as a function of pH, oxygen, nitrogen, and carbon dioxide saturation suggests that $\text{Mn}(\text{H}_2\text{O})_6^{2+}$ is the major species present over the pH range of 2 to 6.3. A mixed precipitate of manganese(III) compounds was obtained at pH greater than 8.

Brewer and Spencer (3C) modified a formaldoxime-colorimetric method to determine manganese in reducing environment waters. Rapid and reproducible color development is achieved by using a mixed ammonia-formaldoxime reagent added singly to the sample. Precipitation is avoided by maintaining the pH in the range of 8.8 to 8.9. Sulfide and dissolved solids showed no interference. A small correction is made for interference due to iron.

Ohmori and Iwasaki (17C) used the persulfate method to determine manganese in water. If the chloride exceeds 8 grams per liter, the manganese is coprecipitated with ferric hydroxide and then determined by the persulfate method. If organic material is present, the persulfate is added in small portions and the solution heated for 1 hour.

Kessick, Vuceta, and Morgan (10C) described a spectrophotometric method to determine manganese dioxide in natural waters. The method is based on the reaction of oxidized manganese at pH 4 with 4,4',4''-methylidynetris (*N,N*-dimethylaniline) to give the dye crystal violet. The absorbance of the solution is measured at 591 nm. Based

on an absorbance of 0.01 using a 5-cm cell, the method is sensitive to 0.13 μg of tetravalent manganese in a 50-ml sample. The sensitivity may be increased approximately 4-fold by extracting the dye with isobutanol-benzene solvent.

Fujinaga and Takamatsu (6C) reported that the determination of chromium based on its catalysis in the reaction between cerium(IV) and tellurium(IV) in dilute sulfuric acid is hindered by manganese(II) and iodide. To avoid this interference, chromium 8-quinolinolate is formed at pH 3 by reflux, and is extracted into chloroform. After successive washing of the organic phase with a pH 1 buffer and 0.5N sodium hydroxide, the organic phase is evaporated to dryness, the residue treated with perchloric acid, and chromium then determined catalytically. Seitz, Suydam, and Hercules (19C) determined chromium(III) in natural waters by measuring chromium(III)-catalyzed light emission from luminol oxidation by peroxide. A continuous analysis is used and is diagrammed in the report. Light emission catalyzed by other metals is quenched by adding EDTA to form complexes that are not active as catalysts. Chromium(III)-EDTA complex is kinetically slow to form. The detection limit is 0.025 ppb. No sample pretreatment is required and analysis time is less than 30 minutes.

Matthews and Riley (11C) developed a neutron-activation procedure for the determination of rhenium in sea water. Rhenium is concentrated by anion exchange using Deacidite FF, eluted with 4M nitric acid, and then submitted to irradiation with thermal neutrons. Rhenium-186 is then separated from other induced activities by solvent extraction using methyl ethyl ketone and an almost specific ion-exchange process (Dowex 1) employing mixed aqueous-organic solvent (acetone-hydrochloric acid). Finally, rhenium is precipitated as tetraphenylarsonium perhenate for determination of chemical yield and counting. Precision was tested by replicate determination on a sample of sea water. The average rhenium content of the sample was 0.057 μg per liter with a coefficient of variation of $\pm 7\%$. Accuracy was examined by adding a known amount of rhenium to a sample. The recovery was 99%.

A fluorometric determination of cerium(III) at 350 nm by a 255 nm ultraviolet excitation is described by Shigematsu *et al.* (20C). Cerium is coprecipitated with ferric hydroxide from a 20-liter sample of sea water. The precipitate is separated and dissolved in 7N hydrochloric acid, and the iron removed by extraction with MIBK. Perchloric acid is added to the aqueous phase and the mixture evaporated to dryness. The residue is dissolved into a small amount of 0.01N perchloric acid and water, and subjected to reversed-phase ion-exchange using poly(vinyl chloride) resin beads treated with di-(2-ethylhexyl)-orthophosphoric acid. The effluent containing perchloric acid is again evaporated to dryness, 0.1N perchloric acid, titanium(III), and water added, and the resulting solution is used for the fluorometric determination of cerium.

A selective and sensitive photometric method for the determination of 0.1 to 15 mg of titanium per ml in highly mineralized waters of volcanic origin was developed by Nikitina and Basargin (14C). The sample is acidified with hydrochloric acid and hydrogen peroxide added. The solution is then boiled for 10 to 15 minutes and brought to volume. Titanium is precipitated as the hydroxide with ammonium hydroxide and the precipitate boiled with sulfuric acid and diluted to a specified volume with water. A 1% tichromin in 5% ascorbic acid solution is added to an aliquot of solution and the solution diluted to a specified volume with 5% sulfuric acid. After 5 to 10 minutes the

absorbance is measured at 443 nm. The authors claim the method to be 12 times more sensitive than the peroxide method.

COPPER, ZINC, LEAD, CADMIUM, NICKEL, COBALT, AND TIN

Miyanaga (21D) used atomic absorption for the determination of 0.01 to 0.05 mg of copper per liter in thermal-spring water. The samples are made up to 0.5M with hydrochloric acid prior to aspiration.

To determine the chemical forms of copper in sea water and the total copper concentration, Odier and Plichon (24D) utilized ac polarography. The shift of the half-wave potential of reduction of copper(II), which depends on the concentration of each ligand and on the pH, makes it possible to establish the formula and the formation constant of each complex. To determine the concentration of copper, the sample is acidified to pH 5 by passing pure carbon dioxide through the solution. Copper concentrations as low as 2.5 μg per liter can be detected.

Trachtenberg (38D) investigated a number of different ion-selective electrochemical sensors. The copper electrode which he used and described showed good response for 0.1 ppm total copper in sea water.

Obraztsov and Bocharova (23D) developed a luminescent method to determine copper in especially pure water. Copper is determined at 40 to 50 °C in an acetate-ammonia buffer of pH 9 by the method of additions using lumocupferron indicator. The sensitivity of the determination is 0.001 μg of copper per ml.

Rychkova, Dolmanova, and Peshkova (30D) described a catalytic method for the determination of copper based on the oxidation of hydroquinone with hydrogen peroxide in the presence of pyridine. The method requires a thorough purification of the reagents, and a constant temperature for the analyzed solutions and reagents.

Stiff (35D) determined copper in water spectrophotometrically with a specific colorimetric reagent 3-propyl-5-hydroxy-5-n-arabinotetrahydroxybutyl-3-thiazolidine-2-thione (PHTTT). The colorimetric reagent is added to a sample of water and after 10 minutes the absorbance is measured at 436 nm. Beer's law is followed up to 1000 μg per liter. Interference by other metals and by cyanide were examined; inhibition of complex formation by cyanide appeared to be almost quantitative. Mercury interferes, but its effect can be overcome by the addition of excess PHTTT reagent. For copper concentrations of 100 and 1000 μg per liter, the standard deviations were ± 1 and ± 8 μg per liter, respectively.

Akaiwa, Kawamoto, and Abe (2D) described a spectrophotometric method for the determination of 0.1 to 1.0 μg of copper per ml in natural water. Copper(II) is extracted with 2-thenoyltrifluoroacetone (TTA) in cyclohexane at pH 5.5. The residual TTA in the organic phase is removed by washing with sodium hydroxide solution containing pyridine which prevents the copper chelate from decomposing during the alkali treatment. The absorbance of the chelate in the organic phase is measured at 340 nm.

A spectrophotometric determination of microgram quantities of copper in water with 4-(2-pyridylazo)resorcinol (PAR) is reported by Kowalska (16D). A 100-ml aliquot of water is acidified with nitric acid, and hydrogen peroxide is added to oxidize ferrous to ferric iron. The PAR reagent is then added and the solution brought up to a pH of 6.5 to 7.7 with sodium hydroxide. After 50 minutes, the absorbance is measured at 510 nm.

Smith (33D) reported that the zinc dibenzylthiocarbamate method for estimating copper in water is not sig-

nificantly different from the bathocuproine method. Reproducibility at 2 concentrations of copper gave standard deviations of 0.001 and 0.002 ppm. Recovery ranged from 93 to 104%. Chloride concentrations greater than 10 ppm interfered with the recovery of copper. A new spectrophotometric method for the determination of copper in sea water is suggested by Balabanoff, Saelzer, and Bartolome (5D). Copper is concentrated and then precipitated with lead diethyldithiocarbamate. The precipitate is dissolved in a small volume of chloroform and the copper complex measured at 436 nm. Bitterlich and Woebling (6D) described a rapid visual field prospecting method for determining copper in water. The method is based on the formation of copper diethyldithiocarbamate at pH 7 and extraction of the complex with isobutanol. The brown alcoholic layer is visually compared to standards prepared similarly. The lower limit of detection is 5×10^{-9} gram of copper per ml. Baker, Teggin, and Hoffman (4D) also described a colorimetric method for determining copper which can be used in the field. Copper is concentrated by batch ion exchange using Dowex 50W-X8 and then triethylenetetramine is added. The solution is adjusted to pH 3.0 and the absorbance of the blue complex is measured. At 600 nm, 0.5 to 3.00 ppm of copper is determined; at 260 nm, 20 to 100 ppb is determined.

Sikorowska and Wichrowska (31D) compared several modifications of the dithizone extraction method and a direct colorimetric method for the determination of zinc in drinking water. The quantity of interfering substances and the use of masking agents to suppress the interferences were also investigated. The results showed that, for routine analysis, the best method is the two-color dithizone method. When the amount of zinc is between 1 and 5 mg per liter, the complex is extracted into chloroform; between 0 and 0.5 mg per liter, the complex is extracted with carbon tetrachloride. A survey of the literature on the determination of stable zinc in sea water is given by Rozhanskaya (29D). Sensitivities of the various methods are compared. A detailed description of the procedures using dithizone are presented with remarks on collection and storage of sea-water samples. The problems existing in the determination of different chemical forms of zinc are discussed. A suggestion is given for the determination of zinc-65 by coprecipitation with ferric hydroxide.

Evans and Sayers (10D) developed a visual estimation and a quantitative spectrophotometric determination of zinc in potable waters with 4-(2-thiazolylazo)resorcinol. Visual estimation is made by adding the reagent to standards and samples and, after 30 minutes, comparing the color of the samples against the standards. Spectrophotometric measurement in the range of 0 to 1 mg/l. is made at 530 nm in 1-cm cells. Possible interferences of iron and copper are discussed.

A rapid and sensitive atomic fluorescence method for the determination of 0.0004 to 0.01 ppm of zinc in boiler feed water is described by Marshall and Smith (19D). A hollow-cathode lamp, a vapor-discharge tube, and a microwave-excited electrodeless discharge tube were the high-intensity line sources examined. The electrodeless discharge tube is the best excitation source to stimulate the zinc fluorescence at 213.8 nm from a sample solution directly sprayed into an air-hydrogen flame.

Zirino (45D) and Zirino and Healy (46D) used pH-controlled differential anodic stripping voltammetry to determine zinc in ocean water.

Kisfaludi, Henry, and Jourdain (13D) described both direct and chelation-extraction procedures for determining lead in water by atomic absorption. For the determination

of 0.3 to 3 mg per liter, the water sample is acidified with hydrochloric acid and then aspirated directly into the air-propane flame of the spectrophotometer. For the determination of 0.003 to 0.5 mg per liter, the sample solution is acidified with HCl, the lead chelated with APDC, and extracted into isopropylacetone. The organic layer is then aspirated into an air-acetylene flame.

Spasojevic and Jovanovic (34D) determined lead in drinking water polarographically. Lead is coprecipitated with zinc phosphate, the solution filtered through a sintered glass crucible, and the precipitate dissolved in warm dilute nitric acid, and lead determined from -0.25 to 0.70 volts. In the range of 0 to 25 μ g, the recovery is 96 to 98%.

Skurnik-Sarig *et al.* (32D) used ultraviolet spectrophotometry at 270 nm to determine lead in natural saline waters. Ashizawa and Hosoya (3D) described a spectrophotometric method to determine traces of cadmium in water. Cadmium from a 1-liter sample is concentrated by batch ion exchange using Amberlite IRA-400. After removal of cadmium from the resin by eluting with acetic acid, the eluate is evaporated to a small volume, and the cadmium determined with dithizone using chloroform to extract the complex. The absorbance of the organic extract is measured at 517 nm.

Atomic absorption spectroscopy and its application to the determination of cadmium are summarized by Yamagata (42D). Sampling methods and analytical methods for river water, soil, and crops and standardization of analytical data are discussed. Yamamoto *et al.* (44D) used a chelation-extraction technique to determine cadmium in water by atomic absorption. Cadmium is chelated with APDC, extracted into MIBK, and then aspirated in the flame of the spectrophotometer. Tonouchi *et al.* (37D) also used atomic absorption combined with solvent extraction to determine cadmium in water. Cadmium is extracted from 0.5N hydrobromic acid solution with 1 volume percent trioctylmethylamine-ammonium chloride, or tri-*n*-octylamine, in MIBK, and cadmium determined in the organic extract after centrifugation.

Rampon and Cuvelier (28D) described an atomic absorption method to determine traces of nickel in sea water. Nickel, 1 μ g per liter or more, is extracted from 500 ml of water at pH 9 to 10 as the dimethylglyoximate with chloroform. The aqueous phase is extracted again with carbon tetrachloride and the organic extracts are combined and evaporated to dryness. The residue is dissolved in 1 ml of warm hydrochloric acid, water is added, and the solution evaporated to dryness. The residue is then dissolved and diluted to 5 ml with 0.1M nitric acid, and aspirated in an air-acetylene flame of the spectrophotometer.

Matueda and Ikeoka (20D) described a spectrophotometric-solvent extraction method to determine soluble nickel below 40 ppb in boiler water and feed water. Nickel is complexed with 1-(2-pyridylazo)-2-naphthol (PAN) and extracted into chloroform. The absorbance of the extract is measured at 570 nm. Insoluble nickel can be determined by the same procedure after it is fused with potassium persulfate and dissolved in water containing hydrochloric acid. Copper, iron, and zinc in concentrations less than 500 ppb do not interfere.

Okac and Nevoral (25D) determined traces of cobalt in water spectrophotometrically with 4-(2-pyridylazo)resorcinol (PAR). Cobalt is first concentrated on Dowex A-1 cation-exchange resin. Interfering cations, nickel and iron, are separated with Dowex 1-X10 anion-exchange resin using hydrochloric acid. The absorbance of the cobalt-

PAR complex at pH 8.3 (borate buffer) is measured at 510 nm. A spectrophotometric method for the determination of cobalt with nitroso-R salt in natural waters is described by Chuiko *et al.* (8D). The method is based on the coprecipitation of cobalt with magnesium hydroxide, reaction of cobalt with nitroso-R salt, and measurement of the absorbance at 530 nm.

Prik and Orlova (27D) used paper chromatography with rubeanic acid to separate traces of cobalt in tap and spring water from nickel and copper. The cobalt rebeanate spot is eluted with acid, the solution evaporated in the presence of hydrogen peroxide and nitric acid, and the dry residue analyzed catalytically. The method is based on the oxidation of pyrocatechol with hydrogen peroxide in the presence of *p*-phenetidine. Blazis *et al.* (7D) also described a method based on the catalytic oxidation of pyrocatechol-3,5-disulfonic acid by hydrogen peroxide for the determination of cobalt in sea suspensions. Techniques are given to remove interferences from other ions. The sensitivity of the method is 2 ng per ml.

Kodama and Tsubota (14D) reported an ion exchange-spectrophotometric method to determine tin in sea water. Tin is concentrated from a 500-ml sample on Dowex 1-X8 anion-exchange resin. The tin is then eluted with 2M nitric acid, evaporated, and redissolved. The ion-exchange and evaporation process is repeated. The residue is then dissolved in water, Pyrocatechol Violet and cetyltrimethylammonium bromide solution are added, the pH is adjusted to 1.6 with 1M sodium acetate, and the solution diluted to 10 ml. After 15 minutes, the absorbance is measured at 662 nm. The concentration of tin in waters from the North Pacific ranged from 0.6 to 2.0 μ g per liter, which was similar to results obtained by activation analysis.

Procedures are described by Paus (26D) for the determination of copper, lead, zinc, cadmium, cobalt, and nickel in natural waters by atomic absorption. Chelation-extraction with APDC-MIBK was found useful. To improve the detection limits, a preconcentration technique employing Dowex A-1 was used. The sampling boat and heated graphite atomizer were also used to determine the above metals directly, eliminating the need to preconcentrate or extract. He stated that with the different techniques it is possible to obtain valuable information about the forms in which the metals exist as well as their concentrations. Kuwata, Hisatomi, and Hasegawa (17D) applied atomic absorption to the determination of cadmium and copper as low as 0.05 and 0.5 ppb, respectively, in river and sea water. Cadmium and copper are chelated with sodium diethyldithiocarbamate at pH 9 and extracted with MIBK. They used MIBK solutions of acetylacetonate-Cd and -Cu complexes as standard solutions for constructing analytical curves, because the complexes of diethyldithiocarbamate with standards of cadmium and copper were relatively unstable. Kanke *et al.* (12D) used dithizone-nitrobenzene extraction and atomic absorption spectrophotometry to determine ppb levels of cadmium, zinc, lead, and copper in water. These elements are extracted from 50 to 1000 ml of water with 25 ml of 0.03% dithizone solution in nitrobenzene at pH 5.3. The extract is then aspirated for each of the elements in an air-acetylene flame. Frei *et al.* (11D) investigated the extraction of pyridine-2-aldehyde 2-quinolyldihydrazone chelates of cadmium, cobalt, copper, nickel, and zinc into isoamyl alcohol and MIBK as a basis for the determination of these metals by atomic absorption. Below pH 6 the extraction is enhanced by the addition of perchlorate. Procedures are given for these metals and then applied to the analysis of

tap water for cadmium, copper, and zinc. Yamamoto *et al.* (43D) described three different solvent extraction methods for determining traces of cadmium, zinc, lead, and copper in water by atomic absorption.

Wieclawski (40D) checked the usefulness of a dithizone column for separating and concentrating lead, cadmium, zinc, manganese, copper and cobalt from lake waters. The collection of water samples, the preparation of the dithizone column, and the determination of the trace metals by polarography are discussed.

Supatashvili and Karsanidze (36D) developed a procedure for successive extraction-photometric determination of lead, copper, nickel, and zinc in one water sample.

A combination of square wave polarography with anodic stripping polarography was applied by Komatsu, Matsueda, and Kakiyama (15D) to the simultaneous determination of zinc, cadmium, lead, and copper in mine water. The effect of diverse anions was also studied. Mal'kov *et al.* (18D) used anodic stripping polarography to determine lead and copper simultaneously in natural and waste waters. The potentials of the anodic peaks of lead and copper were -0.42 and -0.19 V, respectively, and the heights of the peaks were in linear relation to concentration in the region of 0.01 to 60 ng per ml. Zirino and Healy (47D) studied the state and concentration of zinc, cadmium, lead, and copper in the parts-per-billion range in fresh and saline waters by anodic stripping voltammetry. Increased sensitivity is achieved by opposing two hanging mercury drop electrodes in the same cell which eliminates the large residual current and allows maximum signal amplification. Detailed schematic diagrams of both the assembly for differential anodic-stripping voltammetry and the cell are given. A polarographic technique with a sensitivity of 1 μ g per liter is described by Usheva and Gerei (39D) for the determination of zinc and lead in ground and surface waters. The technique is based on coprecipitation of lead and zinc with calcium carbonate from which the lead is separated out in the presence of ammonia and determined with a calcium nitrate electrode; the zinc is determined from ammonium chloride solution. Abdullah and Royle (1D) determined copper, lead, cadmium, nickel, zinc, and cobalt in natural waters by pulse polarography after preconcentration on chelating resin Chelex-100 in its calcium form. The eluate from the resin contains sufficient calcium to act also as a supporting electrolyte. The determination of the six metals is made on a single aliquot of eluate. Muzzarelli and Sipos (22D) used anodic stripping voltammetry with a composite graphite-mercury electrode to demonstrate that chitosan can collect naturally occurring zinc, cadmium, lead, and copper from sea water with high yields. Chitosan columns are used to preconcentrate these metals from 3 liters of sea water. The four elements are selectively eluted with electrolytes suitable for conventional polarography. A relatively simple and inexpensive self-contained device designed as a three-electrode polarograph for field determination of lead, cadmium, zinc, and iron in natural waters has been developed and tested by Coleman, Van Atta, and Klatt (9D). The analyses for these four metals may be completed in less than an hour with results accurate to within ± 0.1 ppm. A detailed description, schematic diagrams, and operating instructions are available from the authors.

Yagodnitsyn (41D) described a photometric determination of zinc and lead in water by means of sulfarsazen. A detailed procedure is given. Details are also given for the determination of manganese and copper, if present.

MERCURY, SILVER, AND GOLD

Brooks and Wolfram (7E) reviewed and compared methods for the determination of traces of mercury in environmental samples and other materials. The preparation of equipment and samples, reduction of blank values, and the elimination of interferences are discussed. Fourteen references are given. A review with 10 references on man-made mercury pollution above background levels, and a method for the determination of mercury in water and other media have been published by Klein (19E). The basic instrument requirements are discussed. Mercury background levels compiled from various sources are given.

Joensuu (16E) built a simple and inexpensive accessory to an atomic absorption spectrophotometer which permits the determination of 1 to 100 ng of mercury in water and other materials. For water, the mercury is reduced with stannous chloride and aerated into a furnace where mercury is amalgamated with gold. The mercury is then removed from the gold by heat, and the mercury vapor determined. Ramirez-Munoz (31E) described in detail the general operation of the nonflame mercury analyzer and the preparation of reagents for mercury determinations at the ppb levels. Analytical procedures for water are included. Kempf and Kneisel (18E) used a commercial mercury vapor analyzer to determine mercury in drinking water. Organic mercury compounds are oxidized with bromine, the excess bromine is reduced with hydroxylamine hydrochloride, and stannous chloride is added to reduce the mercury. The sample is cooled with ice and a vigorous stream of air is blown through it to pass the mercury vapor into the absorption cell. The method is suitable for 0.1 to 0.6 μ g per liter.

Kopp, Longbottom, and Lobring (20E) made a detailed study of the cold-vapor method for determining mercury in water. They evaluated a number of variables including effect of heat, air flow, and water vapor. Oxidation procedures to break down organic mercury compounds were evaluated. Two-hour heating at 95 °C in the presence of potassium permanganate and potassium persulfate is recommended to ensure complete breakdown of organic mercury compounds. Details are given on instrumentation, reagent preparation, calibration, and the procedure. Cavallaro and Elli (9E) also described a flameless atomic absorption method for the determination of mercury in water and other materials. They studied the effects of temperature, time variations in the oxidation step, and possible interferences. Mercury compounds are oxidized with sulfuric acid-nitric acid and potassium permanganate at 50 °C followed by reduction of the excess oxidants with hydroxylamine hydrochloride. Omang (29E) used a flameless atomic absorption method to determine mercury concentrations as low as 20 ng per liter in water. A 1-liter sample is acidified with 10 ml of 1 + 1 sulfuric acid. Five milliliters of potassium permanganate are added, and the solution is left to stand for 24 hours at room temperature to decompose organic compounds containing mercury and to avoid adsorption on the walls of the container. The recommended concentration of permanganate is shown to decompose methyl mercury compounds completely. After digestion, a 50-ml portion of the sample is added to a mixture of distilled water, sulfuric acid, and stannous chloride which has previously been aerated to remove traces of mercury contamination, and mercury is then determined.

