

Water Analysis

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THIS TWELFTH REVIEW of the literature of analytical chemistry applied to water analysis covers a period from October 1964 through September 1966. The present review follows the plan of the previous reviews, the last of which appeared in ANALYTICAL CHEMISTRY for April 1965 (20).

A review of the literature on water pollution control which includes a section on analytical methods is published annually by a Research Committee of the Water Pollution Control Federation. The 1964 review of the literature (18) includes 118 references to analytical methods for determining organic carbon, pesticides, phenolic materials, general organics, cations and anions, various forms of nitrogen, and dissolved oxygen. Ninety-three references are included in their 1965 review (7) and cover topics like dissolved gases, organic acids, phenols, total carbon, isolation-concentration, and others.

Other reviews on water analysis have been published during the past two years. Kitano and Nasu (13) prepared a review with 495 references. A review on various methods of water analysis was published by Mitchell (16). The review contained 51 references. A review of 80 references for determining chlorinity (salinity) in sea water was prepared by Bruevich (2). Coutinet (8) published a review, with appropriate details, of methods for determining potassium, sodium, calcium, magnesium, carbonate, bicarbonate, chloride, and sulfate in water.

A handbook of analytical procedures for water analysis was prepared by Bush and Higgins (3). The handbook also contained sections on radiochemical analysis, determination of uranium in various media, and procedures for determining major and minor constituents of silica rocks and ores. Ceausescu (4) proposed the use of a number of analytical methods, which were taken from standard methods of various countries, for the control of water quality in supply centers. Twenty-eight references are given. Detailed analytical methods as applied to the analysis of hydrothermal fluids were described by Ritchie (19).

Husmann and Malz (12) discussed the use of photometry, gas chromatography, polarography, and paramagnetic oxygen measurements as applied to new methods of analysis of water and waste water. New or improved methods for determining silica, phosphate, oxygen, chloride, and organic compounds in boiler feed and condensate waters were

described by Dzysyuk *et al.* (10). Ballezo (1) discussed, in general, analytical methods used in water analysis and then proposed new methods for determining chloride, bicarbonate, hardness, iron, and fluoride. Instrumental methods of analysis for determining microgram and submicrogram quantities of various elements in water were reported by Monnier (17). A review on the use of spectrophotometry in water analysis was prepared by Weber (21). Eight references are listed. Ceausescu (5) discussed the use of ion exchange in water analysis in a review of 97 references covering the period from 1927 to 1962.

Automated procedures, using an Auto-Analyzer for routine analysis of water samples, were reported by Henriksen and Samdal (11). Semiautomated methods for determining free chlorine, silicic acid, phosphoric acid, and hardness in water have been described by Leithe (15).

The advantages of neutron activation as applied to the analysis of water are discussed by Cerrai (6). Landstrom and Wenner (14) described procedures for determining a number of elements in ground water by neutron activation.

A review of procedures for determining the alkali metals and uranium in sea water by stable isotope dilution analysis was prepared by Dobkowski (9).

ALKALI METALS

Flame photometry is still used as the principal means of determining the alkali metals in water. Rozanova (16A) checked the applicability of flame photometry for determining sodium and potassium in mineral waters. The sample was introduced into the flame in the form of an aerosol. No problems occurred for samples low in total mineral content and containing sodium and potassium concentrations less than 20 mg per liter. Dilutions were used for samples containing high concentrations of minerals. Potassium in brines was also determined by the cobalt nitrite method, but results were 9 to 20% higher than results by flame photometry.

A portable flame photometer was used by Zatsepa (20A) for determining sodium and potassium in various types of natural water. A sensitivity of 0.5 mg per liter was obtained for potassium between 1 and 20 mg per liter, and for sodium between 1 and 12 mg per liter.

The maximum relative error was 7% for sodium and 15.4% for potassium.

A direct flame photometric analysis of sodium and potassium in aqueous extracts was recommended by Azovtsev (1A), instead of a calculation based on the gravimetric determination of sulfates.

Hosokawa *et al.* (6A) determined sodium and potassium in sea water with a filter-type flame photometer using lithium as an internal standard. Lithium is added to a diluted sea water sample, the sample aspirated, and the transmittance adjusted to 100% using a lithium interference filter. The filter is then replaced with a potassium interference filter and the transmittance of the sample measured. Sodium is determined in a similar manner.

A flame photometric method for determining lithium in the parts per billion range after concentration by evaporation was reported by Joensson (7A). The intensity of the lithium emission was increased by adding a 20% mixture of acetone-*n*-amyl alcohol (4:1) to the samples. A special absorption filter was used to eliminate interference by stray light from sodium. Aluminum was used to suppress interferences from alkaline earth oxides. Ion exchange followed by flame photometry was used by Sulcek, Povondra, and Stangl (18A) for determining lithium in mineral waters. A sample of water containing between 5 and 1000 μg of lithium and less than 10 meq total cations was acidified with hydrochloric acid. The carbon dioxide formed was then removed either by heating or by passing a stream of nitrogen through the sample. The sample was then passed through a strong acid cation-exchange resin, the lithium eluted with a mixture of 0.5*M* hydrochloric acid in 80% methanol, and lithium determined by flame photometry.

Collins (4A) described a method for detecting less than 0.01 mg of rubidium or cesium per liter in oil-field waters. Samples which contain very low concentration of rubidium and cesium are first concentrated by either evaporation or ion exchange. The samples are then transferred to separatory funnels, volumes adjusted to 100 ml with water, sodium tetraphenylboron is added, and cesium and rubidium tetraphenylborates are extracted with nitroethane. The cesium and rubidium are then determined by flame photometry.

Podobnik, Dular, and Korosin (14A) described a flame photometric method

for determining lithium, sodium, potassium, calcium, magnesium, and strontium in natural and mineral waters. Interfering anions were removed by ion exchange. Lithium, potassium, and strontium were determined by standard addition, and sodium, calcium, and magnesium determined by dilution. Detection limits of 0.0006, 0.0002, and 0.0008 ppm were reported, respectively, for lithium, sodium, and potassium. Flame photometry was also used by Pulido *et al.* (15A) for determining the alkali metals and the alkaline earths. A sample volume of 500 or 1000 ml was passed through Dowex 50 W-X resin, and the cations eluted with 0.2, 0.7, 1.2, and 2.0N hydrochloric acid. The order of elution with some overlapping was reported as follows: lithium and sodium, potassium, rubidium and cesium, and finally calcium and strontium. Khitrov and Kotlyarov (8A) described a flame photometric system which they used to determine sodium, potassium, cesium, rubidium, calcium, and strontium in sea water.

Normand (12A) compared atomic absorption techniques with colorimetric, flame emission, and activation methods for determining lithium, sodium, potassium, and magnesium in ice cores. Good results were obtained but some drawbacks were evident. Meshkova, Zelyukova, and Poluektov (9A) developed an atomic absorption method for determining rubidium in sea water. With the use of scale expansion the sensitivity was 0.02 μg of rubidium per ml. The sensitivity is also increased by adding 5% butanol. Lithium, strontium, magnesium, barium, calcium, iron, and uranium do not interfere. Rubidium ionization is suppressed by the addition of 1 mg of potassium salt per ml.

Truesdell, Jones and VanDenburgh (19A) analyzed a number of waters for sodium using a sodium-sensitive glass electrode. The sodium in the waters ranged from 10 to 130,000 ppm. The method was compared with a flame photometric method and over two thirds of the results were within 5% of each other. The glass electrode method allowed for a rapid and accurate field measurement of sodium. Nogina and Kobayak (11A) recommended an indirect complexometric method for determining sodium in water which is ideal for use under field conditions. Sodium is first precipitated with zinc uranyl acetate, the precipitate washed, and redissolved in hot water. Ammonium carbonate is added to keep the uranium in solution, and the solution is then titrated with Complexon III after adding ammonium hydroxide and Eriochrome Black T.

Bauman and Tagliatti (3A) reported that the method of determining potassium as the tetraphenylborate is equivalent to the flame photometric method. They recommended it for routine con-

trol purposes. The method is cheaper and is suitable for micro- and macro-determinations. An indirect titrimetric method for determining potassium in sea water was described by Murakami (10A). Potassium is precipitated with sodium tetraphenylborate and the excess tetraphenylborate titrated with benzalkonium chloride. A number of different indicators at various pH's were used.

Pencheva and Stoyanova (13A) described a spectrographic method for determining low concentrations of rubidium and cesium in natural waters. Rubidium, cesium, and potassium are precipitated with sodium tetraphenylborate, and the precipitate separated and dried. The residue is mixed with potassium sulfate and lithium carbonate in a ratio of 2:50:48, packed into an electrode and arced. Standards are taken through the procedure and treated exactly as the samples. The limit of detection in the initial specimen was in the order of $10^{-7}\%$ rubidium and $10^{-6}\%$ cesium. Parallel results obtained with flame photometry were always higher by 8% for rubidium and 9 to 10% for cesium. Potassium did not interfere. Shestov, Shurubor, and Tver'e (17A) concentrated potassium, lithium, rubidium, zinc, and manganese in natural waters with silica gel and then determined the elements by flame spectroscopic analysis. A mixture of fused silicon dioxide and sodium carbonate is dissolved in 100 ml of sample, the sample is acidified, and the precipitate separated and dried. The residue is then powdered and fired in carbon electrodes.

Micro- and semimicroanalysis of mineral waters for sodium and potassium was investigated by Ghimicescu, Musteata-Ghimicescu, and Dumbra (5A). Sodium was determined as the triple $\text{Mg-} \text{UO}_2\text{-Na}$ acetate and potassium determined as the $\text{Na-K-cobaltinitrite}$. Bartalis (2A) used ascending and circular paper chromatography for detecting sodium, potassium, magnesium, and calcium in mineral water.

HARDNESS, ALKALINE EARTH METALS

A review listing 20 references on the history of the determination of the hardness of water was published by Snelders (30B). Luppi and Herce (16B) reviewed the EDTA methods for the determination of total and permanent hardness of water. A statistical appraisal of the manganometric and complexometric methods for calcium, and of the 8-hydroxyquinoline and complexometric methods for magnesium was reported by Kotkowski and Wierzelejska (15B). They stated that the complexometric methods are faster, more precise, and cheaper than the other methods. Sedlacek and Dusek

(28B) investigated various indicators for use in the chelatometric determination of calcium in water. Best results were achieved with glyoxalbis(2-hydroxyanil), Fluorexon mixed with phenolphthalein, and a combination of glyoxalbis(2-hydroxy-5-chloroanil) with methylene blue. Murexide was not so suitable.

Kleber and Franke (13B) used a EDTA complexometric titration method to determine hardness in water. Blaedel and Laessig (4B) developed a continuous potentiometric titrator with direct readout for determining concentrations of calcium and magnesium as low as $10^{-6}M$. A block diagram of the titrator is given. The continuous methodology and techniques described in their paper permit titrations at concentrations where conventional colorimetric and electrometric methods of EDTA are not applicable. The method has been applied to the determination of hardness in stream and lake waters. A simultaneous complexometric determination of calcium and magnesium in water was reported by Handa (10B). Total hardness due to calcium and magnesium is determined by adding a few drops of murexide indicator to a 25-ml sample and titrating with EDTA to a deep purple color. Buffer is added near the end point. A known amount of standard hard water or additional amount of water sample is then added to the above titrated sample, sodium hydroxide added, and the sample again titrated with EDTA. Calcium hardness is then calculated and magnesium is determined by the difference in the value of total hardness and calcium.

Marti and Herrero (17B) proposed an ion exchange separation of calcium and magnesium prior to their determination with EDTA. A sample of water is passed through a column of Zerolit 225 in the Na form. The magnesium is eluted separately with 1M ammonium acetate and the calcium removed by elution with 1M ammonium lactate. The magnesium is then titrated with Na-EDTA using Eriochrome Black T as indicator, and the calcium titrated similarly using murexide as indicator. Corless (6B) described an ion exchange technique for determining calcium in sea water. Calcium is separated from magnesium and strontium using Dowex 50W-X8 resin. Calcium-45 is used as a tracer to monitor calcium elution. The calcium is then titrated spectrophotometrically with EDTA using murexide as indicator.

Ceausescu (5B) determined total water hardness by titrating a 100-ml sample with 0.1N potassium stearate using a methyl orange-phenolphthalein indicator. One milliliter of titrant corresponds to 0.1 meq total hardness.

Meshcheryakov (18B) proposed a trilonometric method for determining

calcium, calcium plus magnesium, and sulfate in water. A sample is first acidified with hydrochloric acid, boiled to remove carbon dioxide, and then cooled. The sum of calcium and magnesium is determined first by titration with trilon using chromogen indicator. Calcium is then determined separately using murexide indicator. To determine sulfate, it is precipitated with barium chloride and then the excess barium, calcium, and magnesium are titrated with trilon using chromogen indicator.

Rouéche and Monnier (26B) described a rapid and direct amperometric method for determining calcium and magnesium in water. A sample is evaporated to dryness, the residue dissolved with concentrated hydrochloric acid and the solution diluted to 50 ml with water. A 2-ml sample is then buffered and the calcium titrated with EDTA until a sharp rise in current is observed.

Ruf (27B) described a flame photometric method for determining calcium in water. Aluminum, iron, and titanium interferences are removed by precipitation with ammonium hydroxide-ammonium chloride solution. Sulfate and phosphate are removed by anion exchange. Pavlik (23B) determined calcium in water by flame photometry. Sulfate, phosphate, and carbonate, which interfere, are removed prior to analysis by ion exchange (Dowex 2-X8). Results obtained by this method agreed with results obtained by volumetric analysis. To determine calcium in atmospheric water, Gorbenko, Krasuskaya, and Kuchkina (8B) first extracted traces of calcium with a 4:1 mixture of 0.01% azo-azoxy BN reagent in carbon tetrachloride and butyl phosphate. The calcium was then reextracted with 0.1N hydrochloric acid and determined spectrophotometrically with glyoxybis (2-hydroxyanil).

A spectrophotometric method for determining low concentrations of magnesium in natural waters based on the development of the blue color formed between magnesium and Azovan Blue dye was reported by Rai, Pande, and Tripathi (24B). Beer's law is obeyed over the range of from 0 to 30 mg of magnesium per liter. Interferences from a number of other elements are negligible.

Konopac (14B) described a polarographic method for determining strontium and barium in the presence of an excess of calcium in mineral spring waters. Calcium is removed by ion exchange and the strontium and barium are determined directly by recording the polarographic curve in a nitrogen atmosphere, beginning from -1.7 volts vs. Hg pool in the same solution. A polarographic method for determining magnesium and calcium in water was reported by Gutman (9B).

Isotopic dilution for determining water hardness was described by Takanaoka *et al.* (32B). The method is readily applicable for calcium concentrations greater than 150 mg per liter, but is somewhat difficult for lower concentrations.

Myasnikov and Shumaev (21B) determined strontium in water directly by flame photometry. The sample and standards, which contained from 0 to 20 mg of strontium per liter were sprayed into the flame of an acetylene burner and the strontium in the sample was determined from a working curve. The detection limit was 0.05 mg per liter while the error of the determination was ± 4 to 6%. An ion exchange flame photometric method was used by Jonsson (12B) to determine strontium. Anions and alkali metals which might interfere were first separated from the sample by an ion exchange operation. Strontium was then determined flame photometrically after adding butanol to sample to increase the intensity of the strontium emission. The method permitted the detection of a 0.1 μ g of strontium per ml with a standard deviation of 3.1%. Aleksandrov, Shermet'ev, and Zakharenko (2B) described a simple standard addition-flame photometric method for determining strontium in waters from oil and gas deposits. Strontium is first determined directly on the water sample and then an amount of strontium nearly equivalent is added to one of two aliquots of sample and a similar amount of distilled water added to the other. Both samples are then analyzed for strontium and the actual strontium content of the sample determined by a given formula. The method permitted the determination of a 0.1 mg of strontium per liter with a relative error of 10%. Standard addition was also used by Osmolovskaya (22B) as a rapid means of determining strontium in sea water. Strontium was determined by measuring the intensity at 4607 Å and subtracting background intensity measured at 4540 Å. The mean deviation was found to be ± 3 μ g/ml.

Several spectrographic procedures for determining strontium in water have been reported. To determine strontium spectrographically, Sednev, Starobinets, and Akulovich (29B) first separated strontium from calcium and magnesium by ion exchange. Thirty to 50 liters of water are passed through sulfopoly-styrene cation exchangers containing 0.16% divinylbenzene, and the strontium is eluted from the resin with Complexon III. Mints and Bylinkina (20B) boiled a 500 ml to 1000 ml sample to dryness, ashed the residue with nitric acid, treated it with sulfuric acid, and then powdered the sample. The powdered sample was arced and the spectra were recorded. The strontium line at 4607 Å and calcium line at 4878 Å were

measured photometrically. Analytical sensitivity in respect to the solid residue was 0.01%. Ichikuni (11B) determined strontium spectrographically after separating the alkaline earth oxides gravimetrically. Only 100 to 1000 ml of water is necessary, whereas 30 to 60 liters are necessary by other methods; and the difference between chemical and spectrographic results is very small. To determine strontium in surface waters spectrographically using carbon electrodes, Dojlido and Jedlewska (7B) evaporated the samples to $1/100$ of their initial volume before measurements were made. The strontium content of the samples, which were analyzed, ranged from 0.27 to 0.65 mg per liter. A simple spectrographic method for determining strontium, calcium, and magnesium in natural waters was described by Berman (3B). A spark discharge between a plate-like and a hemispheric electrode dipped into the sample or standard was used. The standards also contained sodium and potassium in concentrations comparable to the sample. The Cu 3247.5 Å line was used as an internal standard. The results agreed with flame photometric and chemical analysis.