Krause *et al.* (22E) reported on a flameless method to

determine mercury in water. The method, as the ones above, depends on the use of stannous chloride to release the mercury. The mercury vapor is then passed through water, and finally to an optical cell mercury meter for measurement. In this procedure, there is no interference from water vapor at 68 to 70 °F or from carbon dioxide. Concentrations below 1 ppb can be detected by adjusting the aliquot of the sample. Hwang, Ullucci, and Malenfant (15E) determined mercury in water and other materials by a flameless atomic absorption technique. Stannous chloride or hydrazine hydrate is added to the sample to reduce the mercury. The sensitivity is reported to be 2×10^{-10} gram with a relative standard deviation of $\pm 5\%$ at the 2×10^{-7} gram level.

A method for the simultaneous determination of methyl and ethyl mercury chloride and phenylmercuric acetate in water is described by Umezaki and Iwatomoto (38E). In 2N sulfuric acid solution containing approximately 0.4M chloride, only inorganic mercury is reduced by stannous chloride, while in 1N sodium hydroxide solution containing traces of copper(II), both inorganic and organic mercury are reduced. Organic mercury is calculated by difference. The range of the determination is 0.2 to 7 ppb with a relative standard deviation of 2% at 5 ppb. Sulfide, thiosulfate, and silver in alkaline solution interfere.

Similar methods for the determination of submicrogram amounts of mercury in lake water by combination of concentration by dithizone extraction and flameless atomic absorption were developed by Chau and Saitoh (10E, 11E). Mercury is extracted from 500 ml of sample with dithizone in chloroform and back-extracted with hydrochloric acid. Stannous chloride is then added to the acid extract, and the mercury vapor determined by atomic absorption. The sensitivity is 0.008 μg per liter. The standard deviations for lake-water samples containing 0.478 and 0.048 μg of mercury per liter are 0.0087 and 0.0042, respectively.

Doherty and Dorsett (13E) reported that ionic and organic forms of mercury in water at 0.1 to 10 ppb can be determined with a precision and accuracy of $\pm 10\%$ by electrodeposition. Mercury is electrodeposited onto a copper coil at a potential of 3 volts. When electrodeposition is complete, the coil is removed from the sample, rinsed with water, acetone, and air dried. The mercury on the coil is then determined by atomic absorption. They reported that no separate chemical oxidation is required to decompose organic mercury compounds. Duffer (14E) described a similar method for determining both inorganic and organically combined mercury by electrodeposition followed by atomic absorption. Solutions containing 1.0 and 10.0 ppb mercury were analyzed with absolute errors of 0 and 0.2 ppb, respectively, and with standard deviations of 0.1 and 0.2 ppb.

Methods are described by Moffitt and Kupel (25E, 26E) for the determination of mercury in water. Organically bound mercury is oxidized with nitric acid and cysteine hydrochloride and reduced to the elemental state with stannous chloride. The mercury is then driven by an air current through especially impregnated charcoal for approximately 2 minutes and the charcoal samples are analyzed directly with an atomic absorption sampling-boat assembly. The charcoal samples may be stored for later analysis. The minimum detectable quantity of mercury is 0.02 μg .

Bailey and Lo (5E) developed an automated atomic absorption method for the determination of mercury. Com-

ponents of the system include an atomic absorption spectrophotometer with automatic sampler, curve corrector, digital indicator, and digital printer, together with an AutoAnalyzer pump and associated glassware and tubing. Basically, the procedure is the same as the manual version, in that the sample is digested with permanganate to destroy organic matter followed by hydroxylamine hydrochloride and, finally, by stannous chloride. The coefficients of variations for duplicate results at the 2-, 4-, 6-, and 8-ppb levels are 7.6, 6.1, 2.3, and 1.6%, respectively.

Muscat, Vickers, and Andren (28E) and Muscat and Vickers (27E) described atomic fluorescence spectrophotometric methods for the determination of mercury in water. The typical reduction-aeration procedure is described in the first reference. In the latter reference, a silver amalgamator is used to collect mercury prior to the fluorescence measurement. The mercury is released for the final measurement step by heating. The amalgamation procedure has advantages over the reduction aeration procedure: First, all the mercury is present in the cell at the same time with a 10-fold increase in signal over the closed system; second, with the closed system it is difficult to use a carrier gas other than air, but with the amalgamator, argon can be used which results in approximately a 100-fold increase in signal; third, the amalgamator serves to separate mercury from potential interferants; and, fourth, less sample is required. As little as 0.6 ng of mercury can be determined.

Longbottom (24E) described an inexpensive mercury-specific gas chromatographic detector to determine organic mercury compounds in water and other materials. A commercially available mercury analyzer is adapted for use as the detector, and mercury is reduced to its elemental state by combustion in a flame. The detection limit is 0.02 ng of dimethylmercury. With injections of 5-ng amounts of diethyl mercury, the relative standard deviation is 1.72%.

April and Hume (4E) described a capacitively coupled radiofrequency plasma torch in conjunction with a mercury reduction-vaporization cell for emission spectrometric determination of mercury in water. Data presented suggest a limit of detection of 2 ng of mercury with a working range of 10 ng to 10 μg in 10 ml of sample. Lichte and Skogerboe (23E) reported that mercury in water and other materials can be determined by emission spectrometry at 2536 Å with microwave plasma excitation after reduction and vaporization of mercury by previously discussed procedures. The evolved mercury is carried by argon to the plasma. Results appear to be accurate to $\pm 10\%$ or better. They concluded that the absolute sensitivity of the microwave-excitation technique is approximately 2 orders of magnitude better than those reported by April and Hume (4E), and users of atomic absorption techniques. The detection limit is 6×10^{-11} gram.

A spectral technique based on the extraction of mercury from water with a 0.004% solution of dithizone in carbon tetrachloride is described by Shater *et al.* (35E). At mercury concentrations of 0.001 to 0.1 μg per liter, the method is accurate to $\pm 18\%$. By using 20 to 100 volumes of solvent, 99.1 to 99.8% of the mercury is extracted from the water. The mercury-dithizonate is adsorbed on carbon, heated to 400 to 500 °C, which volatilizes the mercury; and the mercury is determined spectroscopically at 2536.5 Å.

Weiss and Crozier (39E) described a neutron activation method to determine mercury in sea water which requires a

0.5-liter of sample. To reduce exposure to the levels of induced radioactivities which would develop from sea salts, the mercury is coprecipitated with copper sulfide from acid solution prior to neutron irradiation. The sensitivity of the procedure, which is defined by a count rate in excess of 3 standard deviations from the background 7 days after the irradiation, is 4 ng. A neutron activation analysis method was developed by Becknell, Marsh, and Allie (6E) for the determination of mercury at levels of 0.05 to 250 μg and was applied to a variety of natural waters, the compositions of which varied from 0.03 to 6.6 ppb. A preirradiation concentration of mercury from 500-ml aliquots of sample is accomplished through the use of SB-2 anion-exchange resin-loaded filter disks. Hydrochloric acid is added to the sample and mercury, as HgCl_4^{-2} , is removed from solution and concentrated by ion exchange on the filter disks which are then irradiated. The mercury content is determined by comparing to standards using the 77-KeV gamma ray from the decay of ^{197}Hg . In solutions containing up to 250 μg of mercury, 100% of the mercury is removed by ion exchange. A chlorine treatment is used to decompose organic mercury compounds.

Carter and Sites (8E) used a spark-source mass spectrometer to determine mercury as well as cadmium and zirconium in water. A probe-type sample changer was added to the spectrometer which enabled a rapid determination of ppb levels of mercury by isotope dilution.

Kartushinskaya and Kolpakova (17E) determined nanogram amounts of mercury in water by anodic stripping voltammetry. The method is based on electrochemical deposition of mercury on a graphite anode from water acidified to pH 1 with nitric acid followed by an electrolytic dissolution of the film in the presence of potassium nitrate and potassium thiocyanate. Mercury (1×10^{-9} gram per ml) is determined with an accuracy of 20% in waters with salinities less than 5 grams per liter.

Osajima *et al.* (30E) described a colorimetric procedure for the determination of mercury in sea water. Mercury is extracted from an acidified sample with dithizone-carbon tetrachloride solution and then back-extracted with 0.1N sulfuric acid containing a small amount of 10% potassium iodide solution. The pH of the aqueous layer is raised to 6 to 6.5, and mercury again is extracted with dithizone-carbon tetrachloride. The organic layer is then shaken with 0.1N sulfuric acid and the absorption of the clear organic layer measured at 490 nm. Simultaneous separation and determination of organic and inorganic mercury compounds in water by dithizone extraction was developed by Suenaga and Shiraishi (36E). The dithizonate of both organic and inorganic mercury salts is quantitatively separated with acidic potassium iodide solution. Organic mercury dithizonate is further decomposed to inorganic mercury with acidic potassium permanganate solution. The effect of chlorides, copper, and zinc is negligible. Mercury concentrations between 0.001 and 0.02 ppm can be determined.

Coyne and Collins (12E) investigated the stability of dilute mercury solutions stored in polyethylene containers and documented the rate of loss under routine conditions of sample collection and storage. Measurement is made by atomic absorption spectrophotometry. The investigation indicated that samples acidified to pH 1 with nitric acid seemed to be the only effective preservative. The effectiveness is realized only if the acid is present in the container before the sample is introduced, so that the sample is acidified immediately upon entering the container.

Wichrowska (41E) compared the rhodanine and dithi-

zone methods for determining silver in drinking water and tested various methods for the removal or masking of interfering elements. For routine analysis in the range of 0.005 to 0.1 mg per liter, the rhodanine method in ethanol or acetone is recommended. Preliminary removal of interfering elements is accomplished with a tellurium carrier in the presence of stannous chloride or by evaporation with nitric acid.

An atomic absorption method was developed by Talaev and Mironova (37E) for the determination of 10^{-4} to $10^{-3}\%$ silver in industrial waters. A photometer without a filter is used. The source is an electrodeless discharge silver lamp, while the source of radiation is a silver hollow cathode lamp. The sensitivity is 1.5 μg of silver per ml, and the relative standard deviation is 11%.

Abdullaev *et al.* (3E) used a simultaneous neutron irradiation of a standard containing 10^{-6} gram of silver per liter and a sample concentrated by evaporation to determine silver in water. The solutions are irradiated at a flux of $1.8 \times 10^{13}n$ per cm^2 -second for 30 hours. The γ -spectrometric analysis is made following a 2-month cooling period with a 40×40 mm NaI(Tl) detector and a AI-100 analyzer.

A method based on fluorescence quenching for the determination of silver in pond water is described by White and Lisk (40E). The method is sensitive to approximately 0.02 ppm of silver.

A spectrochemical determination of gold in natural water is described by Korganova and Polyakov (21E). Gold from a liter sample is concentrated on 500 mg of activated ground coal at the water source. The coal is added to the sample, the mixture is shaken for 2 minutes, and then left to stand for 15 minutes. The coal is filtered off, dried, and ashed together with the filter at 700 to 750 $^{\circ}\text{C}$. Before ashing, an internal germanium standard is added. Samples and standards are placed in a lower electrode anode channel, and the spectra excited with 12 amperes for 30 seconds. Gold 2676 and 2428 \AA and germanium 2740 \AA lines are photometrically measured. The mean square error is $\pm 14.1\%$ with 0.05 to 0.1 μg of gold per liter and $\pm 39.6\%$ with 0.01 μg per liter.

Schiller and Cook (32E, 33E) and Schiller, Cook, and Beswick (34E) used nondestructive activation analysis for the determination of gold in natural waters. Gold is pre-concentrated from water samples on a lead sulfide carrier. The concentrates are irradiated for 8 hours in a neutron flux of 1.75×10^{12} neutron per cm^2 per second followed by γ -spectrometric measurement. Abdullaev, Zakhidov, and Umirbekov (1E) also described a neutron activation analysis of gold in water. Details of the procedure are given. Gold concentrations of 0.5 to 6.27×10^{-11} gram per ml were determined in 10 water samples with an error less than 3%. Abdullaev *et al.* (2E) also determined both silver and gold simultaneously by neutron activation.

VANADIUM, MOLYBDENUM, SCANDIUM, ANTIMONY, BISMUTH, URANIUM, AND RARE EARTHS

Vrbsky and Fogl (24F) described a method for the determination of vanadium in natural waters. The method is based on the oxidation of *N,N*-diethyl-*p*-phenylenediamine by potassium chlorate with vanadate as catalyst. The red oxidation product is measured photometrically. Reaction time, pH, temperature, activators, and range of concentration were investigated. Citric acid mixed with potassium citrate provided an effective activator and a

suitable pH. Reproducibility depended mainly on reaction time and temperature. Oxidation in a boiling-water bath for 10 minutes produced good results. The linear dependence of the absorbance was established for the range of 0.2 to 1.2 μg per 15 ml. Sulfate and monohydrogen phosphate interfered in concentrations higher by a factor of 1000 than that of vanadium. Ferric iron, copper, and ammonium ions affected the absorbance of the red color.

To determine molybdenum in water, Wenger and Hoegl (25F) extracted molybdenum, as the α -benzoin oxime complex, with chloroform. The complex is then destroyed with sulfuric and perchloric acids and molybdenum reacted with toluene-3,4-dithiol. The complex is extracted with isoamyl acetate and molybdenum determined spectrophotometrically at 680 nm. The detection limit is approximately 0.1 μg .

Kim and Zeitlin (6F) studied the use of thorium hydroxide as a collector of traces of molybdenum from sea water before spectrophotometric determination as the thiocyanate complex. This collector offers advantages over ferric hydroxide, which include enhanced sensitivity and accuracy, and elimination of the need for masking iron since thorium ion does not react with thiocyanate. The thorium hydroxide coprecipitate, also, is less bulky than ferric hydroxide and easier to handle and filter. At the optimal pH of 6, the recovery is 99.5%. Kim and Zeitlin (8F) also described a rapid adsorbing colloid flotation method for the separation of molybdenum from sea water. Molybdenum is separated in 5 minutes using colloidal ferric hydroxide, sodium dodecyl sulfate surfactant, and compressed air. The mean recovery for 11.7 μg added molybdenum was 95.3% with a relative standard deviation of 2.6% based on spectrophotometric analysis of the separated molybdenum.

Kim (5F) used neutron activation for the determination of scandium in natural waters. Scandium is precipitated from 5 to 10 ml of sample with ammonia. The precipitate is dried, wrapped in foil, and subjected to irradiation for 10 hours in a quartz ampoule by 1.8×10^{13} neutrons per cm^2 second. Samples with low concentrations of trace elements are stored for 10 to 15 days before they are analyzed; samples containing high concentrations are stored for 20 to 30 days. The content of scandium is calculated according to a 1.12-MeV peak by comparison with a seawater standard containing 4×10^{-11} gram of scandium per liter.

A neutron-activation method is described by Ryabinin *et al.* (20F) for the determination of antimony in sea water. Antimony is coprecipitated with ferric hydroxide and irradiated at 1.8×10^{13} neutrons per cm^2 second for 1 to 2 hours in quartz ampoules shielded by metallic cadmium. The antimony is then measured in a single-crystal NaI(Tl) γ -spectrometer. Ryabinin and Romanov (19F), in an earlier publication, described a neutron activation method to determine antimony in sea water.

Mal'kov and Fedoseeva (10F) determined 0.02 to 0.10 mg of bismuth per ml in natural waters by anodic stripping polarography. The bismuth is separated from calcium, magnesium, iron, aluminum, antimony, and copper by extraction from an ammoniacal solution containing sulfasalicylic acid and Complexon III with sodium diethyldithiocarbamate in chloroform and is then back-extracted into hydrochloric acid. Bismuth is determined in the acid phase by anodic stripping analysis with a mercury-graphite electrode. The error at 0.02 and 0.10 mg per liter is 20% and 5 to 10%, respectively. Mal'kov, Fedoseeva, and Stromberg (11F) described a polarographic meth-

od for the determination of antimony and bismuth. All pentavalent antimony species are converted to the cationic antimony pentasulfide form which reacts quantitatively with sodium diethyldithiocarbamate as in the previously described publication. Zinc diethyldithiocarbamate is used for the separation of bismuth from antimony and the majority of interfering ions. Anodic stripping analysis is used for measuring both ions. The sensitivity of the method is 0.01 μg of antimony and bismuth per ml. The mean relative error is $\pm 20\%$.

A colloid flotation technique is described by Kim and Zeitlin (7F) for the separation of uranium, present as the stable tricarbonatouranyl anion, from sea water. The uranium is coprecipitated with ferric hydroxide at $\text{pH } 6.7 \pm 0.1$. Sodium dodecyl sulfate is then added, and air is bubbled through the sample. Within 2 to 3 minutes, the colloidal ferric hydroxide enriched with uranium floats to the surface as a stable froth which is removed. Uranium is then determined spectrophotometrically using Rhodamine B. Average recovery of uranium from sea water by this method is 82%. An improved method for the separation and determination of uranium in sea water is described by Leung, Kim, and Zeitlin (9F). The flotation technique described above is used; however, the uranium is absorbed on thorium hydroxide instead of ferric hydroxide, and uranium determined fluorometrically at 575 nm with Rhodamine B. Average recovery for 2 to 6 μg of uranium added to 500 ml of sea water by this technique is 90%. Takai and Yamabe (23F) studied coprecipitation, adsorption, ion flotation, and solvent extraction techniques to separate and concentrate uranium in sea water.

A spectrophotometric procedure is outlined by Florence, Johnson, and Farrar (3F) for the determination of uranium in water. Uranium is complexed with 0.05% 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol. The precision of the method is $\pm 0.5\%$ at 50 μg uranium. An extraction photometric method for the determination of uranium(IV) in mine waters was developed by Shchemeleva, Stepenenko, and Kovalenko (22F). The method is based on the reaction of uranyl benzoate complex with Brilliant Green and extraction of the compound with toluene. Beer's law is obeyed in the concentration range of 0.2 to 10.7 μg per ml. Ascorbic acid eliminates ferric iron interference. A methyl blue extraction-photometric method for the determination of uranium in natural water is described by Shchemeleva, Bagdasarov, and Stepenenko (21F). Methyl blue forms a complex with uranyl benzoate which is then extracted with 1:1 benzene-chloroform. The complex has an absorbance maximum at 650 nm. Ferric iron interference is avoided by reduction with ascorbic acid. The sensitivity of the method is 0.2 μg of uranyl ion per ml.

Bilal *et al.* (2F) determined uranium in sea water by neutron activation. The sample is irradiated at 5.1×10^{11} neutrons per cm^2 second followed by γ -spectrometry with a Ge(Li) detector and a multichannel analyzer. Uranium is detectable in concentrations of less than 0.05 ppb. Uranium in water and other materials was determined by Nass, Molinski, and Kramer (14F) by delayed neutron emission counting, with BF_3 tube detectors embedded in a paraffin moderator, 20 seconds after a 30-second irradiation in a 3×10^{13} neutrons per cm^2 second thermal neutron flux. A 0.1 ppm of uranium can be determined.

Bertrand and Linn (1F) described a radioisotope energy dispersive X-ray determination of uranium in ground waters, mine waters, waste waters, and brines. The detection limit is 0.2 ppm. The method utilizes barium sulfate precipitate as a collector for uranium(IV). The 22-keV silver

X-rays from the ^{109}Cd radioisotope source are utilized for excitation of the uranium *L*-series fluorescence. The single-channel analyzer is adjusted to discriminate against all pulses except those from the 13.6-keV uranium *L*-alpha photopeak.

Hashimoto (4F) determined the uranium content in sea water by counting the fission tracks on muscovite immersed in a condensed aqueous solution during the reactor neutron irradiation. Uranium is first coprecipitated with aluminum phosphate and then dissolved in a small volume of dilute nitric acid; an aliquot of this solution in a quartz ampoule is taken up by a piece of muscovite and irradiated. The muscovite is then etched with hydrofluoric acid, and a microphotograph is taken. The fission fragment tracks are counted and compared with standards. Matsuda *et al.* (12F) also described a fission-track technique for the determination of uranium in silicate minerals. They mentioned that the procedure, with a slight modification, is applicable to the determination of uranium in natural waters.

A method for the fluorometric determination of uranium in water is described by Pakalns (17F). A precision of $\pm 5\%$ was obtained at all uranium concentrations.

Nagatsuka, Suzuki, and Nakajima (13F) used neutron activation to determine nine rare-earth elements in surface and coastal sea-water samples. The lanthanide elements are coprecipitated with ferric hydroxide and then irradiated. This group of elements is then separated with lanthanum oxalate and measured by γ -spectrometry with a Ge(Li) detector.

An activation method was studied by Oda (15F, 16F) for the determination of rare-earth elements in hot-spring waters. The rare-earth elements in a 10-liter sample are coprecipitated with calcium oxalate, the precipitate ignited, and then irradiated at a neutron flux of 2×10^{13} per cm^2 second for 5 hours. A lithium-drifted germanium high-resolution semiconductor detector is used for the measurements.

An extraction-spectrophotometric determination of total rare-earth elements in natural waters is described by Poluektov *et al.* (18F). The method is based on the extraction of their mixed ligand complexes with Arsenazo III and diphenylguanidine into butanol. Preliminary separation of other metal ions by extraction using ammonium thiocyanate and diantipyrinylmethane in chloroform is necessary. Sensitivity is $0.007 \mu\text{g}$ rare earths per ml.

BORON, SELENIUM, ARSENIC, PHOSPHORUS, AND SILICA

Liebich, Monnier, and Marcantonatos (13G) used the spectrofluorometric 2-hydroxy-4-methoxy-4'-chlorobenzophenone method to determine boron in natural water. After adding an acid solution of the above reagent, the solution is heated for 40 minutes at 70°C . The solution is then cooled for 30 minutes and the fluorescence measured at 490 nm using an excitation of 365 nm.

Nicholson (20G) determined boron in saline waters spectrophotometrically with Nile Blue A. Chloride concentrations greater than 100 mg per liter interfere, and the chloride in the sample is removed by precipitation with silver sulfate. The boron in solution is then converted to the fluoroborate ion. Nile Blue A reagent is added, and the complex is extracted with *o*-dichlorobenzene. The absorbance of the organic extract is measured at 647 nm. A modified dianthrimide method is described by Levinson (11G) which is capable of detecting as little as 0.01 mg of

boron per liter in river waters. The modified procedure eliminates the overnight digestion with sulfuric acid in which boron may be lost. The water is rapidly evaporated to dryness after adding a few drops of saturated aqueous calcium hydroxide which fixes the boron. Boron is then determined spectrophotometrically after the addition of dianthrimide dissolved in concentrated sulfuric acid.

Bailey and Lo (4G) described a flame-photometric method for the determination of boron in potable waters. The determination is based on measuring the chemiluminescence of the molecular species BO_x in either an air-hydrogen or air-acetylene flame. The detection limits with these flames are 0.004 and 0.06 ppm, respectively. Potential interferences can be removed by ion-exchange chromatography.

Alexandru and Tufoi (2G) determined boron in water by an isotopic-dilution method using a parallel-beam mass spectrometer. The sample is diluted with ^{11}B and deposited on a tungsten blade inside the thermal source of the spectrometer.

Reti (23G) developed two methods for the determination of borates in deep water. Each consists of the same steps, but uses different reagents. The steps are as follows: 1) adjust to a definite pH; 2) add an activator reagent of the same pH; 3) titrate with carbonate-free 0.02N barium hydroxide solution to the same pH, using potentiometric end-point control; and 4) repeat steps 2 and 3 until there is no further change of pH upon addition of the activator. Invert sugar or mannitol is used as activator. A potentiometric method suitable for laboratory and on-board ship determination of boron in sea water is described by Sheremet'eva (28G).