Rao, Khan, and Kamath (25B) used activation analysis to determine stable strontium in sea water. Strontium is quantitatively coprecipitated on calcium oxalate, the precipitate irradiated, $^{87}\text{Sr}^m$ separated by radiochemical methods, and activity counted by a NaI(Tl) detector and compared with a standard treated in the same manner. Afanas'eva and Tsikunov (1B) surveyed the existing methods for the separation of strontium from sea water. They proposed a simplified modification of one of the methods. Complete details are given.

Miller and Komarova (19B) described a highly sensitive method for determining 0.03 to 3 μ g of beryllium per liter in natural waters. The beryllium is coprecipitated on aluminum hydroxide and the precipitate analyzed for beryllium spectrographically, or determined fluorometrically using morin which is ideal for field assaying. Solov'ev and Bozhevol'nov (31B) determined beryllium in waste water with dibenzoyl-methane by measuring the phosphorescence spectrum of the treated sample at the temperature of liquid nitrogen and in a like manner a similar sample with the addition of a known amount of beryllium. The amount of beryllium in the sample is then calculated.

IRON, MANGANESE, CHROMIUM, AND ALUMINUM

Sturla (32C) studied the 1,10-phenanthroline, 4,7-diphenyl-1,10-phenanthrolineisoamyl alcohol extraction and the 2,4,6-tripyridyl-s-triazine-nitroben-

zene extraction methods for determining reactive and total iron in industrial water. Detailed procedures, reagents, and equipment are described. The first method, listed above, has an accuracy of 0.02 ppm in the 0.1 to 2.0-ppm range. The second method has an accuracy of 0.05 ppb in the 0.1- to 100-ppb range. The third method has an accuracy of 0.2 ppb in the 10- to 100-ppb range. O'Connor, Komolrit, and Engelbrecht (24C) evaluated the *o*-phenanthroline method for ferrous iron and found that it gives high values for ferrous iron when the samples have a low ratio of ferrous to ferric iron, that acidification and buffering of natural water samples result in an increase in the observed ferrous iron concentration, and that color of complex is not stable and intensity of color increases with time.

Garbenko *et al.* (11C) detected 2 μ g of iron per liter with bathophenanthroline with a relative error of less than 10% in atmospheric precipitation. Iron is reduced with hydroxylamine-hydrochloric acid, complexed with bathophenanthroline, and the complex is extracted with nitrobenzene. The iron-bathophenanthroline complex is then determined colorimetrically. A rapid colorimetric method for determining iron in water was described by Fadrus and Maly (8C). Dilute hydrochloric acid was added to a 50-ml sample, the sample boiled until there is approximately 40 ml left, and cooled. Hydroxylamine, *o*-phenanthroline, and sodium acetate were added, and the solution was diluted to 50 ml. Comparison was then made with standards containing 0 to 2 mg of iron per liter. Fresenius and Schneider (10C) reported the use of 2,2'-dipyridyl for determining ferrous and total iron in water. An aliquot of sample is buffered to pH 5 to 6, 2,2'-dipyridyl added, and the absorbance of the colored ferrous complex measured spectrophotometrically at 546 m μ . Total iron is determined after reduction of ferric iron to ferrous iron with ascorbic acid at pH 2 to 3. A spectrophotometric method based on the blue-colored complex formed between ferrous iron and 2,4,6-tripyridyl-s-triazine at pH 3.8 to 5.0 was used by Nakashima and Sakai (22C). The method is sensitive to 0.005-ppm iron. Tanno and Nakashima (33C) determined iron in boiler water with 2,4,6-tripyridyl-s-triazine and an automatic recording analyzer. The analysis requires 30 minutes and the range of iron that can be determined is between 30 and 150 ppb.

A double-extraction procedure for determining ferric iron in turbid and colored waters was reported by Gregorowicz and Suwinska (13C). Iron is extracted with ethyl acetate from a neutralized solution containing am-

monium acetate and sodium benzoate and then re-extracted into water acidified with hydrochloric acid. The solution is buffered, Variamine Blue hydrochloride added, and the absorbance of the iron complex measured at 580 m μ . Sono *et al.* (31C) used solvent extraction to determine ultramicro quantities of iron in water. The ferrous-1,10-phenanthroline complex reacts with sodium dioctyl sulfosuccinate to form an electrically neutral compound which is extracted into chloroform. The absorbance of the extract is measured at 515 m μ . Up to 24 ppb of iron obeys Beer's law. In the determination of 3 ppb of iron, the 90% confidence limit is 0.67 ppb.

A kinetic method for determining iron in natural water was described by Birmanantas and Jasinskiene (1C). The reaction takes place at pH 2 to 3 in the presence of Pyrocatechol Violet and hydrogen peroxide. The absorbance of the solution is measured at 413 m μ and the iron content compared to standards. The determination of iron by means of a luminescent catalytic reaction in the presence of stilbexon was investigated by Bozhelol'nov and Kreingol'd (3C). Stilbexon is oxidized by hydrogen peroxide, and the resulting decrease of fluorescence measured. A number of other elements studied caused no interference. Sakurai (27C) determined iron by a continuous coulometric detection method. A sample of water is passed through a Jones reducer and the ferrous iron, which is formed, is determined coulometrically with electrolytically produced bromine.

Shidlovskaya-Ovchinnikova (29C) reported that several methods have been proposed for determining suspended iron in fresh waters. He recommended determining total and dissolved iron and then calculating suspended iron by difference. Shapiro (28C) reviewed the methods of analysis of ferrous iron in natural waters and discussed the sources of error.

A method for determining manganese and iron in one aliquot of sample was described by Kajiwarra and Goto (15C). Manganese and iron are complexed with formaldoxime and the absorbance of the colored complex is measured at 470 m μ . Hydroxylamine and EDTA are then added to the solution and the absorbance is again measured at the same wavelength. The latter measures only the manganese complex. Iron is determined by difference. Nickel and cobalt severely interfere.

Ingols, Esterman, and Enginun (14C) prepared a review of methods for determining manganese in water. Morgan and Stumm (30C) stated that methods used for determining manganese in water should distinguish states of oxidation and physical states, and be capable of determining concentration, less than 50

μ g per liter. They investigated several methods and developed a spectrophotometric method using *o*-tolidine for quantitatively distinguishing manganous ion from total manganese. They also mentioned that the *o*-tolidine and formaldoxime methods, with the use of a membrane filtering technique, have potential for determining the occurrence and distribution of various forms of manganese.

Several catalytic methods for determining manganese have been reported during the past two years. The method reported by Gottschalk (12C) is based on the oxidation of manganous ion to permanganate with persulfate at a temperature of $99 \pm 1^\circ$ C. The reaction is catalyzed by silver. The working curve covers the range between 0.006 and 0.55 mg. Substances precipitated by silver ion or oxidized by persulfate interfere unless excess reagents are added. Manganous ion catalyzes the oxidation of $\text{Et}_2\text{NC}_6\text{H}_5$ by potassium iodate to a yellow dye, and this reaction was used by Oradovskii (25C) to determine manganese in natural waters. The optimum pH is 7 and the optimum temperature for the reaction is 18.6° C. Iron interferes, but is masked with a phosphate-citrate buffer. Between 0.0005 and 0.01 μ g of manganese per cubic centimeter in the sample can be determined.

Blank and Voronkova (2C) determined manganous ion by its catalytic effect on the oxidation of Eriochrome Black T by hydrogen peroxide. The reaction is carried out at pH 10 to 10.5 and at a temperature of 18° to 20° C. The excess Eriochrome Black T remaining is extracted with *n*-amyl alcohol and its absorbance measured. As little as 5×10^{-9} gram of manganese in 30 to 150 ml of sample can be detected.

Matejak, Jurkiewicz, and Pietrow (18C) reported that chloride interferes when manganese is being determined by the ammonium persulfate method, but that the interference is eliminated by the addition of mercuric nitrate. A spectrophotometric method for determining manganese in water with acetaldoxime was described by Kajiwarra and Goto (16C). Acetaldoxime and potassium hydroxide are added to a sample containing less than 4 ppm of manganese, the sample diluted to 25 ml, and after 20 minutes the absorbance of the complex measured. Calcium and magnesium interfere. Sikorowska and Dozanska (30C) compared the mercuric nitrate and mercuric sulfate methods for determining manganese in water and sewage. The latter method was most suitable. Mokievskaya (19C) described procedures for determining the different forms of manganese in sea water.

A polarographic method for determining nickel and manganese in ocean

water was described by Tikhonov and Shalimov (34C). The sample is filtered, nickel and manganese carbamates are formed, the complexes extracted with chloroform, and evaporated to dryness. The residue is dissolved with hydrochloric acid, buffered with ammonium hydroxide-ammonium chloride solution, and a polarogram made.

To determine chromium in sea water, Chuecas and Riley (4C) coprecipitated chromium with ferric hydroxide, separated chromium from the iron by De-Acidite FF anion exchange resin, and measured the absorbance of the chromium-diphenylcarbazide complex. Details of the procedure are given. Chromium-51 was used as a tracer to determine the recovery of chromium. Kovalenko and Petrashen (17C) determined chromium in water by complexing Cr^{+6} with diphenylcarbazide- β -naphthalene sulfonate, extracting the complex with isoamyl alcohol, and measuring the absorbance of the complex at 545 $\text{m}\mu$. An interference study was made and the maximum allowable amounts of other elements are given. Bromine water in the presence of sodium carbonate is used to oxidize any Cr^{+3} to Cr^{+6} .

Delaughter (6C) described an atomic absorption method for determining chromium and molybdenum in brines. Chromium is complexed with diphenylthiocarbazone and the complex is extracted with methyl isobutyl ketone. Molybdenum is extracted with the same ketone after it is complexed with dithiol. With an 800-gram sample of brine 1 ppb of either element can be detected.

An amperometric method for determining chromate in waste water was reported by Dietz and Schwabe (7C). Chloride concentrations greater than 50 mg per liter interfere. Iron should first be eliminated. The advantages and provisions of a continuous amperometric determination of chromate are given.

De Angelis, Chiacchierini, and D'Ascenzo (5C) developed gravimetric and spectrophotometric methods for determining chromium in water. Isocinchomeric acid and Cr^{+3} react at pH 4.5 to form a gray-blue precipitate that can be used to determine chromium gravimetrically. However, if the precipitate is dissolved slowly in water, a red-violet solution is formed which can be measured spectrophotometrically. The latter method is useful in the range of from 2 to 30 mg chromium per 50 ml.

A radio release procedure was developed by Richter and Gillespie (26C) for determining dichromate ion in natural waters. A sample is acidified, and then stirred with a paddle of irradiated silver gauze. Silver ions are stoichiometrically produced in the sample and the activity of the silver ions in

solution determined by scintillation counting. The activity is directly proportional to the dichromate present. The limit of sensitivity is 10 μg of chromium per liter.

A spectrophotometric method for the determination of microgram quantities of aluminum in water was described by Fishman (9C). Aluminum solutions buffered to pH 6.0 form, with Eriochrome Cyanine RC, a red lake which exhibits maximum absorbance at 535 $\text{m}\mu$. Aluminum concentrations as low as 0.01 mg per liter can be determined. Ascorbic acid inhibits the interference by iron. Interferences by fluoride and silica are eliminated by evaporation of the sample with concentrated sulfuric acid.

An extraction method for determining aluminum, chromium, copper, iron, manganese, molybdenum, and nickel spectrophotometrically in pure water was reported by Motojima and Ishiwatari (21C). Details of the procedure are given. With this method 3 ppb of Al, Cr, and Mn and 5 ppb of Cu, Fe, Mo, and Ni can be detected. Nowicka-Jankowska and Golkowska (23C) described methods which are used to determine trace amounts of iron, aluminum, and silica in reactor water. Bathophenanthroline or 2,2'-bipyridine is used for iron. Aluminum is determined by the hematoxylin method, and silica by the molybdenum blue method. Wheat (35C) used atomic absorption to determine Fe, Cr, Ni, Co, Mn, and Cu in water. Techniques used to extend detection limits were extension of the path length, multiple passes through the flame, fuel-rich flames, different fuels, and increased sample uptake by mechanical pumping.

COPPER, ZINC, LEAD, CADMIUM, NICKEL, COBALT, BISMUTH, AND ANTIMONY

Bilikova and Bilik (4D), Bilikova (2D), and Bilikova and Zyka (5D) evaluated several methods for determining copper and zinc in natural waters. The dicupral and oxalaldehydrazide methods yielded the best results for copper, whereas the results obtained by the cupral method were less satisfactory. A modified zincon method utilizing ion exchange was best for zinc, and in a later publication Bilikova (3D) described a zincon method. A sample containing less than 0.6 μg of zinc per ml is passed through Dowex 2-X8 resin and the zinc eluted with 0.1N potassium nitrate. Borate buffer and a zincon solution are then added to an aliquot of eluate, the solution is diluted to 50-ml volume, and zinc is determined spectrophotometrically at 620 to 625 $\text{m}\mu$. Up to 1000 μg of Fe, Mn, Cd, Hg, Pb, Cu, Cr, Co, and Ni do not interfere.

Pavlik (28D) determined copper in water by concentration of the copper on

a strongly acidic cation exchanger, elution of the copper with 5N calcium chloride solution, and determination of the copper spectrophotometrically in the solution of lead cupral in chloroform at a wavelength of 425 $\text{m}\mu$. The sensitivity is 0.001 mg of copper per liter.

Sono *et al.* (34D) described a solvent extraction procedure for determining ultramicroquantities of copper. Copper is extracted from a 400-ml sample with cadmium diethyldithiocarbamate-chloroform solution. The absorbance of the complex is measured at 435 $\text{m}\mu$. Beer's law is obeyed from 0 to 10 ppb. Citric acid is added to the sample to prevent ferrous iron interference. Morimoto and Hirakoba (26D) used thiosemicarbazide to determine between 0.5 and 20 ppm copper(II) in water. A stable blue complex is formed between copper and the thiosemicarbazide in acid solution. The absorbance of the complex is measured at 365 $\text{m}\mu$. Ferric iron also forms a complex with the reagent and must first be reduced. Corbett (9D) investigated the complex formed between copper and 3-methyl-5-hydroxy-5-(α -arabino-tetrahydroxybutyl) thiozolidine-2-thione (MHATT). The complex forms at pH 4.5 to 7.0 and shows maximum absorption at 432 $\text{m}\mu$. The reaction is highly selective and the organic reagent has negligible absorption at 432 $\text{m}\mu$. He used the method to determine copper in water. Casassa (6D) described a semiquantitative method for detecting 0.06 to 1 ppm copper in drinking water. The method is based on the white precipitate formed when 2,3-dimercaptopropanol is oxidized in 1M sodium hydroxide by oxygen or peroxide reagents. The reaction is catalyzed by copper. Ions which are precipitated by hydroxide are first removed by filtration.

Tanno (35D) described a spectrophotometric method for determining traces of copper in boiler water. Copper forms a colored complex with 2-carboxy-2'-hydroxy-5'-sulfoformazylbenzene which shows maximum absorbance at 600 $\text{m}\mu$. Beer's law is obeyed up to 50 ppb. Tanno (36D) then described an automatic recording analyzer which utilized the above method. Results obtained agreed with results obtained by the manual method.

Magee and Rahman (23D) determined copper in sea water by atomic absorption. Copper is chelated with ammonium pyrrolidine dithiocarbamate, extracted with ethyl acetate, and then aspirated in the flame. The detection limit is about 2.5 μg of copper per liter. Cummins and Woods (10D) developed an atomic absorption method using the copper resonance line at 3248 Å and an air-coal gas flame to determine copper in artificial sea water. Sodium, magnesium, and chlorine had little effect on the copper absorption.

Giambastiani, Filho, and Lima (15D) used radioactivation analysis for determining ppb levels of copper and chlorine in high-purity water.

Hickey and Overbeck (19D) developed a chelometric titration method for determining zinc in municipal and industrial waters. Diethylenetriamine-pentaacetic acid (DTPA) is used as a titrant and is 200 times better than EDTA in its effective chelation of zinc over calcium. Up to 1000 ppm of calcium as calcium carbonate can be tolerated. Concentrations as low as 0.2 ppm zinc can be determined. Dithizone is used as the indicator. Isopropanol is added to the sample to keep the dithizone in solution. The interference effects of other substances have been investigated. A highly sensitive direct determination of zinc in sea water using a cathode-ray polarograph operating for anodic polarography was reported by Macchi (22D). Good reproducibility was reported at the 1 µg/liter level. The relative standard deviation was about 4.5%. The method is rapid, specific, and also relatively free from contamination from reagents because no chemical treatment of the sample is required.