A review with nine references is given by Pletnikova (21G) on the determination of selenium in water.

To determine selenium in waste waters, Baird, Pourian, and Gabrielian (5G) employed a flameless atomic absorption technique which utilizes a carbon rod for atomization. A nitric acid-perchloric acid digestion is used to oxidize organic material and solubilize the selenium before injection of 0.5 μl of sample into the carbon rod. Instrument settings are given. Replicate analyses of samples containing approximately 10 μg of selenium per liter gave a relative standard deviation of 6.8%.

To determine selenium in water in the range of 0.02 to 0.10 ppm, Mesman and Doppelmayer (17G) used a methylene blue method with photometric monitoring of the rate of bleaching of the methylene blue solution by a selenium-catalyzed reduction with sodium sulfide. The authors developed a simple photometric device to increase the efficiency of the technique.

Abdullaev, Nishanov, and Sharipov (1G) applied neutron activation analysis to determine selenium in natural waters at concentrations greater than 1×10^{-10} gram per ml with accuracies of ± 10 to 20%. An aliquot of sample, 50 to 100 ml, is evaporated to dryness and irradiated for 8 to 10 hours at a flux of 1.8×10^{13} neutrons per cm^2 second. Following a cooling-off period of 2 to 3 days, the irradiated sample is mixed with 10 mg of selenium carrier and dissolved in a mixture of nitric and hydrochloric acids. The ^{76}Se is precipitated out with hydrazine hydrochloride and analyzed by γ -spectrometry.

Yamamoto *et al.* (33G) described a spectrophotometric method and an indirect atomic absorption method to determine 1 ppb or more of arsenic in water. In the spectrophotometric method, arsine is evolved into a silver diethyldithiocarbamate-brucine-chloroform solution which they claim is superior to silver diethyldithiocarbamate-

pyridine solution. In the atomic absorption method, arsenic is reacted with molybdate reagent, and the molybdoarsenic acid is extracted with MIBK, and molybdenum determined using a nitrous oxide-acetylene flame.

An indirect polarographic method suitable for determining arsenates in mineral waters in the concentration range of 5 to 100 mg per liter was reported by Rozanski (25G). Arsenates are preconcentrated by coprecipitation with ferric hydroxide. The precipitate is dissolved in hydrochloric acid, buffered, and a known amount of uranylacetate added to precipitate uranylarsenate. Excess uranyl ion is then determined polarographically.

An emission spectrographic method for the determination of traces of arsenic in water has been developed by Lichte and Skogerboe (12G). An arsine generator is coupled directly to a microwave-induced excitation plasma. Less than 5 ppb of arsenic can be determined when a 1-ml sample is used.

Reymont and Dubouis (24G) developed an X-ray fluorescence method to determine 1 μ g of arsenic or less. Arsenic is precipitated as the sulfide from a 3.7M perchloric acid solution with a 2% thioacetamide solution containing 200 μ g of molybdenum as coprecipitant. The sulfide precipitation from highly acidic solution minimizes interferences. The precipitate is filtered and the arsenic $K\alpha$ line is counted. Tanaka (31G) simultaneously determined arsenic and lead in water by X-ray fluorescence. The lead and arsenic pyrrolidine dithiocarbamate chelates are extracted at pH 3.0 to 6.5 into chloroform. The extract is then spotted on an ion-exchange paper, and the intensities of arsenic $K\alpha$ and lead $L\beta$ lines are measured. The lead $K\beta$ intensity is subtracted from the arsenic $K\alpha$ intensity. Zinc, copper, iron, and cadmium did not interfere. The relative standard deviation is less than 6%.

A neutron activation method for the determination of arsenic in sea water is described by Ryabinin *et al.* (27G). Arsenic is coprecipitated with ferric hydroxide, placed in quartz ampoules shielded by metallic cadmium, and irradiated at 1.8×10^{13} neutrons per cm^2 second for 1 to 2 hours. The arsenic activity is then measured in a single-crystal NaI(Tl) γ -spectrometer.

Johnson (9G) reported that arsenate in natural waters can be measured by a slight modification of the phosphomolybdate method for phosphate. Arsenate forms a blue complex with molybdate, like phosphate, while arsenite does not. Arsenate is reduced to arsenite by thiosulfate in an acidic medium and the difference in absorbance between a reduced and unreduced sample is taken as a measure of the arsenate present. Phosphate is determined simultaneously. Johnson and Pilson (10G) modified the above method to include the determination of arsenite in water. Iodate in acid solution was found suitable to convert any arsenite present to arsenate. Calculations are given to determine the concentration of phosphate, arsenate, and arsenite.

An indirect polarographic method for the determination of 0.1 to 0.5mM arsenate and phosphate in natural waters is described by Rozanski (26G). Arsenate and phosphate are first coprecipitated with ferric hydroxide. Arsenate is then reduced to arsenic trichloride and extracted with carbon tetrachloride. The arsenic trichloride is re-extracted into hydrochloric acid and oxidized again to arsenate, which is then precipitated as ferric arsenate and dissolved in 25% hydrochloric acid. The ion is then reduced, the solution neutralized, and a known amount of uranyl solution added to precipitate the arsenate. The remaining uranyl ion is determined polarographically. Phosphate is deter-

mined similarly to the arsenate after the extraction of arsenic trichloride.

Winter and Booth (32G) compared three modifications of the single-reagent method for soluble orthophosphate and reported that the method of Murphy and Riley (19G) has the best precision and accuracy, and least bias.

Henriksen (7G) reported that the determination of orthophosphate by reduction of molybdophosphate with ascorbic acid and antimony, and the determination of total phosphorus by oxidation with potassium persulfate, are suitable methods for determining phosphorus compounds in water. He also mentioned that preservation with 1 ml of 8N sulfuric acid per 100 ml of sample is practical if the samples cannot be analyzed within 8 hours after collection. Lur'e and Panova (15G) determined orthophosphate in water colorimetrically with a mixed reagent consisting of sulfuric acid, sulfamic acid, ammonium molybdate, antimony trichloride, and tartaric acid. Four milliliters of the reagent and 1 ml of ascorbic acid solution are added to 100 ml of sample. The absorbance of the color is measured at 670 nm after 5 to 15 minutes.

Chamberlain and Shapiro (6G) studied the specificity of the molybdenum blue method for determining dissolved inorganic phosphate in natural waters. They stated that, except in instances where arsenate is a problem, the molybdenum reactive material present in natural waters may be regarded as inorganic phosphate, although not necessarily as free phosphate ion.

Popovsky (22G) described two procedures for hydrolyzing total phosphorus in fresh waters. In the first procedure, the sample is evaporated to dryness in the presence of perchloric and sulfuric acids, the precipitate taken up in water, and heated at 80 °C for 3 hours. In the second method, the sample is autoclaved with sulfuric acid at 1.7 to 2.0 atmospheres for 8 hours.

Altmann *et al.* (3G) used the stable dye salt formed between malachite green and dodecamolybdophosphoric acid in the presence of poly(vinyl alcohol) as a stabilizing agent in 0.5N sulfuric acid to determine 10 to 1,200 μ g per liter of phosphate in water. The absorbance is measured at 623 nm. Excess malachite green in acid medium forms colorless products within 30 minutes of its addition.

Suranova *et al.* (30G) carried out experiments on preliminary concentration of phosphates on chloride-form anion exchangers in sodium chloride solutions of different acidity. A procedure for determining phosphate in sea water is described. The possibility of silica absorption on anion exchangers is mentioned.

A study was made by Sowden (29G) of the possible interference of silica on the determination of orthophosphate in natural waters by automated methods. This interference might be serious when the concentration of phosphorus is less than 50 μ g per liter. An automated isobutanol-extraction procedure appeared to be free from silica interference. If the silica concentration is determined and a correction for it applied to the apparent phosphorus value, the usual automated method may give acceptable results.

A spectrophotometric method for the determination of phosphate and silicate in natural and industrial waters in the presence of each other is described by Jarabin, Vajda, and Szarvas (8G). The heteropoly acids formed with molybdate are reduced in the presence of oxalic acid with a solution of potassium metabisulfite-paramethyl aminophenol sulfate and the color is measured at 660 nm. In the absence of oxalic acid, the color intensity of the heteropolyblue is proportional to the sum of silicate and phos-

phate. The difference between the two measurements gives the phosphate content.

Mohyuddin and Aglan (18G) used a molybdate blue method to determine as little as 0.001 ppm of silica. Ammonium molybdate is added to a sample followed by tartaric and citric acids to eliminate interference by phosphate, calcium, aluminum, zinc, iron, arsenic, germanium, and magnesium. Hydroquinone is added and the solution develops a dark greenish yellow color which changes to greenish blue upon dilution. At pH 0.5 to 1.6, a persistent deep blue color is developed in 10 to 20 seconds. The color, which is stable for 48 hours, is measured at 770 nm.

Magnuson and Galle (16G) determined suspended silica in water by atomic absorption. The silica is collected on a membrane filter which is then fumed in platinum with 50% sulfuric acid and heated until carbon is oxidized. The sample is then fused with sodium carbonate, dissolved in water, and silica determined.

Looyenga and Huber (14G) proposed a new technique, atomic absorption inhibition titration, for the determination of silicate in waste water. Magnesium absorption in a hydrogen-air flame is monitored while using a standard magnesium solution as titrant. A distinct increase in the magnesium absorption signal marks the end point. The samples are first passed over a hydrogen-form cation-exchange column to remove magnesium and other potentially interfering cations. The method is applicable down to 1 μg of silica per ml.

HALIDES

A review with three references is given by Bellack (5H) on the alizarin visual (Scott-Sanchis), photometric (SPANDS), and the ion-selective electrode method for the determination of fluoride in fluoridated drinking waters. Factors which govern the choice of one of the three analytical methods are also discussed. Continuous monitoring of fluoride also is mentioned briefly. Walker and Smith (27H) discussed the use of continuous fluoride analyzers. Several types of continuous analyzers being marketed make use of either colorimetric, potentiometric, or amperometric principles. The amperometric principle is discussed, and many of the user advantages from actual field experience are given. An amperometric fluoride analyzer is also described.

Nawlakhe (20H) discussed the merits of the colorimetric Eriochrome Cyanine R (ECR), SPADNS, and Alizarin Red methods for determining fluoride concentrations in distilled and undistilled waters in the presence of aluminum and sulfate. Fluoride determined by the SPADNS and ECR methods gave lower results in the distilled samples. High results were obtained with the Alizarin Red method. Gitsova (12H) described a modified Eriochrome Cyanine R-zirconyl nitrate colorimetric method for determining fluoride in drinking water. Wierzbicki, Pawlita, and Pieprzyk (31H) reported that ozone in concentrations greater than 0.1 mg per liter in potable water interferes with the determination of fluoride by the alizarin-zirconium method. Sodium thiosulfate is used to eliminate ozone since excess thiosulfate and its oxidation products do not interfere with the method. Anosova (3H) described a colorimetric method for determining fluoride in high-saline waters. An aliquot of sample is evaporated to a few milliliters in the presence of sodium hydroxide. Quartz powder, silver nitrate solution, and sulfuric acid are added to the residue, and the fluoride is steam distilled at 135 to 140 °C. Fluoride is then determined visually with thorium-alizarin indicator using a color scale of standard solutions con-

taining from 0 to 0.1 mg of fluoride per liter. The cerium-alizarin method for determining fluoride in fresh water was modified by Kletsch and Richards (17H) to determine fluoride in sea water. They used a cerium-alizarin complexon chelate in 20% acetone buffered at pH 4.35 with acetate. The absorbance is measured at 625 nm, and the standard deviation for determining 1,080 μg per liter is 10 μg per liter. The effect of salt in sea water is negligible.

Warner (28H, 29H) reported that fluoride in natural waters, such as estuaries, rivers, or lakes, can be measured with an ion-selective electrode using a standard addition and a high-ionic-strength buffer that fixes the pH near 5.5. Cyclohexanediaminetetraacetic acid is used to free fluoride bound by some complexing ions. Results are accurate to about 0.02 mg per liter. The relative standard deviations range from 1 to 5% depending on type of water and fluoride concentration. Ke and Regier (15H) used an ion-selective electrode to determine fluoride in water. Interfering polyvalent cations, such as calcium, magnesium, iron, and aluminum, are extracted as 8-hydroxyquinoline complexes into a butyl Cellosolve-chloroform mixture. Fluoride is determined in the aqueous phase with the total ionic strength adjustment buffer (TISAB). The procedure is usable for samples containing 0.2 to 6.0 ppm of fluoride with an overall error of less than 0.5%.

A standard-addition titration method for the potentiometric determination of the total fluoride concentration in sea water with a lanthanum fluoride electrode is described by Anfalt and Jagner (2H). Equations for the computer treatment of the titration data are given.

Florence and Farrar (11H) investigated the mercuric thiocyanate method for determining chloride in boiler feed water. They show that, with correct conditions and with a precision spectrophotometer, the limit of detection is 15 ppb of chloride at the 95% confidence level. Rodabaugh and Upperman (22H) described a spectrophotometric method for the determination of part-per-billion levels of chloride in high-purity waters. Chloride is precipitated from 250-ml aliquots of sample in polyethylene bottles with lead nitrate and sodium phosphate. The samples are then centrifuged and the supernates are decanted immediately. The precipitates are dissolved with an iron nitrate-nitric acid solution, and mercuric thiocyanate solution is added to each bottle. The absorbances of the solutions are measured at 463 nm. Data indicate that a precision of 1 ppb is obtainable.

Kobayashi and Takeno (18H) described a visual colorimetric method for determining part-per-billion levels of chloride in water. The method is suitable for field use. Silica gel containing silver chromate is placed in a small-diameter glass tube. When 2 ml of sample are injected into the tube for 5 minutes, the color of the detector reagent changes from red-brown to white depending on the amount of chloride present. The length of the discolored zone is a measure of the chloride concentration. Measurable concentration is 5 to 40 ppm with a relative error of $\pm 10\%$.

Van Loon (26H) described and compared inexpensive liquid-filled and solid-solid connected thermoplastic silver-chloride electrodes with a commercially available crystal membrane type. Similar results were obtained. The electrodes are ideally suited for both field and laboratory analysis of natural waters. Florence (10H) reported that the differential potential of a pair of solid-state membrane ion-selective electrodes is directly proportional to chloride concentration between 0 and 350 ppb. The method is applicable to analysis of high-purity waters. The detection limit is 0.6 ppb with a relative standard de-

viation of 3% at 100 ppb of chloride. Weiss' (30H) described a procedure for determining chloride and bromide with ion-selective electrodes. Description of the electrode systems are given. Al-Khalili and Rubeska (1H) used potentiometric titration with 0.1N silver nitrate and direct potentiometric measurement of chloride activities on the basis of the Nernst equation to determine chloride in water. Bystritskii, Bardin, and Aleskovskii (7H) described an apparatus incorporating a water-cleaning train and a two silver-chloride electrode concentration cell that can be used to continuously determine chloride potentiometrically. The working range is 0 to 0.45 mg of chloride per kg of water.

Meyer *et al.* (19H) discussed both batch and continuous electroanalysis techniques to determine the chloride content of saline waters. Theory of operation, analytical procedures, special cells, and instrumentation are described.

Johannesson (14H) determined trace amounts of chloride in natural waters radiochemically. A sample containing nonvolatile chlorides is treated with a known amount of ^{36}Cl -labeled hydrochloric acid and evaporated to dryness; the radioactivity of the residue is measured. In the range of 10 to 100 μg of chloride, the accuracy is within 3 μg ; and in the range of 1 to 10 μg , it is within 1 μg .

Won and Park (33H) compared the accuracy of the micro method (5-ml buret) with that of the macro procedure (50-ml buret) for the determination of chlorinity.

A semiautomatic potentiometric titration of the total halide concentration in sea water with silver nitrate is described by Jagner and Aren (13H). The titration is monitored with a silver electrode, and the titration data, which are recorded directly on punched tape and typewriter, are evaluated by means of a computer program based on the Gran extrapolation method. The determination has a precision of $\pm 0.02\%$. The time for a single determination including evaluation and editing of data is less than 5 minutes. Samples can also be run simultaneously. Ciabatti, Masini, and Rabbi (8H) reported a potentiometric method for the determination of the salinity of sea water. An analysis is completed in 5 to 10 minutes using silver nitrate titrant and a recording apparatus. The relative standard deviation is 0.33 to 0.34%. Cunningham and Duedall (9H) reported that phenosafranine indicator gives a sharper end point than chromate indicator for chlorinity titrations of sea-water samples containing more than 2% chloride.

Wiggins and Athow (32H) determined chloride in sea water by neutron-capture gamma-ray analysis. A ^{252}Cf source and a 30 cm^3 $\text{Ge}(\text{Li})$ γ -ray detector are used. Linear relation between chloride concentration and number of counts was found for the 5.09-, 5.60-, 6.11-, and 6.39-MeV peaks with probable error ranges of 0.34 to 3.60%.

Babkin (4H) photometrically determined bromide in water after its reaction in acid solution with Chromotrope 2B. The color change is from red to yellow. The method is based on the measurement of the reaction time. Potassium bromate is used as the oxidation agent in place of the conventional mixture of acetic acid and hydrogen peroxide. There is a linear dependency between the concentration of bromide and reaction time for 0 to 20 mg per liter. A spectrophotometric method for determining 0 to 0.6 gram per liter of bromine in sea water is described by Saenger (23H).

Berge and Brueggmann (6H) reported that traces of bromide in the presence of a large excess of chloride in water can be determined by two polarographic techniques. Oxidation of bromide to bromate is accomplished by means of an alkaline hypochlorite solution followed by reduction on a mercury dropping electrode. Alternatively, bromide is

oxidized to bromine using potassium permanganate in acid solution. Excess permanganate is precipitated as manganese oxide, and bromine determined with a platinum electrode.

Radcliffe (21H) described an ion-exchange X-ray spectrographic procedure for the determination of bromide in natural water. An ion-exchange resin contained in a filter disk is used to separate bromide prior to X-ray measurement. Analytical error is estimated at 5% and 0.05 ppm of bromide is the statistical detectability limit when compared with a standard disk containing 100 ppm of bromide.

Schnepfe (24H) determined iodide in sea water spectrophotometrically as the starch-iodine complex without prior separation or concentration of the iodide. In slightly alkaline solution, iodide is first oxidized to iodate with permanganate. Oxidized states of manganese are destroyed with ferric iron in phosphate medium without affecting the iodate. After acidification, iodide is added to react with the iodate in the presence of starch. The iodate content is determined similarly without the addition of permanganate. A 0.1 μg of iodide is determinable in the presence of 500 mg of chloride and 5 mg of bromide. Tsunogai (25H) used a starch-iodine method to determine total iodide in sea water. Details of the procedure are given.

Low concentrations of iodide (5×10^{-8} to $5 \times 10^{-6}M$) in mineral waters with salinities less than 8000 mg per liter were determined polarographically by Khelashvili (16H). Mercurous iodide dissolution currents are measured using a stationary saturated mercurous sulfate electrode. The iodide determination is not hindered by 5000- and 60-fold excesses of chloride and bromide, respectively, nor by sulfate and carbonate normally present in mineral water. The iodide determination is independent of water pH less than 6.5.

SULFATE AND SULFIDE

A spectrophotometric method for the determination of sulfate in water in the 0 to 10 and 0 to 100 ppm ranges was developed by Davis and Lindstrom (4J). The samples are treated with a mixture of hydriodic acid, acetic anhydride, and sodium hypophosphite, and heated in a modified countercurrent reaction apparatus to evolve hydrogen sulfide. The gas is swept by nitrogen into a buffered solution of ferric ion and 1,10-phenanthroline. The ferric ion is reduced to ferrous ion and the ferrous-1,10-phenanthroline complex, which is formed, is measured at 510 nm. Only nitrite and those ions capable of yielding hydrogen sulfide under the same conditions interfere. Sulfonated surfactants do not interfere.

Jagner (9J) determined sulfate in sea water, with a chlorinity of at least 2‰, by photometric titration. Sulfate is titrated in dimethyl sulfoxide solution with hydrochloric acid using bromocresol green as indicator. The end point is evaluated graphically and a complete titration can be performed in less than 15 minutes. Borate, carbonate, and carbonic acid interfere and a separate determination of alkalinity is necessary. Results obtained for sulfate by this method agree with results obtained gravimetrically.

Lambert and Manzo (12J) described an ion exchange-titrimetric method for the determination of sulfate in natural waters. The samples are passed through an ion-exchange column of homogeneously precipitated barium iodate monohydrate, followed by iodometric titration of iodate in the eluate. An indirect titrimetric method for the determination of sulfate in irrigation and drinking

water is described by Galben (6J). The sample is passed through an anion-exchange resin (chloride form), and the chloride in the eluate determined by the Mohr method. The method can be used for waters containing less than 10 mequiv per liter of sulfate and 35 mequiv per liter of chloride.

A titrimetric method for the determination of sulfate in water is reported by De Maeseneer (5J). Details of the procedure are not included in the abstract. Shpeizer, Rubinchik, and Atanova (18J) described an indirect potentiometric method for determining sulfate in natural waters. The method is based on the precipitation of lead sulfate in acetone-water medium and potentiometric titration of excess lead with sodium sulfide using a silver-sulfide indicator electrode. The error of the determination at 15 mg per liter is approximately 15%. With a concentration of 20 mg per liter or more, the error is less than or equal to 3%. Goertzen and Oster (7J) determined sulfate in natural waters and soil-water extracts semiautomatically with a lead ion-selective electrode to indicate the solution potential change at the end point. The titration system includes a constant-flow device to deliver the titrant, a mV meter to measure the potential, and a strip-chart recorder. Sulfate concentrations as low as 0.5 mequiv per liter can be determined.

Bierwagen and Dojlido (3J) used an automated nephelometric method to determine sulfate in water. The method is based on the reaction of sulfate with barium ion and measurement of the turbidity at 520 nm. An EDTA wash solution is used which dissolves the barium sulfate deposit left in the pipes. The range of the method is 5 to 150 mg per liter. Audouze and Bonometti (1J) also described an automated nephelometric method to determine sulfate in potable and polluted waters. Sulfate is precipitated with 2-aminopyrimidine at pH 5 to 7. The error at 2 mg of sulfate per liter is approximately 2%.

Pleskach and Chirkova (15J) and Pleskach, Chirkova, and Fedorova (16J) described flame photometric methods for determining sulfate in natural water. The methods are based on the photometric determination at 493 nm of excess barium ions remaining in solution after sulfate is precipitated with barium sulfate and filtered.

Sulfate between 0 and 500 mg per liter in fresh water was determined radiometrically by Klehr (11J). Sulfate is precipitated with barium chloride containing ^{133}Ba , and then filtered. Either the precipitate or the supernatant liquid is counted. Standard solutions containing 50 and 250 mg of sulfate per liter showed 48 and 253 mg per liter, respectively, by counting the supernatant and 50 and 251 mg per liter, respectively, by gravimetric analysis.

Berge and Brueggmann (2J) used polarography to determine sulfate in sea water. The method is based on the reaction of sulfate, in an ammonia-ammonium chloride buffer, with a hydrochloric acid or perchloric acid solution of barium chromate which liberates chromate ions equivalent to the sulfate concentration. The relative standard deviation at a sulfate concentration of $2.4 \times 10^{-2}M$ is $\pm 0.5\%$.

The determination of sulfide in water, based on its catalytic effect on the reduction of iodine by sodium azide, is described by Naumczyk (13J). The reaction is carried out in an acetate buffer of pH 4.6, and the change in iodine concentration is determined colorimetrically. For the concentration range of 0.02 to 0.15 mg of sulfide per liter, the relative error is less than 10%. Thiosulfate, ferrous and ferric iron, and manganese interfere.

Rees, Gyllenspetz, and Docherty (17J) determined sulfide between 0.5 and 100 μg in condensed steam. A spec-

trophotometric measurement is made at 670 nm after mixing the sample with *N,N*-diethyl-*p*-phenylenediamine and ammonium ferric sulfate. Absolute standard deviations were approximately 3 and 0.08 μg of sulfide at 100 and 1.0 μg of sulfide, respectively. For estimations in the field, the absorbance measurement can be replaced by a colorimetric titration of the final solution with methylene blue solution against a reagent blank as reference.

Sutherland (20J) used a silver-sulfide electrode for the determination of total dissolved sulfide concentrations in waters in the field.

Kakiyama and Komatsu (10J) determined sulfide in industrial water by square wave polarography. Sodium hydroxide is used as a supporting electrolyte. The peak potential of sulfide is the same as that for lead, which is present as an impurity in the sodium hydroxide. To separate the lead wave from that of sulfide, EDTA tetrasodium salt is added to the supporting electrolyte. Anions, such as sulfate, sulfite, thiosulfite, thiocyanate, iodide, bromide, and chloride, do not interfere even in 100-fold amounts. Calibration curves were obtained for 1×10^{-6} to 1×10^{-5} and 1×10^{-5} to $1 \times 10^{-4}M$ sulfide.

Paez and Guagnini (14J) reported that hydrogen sulfide in water in concentrations equal to or greater than 0.1 ppb can be determined by a methylene blue photometric method after isolation in water on an Amberlite IRA 400 anion-exchange resin in the hydroxide form. Elution of the sulfide is made with 4M sodium hydroxide. The eluate is then treated with 4N sulfuric acid, *N,N*-dimethyl-*p*-phenylenediamine, and ferric chloride, and the absorbance measured at 670 nm. The ion-exchange column retains sulfide approximately 10 days without loss. Smirnov (19J) used ethoxyethyl-*p*-phenylenediamine sulfate and ferric chloride to determine hydrogen sulfide in sea water. The blue-colored complex is measured colorimetrically using a red filter. The sensitivity is 0.002 mg of hydrogen sulfide per liter at concentrations of 0.2 to 0.6 mg per liter. Small concentrations of oxygen in sea water do not interfere.

An automated method for the determination of hydrogen sulfide in natural waters, based on the formation of methylene blue by the reaction of sulfide with *N,N*-dimethyl-*p*-phenylenediamine and ferric chloride, is reported by Grasshoff and Chan (8J). A modified Technicon AutoAnalyzer technique is used and is described in detail. The coefficient of variation of replicate determinations of 34 μg of hydrogen sulfide per liter is $\pm 0.8\%$, the sensitivity is 6.8 μg per liter.

A thiomercurimetric technique to determine hydrogen sulfide in natural waters below and above the microgram-per-liter level is described by Wronski (21J). Hydrogen sulfide is extracted from alkaline water samples as triethyllead sulfide into hexanol. Concentrations of hydrogen sulfide greater than 2 μg per liter are determined by titration with *o*-hydroxy-mercuribenzoic acid to a red color using either dithizone or dichlorofluorescein as indicator. Concentrations greater than 0.01 μg per liter are determined by fluorometric titration with tetramercurated fluorescein.

NITRATE, NITRITE, AND NITROGEN COMPOUNDS

A review containing 84 references on methods for determining various forms of nitrogen was compiled by Wagner (46K). The most important methods for determining nitrate and nitrite in surface and waste waters are given by Marculescu (26K) in a literature review containing 96 references. He concluded that the most suitable method for determining nitrate is a colorimetric determination with

sodium salicylate, whereas the most suitable method for determining nitrite is a colorimetric one using sulfanilic acid and 1-naphthylamine. Kamenova and Kazantseva (19K) found that the most suitable method for determining nitrates in carbonate saline soils and ground waters was based upon the reduction of nitrate to nitrite in ammonium hydroxide medium and subsequent colorimetric determination of nitrite by using sulfanilic acid and α -naphthylamine. The use of zinc powder or manganous sulfate as reducing agents and the colorimetric method employing phenoldisulfonic acid were not as satisfactory.

Volokhonskii (44K) determined nitrate in natural water by reducing nitrate to nitrite with hydrazine at pH 9.6 in the presence of a copper catalyst. Sulfanilic acid and α -naphthylamine are added, and the absorbance of the resulting solution is measured. The error of the method is approximately 0.5%, and the sensitivity is 0.01 mg nitrogen per liter. The brucine colorimetric determination of trace amounts of nitrate less than or equal to 1 mg per liter as sodium nitrate in the presence of chloride was studied by Petriconi and Papee (36K). Ranney and Bartlett (37K) described a brucine method for the field determination of nitrate in natural waters. The brucine reagent is stable for at least a year when dissolved in methanol. It was stated that this is the simplest colorimetric method, and that it is more sensitive to low concentrations than the nitrate selective-ion electrode. Nitrate less than or equal to 100 ppm was extracted from potable water and determined spectrophotometrically at 269 nm as described by Burns, Fogg, and Willcox (5K). Silver acetate is added to the sample before the nitrate is extracted with Ph_4PCl in carbon tetrachloride. Silver acetate reduced the blank absorbance and precipitated some interfering anions. Concentrations in excess of 200 ppm for chloride, 20 ppm for nitrite or bromate, and 100 ppm for bromide, iodide, or thiocyanate interfered. Sayato *et al.* (40K) studied the optimum conditions for the phenoldisulfonic acid method for the measurement of nitrate-nitrogen in natural waters. Interference of nitrite and of sulfide are removed by the addition of sulfamic acid and lead acetate, respectively. A proposed graphical technique for compensating for chloride interference in the phenoldisulfonic acid method for nitrate was found to be very satisfactory by Malhotra and Zanoni (22K). The effect of alkalinity of the sample on the determination was eliminated by neutralization with sulfuric acid. Dabrowska (7K) described a spectrophotometric method in which nitrates in natural waters react with chromotropic acid in a sulfuric acid medium. The absorbance of the solution is measured at 420 nm. Interfering chlorides (over 30 mg per liter) are removed with silver sulfate; nitrites at 10^{-3} mg $\text{NO}_2\text{-N}$ per liter are ignored. Ferric ion (below 20 mg per liter) and ferrous ion (below 40 mg per liter) do not interfere.

Several authors discussed the use of ion-selective electrodes to determine nitrate in water. Sommerfeldt, Milne, and Kozub (42K) stated that the advantages which an ion-selective electrode has over the phenoldisulfonic acid method in surface and ground water include a lack of interference from soluble salts and color, and a much wider range of detection without dilution. They obtained greater sensitivity by using a potassium chloride-saturated reference electrode rather than a silver-saturated 0.02N potassium chloride reference electrode. Langmuir and Jacobson (20K) compared the brucine and ion-selective electrode methods for the determination of nitrate in some fresh waters and sewage effluents. Good agreement was achieved between these methods when corrections were made for the latter method. When the corrections were

not made, the results were systematically 1.8 ppm more than the brucine values for samples containing less than 50 ppm of nitrate, but were still comparable for nitrate values greater than 50 ppm. Milham *et al.* (31K) present an ion-selective electrode method for the nitrate determination of nonsaline waters which gives better recovery and reproducibility at 2 to 500 ppm than the Devarda method. A pH 3.0 buffer eliminated chloride, nitrite, and organic anion interferences. Automated potentiometric techniques for the on-site monitoring of anion concentrations in water were discussed by Manahan (23K) and Manahan *et al.* (24K). Characteristics of several new nonglass ion-selective electrodes and of several reference electrodes were studied in detail with particular emphasis upon the nitrate ion-selective electrode. A new high-accuracy standard-addition technique for the potentiometric determination of nitrate in a cell without liquid junction, using the fluoride electrode as a reference, was developed. Algae interfered with the electrode response.

The effects of interfering organics in the ultraviolet determination of nitrate in water at 210 nm are discussed by Morris (32K) and a mathematical method based on the determination of the first derivative ($\Delta E/\Delta \lambda$) of the absorbance curve for the determination of nitrate is proposed. The nitrate concentration is obtained by subtracting the absorbance difference between 205 and 210 nm and 225 to 230 nm from the absorbance difference between 215 and 220 nm. Mertens and Massart (30K) determined nitrate in sea water by ultraviolet spectrophotometry at 210 and 220 nm. The samples are treated with hydrochloric acid to remove carbonate and bicarbonate interferences, as well as to destroy microorganisms. A blank obtained by the reduction of nitrate with Raney nickel is used. This method can be used when nitrate concentrations exceed 1 ppm.

Soares, Pereira, and Antunes (41K) state that the optimum conditions for the spectrophotometric determination of nitrate and nitrite depend on the pH and possible interferences from organic matter, carbonate, bicarbonate, dihydrogen phosphate, and sulfate in the analysis of urban, waste, and sea waters. Nitrate is determined at pH 1 to 2, and absorbance readings are required at both 220 and 270 nm to compensate for organic matter. Nitrite in the absence of nitrate is determined at pH 6 at 220 to 230 nm; when both ions are present, nitrite is determined separately by the sulfanilic acid and α -naphthalenediamine method. Rusanov and Blinov (39K) found that low nitrate concentrations and poor agreement in nitrate and nitrite concentrations in samples of sea water at depths less than 3000 meters were due to the reduction of N^{5+} to N^{3+} by the brass bathometer. To alleviate these problems, the bathometer must be produced of or coated with Viniplast or fluoroplast. A method for the automated determination of nitrate and nitrite in water and sewage was described by Wudzinska and Ponikowska (49K). The colorimetric determination of nitrite is based on its reaction with sulfanilic acid and α -naphthylamine. Nitrate is determined by the same method after reduction with hydrazine sulfate in phenolate buffer.

Nishimura, Matsunaga, and Matsuda (34K) discussed an extraction procedure for the determination of trace amounts of nitrite in fresh water. Sulfanilamide and *N*-1-naphthylethylenediamine dihydrochloride are added to the sample, followed by carbon tetrachloride and dodecylbenzenesulfonate. After extraction, the absorbance of the organic layer is measured at 525 nm. The above method was modified by Matsunaga, Oyama, and Nishimura (28K) to include a back-extraction of the azo dye into an

acetone-hydrochloric acid phase. The absorbance of this phase is measured at 543 nm. Beer's law is followed for 0 to 7.36 μg of nitrite per liter for both fresh and sea water. A spectrophotometric determination of traces of nitrite in sea water by concentration of azo dye on an anion-exchange column was described by Wada and Hattori (45K). The sample is treated with sulfanilic acid and *N*-1-naphthylethylenediamine and drawn through a column of Dowex 1-X8 resin. The azo dye collected on the column is then eluted with 10 to 25 ml of 60% acetic acid before the absorbance is determined at 550 nm. The effects of the amounts of reagents, pH, flow rate, and eluting solvents are discussed. Nitrite-nitrogen concentrations of 0.001 to 0.100 μg -atom can be determined.

Mautner (29K) compared the Nessler, phenol-hypochlorite and salicylate methods for determining ammonia in drinking waters. He concluded, after conducting ammonia determinations on a synthetic water, that the salicylate method had better sensitivity, reproducibility, and accuracy than the others studied. An infrared phenol-hypochlorite method for determining ammonia in natural waters was investigated by Harwood and Kuehn (16K). Nitroprusside is used as a catalyst. This method is simple and does not require accurate time intervals between reagents nor expensive equipment. Matsunaga and Nishimura (27K) described an extremely sensitive indophenol method for colorimetrically determining ammonia in sea water by using chloramine-T and thymol. For samples of low ammonia concentration, the indophenol is concentrated by extraction into *n*-hexyl alcohol before its absorbance is measured. The effect of pH, amount of chloramine-T and thymol, and the standing temperature were investigated. No appreciable interference was observed with some nitrogen-containing compounds, such as urea, hydroxylamine, glutamic acid, glycine, and lysine. Truesdale (43K) determined ammonia and amino acids in sea water spectrophotometrically at 543 nm by a modified, shortened method of Strickland and Parsons. In 13 replicate sea-water samples with ammonia-nitrogen content of approximately 20 μg per liter, the modified procedure yielded results with a relative standard deviation of 1.6%. The spectrophotometric determination of ammonia-nitrogen in natural waters with an indothymol-extraction method was described by Fujinuma, Shimada, and Hirano (10K). Otchenashenko and Rogatskin (35K) compared two ammonia methods used to analyze ammonia-plus-hydrazine-treated boiler waters taken from various stages of the water-steam cycle. At ammonia concentrations greater than 300 μg per kg, the more simple electrical resistivity method yielded results that were comparable to those determined by a Nessler photocolometric method. However, at ammonia concentrations less than 300 μg per kg, the resistivity method yields unsatisfactory results in water containing hydrazine. A technique for determining ammonium-nitrogen in artificial sea-water aquaria is described by Ellender, Armour, and Camp (9K). The method, suitable for measuring ammonia concentrations in the 1- to 20- μg -per-ml range, involves diffusion and colorimetry.

Several authors used Technicon AutoAnalyzers to automate methods for determining ammonia in water. Harwood and Huyser (15K) developed an automated method to determine ammonia over a wide concentration range by altering the reagent concentrations. The most sensitive determination used nitroprusside as a catalyst over a range of 0 to 1 mg ammonia-nitrogen per liter. The effect of sample pH and of certain metal ions was investigated. It was concluded that an adequate buffering system

should be used. An automated procedure for the determination of ammonia-nitrogen in sea water has been adapted by Head (17K) from a manual phenol-hypochlorite method. It has much greater sensitivity than previous phenol-hypochlorite techniques. MacIsaac and Olund (21K) automated an extraction method for the determination of ammonia in sea water. Ten samples per hour can be analyzed with an accuracy of about $\pm 10\%$ at a level of 0.50 μg -atom ammonia-nitrogen per liter. Details of the method are not given in the abstract. Wudzinska (48K) described an automated method for surface waters in which the sample is mixed with phosphate buffer of pH 7.4 and distilled from a bath at 115 °C. The vapors containing ammonia are mixed with hydrochloric acid and treated with Nessler reagent. The standard mean deviation for samples containing 1.0 and 4.0 mg ammonia-nitrogen per liter was 2.2% and 1.2%, respectively. Ten determinations can be made per hour.

Barica (2K) reported on a rapid method of determining ammonium in water with a univalent-cation glass electrode. This method enables direct determination of ammonium in concentrations greater than 0.5 mg per liter without dilution or pretreatment of sample. A constant background of sodium and potassium ions in the samples is required with concentrations of potassium being lower than those of ammonium. For several types of natural and enriched waters with ammonium content less than or equal to 40 mg per liter ammonium-nitrogen, the mean error of the method was $\pm 4.9\%$. The accuracy of the ammonium-selective glass electrodes for determining ammonium (10 to 1000 μg per liter) in boiler-feed water and similar high-purity water samples from power stations was investigated by Goodfellow and Webber (11K). Interfering species in the solution containing triethanolamine-hydrochloric acid buffer cause a deviation from the Nernst equation at low ammonium concentrations (less than 1000 μg per liter). However, by use of a calomel-0.1*N* hydrochloric acid reference electrode, reproducible results are obtained in static buffer solutions containing 10 to 1000 μg per liter of ammonium. Of the other impurities likely to be present in power-station waters, only sodium causes a serious effect. The response time varied from 2 minutes for the 1000- μg -per-liter solution to 8 minutes for the 10- μg -per-liter solution. Diggins and Meredith (8K) discuss several methods which can be used to determine ammonia in on-line analysis systems. A colorimetric method for use in an automatic titrator was developed. Ammonium ion is oxidized at pH 9.2 by hypobromite which is electrically generated from bromide at a platinum electrode. The ranges of the instrument are 0-5 to 0-100 mg per liter. Easily oxidizable metals interfere. An ammonium-potassium electrode method and a gas-permeable membrane method are discussed. The former method is of little use in waters with a high potassium content.

Eleven references are contained in a review by Weiner and Leiss (47K) on methods for the determination of cyanide by distillation and by colorimetric techniques. A tabulated comparison is also made. Gregorowicz and Gorka (13K) reviewed the analytical techniques involved in the determination of cyanide in industrial wastes and surface waters. One hundred and forty-nine references are included. A review, with 51 references, which discusses the analytical methods used to determine total cyanide and free cyanide was authored by Higashiura (18K). Boltz (4K) reported on the role of spectrophotometry in modern trace analysis. The determination of cyanide in water is included.

Roberts and Jackson (38K) concluded that the decom-

position of ferrocyanide can be completely prevented by distilling cyanide in vacuo in the presence of zinc acetate. Cyanide in the distillate is determined by the pyridine-pyrazolone spectrophotometric method. Pentacyanocarbonylferrate and pentacyanonitrosylferrate partially decompose during the distillation. A colorimetric procedure was described by Barkley and Ingles (3K) for the determination of the total cyanide content of effluents from gold cyanidation plants and of water from basins into which they discharge. This rapid method determines cyanide present as hydrogen cyanide, cyanide ion, zincocyanide, cuprocyanide, and nickelocyanide, but not cyanide present in the form of ferrocyanide or cobaltocyanide. It is free from interference by the common constituents of gold-mill effluents. Nelson and Lysyj (33K) developed a method for determining molecular hydrogen cyanide in water. The hydrogen cyanide is trapped in dilute base, and the sparged gas is measured with a rotating gold anode. The apparatus is discussed in some detail. The present detection limit for this method seems to be limited by the techniques of transferring and handling nanogram quantities of the desired species.

Collombel *et al.* (6K) investigated the use of ion-selective electrodes for the determination of cyanide in air, drinking water, industrial waste, and biological media. This very simple and sensitive method permitted a large range of measurement (270 μg to 270 mg per liter). Sulfide and chloride interfere; therefore, hydrogen cyanide must first be isolated by a current of gas if they are present. Bahensky (1K) concluded that the various methods used to determine free and bound cyanide complexes are not accurate enough to distinguish between the two forms. This fact must be considered in the analysis of waste waters with low cyanide concentrations. Two methods for the determination of cyanide were described by Goulden, Afghan, and Brooksbank (12K). The use of an improved absorption column following the distillation step resulted in a detection limit of 5 μg of cyanide per liter. An automated procedure is described that enables 10 samples per hour to be analyzed with a detection limit of 1 μg of cyanide per liter. Distinction is made between simple and complex cyanides by irradiation with ultraviolet light. This irradiation breaks down complex cyanides, including those of cobalt and iron. A manual pretreatment step is described to remove sulfide interference. Thiocyanate interferes when the irradiation step is used.

Manny, Miller, and Wetzel (25K) investigated the ultraviolet combustion of dissolved organic-nitrogen compounds in lake waters. The method combined with sensitive spectrophotometric nitrate plus nitrite-nitrogen and ammonium-nitrogen determinations offers a 100-fold increase in sensitivity over the micro-Kjeldahl method for total dissolved organic nitrogen. This procedure permits quantification of two organic-nitrogen fractions in lake water based on the relative rate and amounts of labile ammonium-nitrogen and refractory nitrate plus nitrite-nitrogen released. Combustion conditions and pH must be carefully controlled to achieve uniform results.

Grunnet (14K) states that concentrations of phosphorus and nitrogen compounds are often so low that large errors can occur during transport and storage due to microbial activity or due to improper washing of the sampling apparatus. Water samples must be either analyzed within 20 minutes or chilled to 0 to 4 °C. For storage of more than 6 hours, samples must be preserved by freezing and (or) addition of 20 mg per liter mercuric chloride. Chloroform and sulfuric acid were unsatisfactory as preservatives. The proper washing of glassware is also discussed.

pH, Eh, ALKALINITY, AND CARBON DIOXIDE

A review with 64 references on the determination of Eh and pH in sedimentary environments (soils and waters) by colorimetric and electrometric methods is given by Langmuir (6L).

A method is presented by Ben-Yaakov (1L) for the accurate calculation of in situ pH from surface measurements on sampled sea water, based on the assumption that total carbon dioxide, total alkalinity, and total boron are invariant with respect to temperature and pressure when expressed in equivalents per kilogram. A compact potentiometric sensor for the in situ measurement of pH, pS^{2-} , and Eh is discussed by Whitfield (15L). Its performance is illustrated by data from a stagnant basin model and from an estuarine survey. Construction details are included. Shpeizer and Charchidi (12L) described a hermetic cell for measurement of pH, pS^{2-} , and Eh of water under field conditions. Breck (2L) described a new approach to Eh that allows determination of the redox level of aerated natural waters, thus avoiding the disadvantages of using electrodes in association with any particular chemical system. Experimental data and theoretical determinations are presented that support the view that the poise of aerated waters is controlled by the oxygen-hydrogen peroxide couple rather than by the oxygen-water couple. Also, it is believed that a small but steady activity of hydrogen peroxide has important functions in the environment.

Schindler and Honick (11L) reported that a Pt-Ag/AgCl electrode method is a marked improvement over previous methods for the investigation of sediment-water interface phenomena. Measurements are made in an essentially sealed system using the corer device described to obtain intact samples of mud-water interfaces. Data are reported for hypolimnetic and lake sediment.

A method for the determination of trace amounts of carbon dioxide was presented by Romanchuk and Nizyayeva (8L). Carbon dioxide is pumped out of a large sample of water into a much smaller volume, where it is determined by titration. The method is satisfactory for determining 0.045 to 15 mg per liter, and the difference between parallel determinations does not exceed 3 to 5%. Sobotha (13L) reported on a simplified carbon dioxide determination in water sources. Fairly good accuracy was obtained by use of a 50-ml sprayer into which was introduced phenolphthalein and the sodium hydroxide titration solution. A graph was used by Sonderegger (14L) to demonstrate the relationship of concentrations of calcium and bicarbonate ions (in moles per liter), and of various pH values to partial pressure of carbon dioxide. It can be used to calculate partial carbon dioxide pressure from standard groundwater analyses in limestone or other carbonate aquifers; however, it applies accurately only to water containing calcium and bicarbonate ions almost entirely. Fossato (4L) compared analytical methods for the determination of alkalinity and total carbon dioxide. A potentiometric method was used for the alkalinity determination and the Van Slyke manometric method was preferred to the indirect pH method. A high-precision determination of titration alkalinity and total carbon dioxide content of sea water by potentiometric titration was outlined by Edmond (3L). The principles of this method are discussed and its development is described. Measurements of alkalinity and total carbon dioxide can be made at sea with an accuracy of $\pm 0.17\%$ and $\pm 0.68\%$, respectively, at the 95% confidence limit. Errors in calculating carbon dioxide and carbonate ion concentrations in water were enumerated by Rozhdov (10L). By using equilibrium constants and ion

balance, the amount of carbon dioxide in moles per liter is calculated by derived equations.

The determination of bicarbonate in water in the presence of anions of other weak acids is reported by Rosopalo and Quentin (9L). In the presence of anions of other weak acids, bicarbonate is calculated by the formula: mg per liter bicarbonate = 1.386 (mg per liter total carbon dioxide - mg per liter free carbon dioxide). The concentration of total carbon dioxide was determined gravimetrically, and the amount of free carbon dioxide was determined by titration with 0.02N sodium hydroxide. The maximum error was -5%. Pennington (7L) used gas chromatography to determine carbonate and bicarbonate. Gas-solid chromatography was carried out with aqueous solutions involving thermal decomposition in a stainless steel tube at 500 °C in a flowing stream of carrier gas. The resultant carbon dioxide was swept into the chromatographic column and separated from the water of the sample. In the concentration range of 10 to 100 μ g (total amount injected), carbonate and bicarbonate gave a linear response vs. concentration. A single analysis required approximately 3.5 minutes. A method is described by Johnson and Michalski (5L) which isolates and concentrates inorganic carbon (bicarbonate and free carbon dioxide) for the accurate and precise determination required in the determination of primary productivity by the 14 C method in extremely soft waters. Lake water was acidified to pH 3.0 and inorganic carbon was absorbed quantitatively in base in a rubber balloon suspended within the sample bottle. Inorganic carbon in the absorbant was analyzed by back-titration with acid and with a carbon analyzer; results of the two methods differed by only 6.5%.