Chakrabarti, Robinson, and West (7D) studied the various factors that affect the determination of lead by atomic absorption and then described a method for the determination of lead in water. To a 25- to 250-ml sample of water containing from 1 to 10 µg of lead, add 4 ml of saturated aqueous potassium iodide solution and enough concentrated hydrochloric acid to make 5% (v/v) of the total volume; extract the lead with methyl isopropyl ketone and measure the absorption of the lead in the extract at 2170 Å.

Nangiot (27D) described a method for determining lead, down to $10^{-7}M$ in water, using differential oscillography. The preferred medium was a mixture of 0.1M tartaric acid and 0.5M ammonium acetate adjusted to pH 5.0 with nitric acid. An automatically operating single-sweep polarograph using anodic stripping for determining as little as $2 \times 10^{-6}M$ lead in water was designed by Ito and Musha (20D). The sample is mixed with the electrolytic solution to make the solution 0.1N in respect to HCl and 0.01% in respect to gelatin. The solution is then deaerated with nitrogen and passed into the cell.

To determine small quantities of cadmium in water, Kuyumdzheva (21D) first removed copper, silver, and mercury by extraction with dithizone-chloroform at pH 2, raised the pH to 5, and extracted cobalt and nickel with dimethylglyoxime-chloroform. The pH is then raised with 10% sodium hydroxide and the cadmium is extracted as cadmium dithizonate with carbon tetrachloride and determined colorimetrically with a green filter at 520 mµ. A dithizone

procedure for determining cadmium was also described by Mal'kov, Kosyreva, and Fedoseeva (24D). The red cadmium complex is compared to a scale of cadmium standards prepared by the same procedure.

A modified dimethylglyoxime method was used by Forster and Zeitlin (11D) to determine nickel in sea water. Nickel is precipitated from 2 liters of water with sodium carbonate, the precipitate dissolved, and the nickel separated by extraction with dimethylglyoxime-chloroform solution at a pH of 8.0. Nickel is then re-extracted into an aqueous phase, oxidized to Ni^{+4} with bromine water, and determined colorimetrically as the nickel dimethylglyoxime complex. The average recovery of nickel determined in this manner is 90%. To compensate for this loss the calibration curve, 0 to 12 ppb nickel, is constructed by adding known amounts of nickel to sea water. Forster and Zeitlin (13D) modified the above method. The nickel after being extracted with dimethylglyoxime-chloroform solution is re-extracted into the aqueous phase with 1:1 hydrochloric acid. The solution is then made basic with concentrated ammonia, quinoxaline-2,3-dithiol reagent is added, and the absorbance is measured at 520 mµ. Miller and Johannes (25D) studied the factors affecting the coprecipitation of nickel and cobalt with cadmium sulfide in the presence of sodium diethyldithiocarbamate. They stated that the method is applicable to the determination of trace concentrations of nickel and cobalt in natural waters.

A detailed procedure for determining cobalt in water spectrophotometrically was described by Golobov and Vakhrameeva (16D). Cobalt is complexed with pyridylazoresorcinol at a pH of 6.8 to 8.2 in the presence of ammonia, sodium citrate, and saturated Trilon B, and the absorbance of the complex measured at 510 mµ. The sensitivity is 0.01 µg per ml with an accuracy of 2 to 5%. Rozhanskaya (31D) determined cobalt in sea water by digesting a liter of sea water with hydrochloric acid. The cobalt after digestion is complexed with β -nitroso- α -naphthol, extracted with toluene, and the absorbance of the complex measured at 530 mµ. The hydrochloric acid digestion gave a 98.9% recovery of cobalt using cobalt-60 as a tracer. The sensitivity is 1 µg of cobalt per liter. Forster and Zeitlin (12D) determined cobalt in sea water by precipitation of the cobalt from 10 liters of water with sodium carbonate. The precipitate is dissolved with hydrochloric acid and the cobalt extracted with dithizone-carbon tetrachloride after the pH is raised to 8.5. At this point citrate is added to complex the iron. The extract is then dried, treated with perchloric acid-sulfuric acid mixture, and redissolved. The colored cobalt complex is developed

with nitroso-R, and the absorbance of the cobalt complex measured at 415 mµ. Standards are prepared by adding known amounts of cobalt to sea water.

A spectrophotometric method for determining 0.1 µg of antimony per liter in sea water was described by Portmann and Riley (30D). Antimony is concentrated from 5 liters of water by coprecipitation with hydrous manganese dioxide and separated from manganese, iron, and other interfering elements by extraction with methyl isobutyl ketone from a solution 5M with respect to sulfuric acid and 0.01M with respect to iodide. The cobalt is then back extracted with 0.4M hydrochloric acid and subsequently determined photometrically with rhodamine B. Antimony-125 is used as a tracer to determine the chemical yield, which amounted to approximately 80%. Solov'eva and Arzamashev (33D) determined antimony in water with Crystal Violet. The method involves the formation in a strongly acidic solution of a complex compound of Crystal Violet with $SbCl_6^-$. The complex is then extracted with toluene and the absorbance of the complex measured in a spectrophotometer. Sheveleva and Buidakova (32D) used brilliant green to determine antimony in waste waters. The complex is extracted with toluene and analyzed colorimetrically.

A spectrophotometric method was described by Portmann and Riley (29D) which determines 0.025 to 1.5 µg of bismuth per liter in natural waters, particularly sea water. Ten liters of sample are acidified with hydrochloric acid and passed through an anion exchange resin. The bismuth is eluted with nitric acid and determined photometrically with dithizone. The average chemical yield, using a radioactive tracer, was approximately 85%.

Several polarographic procedures have been reported for the elements in this group. Ghelberg and Puscasiu (14D) determined lead, cadmium, and manganese to within 0.015, 0.005 and 0.005 mg per liter, respectively, by using a 0.1N hydrochloric acid electrolyte. A 1500-ml sample is evaporated to dryness and the residue redissolved with acid prior to polarographic analysis. Copper, zinc, and cadmium in the presence of each other were analyzed in drinking water by Grebenshchikova (17D) with an ammonium chloride inert electrolyte. Lead was determined with hydrochloric acid and calcium chloride inert electrolytes. The sensitivity of the methods was 10^{-8} to $10^{-4}M$. Virf and Makai (37D) used polarography to determine copper, zinc, and cobalt in some mineral waters. The elements are concentrated from a 500- to 1000-ml sample of water. Polarographic analysis, using standard addition, was chosen by Visintin, Monteriolo, and

Giuseppi (38D) on the basis of specificity and sensitivity. They analyzed lead, cadmium, tin, and zinc in potable waters. Weiss and Fidler (39D) reported on oscillographic determination of 10^{-5} to $10^{-6}M$ concentrations of lead, cadmium, copper, nickel, and zinc in mine waters after preliminary separation of the individual elements by extraction or ion exchange.

Biechler (1D) described an atomic absorption procedure for determining copper, lead, zinc, cadmium, nickel, and iron in industrial waste waters. A 500- or 1000-ml sample is passed through Dowex A-1 resin and the above metals are eluted with 8M nitric acid. The acid solutions were diluted to 4M before analysis because the burner capillaries were attacked by nitric acid solutions more concentrated than 6M. With a liter sample, copper, cadmium, and zinc could be determined to 5 ppb and lead and nickel to 50 ppb. With a 500-ml sample, iron could be determined to 50 ppb.

A photometric method for determining microgram amounts of copper, zinc, and lead in natural waters with xylenol orange was described by Gurkina and Igoshin (18D). The determination is carried out after preliminary extraction of the three elements with dithizone. Copper is separated from zinc and lead by extraction with 0.005% dithizone in carbon tetrachloride in a 0.01N acid medium and determined as the copper-xylenol orange complex after re-extraction with 6N hydrochloric acid. Lead and zinc are then extracted as the dithizonates and re-extracted with hydrochloric acid. Zinc is determined on one portion of the sample with xylenol orange after lead is masked with sodium thiosulfate, and lead is determined on another portion with xylenol orange after zinc is masked with potassium ferrocyanide. Chuiko, D'yachenko, and Shpikula (8D) determined copper, nickel, and zinc in water colorimetrically after concentration of the elements by coprecipitation. Detailed procedures are given.

SILVER, GOLD, AND MERCURY

Warburton and Maher (7E) and Warburton (6E) made measurements on the concentration of silver in precipitation samples in areas where cloud seeding with silver iodide had occurred. Details are presented on the experimental process involved in the concentration of silver, formation of silver iodide colloid, and effects of storage. Samples were pumped through Amberlite CG 120(H) resin columns at a rate of 15 ml per minute, and the silver was eluted from the columns with 0.05M barium nitrate solution at a rate of 0.5 ml per minute. Potassium iodide solution (10^{-4} gram per ml) was then added to

form a colloidal suspension of silver iodide which was detected by its ice-nucleating properties in a 10-liter cold chamber at about $-15^{\circ}C$. Standards of $^{110}AgNO_3$ were used to determine the loss of silver ions on the walls of the container. The lower limit of detection was 4×10^{-8} gram of silver collected on the ion exchange column. Kyuregyan and Eksuzyan (4E) used a dithizone method to detect 1 μg of silver per ml in waters.

A spectrographic method for the determination of gold in natural waters was described by Borovitskii, Miller, and Shemyakin (2E). The pH of a 3-liter sample is adjusted to between 5 and 6. Saturated calcium chloride, mercuric nitrate, and ascorbic acid solutions are added to the sample. Gold ion is reduced to gold metal by the ascorbic acid, and the gold coprecipitates with mercurous chloride which is formed in the presence of excess chloride ion. The sample is left to stand overnight, the transparent solution siphoned off, and the mercurous chloride suspension filtered through an ashless filter. The filter is dried at room temperature and slowly ashed in a muffle furnace by gradually increasing the temperature to approximately $500^{\circ}C$. The ash is then fired in a spectrograph and the intensity of the gold line at 2375.95 Å compared with standards. The sensitivity of the determination from a 3-liter sample is 0.01 μg per liter. Silver, thallium, arsenic, tin, antimony, and tellurium can also be determined simultaneously.

Oka, Kato, and Sasaki (5E) determined gold in sea water by neutron activation analysis. A 100-ml sample is evaporated to dryness, the residue dissolved in 50 ml of 3M hydrobromic acid, the gold extracted with ethyl acetate, and the extract evaporated to dryness. The residue is transferred to a quartz tube with a small amount of aqua regia, and the sample irradiated along with a blank and standards in a neutron flux of 2×10^{13} neutrons/sq cm per second for 72–130 hours. After the samples are allowed to cool for a week, gold carrier and hydrobromic acid are added and the gold is extracted with ethyl acetate. Gold is then reduced with hydroquinone and the gold-198, which is formed during activation, is counted. A quick method of detecting gold in sea water by activation analysis with thermal flux was also described by Cappadona (3E).

To determine mercury in natural waters, Beisova, Generalova, and Fesenko (1E) acidified a sample of water with sulfuric acid to destroy the stable organic mercury compounds and then oxidized any mercurous ions present to mercuric ions with permanganate. After cooling, excess permanganate is destroyed with hydroxylamine and mercury is determined with dithizone.

VANADIUM, TANTALUM, SCANDIUM, ZIRCONIUM, AND URANIUM

Chan and Riley (4F) described the combination of an almost specific ion exchange process with spectrophotometry to provide a simple procedure for the determination of vanadium in sea and other types of water. Vanadium from 3 liters of water is quantitatively coprecipitated with ferric hydroxide in the pH range of 4.0 to 7.5. The precipitate is dissolved and passed through Zeo-Karb 225 ion exchange resin. The vanadium is then eluted with 0.3% hydrogen peroxide solution. Iron and a number of other elements are not eluted by the hydrogen peroxide. After elution, vanadium is determined spectrophotometrically with diaminobenzidine. Vanadium reacts with 8-hydroxyquinoline to form a colored dye which can be extracted with chloroform and was the basis of a method by Mulikovskaya (10F) to determine vanadium in natural water. Vanadium is coprecipitated from a 500- to 1000-ml sample of water with ferric hydroxide, fused with sodium carbonate, and extracted with hot water. The vanadium 8-hydroxyquinoline dye is formed and extracted into chloroform and compared with standards.

Sharova and Chufarova (16F) reported that EDE-10P, basic ion exchange resin, concentrated small amounts of vanadium from waste waters which contained copper, magnesium, manganese, calcium, aluminum, iron, phosphorus, titanium, and silica. These ions are not adsorbed by the resin in the chloride form. Vanadium is then eluted with 1N hydrochloric acid which in turn restores the resin.

Gillespie and Richter (6F) developed a radio-release technique to determine vanadium(V) in water. The sample is acidified to pH 3 or less with either phosphoric or sulfuric acid, and the sample passed through a column containing radioactive metallic silver-110m-110. The vanadate ion oxidizes the silver, and silver ions are released into the solution where their concentration is measured by Geiger or scintillation counting. The sensitivity is 0.1 ppm of vanadium. Chloride interferes but is complexed with 1,3-diethyl-2-thiourea prior to analysis. Iron also interferes and is likewise complexed before analysis with EDTA and fluoride. Lower valences of vanadium can be determined if they are first oxidized to vanadate. Chlorine gas is suited for this purpose.

Pets and Aleskovskii (12F) and Pets, Aleskovskii, and Miller (13F, 14F) described methods for determining tantalum in natural and waste waters. Tantalum is determined after preliminary concentration by carrier precipitation with calcium carbonate or cadmium sulfide. A small amount of ferric salts increases significantly the precipita-

tion of tantalum. Tantalum is then determined colorimetrically with Rhodamine-6Zh. The radioactive isotope tantalum-182 was used to determine the degree of precipitation. A thermal neutron activation method was developed by Hamaguchi *et al.* (7F) for the determination of tantalum in sea water. Tantalum is coprecipitated with ferric hydroxide from 5 to 10 liters of water and the precipitate is air-dried and irradiated for 12 days at neutron fluxes of approximately 3×10^{11} neutrons per sq cm per second. Solvent extraction using $\text{HF-H}_2\text{SO}_4$ -isopropylacetone is used to separate tantalum-182. The activity is then measured on a β -proportional counter. Approximately 0.1 μg of tantalum can be detected. Belopol'skii, Gumbar, and Popov (2F) developed a method for separating traces of scandium in natural sulfate waters; however, zinc and copper interfere and must be removed. They found that scandium could be separated from these two elements by coprecipitating scandium with ferric hydroxide with a 20% solution of pyridine at pH 6.5. If the precipitation is repeated three times, 96 to 98% of the scandium is recovered.

A method for determining trace quantities of zirconium in sea water was reported by Shigematsu *et al.* (17F). Zirconium is coprecipitated with aluminum hydroxide from a large volume of sea water. The precipitate is filtered and dissolved in hydrochloric acid, and zirconium is extracted with TTA in benzene. The organic phase is evaporated to dryness and the organic material destroyed with perchloric acid; zirconium is dissolved in hydrochloric acid and determined fluorometrically with morin. Zirconium recovery is about 95%. A number of sea water samples which were analyzed contained from 0.011 to 0.041 μg of zirconium per liter. An automated procedure to determine zirconium in water in the range of 0.5 to 100 ppb has been developed by Demmitt (5F). It is based on a differential fluorescence technique which employs morin as the indicator in a hydrochloric acid medium. The fluorescence is measured before and after addition of Na_2EDTA to the solution. Further tests and evaluations are still being conducted.

A simple extraction method for field determination of uranium was described by Chamberlain (3F). Sodium chloride is added to reduce the solubility of carbon tetrachloride in water, and Na_2EDTA to complex interfering elements. Uranium is then complexed with diphenylpropanedione and pyridine, and carbon tetrachloride is added to dissolve and extract the complex. The color of the sample is compared with standards. Nemodruk and Deberdeeva (11F) determined uranium in water spectrophotometrically with Arsenazo III. Uranium

is precipitated from a liter of water on ferric hydroxide. The precipitate is dissolved and uranium extracted with a solution of butyl phosphate in toluene. The uranium is back-extracted with water and Arsenazo III, and the absorbance of the complex measured at 656 $\text{m}\mu$. Kuznetsov *et al.* (8F) used organic precipitants in the determination of uranium in natural water.

A method was described by Barker *et al.* (1F) for the determination of low concentrations of uranium in water based on the fluorescence of uranium in a pad prepared by fusion of the dried solids from the water sample with a flux of 10% sodium fluoride, 45.5% sodium carbonate, and 45.5% potassium carbonate. This flux permits use of a low fusion temperature and yields pads which are easily removed from platinum dishes for fluorescence measurements. Uranium concentrations of less than 1 μg per liter can be determined in a sample of 10 ml or less. Liu and Chang (9F) stated that fluorometric measurement is the most sensitive method for determining traces of uranium in waste and natural water, and described a method for its determination.

Physicochemical methods, including ultrafiltration, dialysis, adsorption on ion exchange agents, and electrodialysis, were used by Serebryakova (15F) for determining the forms of uranium occurring in subsurface waters.