OXYGEN AND OTHER GASES

A review with eight references was compiled by Borrowman (6M) on physical methods for the measurement of dissolved oxygen. Included are the dropping mercury electrode, solid electrodes, membrane electrodes, the galvanic probe, diffusion-extraction, and gas-liquid equilibrium methods.

Graefe (17M) used a commercially available oxygen sensor and a newly developed telemetering and digitizing system to develop an apparatus for measuring the concentration of dissolved oxygen even in great oceanic depths. Podlowska (41M) reported on the theoretical and practical aspects of the amperometric determination of oxygen in water and sewage with the use of diaphragm probes. Calibration of the measuring system, correction for atmospheric pressure and salt content, influence of temperature and agitation of the medium, direct action of air on the probe, and interfering factors are discussed. A known amperometric method for the determination of oxygen was modified by Bulawa, Dolbniak-Leonowicz, and Mikowski (7M). The measurement of diffusion current is replaced by determination of potential drop across a known resistor. The use of a polarization device coupled with a pH meter makes this measurement possible. Small platinum electrodes increase the rapidity and the accuracy of measurements. The method can be used for continuous determination of oxygen in liquids. The amperometric determination of dissolved oxygen in water using an electrochemical flow cell with porous silver electrodes was studied by Ostrovidov (38M). The sensitivity of the method is 5 μ g per liter at a filtration rate of 6.2×10^{-3} ml per second. Iron, copper, and silver ions interfere. Edgington (10M) discussed three methods for the determination of dissolved oxygen in sea water in which the precision and accuracy differ because of various levels of care in sample

handling and measurement of sample and reagent blanks. Samples are fixed according to the classical Winkler method. Excess sodium thiosulfate is added to the fixed sample, and an aliquot of the solution is back-titrated amperometrically by the constant-current method under a nitrogen atmosphere. This method may be used for samples containing dissolved oxygen up to saturation. An amperometric method for determination of dissolved oxygen in sea water is outlined by Palermo (39M).

A description is given by Marvet, Raudsepp, and Tenno (32M) of a membrane-protected galvanic cell for the determination of dissolved oxygen in natural and waste waters. The current generated in the cell is directly proportional to the amount of oxygen at the membrane-solution boundary. It is necessary to measure the temperature of the solution to within 0.1 °C in order to achieve an accuracy of 0.05 to 1.0 mg of oxygen per liter. Factors affecting electrode design and performance are discussed briefly. Hellige (20M) described a membrane probe which he found to be superior to the bare thallium probe and to the Winkler method for determining dissolved oxygen in industrial water and process wastes. This probe features a very large electrolyte reservoir, a unique system of replenishing any electrolyte spent during the operation of the probe, a constructional arrangement to overcome sensitivity to pressure, a design and placement of a temperature-correcting thermistor to achieve rapid temperature compensation, and a membrane made of Teflon (Du Pont). Two special stirrers are used in the sample bottle. Kolesnikov and Khramov (23M) described a remote, dissolved-oxygen monitoring apparatus (bathythermooxymeter), its electronic circuitry, method and principle of operation, and remote calibration. The apparatus was used to measure dissolved oxygen in sea water at 70 meters, and the results agreed within 2 to 3% with results determined by a standard method. An electrochemical sensor and a differential scheme are proposed by Flis *et al.* (14M) for continuously determining 0 to 200 μ g of dissolved oxygen per liter in water. The results agreed with those of a thallium instrument and with the results of a chemical method. Calibration curves for the sensor at 20 to 35 °C were plotted. The sensor measurements are reproducible within 5%, the experimental sensitivity is 3 μ g per liter, and the response time is less than or equal to 3 minutes. A method is outlined by Behrens *et al.* (4M) for an on-stream determination of 0 to 10 ppb of oxygen in sea water by polarographic monitoring. Galvanic and gas-phase methods were also evaluated and found to be unacceptable. Sudden pH changes may affect the accuracy of measurement. The presence of sulfur dioxide, chlorine, and oxides of nitrogen interfere. Correction for water salinity can be made. Constructional details and performance characteristics are given by Kuz'min and Basharkin (26M) for a polarographic oxygen analyzer capable of continuous or periodic dissolved-oxygen determinations to within ± 0.1 mg per liter. The apparatus is particularly suitable for the monitoring of dissolved oxygen in natural waters, waste waters, and biological solutions because the diffusional membrane, which can readily be wiped off, traps the microorganisms and, thus, prevents their interference with the oxygen determinations. Okuda, Inoue, and Miwa (37M) reported on a rapid polarographic microdetermination of dissolved oxygen in water with flavine enzyme. Dissolved oxygen up to 100% saturation can be determined in 5 minutes in waters at a temperature of 5 to 40 °C. Mercuric, silver, and cupric ions in concentrations greater than 10^{-5} M interfere and extend the dissolved-oxygen determination time. The response characteristics

of a polarographic oxygen analyzer were determined both theoretically and experimentally by Benedek and Heidegger (5M). They demonstrated that a simplified diffusion calculation of oxygen transfer within the probe provides a reasonable basis for describing instrument response. Talreja, Bhalala, and Rao (50M) compared the Winkler and derivative-polarographic methods for determining oxygen in sea water. Because of interferences, no decision was made on which method is more accurate; however, the polarographic method was found to be twice as sensitive as the Winkler method. Principles, features, and advantages of a polarographic system for measuring dissolved oxygen in polluted water are discussed by Gealt and Metarko (15M). The system consists of a sensor coupled to a solid-state electronic signal conditioner.

The measurement of traces of oxygen in light and heavy water by the thallium method was investigated by Lueck (27M). The thallium column for the continuous determination of dissolved oxygen is based on the difference in conductance before and after interactions between metallic thallium and the dissolved oxygen in water. Oxygen concentrations of less than 5 μg per liter and up to 1,000 μg per liter can be detected. The Siemens analyzer is shown and the operating conditions given.

Kral (24M) used a phase exchanger to determine dissolved oxygen in water in order to overcome difficulties due to dissolution of variable amounts of interfering substances. With the analyzing apparatus operating at 220 V and 50 Hz, 0 to 12 mg of oxygen per liter could be detected with $\pm 5\%$ scale accuracy.

Jones (21M) developed a method for determining dissolved oxygen in small quantities of water by drawing the water directly into a small evacuated sample vessel. Because of the presence of 0.15 ppm of hydrazine, which is added as a de-oxygenator for high-temperature water, oxygen is determined by modified indigo-carmin colorimetric and modified polarographic methods.

A new chromatographic method for the determination of dissolved gases in water and the apparatus used are described by Krylov, Kalmanovskii, and Yashin (25M). Dissolved air in water is estimated by using two columns packed with silica gel MSA with carbon dioxide as the carrier gas. The error is less than 4%. A mixture of helium, oxygen, nitrogen, methane, and carbon dioxide dissolved in water is analyzed on a 1.2-meter column filled with 5 Å molecular sieve, and a 0.8-meter column filled with silica gel KSK. A chromatographic method is proposed by Sutter (49M) for the simultaneous determination of dissolved nitrogen and oxygen in water. After sampling in glass containers, the water is partially displaced by helium, and the nitrogen and oxygen are picked up by the helium and determined by gas chromatography. The oxygen results are compared with those obtained by the Winkler method.

A calibration module for trace oxygen analyzers is described by Edgington and Roberts (12M). It is based on de-oxygenation in a 2-stage spinning-disk stripper by counter-current of feed water and oxygen-free nitrogen gas. Oxygen is added by coulometric generation into the second-stage nitrogen purge gas to obtain dissolved oxygen contents of 0 to 100 ppb accurate to ± 2 ppb. An improved modification of the Winkler procedure for dissolved oxygen was also developed which has a precision and accuracy of better than ± 2 ppb. Edgington (11M) further investigated end-point detection, handling parameters, characterization of interferences, reagent characteristics, and interfering ions to find conditions which will permit optimization of the Winkler determination. The optimized method has a precision of 0.75 μg of oxygen per liter and

an accuracy of 1% in the range from 0 to saturation. Won and Park (55M) compared the accuracy of the microdetermination of dissolved oxygen in water with that of ordinary macrodetermination using the Winkler method. The difference between the macro and micro methods was insignificant, and 97% of the 30 measurements agreed within 5% of the significant level. Smirnov (45M) determined traces of dissolved oxygen (0.04 to 0.14 mg per liter) in sea water by using a modification of the Winkler methods. Extreme care was used in the preparation of initial solutions.

Mor, Beccaria, and Poggi (35M) reported that the interference of ferric and cupric ions on the Winkler method for dissolved oxygen is eliminated by preliminary coprecipitation with potassium hydroxide and zinc chloride in a closed apparatus. Errors in the subsequent spectrophotometric determination of the iodine-starch complex range from 10 to 5% for oxygen concentrations of 0.02 to 10 ppm, respectively, and are less than 1% for higher concentrations. A modified Winkler method for determining dissolved oxygen in sea water containing sulfides is described by Mor and Beccaria (34M). For dissolved oxygen concentrations between 0.2 and 0.8 ppm, the method gives good reproducibility and a maximum error of 7%. Lur'e and Nikolaeva (28M) reported on the determination of dissolved oxygen in water containing nitrites (1 to 15 mg per liter) by a standard analytical procedure based on the Winkler method. The results of the analyses of the samples in which nitrite ions were decomposed by sulfamic acid or urea before the fixation of oxygen on manganese hydroxide are high and less reproducible. No satisfactory explanation was found for this phenomenon.

The complexometric determination of oxygen in aqueous solutions was studied by Gertner, Grdinic, and Parag (16M). The tetraamminecopper(I)-chloride complex, formed in an ammonium hydroxide-ammonium chloride solution containing copper powder, is transformed upon the addition of dissolved oxygen into the tetraamminecopper(II)-chloride complex. This solution is then heated to boiling and titrated with 0.1M EDTA to a yellowish green color with 1-(2-pyridylazo)-2-naphthol as indicator. Reproducibility of the method is good, and no influence from impurities usually present in natural and waste waters was observed. Evsina and Nagibina (13M) described a titrimetric method for determining dissolved oxygen in the presence of both ferric and ferrous ions. Ferrous ion is oxidized to ferric ion with potassium permanganate in acid medium. Soluble oxygen is combined in the form of manganese hydroxide, and higher oxides of manganese are titrated with a solution of hydroquinone. The error is 3 to 5% and the divergence between parallel determinations is 1 to 3% for over 60 samples of mine waters.

Walker, Williams, and Hammer (52M) used the Miller method to determine oxygen in saline lakes because the Winkler method is ineffective in saline waters with enough carbonate to cause effervescence after acidification. Miller's method, although less precise, is effective in well oxygenated waters; however, calcium and magnesium interfere when EDTA hardness is greater than 200 mequiv per liter as calcium ion. The method of Shibata-Miller was modified by Mori and Ishihara (36M) to reduce the interference of various ions, such as ferrous and sulfite. A spectrophotometric determination of oxygen dissolved in water with methylene blue was described by Devdariani *et al.* (9M). Oxidizing substances, especially cupric and ferric ions, are eliminated by passage through a cation exchanger or by complexing with 4 grams per liter of glycine.

Soltysiak and Dembek (48M) outlined a colorimetric procedure for determining dissolved oxygen in water. Dissolved oxygen oxidizes iron(II) (from ethylenediamine ferrous sulfate) at pH 7.5 to iron(III) which subsequently oxidizes *N,N'*-bis(β -hydroxypropyl)-*o*-phenylenediamine to a red-colored product. The absorbance is measured at 525 nm. Concentrations greater than or equal to 0.1 ml of oxygen per liter can be determined. A hydroquinone method for determining dissolved oxygen in water was compared with the Winkler method by Lur'e and Nikolaeva (29M). In the final titration of manganese(III)-phosphate complex, hydroquinone can be replaced by Mohr's salt as a reducing agent. A method for photometrically determining dissolved oxygen in water by its oxidation of phenothiazine dyes that are photochemically reduced to the leuco forms was developed by Hamlin and Lambert (19M). Beer's law is followed at 610 nm for determining 16 to 385 ppb of oxygen by using the photo-reduced sodium EDTA-methylene blue reagent at pH 11.2. Nitrate, nitrite, sulfate, bicarbonate, sulfide, iron, and manganese(II) (each in ppm amounts), and possibly hydrogen peroxide and ozone, interfere. The dye reagent is deaerated with nitrogen and photoreduced with ordinary fluorescent light. Reagent regeneration requires exposure to strong artificial light or sunlight.

Czerniec *et al.* (8M) described a colorimetric method for determining 0.1 to 1.3 mg of ozone per liter in purified potable water. This method is based on the discoloration of methyl red dye by ozone. Strong oxidants, such as chlorine, interfere. Determination of dissolved ozone in potable water was outlined by Wierzbicki and Pieprzyk (54M). Siphon 50 ml of water, containing 0.05 to 2 mg of ozone per liter, into a dark bottle, add 5 ml 1:3 sulfuric acid, 5 ml 0.1N potassium iodide, and 5 ml 0.5% starch. The absorbance is read at 570 nm with reference to a blank.

Moore and McNulty (33M) compiled a review with 11 references describing quantitative total analysis by microcoulometric titration of trace nitrogen in water, trace sulfur in air and combustibles, and trace halogens in solution. Instrumentation capabilities and modes of operation, improvements in detection limits, and applications to pollution problems are discussed.

Rhodes and Hopkins (43M) reported a microcoulometric method for the determination of traces of nitrogen in water. Interfering carbon dioxide produced during sample pyrolysis over a nickel catalyst in excess hydrogen is removed by adsorption on Ascarite. Ascarite also removes acid gases which are produced by pyrolysis of halogens and sulfur. From the ammonia-containing stream, nitrogen (greater than or equal to 0.38 ppm) is determined. An electrolytic calibration technique for determining dissolved nitrogen in sea water by on-stream stripping gas chromatography is described by Atkinson (2M). The system incorporates a stripping chamber, a helium carrier, and stripping gas in a molecular sieve 5 Å packed L-shaped gas chromatograph equipped with a helium-ionization detector. An activated charcoal column chemisorbs the oxygen prior to nitrogen and argon determinations. The overall error is 0.5%.

A low-cost procedure by which approximate concentrations of oxygen, carbon dioxide, and inert gases dissolved in water can be obtained is presented by Post (42M). The technique is specially designed to help fish culturists cope with water supersaturated with gas coming from a pressurized source. Heat is used to drive the gas from a given sample volume of water into a syringe. The carbon dioxide and oxygen are successively chemically adsorbed, and the

reductions in volume of gas noted. The remainder is considered inert gas. Tables are given to assist in computing per cent saturation.

Several instrumental methods for the determination of halogen residuals in water are reviewed by Marks (30M). Spectrophotometric, titrimetric, electrode, electrode depolarization, and polarographic methods are discussed.

Marks (31M) compiled a review with 121 references on 14 analytical techniques used for the determination of residual chlorine in water and waste water.

The use of syringaldazine in a photometric method for estimating "free" chlorine in water was investigated by Bauer and Rupe (3M). Syringaldazine, buffered at pH 7, acts as a chromophoric agent. The reagent is sensitive to hypochlorite, but is insensitive to bound chlorine compounds, such as chloramines. The molar absorptivity is about 65,000 at 530 nm, and the developing color deviates only slightly from Beer's law at low concentrations.

Some known potentiometric and colorimetric methods for determining chlorine dioxide in water solutions are described by Ascik (1M). The colorimetric method with tyrosine at pH 4.6 was acknowledged to be the most selective. The presence of hypochlorous acid, chloramine, and manganous ion does not interfere. Thielemann (51M) studied the use of thin-layer and paper chromatography to separate and identify chlorite, hypochlorite, chlorate, and perchlorate ions. These ions are separated by thin-layer chromatography on silica gel G plates impregnated with a 2:1 acetone-formamide mixture and activated at 120 °C with 60:25:5:10 butanol-acetone-ammonia-pyridine mixture as the developing solvent. The spots are sprayed with bromocresol purple and detected by ultraviolet light. The *R_f* values are 36, 38, 58, and 64 for chlorite, hypochlorite, chlorate, and perchlorate, respectively. Chloride interferes with the detection of hypochlorite. Attempts to separate the anions by paper chromatography were not successful.

Palin (40M) determined chlorine dioxide and chlorite by a modified form of the titrimetric and colorimetric *N,N*-diethyl-*p*-phenylenediamine methods. Malonic acid is added to suppress free chlorine.

Colorimetric methods for the determination of free bromine and bromamines in water were investigated by Sollo, Larson, and McGurk (46M, 47M). Twenty-seven reagents were screened on the basis of their reaction with hypochlorous acid, hypobromous acid, chloramine, and bromamine. Methyl red, phenol red, *N,N*-diethyl-*p*-phenylenediamine oxalate, bromocresol purple, and phenosafranine were selected for detailed investigation, primarily because interferences due to chloramine, iron(III), nitrite, and manganese(III) are low. Methyl orange is suitable for the determination of total bromine in the absence of free chlorine; phenol red is a unique reagent for total bromine with no interference from chlorine or chloramine; diethyl-*p*-phenylenediamine oxalate is suitable for the determination of total bromine in the absence of chlorine; bromocresol purple appears suitable for the determination of free bromine with interference only from free chlorine and tribromamine; and phenosafranine may be useful for high concentrations of free bromine.

Kokoshkin and Ivanitskaya (22M) studied a catalytic method for determining microconcentrations of hydrogen dissolved in water. Substitution of safranine oxygen in a catalytic method improved the hydrogen detection in condensate and super-heated steam (free of hydrazine) to a level of 0.3 µg per kg. In 33 determinations at hydrogen concentrations of 1.4 to 15.2 µg per kg by the catalytic-

oxidation and gas-chromatographic techniques, the results agreed within 0.3 μg per kg.

A new procedure is outlined by Sinel'nikov (44M) which permits the determination of hydrogen peroxide in water up to 3 to 4 hours after sample collection. Luminol is used as a chemiluminescent indicator, and the specificity of this method toward hydrogen peroxide is achieved by using catalase to decompose the hydrogen peroxide. The sensitivity is 0.5 μg of hydrogen peroxide per liter.

Hahn (18M) described an improved gas-chromatographic method for field measurement of nitrous oxide in air and water, using a 5 Å molecular sieve trap. Included is a complete description of the instrumental apparatus. It was concluded that the accuracy of this in situ method for sea water was better than $\pm 3\%$.

A bottle for the storage of water samples prior to analysis for dissolved-gas concentrations was designed by Weimer and Lee (53M) to eliminate contact between the sample and the atmosphere. Analysis of water samples for dissolved nitrogen and methane content indicated that the concentrations of these gases remained unchanged during a 2-day storage period and that the samples could, potentially, be stored for longer time periods.

DETERGENTS

An automatic method based on the formation of a blue-colored complex with methylene blue was discussed by Wudzinska and Ponikowska (13N). The complex is extracted with chloroform, and the chloroform phase passed through a filtration tube and then through the flow-cell of the colorimeter. The absorbance is measured at 650 nm. When determining surfactants in wastewaters, the emulsion formed during the chloroform extraction is removed by diluting the samples with 10% sodium sulfate solution. The determinable concentration range corresponds to 0.05 to 1 mg ceryl benzenesulfonate per liter. Nozhaev (8N) also determined anion-active surfactants in water. Hydrogen peroxide, phosphate buffer, chloroform, and a neutral solution of methylene blue are added to the sample and mixed. After one minute of stirring, sulfuric acid is added. The coloration of the chloroform layer is compared with a series of freshly prepared standards. Sensitivity of the method is 0.05 to 0.1 mg per liter, and the error of determination is less than 20%. The concentration and separation of surfactants from surface waters by transport in the gas-water interface was studied by Wickbold (12N). Surfactants of low concentrations are concentrated for analytical purposes by blowing nitrogen through the test water, transporting the surfactants to the water surface where they are absorbed by an overlying ethyl acetate solvent layer; the procedure is repeated, and the two ethyl acetate phases are evaporated to dryness. The resulting residue is dissolved in methanol and water, and the surfactant determined by a methylene blue method. An attempt was made by Ambe and Hanya (3N) to determine the concentration of alkylbenzenesulfonate in polluted waters in which methylene blue active substances coexist. Methylene blue colorimetry and infrared spectrometry is used. The above complexes are extracted from water with 1,2-dichloroethane, and the absorbance is measured at 655 nm. The solution is then evaporated, and the residue analyzed by infrared spectrometry. The absorption bands at 890 cm^{-1} and 1010 cm^{-1} are characteristic of methylene blue and alkylbenzenesulfonate, respectively.

The specific determination of nitrilotriacetic acid in the ppm range in surface water and sewage was investigated by Wernet and Wahl (11N). The ac polarogram is record-

ed after interfering ions are removed by cation-exchange resin. The method is calibrated by the method of additions and has a detection limit of 1 ppm and less than 0.1 ppm for the original sample and the sample after concentration, respectively. The maximum of the polarographic peak is at -0.8 V. Asplund and Wanninen (4N) used a polarographic method to analyze samples of lake water containing nitrilotriacetate (NTA) in concentrations from 1 to 10 ppm. The NTA is converted to its 1:1 cadmium complex by adding a 100% excess of cadmium. The method can be utilized also when sodium tripolyphosphate and sodium alkylbenzenesulfonate are present in the sample. Two methods for the determination of nitrilotriacetic acid in natural water are proposed by Afghan and Goulden (1N). Differential cathode-ray polarography is used to determine "free" NTA in natural water after the formation of a lead-NTA complex in alkaline medium at pH 8.0. The determination of "total" NTA is based upon releasing NTA from metal complexes under acid conditions. The subsequent addition of EDTA and return of the sample to pH 8 preferentially binds the metals with EDTA. This frees the NTA from metal complexes, and the released NTA is then determined by the lead-NTA method. An automated method for determination of total nitrilotriacetic acid in natural waters, detergents, and sewage samples is described by Afghan, Goulden, and Ryan (2N). This method is based on the formation of a bismuth-NTA complex at pH 2 using twin-cell oscillographic dc polarography. Fifteen samples per hour can be analyzed with a detection limit as low as 10 μg per liter without any pre-concentration of the sample. The coefficient of variation at 100 μg per liter is 1.3%. A method for determining low concentrations of nitrilotriacetic acid in natural waters was developed by Murray and Povoledo (9N). The NTA is converted to the trimethyl ester and determined quantitatively by gas chromatography using methyl heptadecanoate as an internal standard. The lower limit of detection is 25 μg per liter of NTA. Quantitative methods were developed by Warren and Malec (10N) for determining nitrilotriacetic acid, iminodiacetic acid, *N*-methyliminodiacetic acid, glycine, sarcosine, and *N*-oxalyliminodiacetic acid in a variety of water samples and *N*-nitrosoiminodiacetic acid in river water. These constituents are converted to volatile esters, separated, and analyzed on an ethylene glycol adipate gas-chromatographic column. Relative standard deviations usually range from 8 to 13%. A sensitive and selective polarographic method for determining traces of nitrilotriacetate in water samples is described by Haberman (6N). This method is used to determine NTA as an indium(III)-NTA complex for 0.0257 to 2.57 ppm when anion-exchange concentration is used and for 2.57 to 257 ppm without concentration. The relative standard deviation is approximately 10% with the exception of the lowest concentration.

The polarographic determination of surfactants in water was discussed by Linhard (7N). The damping of oxygen maxima, as determined by polarography of oxygen-saturated solutions, is proportional ($\pm 2\%$ maximum deviation) to weight of anionic, cationic, and/or nonionic surfactants in 0.001M potassium chloride solutions containing less than 100 mg of surfactant per liter.

Buerger (5N) discussed the use of gas and vapor-phase chromatography for determining gaseous and volatile constituents in water and effluents. The determination of low-volatility compounds by liquid chromatography is also described. Methods for determining anionic, cationic, and nonionic detergents in effluent water were developed.

PESTICIDES AND HERBICIDES

Gitsova (4P) compiled a review with 128 references on the occurrence and determination of pesticides in natural waters.

Four procedures for extracting chlorinated hydrocarbons from water were examined by Weil and Quentin (7P). Samples of water containing 10 μg per liter of DDT, lindane, heptachlor epoxide, dieldrin, and methoxychlor with and without 500 mg of suspended matter per liter were extracted with petroleum ether by three cold-extraction methods and one hot-extraction method. A direct cold-extraction method using a special glass microseparator showed a recovery of slightly more than the added amount of pesticide in the clear sample and approximately the added amount in the samples containing suspended matter. The other extraction methods gave lower results.

The gas-chromatographic determination of insecticides in the water of the Rhine River was discussed by Hellmann (5P). The determination is performed after enrichment by extraction and thin-layer chromatography. A detailed method for the determination of Thiodan is given. The method can also be used for the determination of lindane and parathion. Quantitative determination is possible by calibration with standard solutions. A rapid method for the determination of trace amounts of chlorinated hydrocarbon insecticides in water is described by Herzel (6P). The insecticides are extracted into toluene and determined by gas chromatography. With 4-liter water samples, the following amounts of insecticides (in ng per liter) can be determined: lindane and aldrin, 5 to 10; DDT and derivatives, 100 to 200; and other chlorinated insecticides, 10 to 30. Bevenue, Kelley, and Hylin (1P) discussed the contamination of water samples by organic solvents, glassware, plastic ware, cellulose extraction thimbles, filter paper, and silica gels with regard to subsequent interference in the gas chromatographic analysis of samples for ppb-amounts of pesticides. It is recommended that the use of plastic ware and filter paper be avoided, that high-purity solvents be used, that glassware be purified by washing with acetone, air-drying, and heating 16 hours at 200 °C, and that silica gel adsorbents be heated 16 hours at 300 °C prior to use.

Colorimetric methods and paper and gas-liquid chromatographic methods were compared by Colas *et al.* (2P) for the extraction and determination of certain phenoxyalkanoic herbicides. A method based on the gas chromatography of methyl derivatives is proposed. The effect of different columns on the separation of difficult pairs is discussed.

Two simple aids designed for the determination of pesticides or insecticides were developed by Croll (3P). A stopper-securing clip was designed to secure dust-proof stoppers in 500-ml reagent bottles. A solvent separator was built consisting of two glass taps for phase separation after the extraction step.

OXYGEN DEMAND AND ORGANIC CARBON

Jones (15Q) compared the total organic carbon method to the COD and 5-day BOD methods for the analysis of plant effluents, receiving waters, and waste waters. Good correlation between the TOC and the BOD and COD is obtained only when the waste is from a single source or is of relatively constant composition. Both the BOD and COD methods are affected by certain oxidizable inorganic compounds while the TOC is unaffected by the inorganics normally found in water. Neither the BOD nor the COD shed light on the biochemical time rate. The conditions

which must be met in reasonably clean waters to obtain accurate results using the COD, 5-day BOD, and potassium permanganate determinations were discussed by Leithe (22Q). He has also stated that the potassium dichromate is far more accurate than the potassium permanganate method for determining small concentrations of organic pollutants. Surface waters of differing saprobic levels were compared by the three methods. COD:BOD relations were closer in oligosaprobic and polysaprobic than in mesosaprobic waters. Leithe (20Q) investigated a method for the simultaneous determination of chemical oxygen demand, organic carbon, and nitrogen in waste water. The sample is boiled for 2 hours with mercuric chloride, silver sulfate, sulfuric acid, and potassium dichromate; and the organic carbon is determined by titration with 0.1N hydrochloric acid. The titrated sample is divided into two parts, and the COD value is determined on one part by titration with ferrous ammonium sulfate with ferroin as indicator. Ammonia is distilled from the other part into a known amount of 0.01N sulfuric acid, and the excess acid is back-titrated with 0.01N sodium hydroxide. Recoveries of more than 95% were obtained for most model substances tried. The analytical detection of organic impurities in both community and industrial waste water was discussed by Leithe (21Q). It was stated that the total amount of impurities can be determined by determining organic carbon or COD. It was also mentioned that 5-day BOD values can be obtained by differential COD determinations at the time of sampling and after 5 days.

A review with 20 references on the BOD test and its meaning was authored by Grady (14Q). Nikitina (29Q) stated that determinations of 5-day BOD by standard methods are frequently erroneous because of the nitrification taking place during the 5-day standing of the samples. Parallel determinations of the 5-day BOD by the standard method and by methods in which the samples are treated with either sulfuric acid or with a 0.1M solution of ammonium chloride showed that all three methods gave the same values. In the presence of nitrification, values for samples in which nitrification was inhibited were about half the values obtained by the standard method. Further studies are necessary to select a method giving the most reliable results. Koivo and Phillips (17Q) discussed the determination of BOD in polluted stream models from dissolved oxygen measurements only. The method described is based on the use of a modified mathematical model of Streeter-Phelps which relates the average daily DO and BOD concentrations in polluted streams under steady-state conditions. The method given provides a least-square fit to the DO measurements. This procedure requires little computer time and memory and can thus be incorporated in on-line water control and management. A method by Pauschmann (31Q) used a Heathkit EC-1 analog computer to calculate the 5-day BOD determination by titration of waters.

The use of a manometric method for determining the BOD in waste water is reviewed by Zakhar'ina and Kuz'min (39Q). Twenty-seven references are included. Schaumburg (33Q) used heavy metals, such as mercury, chromium, and copper, to preserve samples containing organic matter for later BOD measurement. Mercury was chosen on the basis of comparative tests with other metals mentioned. After storage the sample is depoisoned by chelation using Chelex 100 which forms a stable compound with mercury. The poisoning-depoisoning method was comparable to refrigerated storage at 4 °C for glucose,

beet cannery waste, and settled domestic waste water. A new technique for determining the BOD of waste water was outlined by Catroux and Morfaux (9Q). The BOD is determined indirectly by comparing the oxygen requirement of a system before and after seeding with activated sludge followed by incubation. This method is applicable to BOD concentration ranges of 200 to 3000 mg per liter.

Fleet, Ho, and Tenygl (13Q) discussed the automatic determination of COD for polluted waters from the amount of dichromate or permanganate consumed in the digestion step. The excess oxidant reacts, in acidic media, with hydrogen peroxide and the liberated oxygen is measured with a new type of coulometric sensor. Values obtained by this method were compared with values obtained by standard methods. Interferences caused by chloride and nitrite were evaluated and methods for removing the interfering effects are outlined. Lur'e and Nikolaeva (23Q) evaluated two methods for determining dichromate oxidizability. In the first method, potassium dichromate and sulfuric acid are added to a 1-ml aliquot of sample. The solution is heated by the exothermal reaction to 100 °C and the oxidation of organic material is completed in two minutes. The addition of silver sulfate is avoided by the use of an empirical factor: 1 mg of chloride corresponds to 0.15 mg of oxygen. The recovery is 78 to 82%. The second method is not recommended because it is more time consuming and yields only 40 to 50% recoveries. A rapid, potassium dichromate method using two submethods is described by Leithe (18Q) for the determination of COD in water. The first submethod is for 100 to 700 mg of COD per liter; the second, for 1 to 150 mg of COD per liter. Both submethods use ferroin as the indicator and ferrous ammonium sulfate as the titrant. In the latter method, a more dilute solution of indicator and titrant is used. A photometric determination of the green chromium(III) ions is also proposed. Absorbance readings are made at 610 nm with oxalic acid solutions as standards. Beer's law is obeyed. Monakova and Skopintsev (27Q) compared five dichromate oxidizability methods for the determination of organics in evaporated natural water residues requiring from 18.32 to 44.79 mg of oxygen per liter. The results were within 1.7% even though the amounts of potassium dichromate used varied by a factor of 15. The dichromate oxidizabilities determined by the dry method were approximately 2% lower than when determined for unevaporated water. Leithe (19Q) outlined a potassium dichromate method for the determination of COD in water. Almost all soluble substances in domestic and industrial waste and surface waters were oxidized.

A new modification of a determination of permanganate oxidizability of sea water in alkaline medium was described by Skopintsev and Nikolaeva (35Q). A study of 71 samples of water, taken in various parts of the Atlantic Ocean, showed that the average ratio of oxygen from permanganate oxidation in alkaline medium (in mg of oxygen per liter) to organic carbon (in mg of carbon per liter) is 0.43 to 0.62. The ratio of oxygen from permanganate oxidation in neutral medium to oxygen from permanganate oxidation in alkaline medium in sea waters is, on the average, 0.25. Various methods used in the determination of COD with permanganate as oxidant were compared by Karlgren and Ekedahl (16Q) by having samples of water analyzed in different laboratories. A permanganate oxidation method was recommended after this comparison.

An apparatus is described by Opperskalski and Siebert (30Q) in which dissolved organic carbon is determined automatically within 10 minutes using a wet chemical oxida-

tion with subsequent measurement and integration of the carbon dioxide by an infrared analyzer. A wet combustion method using an apparatus for automatic titration with continuous recording of results is proposed by Lyutsarev (24Q) for the determination of dissolved organic substances in sea water. A mixture of silver dichromate, potassium dichromate, and sulfuric acid is used as the oxidant. The error in the determination of carbon is $\pm 5\%$. The determination of organic carbon in fresh water by a peroxydisulfate method is discussed by Bikbulatov, Skopintsev, and Bikbulatova (5Q). Total organic carbon is oxidized with potassium persulfate, in the presence of platinum catalyst, to carbon dioxide which is absorbed in 0.02N barium sulfate solution. Halogens, nitrogen oxides, and sulfur are removed by silver sulfate solution, concentrated sulfuric acid, and silica gel saturated with dichromate. The inorganic carbon is determined separately and is subtracted to yield total organic carbon. Abrahamczik *et al.* (1Q) determined organically bound carbon in water using a silver permanganate oxidation. First, the inorganic carbon is removed by acidification and subsequent heating while bubbling oxygen through the solution. Volatile organic compounds are collected in a cooling trap. The organic carbon is determined by wet oxidation followed by a dry combustion on the decomposition product. The carbon dioxide is absorbed after purification and continuously titrated with tributylmethylammonium hydroxide solution using thymolphthalein as an indicator. Two different reactions for the wet oxidation are given. The sensitivities are 6 mg of carbon per liter for the first method and 0.9 mg of carbon per liter for the second. Malissa and Bleier (25Q) outlined a method for the determination of organic substances in water and waste water. The organic substances are oxidized in a mixture of sulfuric acid and potassium dichromate at 180 °C for 15 to 20 minutes. The resulting carbon dioxide is determined with a conductometric analyzer.

An ultraviolet irradiation and photochemical technique accurate to $\pm 1\%$ in the determination of total organic carbon in water is described by Bikbulatova, Skopintsev, and Bikbulatov (6Q). The method is also suitable for the determination of organic nitrogen and phosphorus. A photochemical method for determining organic carbon in fresh and surface water was investigated by Soier and Semenov (36Q). High-intensity ultraviolet light was nearly 100% effective in the photolytic oxidation of organic carbon to carbon dioxide, which was then carried by a stream of purified air into a Reberg cell containing barium hydroxide, where it was titrated coulometrically. Sensitizers speeded up the ultraviolet-coulometric water analysis to 1 hour per sample. The high-intensity ultraviolet radiation readily broke the complex ester-phosphorus bonds; the analysis thus yielded simultaneously total organic carbon and phosphorus. The total carbon determined in several waters by the ultraviolet-coulometric method was higher than the carbon determined by pyrolytic techniques. Mordasova (28Q) also used ultraviolet radiation to determine organic carbon in sea water. After irradiation, the evolved carbon dioxide is determined by the charge required to generate barium hydroxide which keeps the pH of the absorbing barium chloride-barium hydroxide solution at 9.5. Tests with organic compounds gave a 98% oxidation in 1 hour. Comparison with other methods gave a 5 to 7% difference.

A method is presented by Mikheeva, Bitkova, and Kogan (26Q) whereby acidification of ground water with dilute sulfuric acid to pH 3 permits the determination of

more than 100 μg per ml of total carbon with an accuracy of $\pm 10\%$ in the presence of large amounts of chlorides and carbonates. Continuous automatic analyzers for the determination of total organic carbon and total carbon in water and waste water are discussed by Boucher, Vitale, and Ricci (7*Q*, 8*Q*). The three basic functions of the TOC analyzer are outlined and its flow diagram, showing an optional in-line blender and the most critical functions, are schematically illustrated. The oxidation system which utilizes the fluidized bed reactor is described. The suitability and accuracy of continuous total organic carbon and total carbon analyzers utilizing a fluidized bed reactor have been successfully demonstrated for carbon concentrations of 10 to 1000 mg per liter. The TOC analyzer has been shown to remove efficiently inorganic compounds up to 400 mg per liter as calcium carbonate. Emery, Welch, and Christman (12*Q*) used a TOC analyzer to determine total carbon of reservoir water. Fluctuations in TOC and BOD, at three stations, were plotted and compared with each other. A positive correlation was observed between total and organic carbon and long-term 5-day BOD in the reservoir. The analyzer has two combustion furnaces: one oxidizes all carbon materials at 950 $^{\circ}\text{C}$, and the other only inorganic carbon at 150 $^{\circ}\text{C}$. A single carbon value was determined in 2 minutes with an accuracy of 1% and a sensitivity of ± 1 mg per liter. Sander (32*Q*) described an apparatus for the determination of organically bound carbon in water and sewage samples. The system consists of a combustion tube with a gas burner, a condensate cooler with an absorption device, a reflux condenser with an acid funnel, and wash bottles. Similar apparatus for the continuous measurement of organic carbon in drinking and waste waters were described by Schmitz and Bauer (34*Q*) and Axt (2*Q*, 3*Q*). The method is based on the evaporation and combustion of organic substances at high temperatures with the use of catalysts for the oxidation of carbon to carbon dioxide and the subsequent determination of the carbon dioxide by a selective infrared instrument. Bauer and Schmitz (4*Q*) also used a Beckman Carbonaceous Analyzer to determine organic carbon in river water polluted with waste water from paper industries. The results were compared with a 5-day BOD and potassium permanganate consumption, and a good correlation was obtained. The sensitivity of the determination is 0.5 mg of carbon per liter. An analyzer for the determination of the organic carbon content and oxygen demand of waste water is described by Stenger (37*Q*). In the analyzer, the combustion takes place in a quartz tube at 950 $^{\circ}\text{C}$ in the presence of a cobalt oxide catalyst to ensure complete combustion. The carbon dioxide produced is measured by means of a nondispersive-type infrared analyzer. The TOC analyzer has an extra unit consisting of a tube packed with quartz chips acidified with phosphoric acid operating at 150 $^{\circ}\text{C}$ to differentiate between total carbon and carbonate carbon.

Eggersten and Stross (11*Q*) state that trace organic carbon in water can be determined by a thermal analytical flame detection system previously devised for the thermal stability and volatility characterization of organic materials. A small sample is heated in nitrogen carrier gas in two stages to determine volatile (less than 150 $^{\circ}\text{C}$) and nonvolatile (150 to 550 $^{\circ}\text{C}$) organic carbon with a hydrogen flame ionization detector. Performance of the method is illustrated in tests with 1 to 300 ppm of various types of organic material. The lower level of detection is approximately 0.2 ppm. The analysis time is about 15 minutes. An apparatus and a flame ionization method, capable of

determining organic carbon in water at low concentrations, are described by Croll (10*Q*). The unattended determination time is less than 16 minutes. At carbon concentrations of 0.5, 5.0, and 50 mg per liter in water, the relative standard deviations are ± 38 , ± 7 , and ± 1.7 at instrument standard deviations of ± 3 , ± 0.6 , and $\pm 0.7\%$, respectively. The oxidation furnace tube is constructed of Inconel, and a self-indicating silica gel is used to remove water and ammonia from the methane flow being analyzed. Ratios of organic to inorganic substances contained in suspensions of sea and river waters were determined by Vityuk (38*Q*) with a high degree of accuracy by a technique described. The method is also useful for the determination of carbon dioxide and carbonates in very small samples. The organic fraction is determined by difference between the total deposit collected on a microfilter and the sum of inorganic calcined residue and the carbonate-carbon dioxide content.

ORGANICS

The identification of organic substances in natural waters and the determination of total organic content from total carbon determinations were reviewed by Skopintsev (67*R*). Thirty-nine references were included. A review with 14 references, compiled by Corwin (14*R*), detailed the presence of dissolved organic materials in the ocean, as well as the development of methods to assay the organic content. Attention is called to procedures that eliminate or destroy the volatile material. Methods for determining the volatile organic content of sea water are reviewed, and a method for determining low-molecular weight, oxygen-containing organic compounds is reported. The detection of organic substances in water and the identification of pesticide residues by infrared methods are included in a short review by Ulrich (78*R*) on the applications of some analytical spectroscopic techniques to problems of current interest. A pyrographic method for the analysis and identification of water pollutants is described by Lysyj (46*R*). The technique consists of direct pyrolysis of samples containing organic pollutants; gas-chromatographic separation and hydrogen-flame ionization measurement of pyrolytic fragments produced; and application of mathematical techniques for the interpretation of resulting complex pyrograms. No sample preconcentration or preanalytical handling is required.

A general analytical system for the identification of organic matter in polluted water is described by Rosen (59*R*). Isolation of individual pollutants makes use of solubility group separations, adsorption chromatography, and specific separation techniques. The most rewarding of these have proved to be gas chromatography and spectrophotometry. After separation, a wide array of organic analytical methods can then be used. Goma and Durand (24*R*) described a method for the determination of hydrocarbons in water by gas chromatography. Pretreatment consists of homogenization by ultrasonic-wave treatment in the presence of a surfactant which has either a solubilizing or a stabilizing effect. Kaiser (31*R*) investigated the determination of trace amounts of volatile compounds by reversion gas chromatography and low-temperature gas chromatography with emphasis on the minimization of systematic errors. The sources, theory, and methods for recognizing systematic errors are discussed. Low-temperature gas chromatography with column enrichment dosing several minutes at -60 $^{\circ}\text{C}$ permits the analysis of more than 10^{-2} ppb of organic volatiles in solids and liquids. A method has been developed by Burnham *et al.* (9*R*) for

extracting trace organic contaminants from potable water using macroreticular resins. These resins quantitatively extract weak organic acids and bases and neutral organic compounds from water solutions at ppm-ppb levels. The identification and quantitative determination of individual contaminants are ascertained using a gas chromatograph-mass spectrometer combination and other spectrometric methods. Maier (49R) describes a method by which organic compounds in water are determined by heating oxidizable mixtures according to a definite temperature program. Organic compounds are estimated in sequence as they were oxidized to carbon dioxide. Limitations of the method include water containing volatile materials, water vapor, or azeotropic mixtures. Polar materials are estimated by comparison of their cited oxidation spectra. Foster and Morris (20R) used ultraviolet absorption measurements to estimate the organic pollution of in-shore sea waters. The correlation between the ultraviolet absorption and the dissolved organic content was poor. Appreciable short-term variations in ultraviolet absorbance measurements have been recorded which severely limit the use of these measurements for an indication of marine-organic pollution.

A fluorometric method for the on-board-ship determination of low concentrations of organics in sea water is described by Lyutsarev (48R). The organic content is determined by comparing the sample fluorescence with that of quinine disulfate. Sinel'nikov (66R) discussed a paper-chromatographic method for concentrating organic substances contained in natural waters. The loss of organic substances was 13 to 24% when the inorganic salts were washed off the colored chromatograph. The amount of organic substances concentrated is determined by chromatogram weight loss after treatment with 2N potassium hydroxide. The luminescence of chromatogram areas is more intense with less coloring of water.

Lysyj (47R) discussed a pyrographic instrument for determining water pollutants. After the organic matter is subjected to elevated temperatures in the absence of air, the thermal decomposition fragments are identified by gas-chromatographic or mass-spectral analysis. Parent material is deduced from pyrograms on known matter. Uncoated porous glass and Porapak are used as gas-chromatograph substrates.

Heil (25R) states that the analytical differentiation of chemically similar substances can be carried out on the basis of their individual kinetic reactions. For the characterization and determination of organic compounds in water, oxidation and adsorption kinetic reactions are most suitable.

Pastorelli and Chiavari (55R) used nitromethane or *n*-heptane to extract organic matter from sea water. High-molecular-weight alkanes and phthalates were determined by using a gas chromatograph with a flame ionization detector. Nitromethane extracts were determined with a mass spectrometer. Khailov (38R) outlined a gel-filtration method which provides for concentration and separation of numerous hydrophilic compounds into individual fractions dissolved in sea water and, in particular, of sea-organism metabolites. The concentration of solution with dry Sephadex, the separation of concentrated solution on the columns of neutral Sephadex, and the filtration of each molecular fraction through a Sephadex-ion exchanger are recommended for study of proteins and peptides secreted by single-cell algae in surrounding media. Getseu *et al.* (21R) compared the method of Bakhman with a fractionation method for the qualitative determination of various

organic compounds from aqueous solutions. Kelus and Sikorowska (36R) recommended a freezing method to concentrate organic compounds by a factor of 10. The groups present in water solution can be detected or separated further by liquid-liquid extraction at room temperature. Baker (2R) studied the effect of pH, ionic strength, mixing, and total dissolved salts on the efficiency of recovery of organic solutes from synthetic laboratory solutions and tap waters. Although total dissolved salts and mixing were the principal factors, recovery increased with increased mixing and acidification, and with a decrease in ionic strength and total dissolved solids. A continuous liquid-solvent extractor, designed to utilize organic solvents that are heavier than water, for the continuous extraction of organic materials from water is described by Goldberg, DeLong, and Kahn (23R). A procedure for removing fatty acids from polluted river water was discussed by Chambon and Chambon-Mougenot (12R). These acids interfere with the infrared-spectrophotometric determination of aliphatic hydrocarbons and are extracted with a mixture of carbon tetrachloride and sodium chloride.