BORON AND SELENIUM

Glebovich (3G) developed a colorimetric method for determining boron in natural waters with H-resorcinol. Complexon III is added to complex interfering cations. H-resorcinol is added to sample and standards and the solutions are left in the dark overnight and then the color of the sample is compared with the standards. With a 5-ml sample the sensitivity is 1 μg of boron. If a 100-ml sample is used and the reagents increased proportionally, the sensitivity is increased. A number of water samples analyzed by this method agreed with results obtained by the carmine method.

Vasilevskaya and Lenskaya (8G) determined boron in water with salicylic acid and Crystal Violet. The sensitivity of the method is 0.01 μg of boron per ml. The water sample is neutralized with acid or alkali to pH 6 and passed through a column of KU-2 cation exchange resin. The solution is then neutralized to pH 5 or 6 with alkali, salicylic acid added, and the solution evaporated to dryness to remove excess salicylic acid. Crystal Violet and 0.01M hydrochloric acid are added, the solution is transferred to a separatory funnel, and the complex is extracted with benzene. The absorbance of the extract is measured at 585 $\text{m}\mu$. A spectrophotometric procedure for routine analysis of boron

in water was reported by Saxena, Baser, and Durge (5G). A sample of water is evaporated to dryness and the residue dissolved in 1 ml of water and mixed with turmeric-oxalic acid reagent. The mixture is evaporated to dryness, the residue treated with a known volume of ethanol and filtered, and the intensity of the dye measured at 540 $\text{m}\mu$. Semenov (6G) described a methylene blue extraction method to determine boric acid in sewage and reservoir water. The limit of detection is 0.05 mg of boron per liter.

Sidel'nikova (7G) proposed a modified 3,3'-diaminobenzidine method for determining selenium in natural waters. Selenium, iron, and lead are precipitated with ammonium hydroxide, the precipitate is dissolved with hydrochloric acid, and the selenium is separated from the iron and lead by KV-2 ion exchange resin. Trilon B is added to the eluate to mask other multivalent cations, followed by a solution of 3,3'-diaminobenzidine. After 40 to 50 minutes, the pH is adjusted to 7 to 8 and the yellow selenium complex extracted with toluene. Subsequently, the complex is analyzed colorimetrically. Preliminary tests revealed that the detection limit is approximately 5×10^{-6} gram per liter.

A similar method is described by Chau and Riley (2G) for determining selenium in sea water. Isotope dilution is used to correct results for the small losses occurring during the analysis. For sea water which contains approximately 0.4 to 0.5 μg of selenium per liter, a standard deviation of 0.03 μg per liter was obtained. Morette and Divin (4G) used a 3,3'-diaminobenzidine extraction method to determine selenium in water in which the selenium varied from zero to 74 μg per liter.

A ring-oven technique was used by Biswas and Dey (1G) for determining selenium in water. Selenium is extracted from sea water by distillation with bromine and hydrobromic acid. The separation of selenium from arsenic is achieved with the ring oven by using a magnesia mixture that precipitated arsenic as ammonium magnesium arsenate and it is retained at the center of the filter paper. Washing with 50% aqueous ethanol caused the selenium to migrate to the ring zone. The selenium ring is developed with a 5% aqueous solution of thiourea and the selenium determined quantitatively by ring colorimetry. It was possible to determine selenium in water at a level as low as 0.23 ppm.

Methods were described by Zanoni (9G) to determine selenium in water.

CHLORIDE, BROMIDE, AND IODIDE

Gromoglasov, Subbotina, and Komarova (8H) described a diphenylcarbazone method for determining chloride concentrations between 0.1 and 0.5 mg

per liter in water of high purity. The determination requires constant temperature, pH 2.8 to 3.2, 200 μg mercuric ion, and 2 mg diphenylcarbazone per 100 ml. The absorbance is measured at 540 $m\mu$. Bystrikskii, Aleskovskii, and Degtyarenko (5H) also used diphenylcarbazone for chloride and found that color development is affected by temperature. After diphenylcarbazone is added, the solution is held in a thermostat for 1 hour at 24° C. Mercuric nitrate is added, the sample brought to volume, and the solution again held at 24° C for 40 minutes before the absorbance is measured.

A rapid method for determining chloride in water by liquid detector tubes was developed by Kobayashi and Takeno (9H). A tube approximately 2.0 mm in diameter is filled with a known amount of a mixture of potassium chromate, silver nitrate, and silica gel. To determine chloride, 2 ml of sample are injected into the column with a syringe over a period of 5 minutes. The amount of chloride is determined by measuring the length of the discolored zone in the tube. Between 5 and 40 ppm chloride can be determined within a relative error of $\pm 10\%$. Results obtained by this method agreed with results obtained by the silver nitrate turbidimetry and mercuric thiocyanate methods.

Dyrssen and Jagner (6H) determined the chlorinity of sea water by potentiometric titration with 0.1M silver nitrate. Polished silver rods were used as electrodes and the cell was as follows: Ag/diluted sea water/0.1M KNO_3 /0.1M AgNO_3 /Ag. Aleskovskii, Bardin, and Bystrikskii (1H) described a potentiometric chloride method with a sensitivity of 1 μg of chloride per liter. Silver chloride electrodes are used. One electrode is immersed in an electrolyte free from chloride and the other electrode is immersed in the sample. The emf is then measured. Two concentration cells for the potentiometric determination of chloride in sea water were described and discussed by Koske (10H). The first cell is $\text{Ag}/\text{AgCl}, \text{NaCl}(c'')/\text{M}/\text{NaCl}(c')$, AgCl/Ag in which the electrodes are immersed in sodium chloride solutions of different concentrations and separated by a polyethylene membrane. In the second cell, $\text{Ag}/\text{AgCl}, \text{NaCl}(c)/\text{M}^+/\text{NaCl}(c'')/\text{M}^-/\text{NaCl}(c')/\text{AgCl}/\text{Ag}$ the electrolyte solutions are separated by an ion exchange membrane.

An automatic potentiometric method for determining chloride in sea water was developed by Shimanova and Ponomarev (16H). A silver and a calomel electrode are placed in a beaker containing the sample. The sample is then titrated and at the equivalence point the automated system closes the buret valve.

To determine chloride in water from nuclear power plants, Maley (11H) de-

signed a flame photometer which records the chloride concentration by measuring the sodium content. Complete details of the apparatus and operation are described.

An indirect spectrophotometric method for determining bromide in natural water with a standard deviation of 2 μg for bromide concentrations of approximately 40 μg per ml was described by Elbeih and El-Sirafy (7H). Bromide is oxidized to bromine in acid solution with acetic acid and hydrogen peroxide. The temperature of the reaction is kept between 40° and 50° C. A known amount of Chromotrope 2B is added and the bromine allowed to bleach the color for 30 minutes. The reaction is then stopped by buffering the solution to pH 5. The absorbance of the residual color is measured at 511 $m\mu$. The color is stable for several days. Most elements which occur naturally in water can be tolerated.

A review with 38 references on the determination of iodide in water was prepared by Pavlova and Shishkina (13H). The sodium thiosulfate method was the preferred method to determine iodide.

An indirect catalytic method for determining as little as 0.005 μg iodide in water was developed by Mano (12H). Iodide catalyzes the reduction of ceric salts by arsenite in acid solution. The reaction can be stopped by adding excess ferrous iron which immediately reduces the free Ce^{+4} . The ferric iron which is found is then determined colorimetrically with thiocyanate. Another feature of the method is the addition of sodium chloride to the reaction which increases the catalytic effect of iodide and also inhibits silver and mercury interference. Yonehara (18H) described a catalytic method for determining iodide and iodate in sea water. Both ions show the same effect on the color fading of a solution of ferric thiocyanate. To determine iodide and iodate, total iodine is first run, and then iodide is extracted with carbon tetrachloride from a separate sample after it is oxidized to free iodine. Iodate is then determined and iodide calculated by difference.

Bobek and Kolczak (4H) adapted the alkaline method to determine iodide in water. Two samples are taken for analysis. A known amount of iodide is added to one sample and an equal volume of water to the other. Sodium arsenite is added and the samples equilibrated at 25° C for 10 minutes at which time ceric sulfate is added. The absorbance of the resulting solutions is measured at 435 $m\mu$. Ramanauskas (14H) stated that tetramethyldiaminodiphenylmethane is oxidized by iodine set free from iodide with chloramine B to a blue quinoid derivative. The absorbance of the blue quinoid is measured

at 584 $m\mu$ or compared to copper sulfate standards. He used this reaction to determine iodide in natural waters. Chloride and up to 0.5 mg of bromide per liter do not interfere.

A simple, radiometric procedure for determining microgram quantities of iodide in natural water was developed by Richter (15H). The principle of the procedure is as follows: If a benzene solution of radioiodine of known specific activity is shaken with a sample of water containing iodide, the radioactivity will distribute itself in both phases in direct proportion to the mass of iodine or iodide in the individual phases. Measuring the radioactivity of each phase and knowing the quantity in the benzene phase permits calculation of the iodide in the aqueous sample. The reaction is specific for iodide and iodine species in the absence of oxidizing or reducing agents and precipitating or complexing agents. The lower limit of detection with this method is 2 μg of iodide per liter.

A potentiometric method for determining iodide in water was described by Atanasiu, Lazar, and Constantinescu (2H). Iodide is first concentrated by electrolysis in which it is oxidized to iodate. After electrolysis the iodate is reduced to iodine and concentrated by ion exchange (Dowex 1). The iodine is eluted with sodium sulfite and determined potentiometrically.

A polarographic method for determining iodide was reported by Yazhemskaia and Shtukovskaya (17H). Iodide is concentrated by evaporation; the solution is made basic with potassium carbonate and evaporated to dryness. Organic material is then removed by heating at 400° C. The residue is dissolved in water, iodide oxidized with HBrO_3 , and the excess of this reagent and oxygen removed by the addition of sodium thiosulfate. Iodide is then determined polarographically in gelatin solution using a dropping mercury electrode.

A spectrographic method for determining iodide, bromide, and chloride in natural waters was developed by Belobragina and Miller (3H). Details of the procedure are given.

FLUORIDE

Cox and Dirks (4J) compared the cerrous-alizarin-Complexon and the zirconium-Eriochrome Cyanine methods for determining fluoride in fluorinated drinking water and stated that the alizarin method seemed simpler to use. The zirconyl chloride-sodium alizarin-sulfonate and zirconyl chloride-Eriochrome Cyanine R methods and the effects of sulfate and bicarbonate on these methods were studied by Debiard and Dupraz (5J). The latter method gave better results; was simple, rapid, and sensitive; and did not require distil-

lation to isolate fluoride. Cooke, Dixon, and Sawyer (3J) investigated a number of fluoride methods and found that most suffer from aluminum interference. They then described two improved methods. The first was a modification of the visual method of Megregian and Maier. A color disk is available which offers improved color discrimination over the range of 0.5 to 1.3 ppm fluoride and much greater accuracy in the 1.0- to 1.3-ppm range. Color development was reduced from 1 hour to approximately 20 minutes. The second method was a spectrophotometric method based on the bleaching of the zirconium Solochrome Cyanine R complex by fluoride ions. Distillation in most cases was not necessary. Ruzicka and Mrklas (12J) compared a number of well known visual fluoride methods and found none precise for waters with high concentrations of aluminum.

Baeumler (1J) for several years has used the SPADNS method for determining fluoride in waters where the phosphate content is low and no aluminum is present. In the absence of these interfering ions the method works well.

Palin (10J) devised a simple method to eliminate aluminum interference in existing direct colorimetric fluoride methods. The sample is first treated with a weakly acidic cation exchange resin. The reaction between fluoride, cerous ion, and alizarin complexon was employed by Jeffery and Williams (7J) to determine fluoride in precipitation waters. Interfering elements are first separated by cation exchange or by distillation from perchloric acid solution. The results obtained agreed well with results obtained by a method involving distillation from perchloric acid and subsequent titration with thorium nitrate solution using alizarin red S as indicator.

Zolotavin and Kazakova (14J) also used the reaction of fluoride with alizarin complexon and cerous ion to determine fluoride in natural and waste waters. Iron and aluminum interference is eliminated by cation exchange. The absorbance of the final solution is measured spectrophotometrically at 617 m μ .

A similar alizarin complexon method for determining fluoride in sea water was described by Murakami and Uesugi (9J). Lanthanum ion is used instead of cerous ion. The absorbance of the color solution is measured at 610 m μ . Beer's law is obeyed up to 1.0 ppm fluoride. The tolerance limits for other elements normally found in sea water are given.

A spectrophotometric method to determine fluoride in mineral waters with zirconium-Pyrocatechol Violet complex was described by Ruzicka (11J). Fluoride is steam distilled with perchloric acid. The distillate is then acidified with hydrochloric acid, mixed with zirconium reagent and Pyrocatechol

Violet, and the absorbance of the resulting solution measured at 580 m μ .

Zolotavin and Kazakova (13J) determined fluoride in natural and waste waters by ion exchange and titration. A 100-ml sample is acidified with hydrochloric acid and the sulfate precipitated with barium chloride. The filtrate is then passed through a KU-2 cation exchange resin. Calcium chloride and sodium alizarinsulfonate are added to the eluate and the solution is acidified with hydrochloric acid and titrated with thorium nitrate which has been standardized against known concentrations of fluoride. Ghimicescu, Musteata-Ghimicescu, and Dumbrava (6J) used thorium nitrate titration to determine fluoride in mineral waters. The fluoride is initially steam distilled with perchloric acid.

An AutoAnalyzer was used by Chan and Riley (2J) to determine fluoride in sea water and other natural waters. A flow diagram of the system is shown. The spectrophotometric procedure used with the above system is based on the blue complex formed in the presence of fluoride when the sample is treated with a buffered reagent containing the lanthanum chelate of alizarin fluorine blue. Satisfactory results were obtained in the range 0 to 1.5 μ g of fluoride per ml.

A consecutive determination of sulfate and fluoride in water was described by Li and Huang (8J). Sulfate was precipitated from a sample with lead nitrate, filtered, redissolved, and the lead titrated with EDTA using dimethylphenol orange as indicator. The filtrate was passed through a cation exchange resin, and the fluoride in the eluate determined colorimetrically with thorium-dimethylphenol orange.

SULFATE, SULFITE, AND SULFIDE

Kleber and Franke (8K) discussed the disadvantages of determining sulfate in water by EDTA methods and recommended the conventional gravimetric method. Koval'tsov and Kononov (9K) prepared a review of the volumetric and physical-chemical methods for determining sulfate in water. The object of the review was to find suitable methods for determining sulfate with automatic analyzers. Turbidimetric and nephelometric methods were suitable. Kanwar and Chopra (7K) compared a rapid volumetric EDTA method with gravimetric and turbidimetric methods for determining sulfate in irrigation waters. The results obtained by the EDTA method were intermediate between the other two methods.

Page and Spurlock (14K) investigated three indirect titrimetric procedures to determine sulfate in sea water. The cations are first removed by ion exchange. Sulfate is then precipitated with barium chloride and the excess

barium in the filtrate is titrated with one of the following: EDTA-NH₄OH reagent or disodium EDTA, disodium EGTA, or standard sodium hydroxide. In the latter method the cation-free sea water is partially evaporated and sulfate is precipitated with benzidine hydrochloride. The benzidine sulfate is redissolved and titrated with sodium hydroxide. Results by the sodium hydroxide and EDTA-NH₄OH methods agreed fairly well with results obtained by the conventional gravimetric method.

Guerello (4K) also determined sulfate indirectly by precipitating sulfate with a known excess of barium and titrating with EDTA. Calcium and magnesium interfere but can be complexed with cyanide and triethanolamine. Kral (10K) critically reviewed the volumetric sulfate method and recommended that sulfate be precipitated with excess barium chloride and excess barium titrated with Chelaton III using Eriochrome Black T as indicator. This gives the total calcium, magnesium, and excess barium in the sample. Therefore, another sample is titrated for calcium and magnesium, and sulfate is determined by difference. Bicarbonate and free carbon dioxide are removed by boiling an acidified sample. Phosphate interferes and is removed by precipitation as silver phosphate. Heavy metals are masked with potassium cyanide and aluminum with triethanolamine.

A complexometric process for determining sulfate in drinking water by adding excess barium chloride and titrating the excess barium was also reported by Hoevers (6K). Riego (16K) described an indirect complexometric method to determine sulfate in sea water. Chloride, phosphate, and carbonate are precipitated with silver nitrate using potassium chromate as indicator. The precipitate is filtered and potassium chromate in the filtrate is reduced to chromic salt to avoid later precipitation of lead chromate. Lead acetate is then added, and the lead sulfate precipitate which is formed is mixed with EDTA, ammonium hydroxide, and ammonium hydroxide-ammonium chloride buffer. When the lead sulfate precipitate is dissolved, the solution is titrated to a violet end point with zinc chloride using Eriochrome Black T indicator. A formula is given to determine the amount of sulfate present.

Lehocky (12K) determined sulfate directly in water, after removal of interfering cations by ion exchange, by titration with lead nitrate using solid diethylenetriamine as indicator. In the concentration range 5 to 400 mg of sulfate per liter, the precision is 2 to 3%.