Three methods and necessary equipment are described by Strafelda (70R) for the automatic determination of phenol in waste waters: 1) Direct-phenol oxidation on a gold stationary electrode in stirred flowing solutions to which sand is added to automatically regenerate the active surface of the measuring anode; 2) direct measurement of total phenol content from its reaction with bromine continually generated in flowing samples by constant current (the bromine decrease, measured by a biamperometric-electrode system, is in proportion to the phenol content); and 3) continuous coulometric titration by bromine which is generated by an electronic control unit according to the instantaneous concentration of phenols in the flowing sample. The effective concentration ranges for the three methods are 1×10^{-7} to $5 \times 10^{-6}M$, 1×10^{-8} to $5 \times 10^{-6}M$, and up to hundreds of milligrams per liter, respectively. A procedure is presented by Eichelberger, Dressman, and Longbottom (18R) for the isolation of many phenols from carbon-chloroform extracts prior to chromatographic identification. Applications of the method to water grab samples, as well as qualitative and quantitative determinations are discussed. Kunte (43R) studied a thin-layer and gas-chromatographic method for the determination of phenolic compounds in water. With this method, 15 different compounds can be qualitatively and quantitatively determined at the 0.2- μ g-per-liter level. Starkey (69R) has developed a continuous liquid-liquid extraction system to selectively isolate phenols and eliminate major interfering substances. Stable reagents have been found to reduce spectrophotometric blank color and to extend sensitivity. This method is suitable for the determination of phenols at a level less than or equal to 1 ppb. An ultraviolet spectrophotometric method for the determination of monophenols in drinking water was presented by Cazaceanu (10R). The absorption spectra showed a maximum for *p*-cresol at 245 nm. Curves are presented for concentrations of less than 10 and 10 to 60 μ g per liter. Split-beam and dual-beam ultraviolet photometric schemes are described by Saltzman (61R). Their successful use in the continuous monitoring of phenols in plant effluents is discussed in relation to minimization of plant-yield losses and water pollution. Cazaceanu (11R) discussed a method for determining phenols in water which involves concentration using cationic resins. The phenols are eluted as phenoxides and determined by using a modified method with Pyramidon. Nitrogen-containing

substances and oxidants, such as chlorine, do not interfere. The method is sensitive to 1 μg of phenol. Thielemann (73R, 74R) studied the problems encountered when using several different colorimetric and bromometric methods to determine steam-distillable phenols in water. When determining phenols in water with *p*-nitroaniline, cresols and xylenols gave false positive tests equivalent to 10 to 146% phenol. Aromatic amines (1 mg per liter) also gave false tests equivalent to 0.03 to 0.40 mg per liter. In the bromometric determination, α - and β -naphthylamine also gave false positive tests for phenols. Kawahara (35R) investigated a method in which mercaptans, phenols, and organic acids are converted to pentafluorobenzyl thioethers, ethers, and esters, respectively, in order to eliminate possible organic interferences. Subnanogram amounts of these compounds can then be separated qualitatively and determined quantitatively by electron-capture gas chromatography.

Methods for the gas-chromatographic identification and determination of fuel oil, gasoline, solvents, and pesticides in polluted natural water are presented by Goeke (22R). Jeltse and Den Tonkelaar (29R) compared gas chromatographic and infrared spectrometric methods for the determination of the hydrocarbon content of aqueous solutions of crude oil and six refinery products. Gas chromatography proved to be the most suitable method to determine the hydrocarbon content of water; in addition to a more complete total measurement, it yields data about the individual compounds. The infrared analysis, however, reveals the occurrence of dissolved polar compounds originating from mineral oil. Gas-liquid chromatography was used by Lure, Panova, and Nikolaeva (45R) to identify petroleum pollutants in water. Krivitskaya (41R) described a detailed turbidimetric-chromatographic method for determining petroleum impurities in water. A flame-photometric sulfur detector was used by Adlard and Matthews (1R) for the rapid identification of hydrocarbon pollutants in waters. The system is based on the splitting of a gas-chromatographic stream and the use of a S-selective flame-photometric detector in conjunction with a flame-ionization detector in the analysis of the split streams. The separation and determination of C_{1-5} aliphatic acids in ground waters of oil- and gas-bearing regions by liquid-gas chromatography was described by Mel'kanovitskaya (52R). Caproic acid is used for desorption of the C_{1-5} acids from the column packing. The operating conditions are outlined. Levy (44R) examined a variety of common petroleum products, including distillate and residual fuel oils and lubricating oils, with ultraviolet spectroscopy. The various petroleum products exhibit different absorptions at 228 and 256 nm. These results suggest a basis for an analytical procedure which may be useful for the identification of oil spills. The determination of oil in a steam condensate was investigated by Imanuilov (27R). The oil is extracted from the condensate with carbon tetrachloride, and the infrared absorption is measured at 2866, 2873, and 2941 cm^{-1} . The absorption of carbon tetrachloride is used as a standard. Experimental error is $\pm 10\%$. Makotkina and Yakobson (50R) developed a spectrophotometric method for the determination of oil traces in deaerated water. The absorbance is measured at 320 nm after extraction with carbon tetrachloride. A rapid luminescent-chromatographic method for determining petroleum products in natural water is presented by Semenov, Stradomskaya, and Zurina (64R). The petroleum products in the sample are extracted with chloroform and separated by thin-layer chromatography. The nonpolar and weakly

polar substances, largely hydrocarbons, are extracted with chloroform and determined by fluorescence. A method is given by Bitkova and Kogan (4R) for the determination of volatile organics, especially phenol, in petroleum-bearing strata ground waters. Purified chloroform is used as the extracting agent. The feasibility of detecting oil pollution by means of sophisticated remote sensors, such as ultraviolet and infrared scanners and microwave radiometers, is discussed by Chandler (13R).

Tiunova (76R) described an extraction method for the spectrophotometric determination of benzene, isopropylbenzene, and α -methylstyrene in waste waters. After extraction with *n*-octane, the absorbance is measured and the concentration of each compound determined by means of equations with an error of $\pm 7\%$. Boldina (7R) discussed the chromatographic separation and determination of benzene and isopropylbenzene in water. The concentrations of the two compounds are determined colorimetrically after nitration, chromatography, and color-development steps. Wasik and Tsang (80R) state that benzene can be determined at the ppb level in water by isotope-dilution analysis combined with gas-liquid chromatography using perdeuterated benzene as the isotope. Zhuravleva and Karyakin (82R) presented a rapid phosphorescence method utilizing a spark phosphoroscope and photoelectric recording to determine benzene extracted from subsurface water with cyclohexane. The quasilinear spectrum of benzene in cyclohexane at 348 to 416 nm is obtained at liquid-nitrogen temperature with a sensitivity of $1 \times 10^{-5}\%$ and an error of 15 to 20%. The method can be used also for the determination of toluene and pyridine in ground waters. Bradley *et al.* (8R) used laser Raman spectroscopy to detect 20 ppm of carbon disulfide or benzene in aqueous solutions. A high-resolution Raman spectrometer, cooled detectors, and photon-counting techniques were employed.

Various methods reported in the literature for separating and determining free amino acids in sea water were compared by Bohling (6R). Experimentally, the sea-water samples were concentrated by evaporation and filtration. Sorption of the amino acids onto cation-exchange resin, followed by elution, recovered 80% of the amino acids present. Reproducibility was good. The individual amino acids were separated chromatographically, and nanogram quantities determined after ninhydrin development. A new gas-chromatographic method was used by Pocklington (58R) to determine free amino acids in ocean waters. A complete separation of small samples was achieved quickly and quantitatively. A one-step trimethylsilylation reaction was used to prepare volatile derivatives for injection in the gas chromatograph. Duplicate injections agreed with a mean coefficient of variation of 1.5%, and linearity of response over the range of 0 to 10 nanomoles was established. Paluch and Stangret (54R) modified generally accepted methods in order to determine amino acids in cellulose-sulfite polluted waters. Both qualitative and quantitative determinations were made using ion extraction and sorption on cation resins followed by paper chromatography and elution with organic solvents. Maurer (51R) used thin-layer chromatography to identify amino acids in natural water.

A colorimetric method for determining more than 0.25 mg of humic acid per ml in ground waters is described by Kuleshova (42R). A method for determining naphthenic acid is also discussed. Irimescu, Teja, and Laureescu (28R) investigated a rapid turbidimetric method for determining humic substances in industrial waters which is based on

the formation of suspensions of lead humate. The mean relative error is less than 1.5% and the other water components do not affect the determination. A photometric method is given by Solodovnikova and Yankina (68R) for determining humic and fulvic acids separately in natural waters. Several metal humates were also determined by comparing electron microscopic photographs of natural humate compounds with those of synthesized metal humates.

A detailed description is given by Novotny (53R) for determining pyridine bases, aromatic hydrocarbons, and ketones in waste water by direct-injection gas chromatography. It is possible to determine pyridine and its homologs at concentrations of more than 40 mg per liter. Ketones and aromatic hydrocarbons can be determined at concentrations of more than 100 mg per liter. At lower concentrations, the determination of α -picoline and benzene in the presence of other compounds is uncertain.

A spectrophotometric method for the determination of carbonyl compounds in natural waters is described by Semenov and Kishkinova (62R). The absorbance is measured at 430 nm and it is possible to determine 0.05- μ g equivalents of carbonyl in 20 ml of water. Interferences are discussed. Karyakin, Chirkova, and Anjkina (33R) presented a luminescence method for determining acetone in water. The decrease in the intensity of luminescence of uranin dye is a function of acetone concentration. Hydrocarbons do not interfere, but organic acids influence the reaction. This method can determine approximately 10⁻³% acetone in water with a reproducibility of 5%.

A method is given by Djoric and Kontic (16R) for determining small concentrations of film-forming amines in steam and condensate systems. A rapid, direct, and sensitive colorimetric method is presented by El-Dib (19R) for the determination of aniline derivatives originating from phenylcarbamate, phenylurea, and aniline herbicides in surface waters. The method is based on the diazotization of the primary aromatic amines and coupling with resorcinol or α -naphthol. The aniline derivatives can be determined at the 0.1-ppm level. A colorimetric technique is described by Drobot, Rubchinskaya, and Mikhailova (17R) for use in determining piperidine and morpholine concentrations in power-plant waters and steam. The determination is based on the formation of copper dithiocarbamates with piperidine or morpholine followed by extraction with chloroform. Manual and AutoAnalyzer fluorometric methods employing alkaline ninhydrin were used by Whitledge and Dugdale (81R) to determine creatine in sea waters.

A spectrophotometric method for determining urea (6 to 60 μ g per 200 mg) in natural waters is described by Semenov and Pashanova (63R). An accuracy of $\pm 5\%$ is achieved in extracts containing biacetyl monooxime urea condensation products. Interfering humus or humic acids are removed by extraction with butanol. Croll and Simkins (15R) determined acrylamide in water by using electron-capture gas chromatography. The bromination of water containing acrylamide yields α,β -dibromopropionamide, which can then be determined with a relative standard deviation of ± 5 and $\pm 10\%$ for 0.25 to 500 μ g per liter, respectively. Operating conditions are listed.

A rapid, colorimetric method for determining hydrazine in water, based on the use of *p*-(dimethylamino)benzaldehyde, is outlined by Kostrikina (40R). Hydrazine is determined by difference between a sample containing hydrazine and possible interfering substances and a sample from which only the hydrazine has been removed.

An ultraviolet spectrophotometric method for the determination of furfural in waste water was discussed by Sadykhov, Zeinalov, and Khaldei (60R). The data obtained by spectrophotometric and colorimetric methods differed by $\pm 0.003\%$ absorbance. Colorimetric and ultraviolet methods for the determination of furfural in water and asphalt were examined by Kelus and Wawiernia (37R). With the instrumentation available, 0.04 mg of furfural per liter could be detected at 520 nm after reacting furfural with aniline in acetic acid. If iron is present, furfural must be separated by distillation.

The determination of soluble carbohydrates in sea water by partition chromatography after desalting by ion-exchange membrane electrodialysis is discussed by Josefsson (30R). The desalted samples are evaporated in vacuo and results compared by liquid-liquid and gas-liquid chromatographic methods. The former method is superior for the determination of different sugars. Eight monosaccharides were determined in the range of 0.15 to 46.5 μ g per liter. Small amounts of sorbose, fucose, sucrose, ethylene glycol, and glycerol were also detected.

Fluorometric methods for determining chlorophyll in vivo were discussed by Karabashev and Zangalis (32R).

A fluorometric method for the determination of lignin-sulfonates in natural waters is described by Thruston (75R). The effect of calcium, ammonium, and magnesium ions is negligible. The molecular weight of the ligninsulfonates affects fluorescence. Salinity and pH show no appreciable influence, and temperature does not interfere if sample and standard are kept near the same temperature. Khazova and Bogomolov (39R) outlined a method for determining dimethyl sulfoxide in waste waters. It is based on a potentiometric titration with perchloric acid in dioxane with acetic anhydride as the solvent. The determination error ranged from 10% for 0.1% dimethyl sulfoxide to 0.7% for a sample containing 5.02% dimethyl sulfoxide.

Siddiqi and Wagner (65R) presented a method for the determination of 3,4-benzpyrene and 3,4-benzofluoranthene in rain water, ground water, and wheat. It involved extraction with benzene, saponification with potassium hydroxide, chromatographic separation, detection with ultraviolet light, elution, and photometric determination. Thielemann (72R) describes a photometric method for determining trichloroethylene (0.1 to 100 mg per liter) in industrial waste waters. The sample is mixed with pyridine and heated with sodium hydroxide before the absorbance of the pyridine layer is measured at 430 nm against water. Other halogenated hydrocarbons do not interfere.

A rapid, photometric method for the determination of tetramethylthiuram disulfide in waters is discussed by Bilikova (3R). The tetramethylthiuram is extracted with chloroform, and cuprous iodide in methanol is added to a portion of this extract. The absorbance is measured at 440 nm.

Tan, Clemence, and Gass (71R) used a gas-chromatographic method to determine fecal water-pollution sterols. Functional derivatives and isomers of progesterone were resolved by this method.

Bogacka (5R) employed 4-aminoantipyrine for the colorimetric determination of chlorophenoxyalkanoic acid derivatives in water. It is possible to determine 0.02 to 0.08 mg per liter of 2,4-dichlorophenoxyacetic, 2-methyl-4-chlorophenoxyacetic, and 2,4-dichlorophenoxypropionic acids. Treguer, Le Corre, and Courtot (77R) determined free fatty acids in sea water indirectly. The fatty acids are extracted with chloroform, quantitatively complexed with triethanolamine-copper complex, and the copper deter-

mined by atomic absorption spectroscopy. The determination of short-chain fatty acids by gas-liquid chromatography was investigated by Van Huyssteen (79R). Complete separation of both the normal and iso acids (from C₂ to C₆) was achieved. Results were linear over the concentration range of 0 to 2500 mg per liter for C₂ and C₃, and 0 to 1000 mg per liter for the C₄₋₆ acids. Patil (56R) described a chromatographic method to estimate the concentration of oxalic acid in water. A method to obtain the dissolved constituents of nucleic acid from sea water by adsorption on in situ precipitated barium sulfate was discussed by Pillai and Ganguly (57R). The ultraviolet spectrum and the molar absorptivity of the hydrolyzate liberate were compared with standard DNA and RNA hydrolyzates. The effect of varying the conditions was also investigated. Kawahara (34R) presented an electron-capture gas chromatographic method by which trace quantities of weak acids, as the fluorinated derivatives, can be readily determined in the presence of gross amounts of interference. Trace amounts of phenols and mercaptans can also be determined by this method. From one carbon-adsorption sample, four organic weak acids were identified tentatively.

A method is described by Hoffsommer and Rosen (26R) for the quantitative determination of TNT, RDX, and tetryl in sea water by vapor phase chromatography with a ⁶³Ni electron-capture detector. A benzene-extraction procedure is employed. The method is capable of detecting TNT, RDX, and tetryl at the levels of 2, 5, and 20 parts per trillion, respectively.

RADIOACTIVITY AND ISOTOPIC ANALYSIS

Methods for measuring the trace-element and radioisotope content in natural waters and the uses of these measurements for studying biogeochemical processes in fresh and salt water systems are discussed by Perkins and Rancitelli (36S). Mundschenk (34S) calibrated a NaI(Tl) scintillation detector for the absolute counting of radionuclides in river-water samples. Two procedures for analyzing complex photopeaks are described. The sensitivity of detection is approximately 10⁴ to 10⁵ greater than the maximum permissible concentrations.

The theory of the measurement of the γ -activity of sea water by using an immersion detector is presented by Vinogradov and Vinogradova (55S). Equations for fluxes of primary and secondary γ -rays entering an immersion scintillation detector from a homogeneous solution have been derived and were compared with experimental results. Good agreement was obtained. The ratio of fluxes of secondary to primary γ -rays is calculated for several γ -ray-emitting radioisotopes in solutions. Means are outlined by Kas'yanov, Zemlyanoi, and Klimenko (26S) for optimizing the design of a polystyrene scintillator useful for the counting of low β -activities in ocean waters. A scintillator is described which has a counting efficiency of 75% for ⁹⁰Sr + ⁹⁰Y at a background count of 5.5 counts per minute.

Daly and Gabay (10S) used a γ -spectrometer with a NaI(Tl) detector in conjunction with a multichannel analyzer to rapidly screen environmental samples. Only the peak channel is used to compute the activity of a particular radionuclide. Compton subtraction is utilized to obtain the net counting rate in each peak due to photoelectric interaction. Riel and Duffey (40S) presented a technique for measuring the concentration of radioisotopes in water. It consists of: 1) determination of pollutant transport by using multiple radioactive tracers together with radiation detectors sensitive to approximately 100 pCi per

liter, 2) determination of radionuclide release by using an underwater γ -spectrometer sensitive to 1 pCi per liter, and 3) determination of environmental radioactivity levels by using an in situ sampler for large-volume samples. The preliminary results obtained for ¹³⁷Cs in various environmental samples are presented.

Fifty-eight references are included in a review by Cheslet (8S) on the current methods for the determination of ¹⁰³Ru-¹⁰⁶Ru, ⁵⁵Fe, ⁵⁴Mn, ⁵¹Cr, Zr-⁹⁵Nb, and their stable counterparts. Tassi Pelati and Mezzadri (52S) used radioactive tracers to improve the G. H. Hamada method for the determination of total β -radioactivity in sea water. Kautsky (27S) discussed the monitoring of surface radioactivity in sea water by using a NaI(Tl) γ -counter with a rate meter. Water flows continuously through a chamber past the detector. The limit of detection is 150 to 200 pCi per liter when based on ⁴⁰K measurements. The concentration of dissolved radioactive contaminants in water was determined by Eichholz and Galli (13S). A preweighed amount of mixed ion-exchange resin is slurried with the water sample followed by γ -ray counting or spectrometry. More than 85% of the trace elements are extracted in approximately 15 minutes. Ruf (42S) studied single-nuclide determinations in water and hydrobiological material with radiochemical and γ -spectrometric methods. The problems encountered in the identification of individual radioactive species at low concentrations are discussed.

Marques and Grade (32S) described a method to concentrate the radionuclides from large amounts of natural water and to separate ¹³⁷Cs from ⁹⁰Sr. Cesium is precipitated with potassium nickel hexacyanoferrate(III) while the ⁹⁰Sr remains in solution. The final cesium purification is performed with molybdosilicic acid. Viewpoints with regard to sampling for determining total radioactivity, its measurement, and special β -spectrometric methods are presented by Huebel (21S). In addition to surveying surface water, it was necessary to sample mud and hydrobiological materials. Various sampling techniques and commonly used measuring techniques are also discussed.

Hodge, Folsom, and Hansen (20S) described in detail the method used at Scripps Institute for determining ¹³⁷Cs and ¹³⁴Cs in sea water. Results obtained in 1971 by this method were compared with results from methods used at 56 other institutions. The methods used at Scripps Institute for the determination of radioactive and stable cesium in sea water were also discussed by Folsom and Sreekumaran (16S). The procedures for precipitation of cesium from large water samples, in situ concentration of cesium by ferrocyanide absorbers, and measurements of stable cesium are presented. Two one-step separation procedures using either ammonium dodecamolybdophosphate or potassium cobaltihexacyanoferrate for the separation of radiocesium from sea and fresh waters are described by Dutton (12S). Cesium-137 and cesium-134 are measured by γ -counting the dried material. Cesium-137 at levels down to 1 pCi per liter can be determined. The preparation and characteristics of ammonium molybdophosphate and potassium or ammonium hexacyanocobalt ferrate compounds supported in silica gel, and their application to the determination of ¹³⁷Cs in natural waters, were outlined by Terada *et al.* (53S). An ammonium molybdate procedure for determining ¹³⁷Cs in sea water was presented by Bauman (4S). Following precipitation and filtration, the ¹³⁷Cs on the filter cake is determined with a 256-multichannel analyzer using a NaI(Tl) crystal. An indirect method employing yellow perch and largemouth bass as biological factors was developed by Kevern (28S).

to measure low-level cesium-isotope concentration in a fresh-water lake. Cesium-137 is determined on a solid scintillation well-type γ -counter after preconcentration on an ion-exchange column. The water is analyzed for ^{137}Cs and the fish for ^{137}Cs and total cesium. With the specific activity of the ^{137}Cs in the fish and the ^{137}Cs concentration of the water, the total cesium concentration is calculated for the water.

A rapid procedure for determining ^{90}Sr and ^{137}Cs in surface waters is discussed by Aleksan'yan (3S). The concentration of these radioactive substances can be determined from one sample with a yield of 90% and 80% for ^{137}Cs and ^{90}Sr , respectively. Rapid, simple, and inexpensive methods for the determination of ^{90}Sr and ^{137}Cs are recommended in a review compiled by Bauman and Juras (5S). Twenty-six references are included. A rapid procedure for estimation of radiostrontium in water samples is discussed by Haber and Stuerzer (18S). SA-2 filters and an extraction procedure are used to separate strontium ions from barium ions. Approximately 60 minutes are required for the determination, and the strontium recovery is approximately 90%. Silant'ev, Chumichev, and Roslyi (46S) presented a method for the determination of ^{90}Sr which is based on the use of a highly sensitive β -spectrometer. A considerable reduction of the water volume subjected to chemical treatment is achieved. Data on the sensitivity of the apparatus are presented. A method for determining the ^{90}Sr concentration in sea water according to the ^{90}Y daughter isotope was developed by Silant'ev, Chumichev, and Vakulovskii (47S). Yttrium-90 radioactivity is determined using a low-background β -spectrometer.

By optimizing the dimensions of a cylindrical scintillation counter, Szy and Urban (51S) were able to measure small amounts of ^{226}Ra and ^{222}Rn in food, water, and the atmosphere. A direct γ -spectrometric method for the measurement of radium and radon in spring waters is discussed by Bizollon, Moret, and Molgatini (7S). Counting of the γ photons was continued until radioactive equilibrium was reached between radium, radon, and the short-life radon daughters. A study of the activities as a function of time permitted the calculation of the activity due to radium as well as radon. There was good agreement between the results obtained by this method and previously reported values.

Three methods are described by Magri and Tazioli (31S) for measuring radon during hydrogeological investigations. The first method, which is good for waters rich in radon, is a liquid scintillation-counting method with a minimum of handling required in sampling and preparing the samples for analysis. The second method is a scintillation-counting method in which water passes through activated carbon for two hours. The detection limit is 0.3×10^{-10} Curie per liter. The third method is a liquid scintillation method. It cannot be used directly in situ; the minimum measurable amount is 0.6×10^{-10} Curie per liter. An apparatus for measuring small quantities of ^{222}Rn in air or in water by using adsorption on activated carbon was developed by Delos (11S). The measurement of radon dissolved in sea water was made for the purpose of studying the radon flow at sea-air interfaces.

Prochazka and Jilek (38S) described a liquid scintillation-counting technique yielding low-activity (10^{-10} Curie per liter) data accurate to 5% to 10% at a 90% confidence level in 400-minute countings. This method is used to measure Ra, natural U, $^{210}\text{Pb} + ^{210}\text{Bi}$, ^{210}Po , Ra + U, and Ra + Pb + Po. A procedure is described in detail by

Johnson (24S) for determining the β -active isotope ^{228}Ra in natural waters. Extensions of the method for determining the α -emitting radium isotopes are discussed. In the final step, ^{228}Ra is determined by counting the yttrium hydroxide precipitate which contains ^{228}Ac , daughter of ^{228}Ra .