Ion exchange followed by titration with barium chloride was used by Handa (5K) to determine sulfate in water. Disodium tetrahydroxyquinone indicator (THQ) and a few drops of an inert

dye (xylene cyanol FF, indigo carmine, or methylene blue) are used to detect the end point, which is difficult to see with only the indicator (THQ). The end point with the dye is the change in color from an olive green to a gray violet. A cation exchange method for determining sulfate in ground water was reported by Lees (11K).

Pavlovich, Oshchepkova, and Solov'eva (15K) developed a rapid method to determine free sulfuric acid in mine water. Ferrous iron is oxidized to ferric iron with hydrogen peroxide and then complexed with ammonium oxalate. Sulfuric acid is then titrated with 0.1N sodium hydroxide using methyl red indicator. The total free sulfuric acid is the sum determined by titration and the amount consumed by the oxidation of ferrous iron. A formula is given to calculate the latter. Ferrous iron is determined separately.

Babkin, Veber, and Averbukh (1K) described a photometric method to determine sulfate in water. Barium sulfate suspension and an acid solution of barium chromate are added to a sample. The sample is mixed, and a 40% solution of sodium acetate is added. The precipitate is then centrifuged and the absorbance of the chromate in the filtrate measured with a photocolormeter. At the 75 mg of sulfate per liter level, the precision is ± 1 mg per liter. Calcium, magnesium, and bicarbonate do not interfere. Sykulska and Drazkiewicz (18K) used barium rhodizonate to determine sulfate colorimetrically in purified water. Various techniques of concentration were used.

A review of the methods for determining sulfide compounds in water and sewage was published by Dojlido (3K). Nusbaum (13K) modified the standard colorimetric method for determining sulfide in water and waste water. The stability and reproducibility of reagents are improved by the use of *p*-aminodimethylaniline oxalate as the colorimetric reagent and $\text{Na}_2\text{S} \cdot 3\text{H}_2\text{O}$ for preparation of standard solutions. A direct potentiometric method for determining hydrogen sulfide in mineral waters with an accuracy of 7 to 10% was described by Shpeizer and Zaidman (17K). Chloride, sulfate, sulfite, carbonate, benzene, phenols, and resins did not interfere. Details are given for preparing the electrodes.

Indirect procedures for determining mixtures of sulfide, sulfate, and sulfite in mineral waters were reported by Burriel-Marti and Alvarez-Herrero (2K). To determine sulfide, an excess of 0.1M copper nitrate is added to precipitate sulfide and the excess copper in the filtrate is titrated with EDTA using murexide as indicator. Sulfate is determined after an acidified aliquot of water is boiled to remove sulfide and sulfur dioxide. The sulfate is precipi-

tated with excess barium chloride, and the excess barium in the filtrate is titrated with EDTA. To determine sulfite, another aliquot is treated with bromine to oxidize all the sulfur to sulfate, and total sulfate is determined as above. Sulfite is then determined by difference.

SILICA, PHOSPHORUS, AND ARSENIC

Matveev and Bashmakova (16L) used the yellow molybdosilicate complex to determine silicon in waters with low mineralization. The color is compared with standard potassium chromate solutions. The samples can also be analyzed colorimetrically if the yellow complex is reduced to the blue form with 1 - amino - 2 - naphthol - 4 - sulfonic acid. Grasshoff (10L) described a yellow molybdosilicate method for determining dissolved reactive silica in sea water. A sample of water is filtered, monochloroacetic acid and molybdate reagent added, the sample stored for 6 hours or more, and the absorbance of the complex measured at 390 m μ . Fresenius and Schneider (9L) determined silica in mineral water by the molybdosilicate method after converting polymeric silicic acid to monomeric silicic acid. Polymeric silicic acid on treatment with hydrofluoric acid yields silicon fluoride which with boric acid gives monomeric silicic acid. Novoselov and Simolin (21L) used a *p*-methylanilinephenol sulfate-sodium sulfite mixture to reduce the yellow molybdosilicate complex. The reduced blue solution is determined spectrophotometrically. The method was used to determine silicates in sea water.

A simple extraction procedure for determining silica in sea water was described by Schink (25L). Ammonium molybdate-sulfuric acid reagent and sulfuric acid were added to an aliquot of sample. The sample was mixed, transferred to a separatory funnel, and the complex extracted with ethyl acetate. The absorbance of the organic phase was then measured at 3350 Å. Turbid waters did not have to be filtered prior to analysis. Kanie (14L) used ion exchange to separate silica in high purity water, and then determined silica by the molybdenum blue method. An aliquot of sample, hydrofluoric acid, and amberlite IRA 402 (OH-type) are shaken together for 30 minutes in a polyethylene separatory funnel. The resin is filtered through a nylon cloth, and silica is desorbed with a saturated solution of boric acid.

Webber and Wilson (31L) used the molybdate blue method of Morrison and Wilson (19L) to determine silica in boiler water. Tests were performed to confirm the suitability of the method in the presence of phosphate and other sub-

stances. The phosphate effect was small for silica concentrations exceeding 0.1 ppm. A method for determining total silica in water was reported by Kostrikin, Shtern, and Dzysyuk (15L). A sample of water was evaporated in a platinum dish, fused with sodium carbonate, and the residue dissolved in hot water. The solution was transferred to a volumetric flask, sulfuric acid was added, it was brought to volume with water, and silica was determined by the molybdenum blue method.

Wilson (32L) described a continuous automatic method to determine reactive silica in boiler water with the molybdate blue procedure. A Technicon AutoAnalyzer was investigated in respect to response time, calibration curve, precision, stability of response, and effect of phosphate. The instrument performed reliably and appeared to be suitable for continuous analysis.

Brewer and Riley (2L) determined silicate in natural waters in the range 0 to 4 mg per liter with a Technicon AutoAnalyzer. Again, the method is based on the reduction of silicomolybdic acid to the molybdenum blue complex. Oxalic acid is used to prevent interference from phosphate. Tanno (28L) also described an automatic recording analyzer based on the molybdenum blue method for the determination of silica concentrations greater than 20 ppb.

Jones (13L) compared several methods of determining inorganic phosphate in sea water and recommended the method of Murphy and Riley (20L) as a standard procedure for estimating phosphate in sea water. The method involves adding a measured amount of a single-solution reagent to an aliquot of the sample and, after 10 minutes, determining the absorbance of the intense blue complex. The reagent is prepared by dissolving appropriate amounts of ammonium molybdate, ascorbic acid, and antimonyl potassium tartrate in dilute sulfuric acid. This reagent solution is unstable and must be prepared fresh.

Fishman and Skougstad (7L) described a simplification of the method of Murphy and Riley and adapted the procedure to the field determination of phosphate in natural waters through the preparation of a set of permanent color standards against which samples may be compared, and a single-solution reagent prepared immediately before use by dissolving a measured amount of a mixture of dry chemicals consisting of ammonium molybdate, ascorbic acid, and antimonyl potassium tartrate in dilute sulfuric acid. Watanabe and Olsen (30L) reported that the method of Murphy and Riley is accurate for determining phosphate in water or sodium bicarbonate aqueous soil extracts. The color of the complex developed with ascorbic acid is stable for 24 hours whereas that developed by stannous

chloride is liable to interference by organic matter in soil extracts.

Chan and Riley (3*L*) used a Technicon *AutoAnalyzer* to determine 0 to 90 μg of phosphate in spiked sea water by the method of Murphy and Riley. Twenty samples can be analyzed in an hour.

Ambuehl and Schmid (1*L*, 26*L*) described a molybdenum blue procedure for determining small amounts of phosphate and total phosphate in inland lake water. Stannous chloride is used as the reducing agent. Data on accuracy are given. Hartmann and Stenzel (11*L*) determined phosphate in drinking water by a molybdenum blue method. Polyphosphate is first hydrolyzed with sulfuric acid at 95° C. The molybdate reagent consisting of ammonium molybdate, hydrazine sulfate, and sulfuric acid is then added and the solution heated for 35 minutes at 95° C. The absorbance of the blue color is measured at 750 $m\mu$. Edwards, Molof, and Schneeman (6*L*) used ascorbic acid as the reducing agent in the molybdenum blue method to determine orthophosphate in fresh and saline waters. They also found that 5% alcohol in the color development mixture extended the linear color development range and eliminated precipitation in sea water. Ninety-nine percent color development occurred within 4 minutes. Umnova (29*L*) also used ascorbic acid as a reducing agent in the molybdenum blue method to determine phosphate in industrial waste water. Total phosphate is first hydrolyzed by boiling a sample with potassium permanganate and sulfuric acid.

A spectrophotometric method for determining phosphate in water based on the reaction of phosphate and vanadate-molybdate reagent was described by Moeller (17*L*). A sample of water is evaporated to dryness and calcined for 1 hour at 450° to 550°. The sample is evaporated twice more with water and hydrochloric acid, and then dissolved with water and hydrochloric acid. The solution is diluted to a known volume passed through an anion exchange resin, and vanadate-molybdenum reagent added to a portion of the eluate. After 1 hour the absorbance of the solution is measured at 366 $m\mu$. Silicic acid removal is not complete and the phosphate values are high by 0.05 ppm. Proft (24*L*) also used a vanadate-molybdenum reagent to determine phosphate in water and waste water. All forms of phosphate are first brought into solution by evaporating an aliquot of sample on a hot plate with perchloric and nitric acids. The sample is then cooled, transferred to a volumetric flask, and vanadate-molybdenum reagent is added; the mixture is diluted to the mark and phosphate determined spectrophotometrically.

Won (33*L*) described a spectrophoto-

metric method to determine orthophosphate in natural waters. An aliquot of sample containing from 0.3 to 1.2 μg of phosphate is transferred to a separatory funnel, and ammonium molybdate and perchloric acid are added. After at least 5 minutes a known volume of butyl acetate is added and the mixture shaken. A portion of the organic phase is transferred to a flask, and stannous chloride in 5.5*N* hydrochloric acid and copper sulfate in 5*N* perchloric acid are added. The mixture is heated, mixed, cooled, and ammonium thiocyanate added. The absorbance of the organic phase is then measured at 460 $m\mu$.

A phosphomolybdate extraction procedure utilizing benzeneisobutanol was used by Morkowski (18*L*) to differentiate the various forms of phosphorus in water and waste water. Details of the procedures are discussed.

An automatic method for determining orthophosphate and acid-hydrolyzable phosphates in fresh and saline waters with the Technicon *AutoAnalyzer* was described by Henriksen (12*L*). The method is based on the extraction of phosphomolybdate with isobutanol and color development with stannous chloride. A schematic and details of the procedure are given. The lowest detectable concentration of phosphorus achieved with this method is 1 μg of phosphate per liter.

Cosoveanu (4*L*) preferred determining phosphate in boiler water with ammonium molybdate, sodium sulfite, and hydroquinone rather than by the conventional reduction of phosphomolybdic complex with stannous chloride or metallic tin. Pavlik (22*L*) described methods for determining phosphate in natural waters. For the concentration range 0.05 to 1 mg per liter a molybdenum-tungstate reagent is added directly to the sample, the complex reduced, and the solution analyzed colorimetrically at 700 $m\mu$. For the concentration range 0.001 to 0.1 mg per liter, phosphate is concentrated by anion exchange (Dowex 2-X8) and then determined colorimetrically.

Fresenius and Schneider (8*L*) determined arsenic in water with silver diethyldithiocarbamate. A sample is acidified with sulfuric acid, As^{+5} reduced to As^{+3} with potassium iodide and stannous chloride, and the As^{+3} converted to AsH_3 in the presence of copper-coated zinc. Arsine is then absorbed by silver diethyldithiocarbamate in pyridine, and the absorbance of the red color measured at 510 $m\mu$.

Conversion of As^{+5} to As^{+3} with ascorbic acid and cocrystallization of As^{+3} with thionalide in a 0.05*N* sulfuric acid medium was proposed by Portmann and Riley (23*L*) for the recovery of microgram amounts of arsenic from sea water. Arsenic is then determined by measuring the absorbance of the molyb-

denum blue complex by a modification of the single-solution method of Murphy and Riley (20*L*).

A spectrophotometric method for determining trace amounts of arsenate, arsenite, and phosphate in natural waters was described by Sugawara and Kanamori (27*L*). Phosphate and arsenate are extracted as molybdophosphate and molybdoarsenate with different solvents. Arsenite is then oxidized and extracted. The respective fractions are then treated with sodium hydroxide to liberate molybdenum, which is then measured spectrophotometrically as the thiocyanate.

Davidyuk (5*L*) proposed a new polarographic method for determining arsenic in natural waters. Details of the procedure are given.

NITRATE, NITRITE, AND NITROGEN COMPOUNDS

A statistical comparison of the brucine, chromotropic acid, and phenoldisulfonic acid methods for determining nitrate in water was conducted by Jurupe Ch. (17*M*). No significant difference between the methods was detected. However, the brucine method was recommended when a rapid analysis is desired, and when the sample is low in nitrate and high in chloride. But for better precision when time is available, the phenoldisulfonic acid and chromotropic acid methods were suggested in order of preference.

Waters (27*M*) compared a number of methods for determining nitrate in water and found that a considerable lack of information existed on the range, precision, and accuracy of present methods. The brucine and indigo carmine methods for nitrate in water were evaluated by Fadrus and Maly (10*M*) with regard to precision and reliability.

West and Ramachandran (28*M*) described a chromotropic acid method which will tolerate over 4000 mg of chloride per liter, and which eliminates other sources of interference. Murai *et al.* (23*M*) modified the traditional aniline procedure to establish a simple and reliable method for determining nitrate in the presence of nitrite. Grasshoff (13*M*) determined nitrate in salt and potable waters by reducing nitrate to nitrite by a cadmium amalgam in a Jones reductor, followed by diazotization with sulfanilamide and coupling with 1-naphthylethylenediamine. The absorbance was measured at 543 $m\mu$. No interferences were observed from other ions in salt water.

Ostrowski and Nowak (24*M*) determined nitrate in sea water by a colorimetric reaction utilizing diphenylbenzidine. The use of both diphenylamine and diphenylbenzidine was investigated by Hsiung (16*M*) in an effort to improve the sensitivity and accuracy of the

nitrate determination. A simplified method using a mixture of the two reagents was developed, and gave more accurate values for nitrate in sea water samples than either reagent alone. It may be applied in the field as well as in the laboratory.

A sensitive method for determining nitrate in water by reaction with 2,6-xylenol and extraction into toluene was described by Andrews (1*M*). The absorbance is measured spectrophotometrically at a wavelength of 432 m μ . Interferences from nitrite and chloride are eliminated by the addition of sulfamic acid and mercuric sulfate, respectively. A standard deviation of ± 0.013 mg of nitrate nitrogen per liter was obtained in recovery tests on natural waters. Bloomfield, Guyon, and Murmann (4*M*, 5*M*) discussed two similar methods for determining nitrate in water based on the interference which nitrate exhibits in the formation of colored complexes of heptavalent rhenium with α -furil dioxime and *syn*-phenyl-2-pyridyl ketoxime in acid solution in the presence of stannous chloride. The effects of a large number of diverse ions were tested. Most common ions do not interfere.

An automatic method for determining nitrate in sea water using a Technicon AutoAnalyzer was reported by Brewer and Riley (8*M*). The sample was mixed with an EDTA reagent (pH 6.5-7.0) and passed through a tube of cadmium filings. The resultant nitrite was determined by diazotization with sulfanilamide and coupling with *N*-naphthylethylenediamine according to the method of Bendschneider and Robinson (3*M*). Allowance is made for the presence of nitrite in the sample. About 30 samples per hour can be analyzed. The method has a coefficient of variation of 1.6% at the 200 μ g per liter concentration level. Henriksen (14*M*) described an automatic method for determining nitrate and nitrite in fresh and saline waters using a Technicon AutoAnalyzer. The AutoAnalyzer system was shown schematically. The method is based upon the diazotization procedure originally published by Mullin and Riley (22*M*) in which hydrazine is used to reduce nitrate to nitrite in the presence of a copper catalyst, followed by diazotization with sulfanilic acid and coupling with 1-naphthylamine. Increasing the temperature from 20° to 70° decreases the time required for reduction of nitrate to nitrite from 24 hours to 15 minutes.

A study of the nitron method for the gravimetric determination of nitrate in sewage effluents was made by Quon and Lagvankar (25*M*).

Dey (9*M*) determined nitrite in water colorimetrically by a procedure involving diazotization with *p*-aminobenzoic acid and coupling with *N*-naphthylethylenediamine dihydrochloride in the pH range of 1.7 to 2.2. The color intensity

is a linear function of concentration in the 0.005 to 0.15 ppm nitrite range.

Richards and Kletsch (26*M*) determined ammonia and labile amino compounds in fresh and sea waters by oxidation to nitrite with sodium hypochlorite in strongly basic solution. The resulting nitrite is diazotized with sulfanilamide, coupled with *N*-naphthylethylenediamine and analyzed spectrophotometrically at a wavelength of 540 m μ . In the procedure reported by Konnov (18*M*), the pH of the sample is adjusted with a borate buffer and the ammonia distilled under reduced pressure. This is followed by addition of sodium hypobromite, after which the color is developed with α -naphthylazo- β -naphthol-3,6-disulfonic acid. A standard curve is prepared by using ammonium chloride. As little as 1 μ g of nitrogen per liter can be detected.

A method for determining total nitrogen in diluted water solutions was proposed by Gertner and Grdinic (11*M*). Nitrogen is freed for analysis by decomposing the organic matter in sulfuric acid solution containing potassium persulfate and hydrogen peroxide, using silver as the catalyst. The method takes less than 2 hours and works irrespective of the manner in which nitrogen is bound in the organic molecule.

Gonter and Schmitt (12*M*) reported a procedure for determining cyanides in effluents from coke oven and blast furnace operations. A reflux distillation procedure employing cuprous chloride as a catalyst was used for removing interfering materials and for conversion of the cyanides into sodium cyanide. The cyanide was then determined by titration or colorimetrically. Several electrode systems were suitable for the electrometric titration of sodium cyanide in alkaline solution. The benzidine-pyridine and the pyridine-pyrazolone colorimetric methods were evaluated independently by 10 laboratories. The results obtained indicated that more precise results can be obtained by using the benzidine-pyridine procedure.

Hissel and Cadot-Dethier (15*M*) isolated free cyanides from complex cyanides and thiocyanates with Amberlite IRA-400 in the hydroxyl form. After elution with 1% potassium nitrate, free cyanide was measured colorimetrically with pyridine-pyrazolone reagent. Boye (6*M*) used a method for determining cyanides in waste water from gas works and coke oven plants by a modification of the method reported by Boye and Scholz (7*M*). The sample was acidified with citric acid and the evolved HCN absorbed in sodium hydroxide solution. The cyanide was then determined colorimetrically with pyridine and benzidine, or by titration with silver nitrate. The modifications consisted of clarifying the effluent with trisodium phosphate, precipitation of sulfides with

lead carbonate, and sweeping out the apparatus with air instead of nitrogen. Bahensky (2*M*) described a rapid colorimetric method for estimating cyanides in waste waters using chloramine T and 1-phenyl-3-methyl-5-pyrazolone.

Lur'e and Panova (19*M*) used both sulfanilic and barbituric acids to determine cyanides in waste waters. Ferrocyanides in concentrations of less than 10 mg per liter did not interfere. The detection limit was approximately 0.01 mg per liter. The decomposition of ferro- and ferricyanides which occurs during the determination of free cyanides by distillation methods was studied by Mrkva (20*M*). By distillation, HCN is freed from these compounds at pH 7. The amount of decomposition depends upon the pH value, method of distillation, amount distilled, and concentration of the complex cyanides. The distillation methods were unsuitable for determining free cyanides in samples containing ferro- and ferricyanides.

Mrkva and Mica (21*M*) developed a method for the determination of thiocyanates in phenol waters. An excess of lead carbonate powder is added to the sample to precipitate sulfides. After filtration, the pH is adjusted and the sample heated with a germanium sulfate-ammonium sulfate solution to precipitate cyanides. The sample is again filtered, decolorized with sodium persulfate, and precipitated by boiling with cupric sulfate. The cuprous thiocyanate precipitate thus formed is filtered and washed. Nitric acid and ferric ammonium sulfate are added to redissolve the precipitate and the red solution is titrated with 0.05*N* silver nitrate until it is colorless.

OXYGEN DEMAND

A critical review of the various methods for the determination of biochemical oxygen demand (BOD) in river water and sewage was published by Niemitz (11*N*). Young and Clark (16*N*) prepared a review with 12 references on the history of the BOD test.

Wolfner (15*N*) determined BOD values in effluents by using dichromate oxidation. The sensitivity of the method was increased by adjusting the amounts of the reagents used to the expected BOD. Filatova (5*N*) reported that 5-day BOD determinations on biologically purified effluents in which nitrification has begun give high results because nitrogen-containing compounds are oxidized. To avoid this difficulty, the methylene blue method is recommended over the one involving pasteurization. Initial values of BOD in rivers strongly contaminated with sewage were determined by Sedzikowski (13*N*).

A barometric vibration method for BOD was described by Burchard (1*N*). It consisted of measuring the change in

pressure of the atmosphere in a closed 903-ml vessel containing the sewage, which is vibrated to provide continued mixing and contact with the air. Results were comparable after 5 days' vibration with volumetric BOD measurements.

An improved apparatus for determining BOD of polluted water was reported by Young, Garner, and Clark (17*N*). A standard 1-liter narrow mouth reagent bottle is used as the reaction flask. Claimed advantages are reduced cost, simplicity of operation, and obviation of a large head space which was a source of error with varying external barometric pressure. A detailed description and scale drawings are given. A rapid polarographic technique for BOD determinations was reported by Krishnamurty (10*N*).

Fitzgerald (6*N*) reported a study on the measurement of BOD on several species of algae, alone, and in association with bacteria under 5 days of incubation in the dark at a temperature of 20°. The object was to determine if algae have an effect on BOD measurements under standard conditions. Eighteen references were cited.

A procedure for measuring low chemical oxygen demand (COD) by the dichromate method in the presence of high chloride concentrations was described by Cripps and Jenkins (3*N*). The method relies on the formation of undissociated mercuric chloride which is only slightly oxidized under standard COD conditions. The limitations of the Cripps and Jenkins method were studied by Burns and Marshall (2*N*), and a revised procedure was developed to correct for sodium chloride concentrations as high as 3%. The improved method is reported to give the most accurate COD measurements presently possible.

Pitter and Se-Kvan-Son (12*N*) compared the dichromate and iodate methods for determining COD for different boiling times with and without silver sulfate as a catalyst. The dichromate method with silver sulfate gave higher results than the iodate method. Without silver sulfate, both methods gave comparable results. The dichromate method was easier to carry out and responds best to the organic pollution of sewage and industrial wastes. El-Dib and Ramadan (4*N*) investigated dichromate as compared to ceric sulfate for COD and concluded that dichromate is far superior, especially if silver ion is used as a catalyst.

Gaudy and Ramanathan (7*N*) reported a colorimetric adaptation of the standard dichromate procedure. Dichromate is added to the sample as an oxidant, and the excess determined colorimetrically at 440 mμ. The method is rapid and yields results comparable to the standard titration technique. Hankus (8*N*) and Jakubowska (9*N*)

each investigated several methods for determining the oxidizability of various waters. Both investigators agree to the superiority of the dichromate method.

A titrimetric method for oxygen demand using chloramine-T was described by Steinmetz and Losonczy (14*N*). Chloramine-T and HCl are added to the sample, which is then kept in the dark for 30 minutes. Potassium iodide is added, the sample left in the dark for 5 minutes, and the excess chloramine-T titrated with standard sodium thiosulfate solution. Five types of waters were tested and the results compared with those obtained using the permanganate method.

OXYGEN AND OTHER GASES

A routine method for the determination of oxygen in water or in gas mixtures was developed by Takahashi, Sakurai, and Sakamoto (44*P*). The sample solution or gas was passed through a Jones reductor and the liberated Zn^{+2} was measured polarographically. An acetate buffer (pH 6) was mixed with the continuously flowing sample solution and used as the supporting electrolyte. The calibration curve was linear in the concentration range of 0.1 to 7.0 mg of oxygen per liter in water under the optimum conditions of pH, flow rate, and column length. A Pt/NaCl/AgCl/Ag cell for determining dissolved oxygen with a mean accuracy of 2% was reported by Khaidarov (18*P*). The cell is covered with a polyethylene membrane and yields a current which is proportional to the oxygen concentration. The principal limitations are the difficulty of temperature compensation and the fact that the platinum cathode must be polished and the instrument periodically standardized. Shol'ts and Ostrovskii (36*P*) described an improved polarographic cell for the quantitative determination of dissolved oxygen which features ease of construction and handling. The basic model was that of Ostrovskii and Gel'man (27*P*) where thermostating was obtained with a flow of liquid in the cell and mixing was done by a magnetic stirrer placed above a film covering the electrodes; the current fluctuations due to temperature variations were almost completely eliminated with a semiconductive thermoresistance. Duxbury (6*P*) reported the use of a galvanic-type cell for use in field determinations of oxygen. It is a modification of the Mancy, Westgarth, and Okun (22*P*) instrument, and its basic construction and calibration are given. The use of a lead anode and silver cathode eliminated the oxidizing procedures required with platinum electrodes.

A simple, all glass apparatus was constructed by Slechtova and Novacek (38*P*) for determining dissolved oxygen in waste waters. The device consisted

of two cells and operated according to Henry's law. Results compared favorably with results obtained by the Winkler method using permanganate.

A coulometric technique for determining dissolved oxygen in natural waters was reported by Healy (13*P*). Burkert (3*P*) described a recording apparatus for the determination of oxygen in boiler feed water. It depends on the reaction $\text{O}_2 + 4\text{NO} + 2\text{H}_2\text{O} = 4\text{HNO}_2$ and measures the change in conductivity brought about by this reaction. Kabanova and Zalogina (16*P*) used the method of Wright and Lindsay (53*P*) for determining trace amounts of oxygen in water. The method is based on the change in specific conductance of the sample after passage through a thallium column. The change is proportional to the dissolved oxygen content. Oxygen concentrations between 10 and 200 μg per liter can be analyzed.

Gillespie and Richter (11*P*) constructed an apparatus for the determination of as little as 1 part-per-trillion dissolved oxygen in sea water, streams, ponds, and drinking water. The analyzer consists of a column of ^{204}Tl (half life 3.6 years, 0.75-mev B, ≈2% electron capture) deposited on copper turnings. Water to be analyzed passes through the column forming Tl^+ ions, and simultaneously the ^{204}Tl is released and detected by a Geiger-Mueller tube. The counting rate is a direct measure of the dissolved oxygen concentration. A battery powered and transistorized portable instrument has been constructed.

Toedt (46*P*) reviewed and discussed difficulties which arise in the continuous electrochemical determination of oxygen in impure water. The major limitations are the deposition of calcium carbonate and the formation of foam on the electrode surface. Two improvements of known methods are given. In the first method the analyzed water is continuously acidified to maintain a pH which prevents the deposition of calcium carbonate. The second method is based on the fact that deposition of calcium and magnesium carbonates vanishes when the current is switched off for some time. An apparatus was constructed which makes possible the continuous determination of oxygen by periodic interruptions of the current. With measurement intervals of 15 seconds and pauses of 45 seconds, the correct and constant values of oxygen content were obtained even in water having a high content of calcium and magnesium carbonates and even over periods of several days.

A review of the various colorimetric methods for determining dissolved oxygen in boiler feed water was given by Shtern (37*P*).

Ungureanu (47*P*) reported two photometric methods for determining dissolved oxygen in water: One is based on the oxidation of the leuco-indigo com-

pound (intense yellow) in alkaline solution into the indigo carmine compound (intense blue), and the other utilizing the oxidation of Ce^{+3} in alkaline media to Ce^{+4} by using *o*-toluidine as indicator. The first method was more rapid than the second, but was more prone to errors because of the lower stability of the leuco-indigo as compared to that of the Ce^{+3} salts.

Sorgatz and Dubois (41P) used a cerous chloride photometric method for determining oxygen in condensate and feed water. A special bottle is used to exclude errors in sampling and in performing photometric determinations.

Ivanitskaya and Mostofin (15P) determined low concentrations of dissolved oxygen in water of thermoelectric stations by colorimetric analysis with Safranin T, whose leuco compounds are nearly colorless and do not form intermediates during oxidation. A sampling device and an apparatus for reducing Safranin with zinc amalgam were described.

Ostrowski, Jasinska, and Zolendziowska (28P) reported the use of *N,N'*-bis(2-hydroxypropyl) - *o* - phenylenediamine for determining dissolved oxygen in water. Manganous ion (added as MnCl_2) in basic solution is precipitated as $\text{Mn}(\text{OH})_2$ and promptly oxidized by oxygen in the solution to Mn^{+3} . *N,N'*-Bis(2-hydroxypropyl) - *o* - phenylenediamine in 25% HCl is then added and the color which develops measured at 533 $\text{m}\mu$. Beer's law is followed in the range of 0 to 5 ppm, and the method is claimed to be faster and to give better precision in this range than the Winkler titration.

Ozone between 0.02 and 1.00 mg per liter in water in the presence of other oxidants was determined by Koppe and Muhle (19P). At pH 2 in phosphoric acid-dihydrogen phosphate media, ozone oxidizes *N,N,N',N'*-tetramethyl-*p,p'*-diaminodiphenylmethane and *N,N,N',N'*-tetramethyl-*p,p'*-diaminotriphenylmethane to the corresponding fluorene which is extracted into chloroform and measured at 492 $\text{m}\mu$. A small amount of permanganate eliminates interference from MnO_2 , Cl_2 , and H_2O_2 .

Several investigators suggested modifications of the Winkler method. Malik (20P) used a redox titration with 1,10-phenanthroline as indicator for greater convenience and reliability as compared to the classic method. Felicetta and Kendall (8P) reported a "rapid permanganate" modification of the Winkler test and galvanic cell methods for determining dissolved oxygen in spent sulfite liquor.

However, the modified Winkler procedure was not satisfactory because of a tendency to yield low results. Rabinovich and Sherman (33P) described a Winkler modification for determining dissolved oxygen concentra-

tions in small biological cultures and sludge solutions.

Carpenter (4P, 5P) examined the potential errors in the various techniques used for the Winkler method and developed a new technique for improved accuracy. A special titration apparatus is illustrated. Errors in sampling for dissolved oxygen were studied by Montgomery and Cockburn (24P). The effects of sample handling were evaluated by Porges (32P), and precautions and recommendations were given for the field treatment of samples to provide a more accurate and reliable analysis.

Ross (35P) described a method which is under consideration as a British standard for determining oxygen in fresh or saline waters. The method involves the sodium thiosulfate titration of the iodine released after the addition of alkaline iodide reagent, manganous sulfate, and sulfuric or phosphoric acid. The complexometric method of Malik (21P) utilized the oxidation of manganous ion to the manganic ion, and titration of the deep violet complex formed between manganic ion and ethylenediaminetetraacetate with standard ferric alum solution. The end point may be detected visually or potentiometrically.

Park and Catalborno (30P) determined dissolved oxygen in sea water using a modification of the gas chromatographic method of Swinnerton, Linnebom, and Cheek (42P). Argon is used as the carrier gas. Gaunt and Shanks (9P) reported a chromatographic method for the microdetermination of oxygen in boiler feed water using hydrogen as the carrier gas. The calibration curve is linear with respect to peak height or peak area over the 0.000- to 0.015-ppm range.

In discussing the determination of trace concentrations of oxidizing and reducing compounds in liquids and gases, Teske (45P) described an apparatus and the electrochemical basis for the continuous measurement of sulfur dioxide, hydrogen sulfide, hydrazine, oxygen, ozone, and hydrogen in flue gas, natural gas, boiler make-up water, sewage, and the atmosphere. A comparison was made with other methods. West (51P) reported a mass spectrographic method for determining dissolved gases in water in the 5- to 30-ppb range. Methods and apparatus diagrams were given by Vignet *et al.* (48P) for the determination of total gases dissolved in water at a pressure of approximately 200 kg per sq cm.

An amperometric titration using sodium arsenite for determining free residual chlorine was described by Ghelberg (10P). The current indicator was a cell containing the Au/Cl water, KCl, AgCl/Ag electrode system immersed in the water. A dual polarizable electrode apparatus consisting of two platinum electrodes, a mercury cell, a microammeter, a resistor to fix the potential, and

an a.c./d.c. stirring motor was used by Morrow (25P) for the phenylarsene oxide titration of residual chlorine. Precision, sensitivity, and interference levels are similar to those for conventional amperometric titrations, but more rapid titrations are possible. The unit may also be battery operated for field use. Two coulometric titration methods were described by Takahashi, Sakurai, and Namiki (43P). A dead-stop end point detection method was used and results were superior to those obtained by the potentiometric titration method.

Williams (52P) used *o*-toluidine for measuring residual chlorine in swimming bath waters; however, Palin (29P) stated a preference for the titrimetric version of the diethyl-*p*-phenylenediamine method. A colorimetric method for determining free chlorine in polluted waters based on the bleaching action of chlorine on methyl orange was developed by Solis and Larson (40P). Absorbance is measured at 510 $\text{m}\mu$. Results agreed well with those obtained by the amperometric titration method. Webber and Wheeler (50P) reported a modification of the absorptiometric method of Asmus and Garschagen (1P) for determining chlorine in water.

Methods for determining free chlorine in water were evaluated by Nicolson (26P) on the basis of reproducibility, specificity, sensitivity, limit of detection, recovery of chlorine, effect of temperature, stability of reagents, simplicity, and convenience. Of the eight methods studied, the procedure based on the reaction of *N,N*-diethyl-*p*-phenylenediamine with chlorine to form a colored oxidation product was judged best in the presence of combined chlorine, if the color is measured immediately. In the absence of combined chlorine, a method based on the Zincke-Koenig reaction in which an alkali metal cyanide reacts with chlorine to form cyanogen chloride, which in turn reacts with an amine (barbituric acid) in pyridine to form an intensely colored solution, was selected as the best laboratory method. Titrimetric procedures were less reproducible than spectrophotometric procedures. Thirty-eight references are listed.