A method is presented by Gorbushina *et al.* (17S) for the determination of uranium isotopes in water by scintillation and extraction. The optimum measuring conditions and the sensitivity of the method were studied. Szy and Toth (50S) outlined a procedure for determining the $^{234}\text{U}/^{238}\text{U}$ ratio in natural waters with an α -scintillation spectrometer. The uranium is coprecipitated with ferric hydroxide, and the precipitate is purified by removing silicon dioxide, iron, interfering radionuclides, and organic substances, followed by electrolytic deposition on a polished stainless-steel cathode. Sarganova *et al.* (49S) tested the known methods for determining low-level uranium concentrations in formation waters to select the most effective one. The adsorption-on-activated-carbon method is the simplest, but it was found unsatisfactory for waters with high mineral content because of decreased adsorption capacity. The most satisfactory procedure involved the use of iron hydroxides followed by reprecipitation of iron with ammonium carbonate and extraction with tributylphosphate. A 3-fold re-extraction of uranium from tributylphosphate by ammonium oxalate resulted in 100% extraction of pure uranium. Bilal *et al.* (6S) determined uranium in sea water by thermal neutron irradiation followed by γ -spectrometry with a Ge(Li) detector and a multichannel analyzer. The sample constituents were then separated into anion, cation, and neptunium fractions by counterion migration. The neptunium oxalate complex is readily separated from the other cations and can be determined quantitatively by γ -spectrometry. Uranium is detectable in amounts of less than 0.05 ppb.

A small computer program is used by Silver (48S) to estimate the concentration of soluble trivalent, tetravalent, pentavalent, and hexavalent plutonium in natural waters. This program, which requires the potential and pH of natural waters, can also be used to approximate the proportion of plutonium that is present as a polymer in equilibrium with soluble plutonium. A radiochemical procedure is described by Wong (57S) for the determination of plutonium in large samples of sea water, sediments, and marine organisms. A sensitivity of 0.004 dpm per 100 liters of sea water and 0.02 dpm per kg of sediment was achieved. A ferrous hydroxide coprecipitation method is used for the concentration of plutonium in sea water. Factors influencing the recovery, contamination, and blank activity are discussed.

An analytical procedure for the spectrophotometric determination of thorium in ores and waters was presented by Pakalns and Florence (35S). The direct determination of thorium in water involved the use of chromogenic reagent Arsenazo III. A precision of $\pm 0.5\%$ at the 50- μg thorium level was obtained. Kamiya *et al.* (25S) determined thorium in natural water and iron ore by neutron activation analysis. Protactinium-233 and protactinium-231, which was added as a tracer, were electrodeposited on a gold-plated copper cathode following the irradiation procedure. The contents of the samples were determined by comparison of α - and β -activities of the plate with those of standards obtained by irradiation, separation, and electrodeposition of a known amount of thorium. A method for determining radioactive cerium in water samples is discussed by Haber and Stuerzer (19S). The separation of

cerium from other radionuclides is discussed in detail. The β -activity of the cerium is measured after precipitation with iodate and drying at 220 °C. Cerium recovery is at least 90%.

Shannon and Orren (45S) presented a procedure for determining polonium-210 and lead-210 in sea water. Sampling and separation procedures are included. The sample is counted on a conventional photomultiplier assembly. Polonium-210 is also counted on a multichannel α -spectroscopy semiconductor detector. A linear calibration curve was obtained for 0.04 to 600 pCi per liter of added $^{210}\text{Pb} + ^{210}\text{Po}$ (in equilibrium). The chemical recoveries are 92 and 85% for ^{210}Po and ^{210}Pb , respectively. A method for determining ^{60}Co in waters at levels of more than 0.5 pCi per sample is presented by Claassen (9S). After the cobalt is separated, first as the hydroxide and then as the thiocyanate complex in methyl isobutyl ketone, it is counted by a liquid scintillation system at 80% efficiency. Separation factors achieved for six isotopes are generally more than 2000. Preston and Dutton (37S) reviewed methods for the determination of selected cobalt nuclides in marine materials. Methods for analyzing samples from the marine environment for these radionuclides are discussed with special reference to the control of radioactive-waste disposal. Analytical methods for the separation and subsequent measurement of stable cobalt in sea water, biological materials, and sediments are also reviewed; the relative merits of techniques such as neutron activation, atomic absorption, and ultraviolet spectrophotometry are discussed. Seventy-nine references are included.

Rauert (39S) compiled a review with 139 references on measurements of tritium and carbon-14 in hydrological investigations. The principles of methods and their hydrological application are discussed. The methods for deuterium and oxygen-18 measurements in water samples are reviewed by Moser and Stichler (33S). Forty-two references are included. The methods and procedures used by the U.S. Geological Survey to determine tritium activity in natural waters are described by Schroder (44S). Liquid scintillation techniques are used to detect levels ranging from 150 to 400 tritium units. Knowles and Baratta (29S) established a standard deviation value for the determination of tritium in water. It was based on the values submitted by five laboratories for tritium amounts ranging from 51.8 to 331.4 nCi per liter. A standard deviation of 10% was established for a single determination of tritium when present in amounts of more than 5 nCi per liter. A method for estimating the standard deviations in measurements of low-level tritium in water samples is presented by Sauzay and Schell (43S). Electrolysis is used to effect an 18-fold enrichment. The final samples after distillation are counted in a liquid scintillation counter. Optimization of the system is discussed, as well as the fitting of the calibration line to the range of measured values. Wallick and Knauer (56S) describe a simplification of the benzene synthesis method for the determination of tritium which produced results which agreed with results from other laboratories. The theory of the synthesis of benzene, a schematic diagram of the apparatus, and factors which affect the determination of tritium are discussed. The results of determinations of tritium in natural waters made by 35 laboratories are compared by Florkowski, Payne, and Sauzay (15S). Three water samples in a concentration range of 10 to 250 T.U. were used for inter-comparison. Analyses were effected by liquid scintillation and gas counting techniques with and without electrolytic enrichment. Hughes and Holmes (22S) outlined the deter-

mination of tritium in rain, river water, and underground water. Electrolytic enrichment of rain-water samples and internal gas proportional-counting were essential for accurate results.

An apparatus is described by Lohez, Hagemann, and Botter (30S) which can analyze 32 samples of water for hydrogen isotopes in less than 24 hours. The D/H ratio is measured to a precision of 0.5% in samples with a natural concentration of 150 deuterium atoms per million hydrogen atoms. Details on the sampling, automatic introduction of the sample, and automatic measurement of the isotopic ratio are given. Thurston (54S) discussed a new sampling concept to convert liquid water to vapor without changes in the deuterium-to-hydrogen ratio. It has made possible the construction of an automated mass spectrometer system for determining the deuterium-to-hydrogen ratio of water samples. A precision of ± 0.1 ppm at the natural concentration level is achieved with a cycle time of 2 minutes. Neutron activation and photoneutron techniques were developed by Abdullaev, Zakhidov, and Umirbekov (2S) for the determination of deuterium in ground waters. Deuterium concentrations of 0.98 to 1.03 relative units were determined on samples with an error less than 3%. Abdullaev and Umirbekov (1S) used a ^{24}Na γ -source, with an initial activity of 150 mCi produced by neutron activation of sodium carbonate, to determine the deuterium content in four natural waters and three gas condensates. The error was less than 3%.

A new technique of sample preparation for the ^{18}O assay of water is described by Roether (41S). It is based on the principle of equilibrating the water samples with carbon dioxide gas before determining the isotopic concentration in a mass spectrometer. The handling time is reduced to about 10 minutes per sample by operating several samples in parallel, and by pumping the air from the equilibration vessels through capillary tubes without freezing down the water samples. The standard reproducibility of sample preparation is better than $\pm 0.3\%$ and no difference in isotopic composition compared to that of samples prepared in the classical manner has been detected. Ereemeev (14S) determined ^{18}O in sea and ocean waters and isotopic exchanges between the ocean surface and the air with an error of 0.02 to 0.03% by the use of a novel sampling and decomposition technique in conjunction with a modified mass spectrometer.

Ikeda *et al.* (23S) discussed the separate determination of radioactive iodide and iodate forms by utilizing an isotopic exchange reaction. The sample is divided equally and the pH of the respective halves are adjusted to 1 and 4. Iodide carrier and carbon tetrachloride containing iodine carrier are added to each half and mixed before the radioactivity of the carbon tetrachloride phase is measured. At pH 4, only radioactive iodide exchanges with iodine in the carbon tetrachloride phase; at pH 1, iodide carrier reacts with radioactive iodate to produce iodine, and then radioactive iodate together with the iodide form becomes determinable.

MISCELLANEOUS

A review is given by Nika (52T) on the principle of operation of automatic analyzers and on the main features of instruments commercially available. Fundamental properties of batch and continuous analyzers are compared. Several investigators, Stephens (70T), Atlas *et al.* (5T), Wilhelms and Bernhardt (78T), Abry (2T), Andren (3T), and Schlumberger (64T) used Technicon AutoAnalyzer systems to determine a number of constituents in fresh water, industrial water, and sea water.

Processes in water analysis, such as distillation, extraction, titration, titration with dosage of reagents, and titration with compressed air, were thoroughly investigated, and time studies to mechanize the analyses on the basis of automatic photometric colorimetry were made by Springer (69T). Apparatus were also constructed for serial analysis.

Gjessing (31T) described a semiautomatic method for simultaneous determination of pH, conductivity, turbidity, and color. By means of an electronically controlled pump connected to a 3-way stopcock, each of the instrument cells is automatically emptied, washed, and filled prior to measurement.

Maeda *et al.* (44T) reviewed the atomic absorption determinations of Cr, Zn, Mn, Cd, As, Fe, Mg, Ca, Cu, Pb, and Hg in water. Twenty-one references are given. The use of atomic absorption for the determination of individual elements appears throughout this review. However, several publications have dealt with multiple-element analysis in natural water and sea water using direct and extraction procedures. Elements that have been determined include Cd, Cr, Co, Cu, Fe, Pb, Mn, Hg, Ni, Si, Zn, Ca, Mg, Na, K, Sr, Li, Rb, Ba, Bi, Se, Sb, and As [Willey *et al.* (79T), Kaiyama and Yasuda (37T), Panday and Ganguly (57T), Nix and Goodwin (53T), and Orren (56T)].

Flameless atomic absorption methods for a number of elements have appeared in the literature. The direct determination of Al, As, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn in natural waters with a heated graphite atomizer was described by Fernandez and Manning (24T). Sensitivity, detection limits, precision of replicate analysis, matrix effects, and recovery tests were investigated. Omang (55T) reported that good results were obtained for Al, Fe, Mn, Cu, and Cd determinations in natural waters by a graphite crucible method. Segar and Gonzales (66T), and Segar (65T) evaluated the heated graphite atomizer for the direct determination of trace elements in sea water. Extraction techniques, which eliminate interferences from major elements, were also investigated.

Copeland, Olson, and Skogerboe (16T) compared pneumatic and ultrasonic nebulization for the atomic absorption analysis of several metals in natural water, and reported that the ultrasonic technique gave better sensitivities.

Galle (30T) determined Mn, Fe, Co, Ni, Cu, Zn, and Pb in oil field brines by atomic absorption. These elements are separated from the alkali metals using Dowex A-1 chelating resin. A method is also described for the extraction and determination of Ca, Mg, and Sr. An atomic absorption determination of Ca, Mg, Fe, Cr, Mn, V, and Mo in brine by soda electrolysis is described by Kurihara *et al.* (41T).

Spectrographic procedures for the determination of a number of trace elements in natural waters and formation waters are described by Fomina and Kuznetsov (27T), Chesterikoff (14T), Zhigalovskaya *et al.* (82T, 83T), and Eremenko and Mel'nikov (21T, 22T).

Romanov and Eremeeva (62T) used an arc discharge in an aerosol atmosphere for the spectrochemical determination of alkali and alkaline-earth elements. A specially developed apparatus for analysis of sea water by a spark-spray method is described.

Zhigalovskaya (81T) studied techniques for introducing samples into a discharge plasma during the spectral analysis of metals in river and rain water. Fixing the samples to the electrode with a 50% solution of "Tsapon" lacquer

in acetone was the most satisfactory technique.

A review with 24 references on the determination of cadmium, lead, mercury, and other heavy metals by activation analysis was compiled by Guinn (34T). Salmon and Creevy (63T) described an on-line computer system for activation analysis of air, water, and soil which simultaneously accumulates γ -ray spectra from several detectors, performs appropriate data transfers, and carries out numerical analysis. Wainerdi, Fite, and Kuykendall (77T) discussed the limitations and capabilities of nuclear analytical methods and systems for the measurement of trace constituents in the environment. Activation methods are of particular interest in pollution control because substances such as mercury, arsenic, lead, and bromide lend themselves to microanalysis using neutron, charged-particle, or photonuclear techniques.

Cram (17T) combined chromatographic separation and neutron activation to determine trace-level metals in fresh water. Chromatographic separations of volatile metal-organic compound complexes were developed for post-irradiation separations. Eluate recoveries for Cr, Mn, Fe, Be, Lu, Gd, and Cu ranged from 52 to 98%. A number of other investigators have described activation procedures to determine trace elements in natural water, sea water, and hot springs [Abdullaev *et al.* (1T), Bankert, Sauter, and Bloom (6T), Battiston and Moauro (7T), Bhagat *et al.* (9T), Fiarman and Scheier (25T), Gohda (32T), Kawakami and Oda (38T), Ljunggren *et al.* (43T), Mamuro (45T), Mamuro *et al.* (46T-48T), Tanner, Rancitelli, and Haller (73T), and Thatcher and Johnson (74T)].

Florkowski, Holynska, and Niewodniczanski (26T) investigated the feasibility of using radioisotope X-ray fluorescence analysis for measurement of metals in water. The limit of detection for several metals was established as 5 ppm with the use of ^{238}Pu , ^{109}Cd , and ^{241}Am sources. They propose an extraction procedure which would reduce the detection limit to less than 1 ppm. Hellmann and Griffatong (35T) used X-ray fluorescence to determine heavy metals in waters after preconcentration on Amberlite IR 120 and precipitation with 1% sodium diethyldithiocarbamate. Fujiwara, Okashita, and Tsukiyama (29T) applied direct X-ray fluorescence to the determination of 0.2 ppm or more of arsenic and mercury, and 0.3 ppm or more of lead in water. Armitage (4T) reported on the determination and distribution of various trace elements in natural waters by X-ray fluorescence spectroscopy.

Methods for the polarographic determination of sodium, potassium, calcium, and magnesium in sea waters are given by Berge and Brueggmann (8T). Good results were obtained in the indirect sodium determination using the first uranium half-wave of a sodium magnesium uranyl acetate precipitate. EDTA is used to mask magnesium and calcium interferences. The direct potassium determination depends upon a quantitative precipitation of the excess sodium. Replacement reactions were used in a simultaneous determination of calcium and magnesium. The application of phase-selective ac polarography for analysis of heavy metals in water was examined by Poole (59T), and Sturrock and Poole (71T). Optimum conditions for determining the individual ions are given. Conditions are also given for analyzing various multicomponent mixtures. Methods for eliminating interferences, such as surfactants, are discussed. Es Souabni and Nangriot (23T) used differential oscillopolarography to determine several trace metals in mineral waters after isolation of the metals by solvent extraction. Copper, lead, cadmium, and zinc are extracted at pH 8.5 to 9 using dithizone-

chloroform and determined in 0.5M ammonium acetate-0.1M tartaric acid. Iron is extracted at pH 1 with cupferron into chloroform and determined in 0.3M triethanolamine-1M sodium hydroxide. Manganese and cobalt are extracted at pH 5.5 with diethyldithiocarbamate into chloroform and determined in ammonium hydroxide-ammonium chloride solution after addition of dimethylglyoxime for cobalt. Frazier (28T) mentioned that pulse polarography should be considered more often for water analyses because many metals can be determined in the range of 10 μ g per liter. Sensitivity is 2 orders of magnitude greater than a nonpulsed system.

Van Atta (76T) described in detail a field kit utilizing a dropping mercury electrode and a trisodium citrate medium for the determination of lead, cadmium, zinc, and iron in natural waters.

Crocker and Merritt (18T) used spark-source mass spectrometry to determine trace elements in water. Thirty or more elements were determined at concentrations of one part in 10^{11} parts of water and higher. The main points of the procedure are freeze-drying for sample pre-concentration, low temperature oxygen plasma ashing to eliminate organic ion interferences, sample presentation to the spectrometer in counter-electrodes tipped with a homogeneous conducting ash-carbon mixture, and newly perfected electronic detection and measurement of the mass-resolved ion beams. Klose (39T) analyzed powdered residues of some thermal and surface waters by mass spectrometry and ultraviolet transmission. Dmitriev and Kitrosskii (20T) prepared a review with 12 references of mass spectrometry and gas chromatography method sensitivities in determining background levels of 27 contaminants in potable and waste waters, and other materials.

Reimann (60T) reported that the reaction of dithizone with certain heavy metals can be utilized in a thin-layer chromatographic technique. Production of specific colors is characteristic of metal-dithizonate complexes. Prior treatment of samples selectively eliminates specific ions, and adjustment of pH simplifies identification of sought-for metals. Yamane *et al.* (80T) described procedures for concurrent determination of Hg, Bi, Cu, Zn, Sn, Mn, Fe, Co, Ni, Cd, Pb, Al, Be, and Cr in water by the combination of dithizone, oxine, 2-methyloxine extraction and silica-gel thin-layer chromatography. Details of the separations and identifications are given. Cassidy, Miketukova, and Frei (13T) reported on the fluorescent detection of nanogram amounts of metal ions in water by thin-layer chromatography. The ions are separated as their diethyldithiocarbamate chelates and detected by a ligand exchange reaction between a metal calcein complex and the diethyldithiocarbamate chelate. The method shows promise as a field tool.

Kaiser (36T) described a computerized gas-chromatographic method to determine hydrocarbons, organic halogen and nickel compounds, and sulfur compounds in water and air in the range of 0.01 ppb to several hundred ppm. Enrichment and sampling are performed in the field.

Carlson and Keeney (12T) surveyed the literature for all presently available selective-ion electrodes with special stress on problems associated with reference electrodes. Details of published procedures are given when applicable to soil and water analysis. Mascini (50T) reviewed the different kinds of ion-selective membrane electrodes applicable to water analysis. The preparation technique and results obtained with the electrodes sensitive to halides, sulfide, copper, and cyanide are described. Manahan *et al.*

(49T) explored the use of ion-selective electrodes, particularly the nitrate electrode for the analysis of ionic species in natural aquatic systems. They stated that the electrodes should not be used when substantial interferences are present and that they preferred standard addition techniques. Reynolds (61T) reported that water derived from glaciers is low in total dissolved solids but contains significant amounts of finely powdered rock. These suspended solids interfere seriously with atomic absorption and colorimetric procedures, but do not affect selective-ion electrodes. Procedures are described for the determination of sulfate, chloride, calcium plus magnesium, potassium, and sulfate using selective-ion electrodes.

Three methods for concentration of trace elements in natural waters are described by Novoselov and Distanov (54T): 1) evaporation with subsequent spectral analysis of the precipitate for all compounds, 2) extraction of zinc, lead, and copper dithizonates and copper, cobalt, and nickel diethyldithiocarbamates with subsequent photometric measurement, and 3) ion-exchange concentration of germanium on EDE-10P anion exchanger with photometric determination, and lithium, rubidium, and cesium in a KU-2 cation exchanger with subsequent determination by flame photometry.

Dingman *et al.* (19T) used polyamine-polyurea resins to concentrate and separate trace metal cations by complexation. Copper, cobalt, and nickel are quantitatively concentrated, recovered, and determined by atomic absorption from known aqueous solutions having concentrations as low as 4 parts in 10^{10} with concentration factors as high as 1000. High concentrations of alkali and alkaline earth metals do not interfere because they are not complexed by the resins. Oxidized carbon prepared from commercial activated carbon was used by Suranova, Grabchuk, and Chernenko (72T) to concentrate heavy metal cations from natural water.

Sokolovich, Lel'chuk, and Detkova (68T) described an extraction-photometric determination of the sum of heavy metals in demineralized water. The metals are complexed with sodium carbamate, double-extracted with chloroform, and converted to the copper-carbamate complex with copper sulfate. The absorbance of the copper complex is measured at 453 nm. Lel'chuk, Sokolovich, and Detkova (42T) also described a dithizone titration technique. Details of the procedure are given. A photometric determination of boron, molybdenum, manganese, copper, and zinc in the water of fertilized fish ponds was reported by Ushakova (75T).

Naidu and Sastry (51T) determined calcium, magnesium, iron, and aluminum in waters and other materials by complexometric titrations from a single aliquot of sample. Iron and aluminum are precipitated and separated as hydroxides, and redissolved with 1N hydrochloric acid. Iron is titrated with EDTA at pH 1.5 to 2.5 using Variamine Blue as indicator; aluminum is back-titrated with zinc sulfate at pH 5 to 6 after addition of excess EDTA using Xylenol Orange as indicator. The sum of calcium and magnesium is determined by titration with EDTA at pH 10 using Eriochrome Black T as indicator. Magnesium is determined after addition of sodium hydroxide by titration of liberated EDTA with calcium chloride using calconcarboxylic acid as indicator. The average error is 0.3%.

Bidzinska and Plochniewski (10T) discussed the effect of sampling methods and the time and conditions of storing water samples on the results of chemical analysis. Determination of iron, manganese, nitrogen compounds, ox-

dizing capacity, and gas content should be conducted in the field unless they are satisfactorily preserved.

A portable water-quality kit for the determination of dissolved oxygen, specific conductance, pH, and temperature in situ to depths of at least 35 meters is described by Slack (67T). The kit weighs 11.3 kilograms. An accessory package contains an extension cable, water sampler, Secchi disk, water-color set, and supplies. Accuracy of the instruments under field conditions is as follows: pH, ± 0.1 unit, temperature ± 0.6 °C, DO, ± 0.3 mg/liter, specific conductance, ± 5 μ mhos for dilute waters and ± 10 μ mhos for relatively concentrated waters. A description of field instruments for monitoring river and estuary water is given by Briggs (11T). A membrane electrode is described for measurement of dissolved oxygen. Suspended solids are measured by turbidimetric or nephelometric photoelectric techniques. Organic carbon is measured by a spectrophotometer at 253.7 nm. A diffusion electrode can be used to measure ammonia.

An apparatus and method for the accurate and rapid measurement of sea-water density are discussed by Kremling (40T). From resonance frequency changes caused by the sea-water sample, the sea-water density can be determined to an accuracy of $\pm 2.5 \times 10^{-6}$ gram per cm³.

Pekarkova (58T) compared the determination of suspended solids in drinking, surface, and waste waters by membrane filtration, asbestos and paper filtration, centrifugation, and evaporation. A membrane 0.4- μ m filter is recommended for below 20 mg per liter of suspended solids. A 0.6- μ m filter is suitable for wastes and sewage up to 200 mg per liter, and a 3- to 4- μ m filter can be used for wastes with 1300 mg per liter.

An electronic method for determining dissolved solids in water is described by Gorbunova (33T).

Christian, Matkovich, and Schertz (15T) used atomic absorption, anodic-stripping voltammetry, and flame-emission spectrometry to determine trace elements in water. Chelation-extraction techniques are used for determining metals by atomic absorption, while the polarographic technique is used for poorly developed metal chelates. More than 60 elements can be determined successfully by flame emission.

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Mercury, Silver, and Gold

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Radioactivity and Isotopic Analysis

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