Kegel (17P) pointed out that errors in the titrimetric determination of free carbon dioxide in water are caused by the presence of calcium bicarbonate. A new method was developed based on the measurement of pH before and after the addition of a small amount of inorganic acid. Haesselbarth (12P) evaluated the relative errors of the Kegel procedure and calculated the systematic and accidental errors involved in the acidimetric titration of carbon dioxide in water. Comparative investigations showed good agreement between the two methods after slight improvements in the Kegel procedure in the concentration

range of 0.5 to 1.0 meq of alkalinity per liter and up to 20 mg of free carbon dioxide per liter.

Rao (34P) used a conductivity technique for determining total carbon dioxide in dilute tropical waters in the 40 to 100 mg per liter concentration range. Park, Kennedy, and Dobson (31P) investigated the gas chromatographic and pH-alkalinity methods for measuring total carbon dioxide in sea water and found that both methods give comparable results. An infrared gas analyzer was used by Waterman (49P) for determining carbon dioxide in surface ocean water.

Matveev and Zavodonov (23P) determined carbon dioxide in snow and ice by allowing a weighed sample to melt in a stoppered flask with barium hydroxide and phenolphthalein indicator. After 6 to 10 hours, aliquots are titrated with standard hydrochloric acid solution. Directions for determining the changes in carbon dioxide concentration from changes in pH were given by Beyers *et al.* (2P). The method involved titration with gaseous carbon dioxide using distilled water as carrier to obtain curves relating pH value to carbon dioxide concentration. Detailed instructions were given for preparing the carbon dioxide saturated water, for titrating the water sample, and for computing the total changes in carbon dioxide content.

Herrmann (14P) reported a proposed ASTM method for determining carbon dioxide in water-formed deposits. The sample is treated with acid and the loss in weight is taken as carbon dioxide.

An indirect method for determining dissolved hydrogen based on the reaction between hydrogen and oxygen in the presence of a platinum catalyst was described by Faber and Brand (7P). Two identical samples are collected. A dissolved oxygen determination is performed on the first sample. A platinum catalyst emulsion is added to the second sample and the dissolved oxygen determined. The decrease in the oxygen content is a measure of the dissolved hydrogen in the sample. In samples having a deficiency of oxygen, a known amount of hydrogen peroxide is added to the second sample to facilitate the oxidation. A simple method for determining hydrogen in condensed steam was reported by Smit and Ehrenburg (39P). The oxygen-free sample is divided into two branches, each containing a platinum electrode. A known hydrogen concentration is introduced into one branch, and the unknown concentration is determined from the potential difference.

DETERGENTS

Three reviews of determinations of detergents in water and sewage were reported. Heinerth (2Q) described 21 references on nonionic surfactants,

mostly on colorimetric methods, not one of which gives an entirely satisfactory method. A thin layer chromatographic method was judged best because the constitution of the ethoxylate under analysis does not have to be known *a priori*. However, this complicated method is not suitable for routine determinations. Krygielowa and Szmalowa (5Q) reviewed 40 references on anionic detergents, and Pitter and Hyankova (8Q) reviewed 24 references.

Several investigators described methods for the determination of anionic detergents in water with Testa (10Q, 11Q) reporting two automatic ones. Both methods involve the extraction in chloroform of the colored complex formed between the detergent and methylene blue and the spectrophotometric reading of the chloroform coloration at 660 m μ . Samples should contain less than 12 ppm of detergent. Sodergren (9Q) used an automatic method for the determination of anionic detergents in water. Peintre and Romens (7Q) determined traces of anionic detergents in water by fixing them on Amberlite IRA 68 (OH form). The exchanger was eluted with a mixture of acetone and 0.1N sodium hydroxide and the detergent-methylene blue complex was measured spectrophotometrically.

Panowitz and Renn (6Q) outlined a modification of the Longwell-Maniece method for the determination of anionic detergents. The essential steps of chloroform handling are carried out in a large glass hypodermic syringe. The device consists of a 50-ml Luer-loc-type syringe, mounted horizontally in a Teflon cradle, and coupled to a 60-rpm CCW gear motor. By using a calibrated syringe, a controlled volume of reagents can be drawn into the reacting system. Another modification of the Longwell-Maniece method, described by Kobayashi, Kanno, and Takuma (4Q), reduced the interference of anionic detergents. To a 50-ml water sample containing 0.02 to 0.14 mg of ABS, 25 ml of hydrochloric acid was added and boiled for 1 hour with a water-cooling condenser. After neutralization with sodium hydroxide to the phenolphthalein indicator end point, the Longwell-Maniece method was used. Kambara and Hasebe (3Q) determined alkylbenzenesulfonate in water by solvent extraction-alternating current polarography. The range of the method covers from 0.07 to 0.028 mg ABS per 10 ml chloroform, and the time required for a single determination is about 25 minutes.

Arpino (1Q) discussed some analytical techniques, particularly the colorimetric and spectrophotometric ones, for the determination of surface active agents. Wayman and Miesch (12Q) compared two field methods using a modified methylene blue technique and the dye Toluidine Blue O as the complexing

agent *vs.* a laboratory method using methylene blue as the complexing agent. The precision decreases in the order: methylene blue laboratory method, modified methylene blue field method, and the dye Toluidine Blue O field method. Significant bias was detected only in the latter.

PESTICIDES AND HERBICIDES

Van Valin and Kallman (11R) investigated the use of activated carbon for the adsorption of chlorinated hydrocarbons from dilute solutions. Warnick and Gaufrin (12R) used electron capture gas chromatography to obtain reproducible results and measurements of both organic phosphorus and chlorinated hydrocarbon pesticide concentrations of less than 1 ppb in water. Using a flame ionization or electron capture detector unit, May, Hindin, and Dunstan (8R) obtained gas chromatographic analyses of pesticides in water. For pesticides containing chloride, a microcoulometric detection unit was used to avoid interference. Most water samples had chlorinated hydrocarbon contents in the range 1 to 10 ppt.

Lamar, Goerlitz, and Law (7R) described the advantages, disadvantages, interferences, and application of an electron capture gas chromatographic procedure for the identification and measurement of chlorinated organic pesticides in water. Aldrin, dieldrin, *o,p*-DDT, *p,p*-DDT, endrin, and lindane were studied. Concentrations in the parts-per-trillion range can be determined.

Baker (2R) reported on a preliminary study of freezing combined with flame-ionization detection chromatography for concentrating and determining phenols, cresols, and chlorophenols in water. The freeze technique is rapid and may be done with simple, relatively inexpensive equipment. Contaminants are not subject to chemical modification as they would be in distillation or extractive procedures. At a critical dissolved-solids content, spontaneous ice crystallization precludes further separation.

Sanderson and Ceresia (9R) described a continuous liquid-liquid extractor that can determine chlorinated aromatic pesticides in parts per billion. It has an efficiency of 95%, is gravity fed, easily controlled, and is safe to operate overnight.

Calderbanks and Yuen (3R) used a combination ion exchange and spectrophotometric method to determine paraquat residues in food crops and water. Sensitivity is 0.01 ppm.

Atabaev and Khasanov (1R) reported a determination of small amounts of aldrin in water. The method is based on the reaction of aldrin with phenyl azide. The resulting aldrin phenyldihydrotriazole, when treated with diazo-

tized 2,4-dinitroaniline in an acid medium gave a pink color that was measured colorimetrically. The intensity of the color and its stability were improved by the addition of 0.50 ml of sulfuric acid (2:1).

In an examination of surface waters and sewage effluents for organochlorine pesticides, Holden and Marsden (5R) obtained a concentration factor of 800 by extraction of a 1-liter stream sample with 50 ml of redistilled hexane followed by evaporation to less than 1 ml. The extract was analyzed by gas-liquid chromatography in an all-glass apparatus equipped with electron capture detectors. Dieldrin and DDE were the two residues most commonly found. Their minimum detectable concentration was 0.5 gram per 10^{12} grams of water.

Katz (6R) reported determinations of several substituted urea herbicides in surface waters. The herbicides are extracted in chloroform and hydrolyzed under reflux conditions with 6N hydrochloric acid. The resulting aniline is diazotized and condensed with *N*-(1-naphthyl)ethylenediamine to form a magenta dye, which is extracted with normal butyl alcohol and measured colorimetrically at 555 m μ . Levels as low as 0.02 ppm of linuron, diuron, and neburon, 0.03 ppm of monuron, and 0.04 ppm of fenuron can be determined. Faust and Hunter (4R) described chemical methods, ion exchange chromatography, gas-liquid chromatography, and steam distillation methods for the determination of aquatic herbicides. Sigworth (10R) noted that coagulation studies with alum and iron salts indicate that these methods do not remove sufficient quantities of herbicides and pesticides from water. Activated carbon is suggested as a means of purification.

ORGANICS

Methods for the determination of organic materials in water are listed for four broad groups: acids, organic carbon, phenols, and miscellaneous.

Starikova and Yablokova (8S) determined amino acids in sea water by preparing chromatograms on No. 4 paper. Spots were cut out, extracted with methyl alcohol, and determined at 540 m μ . Error is $\pm 4\%$. Optimum amount of amino acid for the determination is 2.5 to 12.5 μ g. Maistrenko (60S) developed a method for the determination of α -amino acids in river waters. After a 0.5- to 1-liter sample was concentrated to 1 or 2 ml and treated with citrate buffer to a pH 2.5 to 4.0, 50 mg of ninhydrin and 3 drops of 10% isopropyl alcohol were added. This mixture was transferred to a Parnas apparatus with 10 ml of water and heated 10 minutes. Ten milliliters of 30% sodium hydroxide were added to the mixture and ammonia was distilled and determined. The

method was used with and without preliminary hydrolysis. In waste water analysis Deyl and Rosmus (20S) used centrifugal paper chromatography to determine aliphatic acids as sodium or ammonium salts, acid hydroxamates, and 2,4-dinitrobenzyl esters; phenols as arloxyacetic acids; and amino acids.

Verigo and Mamontova (39S) described a potentiometric method for determining 10 to 100 mg of free butyric acid per liter in waste water containing high quantities of sodium chloride and hydrochloric acid. Fifty to 200 ml of sample were neutralized with 20% sodium hydroxide, acidified with concentrated phosphoric acid, and the butyric acid was steam distilled into a 200-ml flask. The distillate was boiled 30 minutes under a reflux condenser, cooled to room temperature, and titrated with 0.01N potassium hydroxide potentiometrically using normal glass and standard calomel electrodes. Relative error is 5 to 10%.

Stradomskaya and Goncharova (83S) reported the use of chromatography on silica gel for separate determinations of butyric, propionic, and acetic acids in natural water. The procedure consists of adding the sample to a silica gel column, eluting with three column volumes of 1% alcohol in CHCl_3 , and adding thymol blue indicator dropwise to collected fractions. Butyric acid is eluted first, followed by propionic acid; 2% butyl alcohol in CHCl_3 elutes acetic acid. Recoveries are: butyric acid, 95 to 103%; propionic acid, 94 to 112%; and acetic acid, 116 to 118%. Lur'e, Alferova, and Bondareva (57S) used silica gel in the determination of low molecular weight fatty acids in waste waters. Silica gel was mixed with nearly its own weight in water and with benzene and placed in a column. Dry silica gel, 0.5 gram, was placed on top of this layer. The sample of waste water was neutralized to phenolphthalein, filtered, and evaporated to dryness on a water bath. The residue was dissolved in *N* sulfuric acid and then 1 ml of this solution containing 15 to 30 mg of acids placed at the top of the column. Kapronic and valeric acids were eluted with benzene, butyric acid with 2% butyl alcohol in benzene, propionic acid with 5% butyl alcohol-benzene, acetic acid with 20% butyl alcohol-benzene, and formic acid with 30% butyl alcohol-benzene. The effluents were divided into fractions of 2 ml, diluted with 2 ml of neutralized ethyl alcohol, and titrated with 0.02N alcoholic potassium hydroxide with phenolphthalein as indicator. Relative error was approximately 3.5%.

Several researchers described methods for determining humic acid in water. Obenaus (68S) determined humic acid by titration with potassium permanganate. Chalupa (15S) reported a procedure in which 250 ml of sample containing 5 ml of concentrated sulfuric acid

was shaken with 25 ml of amyl alcohol for 30 seconds. The extract was separated and then shaken for 10 seconds with 0.5% sodium hydroxide. Ten minutes later, the alkaline layer was filtered and its absorbance measured at 420 m μ .

Lamar and Goerlitz (54S) used gas, paper, and column chromatography and infrared spectrometry to study the characteristics of organic acids in naturally colored surface water samples. The acids were removed from the water by either continuous extraction with *n*-butanol or by vacuum evaporation at 50°. The study indicated the presence of 20 or more carboxylic acids, of which 13 were identified. Approximately 10% of the acids were volatile or could be made volatile for gas chromatographic analysis. Concentrations of the gas chromatographically volatile acids ranged from trace quantities to about 590 ppb. Paper chromatography indicated a mixture of many similar or related compounds but no distinct separations were obtained. Many fractions eluted by column chromatography provided similar infrared spectra. Infrared analysis of column chromatographic fractions indicated that hydroxy carboxylic acids were predominant and included olefinic, aromatic, hydroxy, and carboxylic groups.

Faehrich and Soukup (25S) reported on the ultraviolet absorbance characteristics of organic substances in some river waters. Most of these waters showed distinct minimum absorbance at 260 m μ and maximum values at 280 m μ . Equations are given for calculating the partial absorbance modules of lignin and humus in water samples.

Dement'eva and Vasilevskaya (18S) used gas-liquid chromatography to determine C_1 to C_8 fatty acids in water. Fluoroplast-4 powder treated with a solution of stearic acid and SKT (silicone) rubber in a mixture of ethyl alcohol and benzene was used as the packing material. An internal standard and correction factor are employed when the water content is high. The time of analyses was 25 to 30 minutes, error 0.1 to 1.0%.

Sedlacek (76S) modified Montgomery's colorimetric method for the determination of fatty acids. To a 2-ml sample of filtrate obtained by coagulation and filtration of sludge waters were added 5 ml of acidic ethylene glycol. After 10 minutes' heating on a water bath and cooling to room temperature, 2 ml of a 10% solution of hydroxylamine-hydrochloric acid and 8 ml of 4.5N sodium hydroxide were added. The solution was then mixed with 20 ml of ferric chloride solution and diluted to 100 ml. After 10 minutes, the absorbance of the solution was measured. The method is more precise and simple than the original method and that based on distillation.

Using a modification of the Barker and Summerson method for the determination of lactic acid, Barnes and Finlayson (5S) estimated concentrations of lactic acid in sea water solutions and homogenates. The modification consisted of removing bromide ion, which interferes with the determination of lactic acid in sea water, by precipitation as thallos bromide before proceeding with the Barker-Summerson method as indicated by Umbreit, Burris, and Stauffer (87S).

Dudova (22S) evaluated titrimetric and turbidimetric determinations of naphthenic acids in underground waters. Both methods are unsatisfactory because fatty, humic, and other organic acids are also included in the results. Bykova (11S) used the capillary-luminescent method to determine naphthenic acids in water. Naphthenic acids were extracted from 150 ml of analyzed water with 15 ml of chloroform at pH 3 and transferred to a paper strip 50×0.8 cm by a capillary lift of extraction accompanied by evaporation of solvent. The optimal concentration of organic substances corresponds to the zone of capillary extraction of less than 10 cm. The paper was transferred to a test tube containing 3 to 4 ml of nonluminescent sodium ester and then the same contacted end of the paper was immersed into the sodium ester, closed hermetically, and chromatographed for 25 to 30 hours. After the paper was dried in ultraviolet light, the boundaries of zones and the luminiscent color of naphthenic acids were determined. Contents of naphthenic acids were determined from a standard curve. The method is more convenient and sensitive than the turbidimetric method. Reproducibility is satisfactory.

Potentiometric determination of organic acids in natural waters was reported by several investigators. Semenov, Bryzgalo, and Datsko (77S) concentrated organic acids by passing 100 to 250 cc of water through a column containing either the cation exchanger KU-2 or the anion exchangers AN-22 and AV-17. The acids are eluted with 100 cc of 2N ammonium carbonate, the eluate is treated with 2 cc of sodium hydroxide, and the solution is evaporated to dryness on a water bath. The residue is dissolved in 0.5 cc of water, treated with thymol blue indicator, neutralized to pink using N hydrochloric acid, transferred into a centrifuge tube, diluted to 1 cc with water, and heated on a boiling water bath for 1 minute. The organic acids are centrifuged with 2 cc of ether and the extract is added to 0.5 cc of butyl alcohol; this solution is titrated potentiometrically with BuONa in butyl alcohol. Bykova (12S) used steam distillation followed by potentiometric titration with 0.01N barium hydroxide to determine organic acids in

underground waters. Corrections were introduced for the control and for the degree of mineralization of the water. Several organic acids were identified from the shape of the titration curve. Phenols evaporated with steam can be determined in an aliquot of the distillate. Bryzgalo and Semenov (10S) studied possible errors in the determination of volatile and nonvolatile organic acids in natural and contaminated waters. Volatile acids were distilled from a water sample acidified with phosphoric acid. The acids were concentrated using anion exchangers AV-17 or Dowex 1X4 and determined by potentiometric titration in nonaqueous media. The same sample was analyzed again to determine the total amount of volatile and nonvolatile acids. Distillation in a vacuum at 15° and cooling the vapor in liquid air prevented analysis errors.

Pohland and Dickson (72S) used column chromatography to determine volatile acids in waste water. The acids of a filtered sample of sludge are absorbed on 100-mesh silicic acid in a Gooch or fritted glass crucible. The acids are eluted with a mixture of chloroform, normal butyl alcohol, and sodium bisulfate solution. The quantity of eluted acids is determined by titration with 0.02N sodium hydroxide in absolute methyl alcohol. Recovery of the acids is $96.6 \pm 3.5\%$. Andrews, Thomas, and Pearson (2S) applied gas chromatography to the determination of volatile acids in sewage sludge. Goncharova and Stradomskaya (35S) reviewed 53 references on methods for the separate determination of organic acids in natural waters.

Several investigators reported determinations of organic carbon in water. Tuerkoelmez (85S) compared the results of the determination of organics by potassium permanganate and potassium dichromate consumption at different concentrations as a function of the boiling time, and by determining the carbon content by the wet incineration method and the persulfate process. Experiments were made using many organic chemical compounds and samples of industrial waste water. The wet incineration process gave the best results. The persulfate method is not suitable for industrial waste waters containing volatile organic components. An average of only 30 to 40% of the organics is determined by the potassium permanganate method, whereas 90 to 96% of the total organic matter content is determined by the potassium dichromate consumption. Kramig and Schaffer (51S) devised a technique for the rapid determination of organic pollution. A sample collected in a polluted stream is sent through a combustion tube with a flow of oxygen to convert all the carbon to carbon dioxide. The carbon dioxide is then determined by infrared spectrometry. Height of

peaks obtained is compared with peaks obtained with known amounts of organic materials. Depending upon sample size and instrument sensitivity, as little as 0.1 mg of carbon per liter of solution can be measured. Because total carbon is measured, inorganic carbon must be eliminated. Schaffer *et al.* (74S) found that the infrared carbon analyzer used to measure small concentrations of carbon in water could be adapted to the measurement of carbon in a variety of waste samples, particularly those taken at various stages throughout waste treatment processes. Measurements were obtained 15 minutes to 1 hour after sample pickup. Data clearly show correlation of total organic carbon measurements with BOD and chemical oxygen demand.

In a study of the potassium dichromate method for total organic material in water, Bolberitz and Hegyessy (8S) found that extending the reaction time significantly raised the characteristic decomposition of the dichromate; however, increasing the temperature to 190° caused virtually no decomposition. Of the catalysts, only silver sulfate was satisfactory.

Brodevskaya (9S) noted that in the preparation of underground water samples for the determination of organic carbon, large amounts of an acidic volatile component are lost. He found that this component can be retained by neutralizing the sample with borax before boiling it down.

Van Hall and Stenger (88S) developed a method for the rapid determination of total carbon in aqueous solutions in concentrations equal to or less than 2 mg per liter. The method involves combustion of a microsample in oxygen in a heated tube followed by measurement of the carbon dioxide produced. The latter is accomplished by the use of a nondispersive infrared analyzer sensitized specifically for carbon dioxide. The signal from the analyzer is recorded on a strip chart. Another combustion method for the determination of total carbon in water was reported by West (92S). As little as 0.004% by weight of carbon in a 10- μ l sample was determined. A rate of 10 to 12 samples per hour was achieved by the injection of a liquid sample from a syringe onto a hot copper oxide bed that was continuously swept by the carrier gas. The relative standard deviation was 3.1% at the 0.3 wt % carbon level and 8.2% at 0.004 wt % carbon level.

Organic carbon in sea water received the attention of several investigators. Oppenheimer, Corcoran, and Van Arman (69S) described a sensitive and simple method for the determination of particulate or total organic carbon by wet combustion, gas collection, and chromatographic analysis of the carbon dioxide and monoxide gases liberated. The method was calibrated with glucose,

was accurate to 1%, and had a lower limit of sensitivity of 0.05 mg of carbon per sample.

Two teams of investigators described similar methods for the determination of dissolved organic carbon in sea water. In both methods 5-ml samples were freed of inorganic carbonate and oxidation of the organic carbon was carried out in sealed glass ampuls. Fredericks and Hood (29S) flushed the resulting carbon dioxide gas through a gas chromatograph with helium as the carrier gas. The signal was recorded on a strip chart recorder. Chromatographic analysis time was approximately 11 minutes per sample with a precision of ± 0.1 mg of carbon per liter. The organic content of the sample was determined by measurement of the peak area using an appropriate carbon dioxide calibration curve. Menzel and Vaccaro (64S) passed the carbon dioxide gas through a nondispersive infrared analyzer with nitrogen as the carrier gas. The signal output was recorded. By using appropriate calibration curves, the carbon content can be determined from the height of the peak. Approximately 100 samples can be analyzed per day with a precision of ± 0.1 mg per liter.

A comparison method is also described for the determination of particulate carbon by high temperature combustion after concentration of the sample on a glass fiber filter. The precision of this method is ± 10 μ g of carbon in a range of 0 to 500 μ g of carbon. Approximately six samples can be analyzed in an hour. Szekiela and Kreg (84S) described an apparatus that permits the determination of particulate organically bound carbon in sea water within 5 to 6 minutes aboard ship. Sea water is passed through a glass filter and the dried filter and retained organic matter are burned in oxygen at 550° in a combustion train. The carbon dioxide is finally absorbed in 0.01N sodium hydroxide and titrated conductometrically. Calibration curves are made with known compounds.

Kreg and Szekiela (52S) used a special microscale apparatus to determine organic carbon in sea water. A 5-ml sample was added to the apparatus and oxidized by a hot solution of silver dichromate-potassium dichromate-sulfuric acid. The evolved gases were carried with oxygen through decomposed silver permanganate at 550° for complete oxidation to carbon dioxide, dried with magnesium perchlorate, freed of oxides of nitrogen by special manganese dioxide, and measured by the decrease in the conductance of 0.01N sodium hydroxide by using a recorder to follow the conductance. For 10 to 30 μ g of carbon the error is 0 to 2 μ g. A single analysis requires 20 minutes.

The determination of oil products in water is outlined in several papers.

Bykov and Lakeeva (18S) used a modified pycnometric method to determine oil concentrations in waste water. By this modified method, a sample of water is extracted three times with 20 ml of carbon tetrachloride; the extract is dried 1 hour with 10 grams of calcium chloride and finally filtered. After evaporation to 7 ml on a water bath, the extract is placed in a 10-ml pycnometer, diluted to 10 ml with carbon tetrachloride, and allowed to stand 20 to 30 minutes at $20^\circ \pm 1^\circ$. A formula is given to calculate the quantity of oil products. Nagibina and Ershova (66S) noted that determination of petroleum products in industrial water by extraction, evaporation of the solvent, and weighing the residue can give different results depending on whether ethylene oxide, petroleum ether, or benzene is used. Deviations were smallest where the effluents contained only petroleum distillates and nitrobenzene but were quite large with effluents from synthetic fatty acid and alkyl-phenol plants.

Fastabend (26S, 27S) described two methods for the determination of oil in water by infrared spectrometry. One paper gave a general method. The other reported that mineral oil contamination of ground and surface waters can be determined rapidly with adequate accuracy by taking a single extraction of 1 liter of water with 50 cc of carbon tetrachloride, taking the infrared spectrum, and measuring the absorbance of the CH bands between 3 and 3.5 μ . The mean error is about $\pm 9.2\%$ for 30 ppm.

Ladendorf (53S) discussed the disadvantages of determining the content of different oils in waste water by the Robert Taft Center-developed method and by a German standard method. A universally usable method based on the density of an extract of a waste water was developed. The accuracy and the reproducibility are better than those of the infrared method. A disadvantage is that concentrations less than 0.3 ppm cannot be determined. Giebler, Koppe, and Kempf (32S) described a gravimetric method for determination of oils and fatty materials in water. By this method, a mixture consisting of 10 ml of 10% complexon III solution, 500 ml of the water to be treated (vacuum-distillation residue of 1 to 100 mg), 50 grams of sodium chloride, and carbon tetrachloride equivalent to 1% of the water sample is shaken for 1 hour in a funnel wet on the inside with 10 ml of sodium hydroxide solution. The mixture is allowed to stand for 15 minutes and then the lower layer is transferred to a centrifuge tube. After centrifugation for 5 minutes at 3500 rpm, the lower phase is transferred quantitatively to a filter and vacuum distilled at 20° under definite time and pressure conditions. Accuracy of the method is 15% of the carbon tetrachloride-extractable material. The

same investigators (33S) described a thin-layer chromatographic determination of mineral oil in water. Chromatograms are developed on plates, dried 5 minutes in air, and then sprayed uniformly with 7 ml of 1% phosphomolybdic acid in *tert*-butyl alcohol. Fuel and lubricating oils give blue-gray spots. The amount of oil is estimated by comparison with chromatograms for known amounts of oil. The method permits determination of 0.02 mg of mineral oil per liter of water. Golubeva (34S) outlined a determination of petroleum and petroleum products in water. The water is acidified with sulfuric acid and saturated with sodium chloride and then extracted with diethyl ether. After 5 hours drying over sodium sulfate or calcium chloride, the diethyl ether is evaporated in a tared special flask on a 45° to 50° water bath. Colorimetric, nephelometric, or luminescence methods may be used to complete the analysis.

Several investigators reported chromatographic and instrumental determinations of phenols and related materials in water: Baker (4S) and Scholz (75S), gas chromatography; Goren-Strul, Kleijn, and Mostaert (36S), gas-liquid chromatography; Bidlo (7S), Koch (48S), and Lur'e and Nikolaeva (58S), paper chromatography; Dyatlovitskaya and Maktaz (23S), thin layer chromatography; Skrynnikova, Govorova, and Matveeva (80S), coulometric and colorimetric; Babkin and Spitsin (3S), Barskii and Noskov (6S), Chalupa and Dvorakova (16S), Csanady (17S), Goto, Kawahara, and Sato (37S), Herrig (41S), Parfenov, Shaginyan, and Ardzhanov (71S), and Zege, Shenderovich, and Stoma (100S), colorimetric. Fleszar (28S) described a method for the determination of nitrochlorobenzenes in water. A 2-liter sample is prepared by shaking with approximately 0.03 gram of activated carbon for 60 to 80 minutes. Then the sample is filtered under vacuum, the carbon is extracted with 15 ml of dimethyl ketone, kept at its boiling point for 90 minutes, cooled, and washed three times with a 1:1 dimethyl ketone-water mixture. Washings and extractions are combined with 5 ml of reagent solution (0.2M $C_5H_5N \cdot HCl$ in C_4H_9N mixed 2:3 with 7% barium chloride and diluted with water to 50 ml) are added. Five milliliters of solution are treated with nitrogen to remove oxygen and scrubbed with an alkaline solution of pyrogallol and dimethyl ketone; the polarograph curve is recorded from 0.2 to 0.9 volt at an applied potential of 4 volts vs. an internal mercury electrode. To the analyzed solution is added 0.5 ml of standard containing 0.02 gram of nitrochlorobenzene per liter in 1:1 dimethyl ketone-water mixture and the process is repeated.

Lewis (55S) described a continuous

phenol analyzer. The sample is converted to sodium phenolate, which is continuously decanted in a separatory funnel, filtered through $\frac{1}{8}$ -inch Raschig rings, and cleaned through glass wool. Hydrochloric acid is added to reform the phenols before the sample enters a 2.5-cm cell. A collector rod transmits the light from a hydrogen lamp to a chopper mirror which, with corner mirrors, alternately passes the light rays through the sample and reference cells. A relay lens forms the light on a photomultiplier tube. Chemical and interference filters absorb unwanted radiation. The difference in intensities striking the photomultiplier is proportional to the phenol content. Electronic adjustments automatically reset the amplifier to zero when periodically calibrating.

From a study to find a rapid and accurate method for the determination of pentachlorophenol in water, Uede, Nagai, and Osafune (86S) reported that Numata's 4-aminoantipyrine method and the colorimetric method following oxidation with nitric acid are the best methods to use. From his survey of the different methods to determine phenol and detergents in waste water, Malz (61S) reported that the photometric methods and the spectrometric methods using *p*-nitroaniline and 4-aminoantipyrine are the most convenient ones for phenols within the concentration range 0.001 to 100 ppm. For detergents, only the anionic ones that react with methylene blue can be determined accurately.

Melpolder *et al.* (63S) developed a method for the determination of non-ionic detergents in water and sewage; it involves passage through an ion exchange resin to remove any ionic material, passage through a carbon column (a little deactivated) to remove organic material, and foam fractionation. The method gives 80% recovery of nonionic material from river water, more than 90% recovery from sewage effluents, and between 86 and 100% recovery from crude sewage. However, the method is time-consuming and a day is required to complete a single determination.

Higashiura (42S) described a determination of para-substituted phenols in waste water. Phenols other than the para-substituted ones react with 4-aminoantipyrine to form dyes. Thus, a mixture of phenols was first reacted with this chemical and then passed through an ion exchange column containing the OH form of Dowex 1-X8. The formed dyes from the unsubstituted phenols pass through the column but the para-substituted phenols remain within. Elution with methyl alcohol plus normal sodium chloride (1:1) and methyl alcohol plus 1.7N acetic acid (1:1) removes the para substituted phenols which are diazotized with sulfanilic acid and determined photometrically at 495 m μ *p*-Cresol, 2,4-xyleneol, 3,4-xyleneol, and *p*-

ethylphenol were determined by this method.

Palaty and Deylova (70S) noted that when 4-aminoantipyrine and *p*-aminodimethylaniline are used in the determination of phenols in water, certain substances, if present, interfere. The substances include aniline, sodium sulfide, sodium cyanide, sodium sulfite, sodium thiosulfate, and ammonium thiocyanate. The investigators recommend distillation of an acidified sample with heavy metal salt.

Dozanska and Sikorowska (21S) compared the 4-aminoantipyrine and 2,6-dibromoquinone chlorimide methods for determination of phenols in water and sewage. Fujii and Shimamine (30S) applied the 4-aminoantipyrine method to the determination of phenol in drinking water.

Kondo and Murakami (49S) found that a mixed solvent containing 30 to 60% acetophenone in benzene gives greater than 95% extraction of phenols in water.

In addition to the methods for the determination of organic acids, organic carbon, and phenols, numerous methods were reported for a variety of organic materials. Nemtsova, Semenov, and Datsko (67S) described a spectrophotometric determination of volatile amines at 410 m μ . Kalalova (45S) reported a determination of hexamethylenetetramine in the presence of formaldehyde in waste waters. The total formaldehyde is determined in 10 ml of sample that is refluxed with 20 ml of 1% sulfuric acid at 60° for 30 minutes, cooled, and diluted to 50 to 100 ml. To a 10-ml. aliquot, 1 ml of concentrated hydrochloric acid and 5 ml of Schiff's base are added. After 1 hour the color is measured with a green filter. To determine hexamethylenetetramine, 10 ml of sample are refluxed with 2 ml of 1% sodium hydroxide and 0.2 ml of 1.2% hydrogen peroxide at 60° for 30 minutes to remove the free formaldehyde. The hexamethylenetetramine is then oxidized with 20 ml of 1% sulfuric acid for another 30 minutes and determined in concentrations 0 to 100 mg per liter.

Konenko and Yarysh (50S) outlined a determination of residual concentration of 3-(*p*-chlorophenyl)-1,1-dimethylurea in reservoir waters after their treatment with monuron. A 100- to 200-ml sample and 50 to 100 ml of 30% sodium hydroxide are boiled 1 hour under reflux, after which 80 to 85 ml are distilled into 5 ml of 6N hydrochloric acid and made up to 100 ml; 50 ml are treated with 1 ml of 1% sodium nitrite, 1 ml. of 10% sulfamic acid, and 1 ml of 2% *N*-(1-naphthyl)ethylenediaminehydrochloride. The violet color is measured spectrophotometrically. A standard solution of *p*-chloraniline is treated similarly.

Masek (62S) reviewed sources of carcinogenic polycyclic hydrocarbons in

waters and the methods of detection and separation of these substances.

Yurko and Volkova (95S) determined acrolein content of waste water. Filtered water is treated with ammonia to remove iron salts and then mixed with hydrochloric acid and a 96% alcohol solution; 19.5 ml solution is treated with 2 ml of a 0.2% tryptophane solution in hydrochloric acid at 45° to 50° for 15 to 20 minutes, and the determination is made colorimetrically.

Adamovsky (1S) described a benzene determination based on the desorption of benzene from waste water by nitrogen as carrier gas, which is then passed into a nitration mixture; the resulting *m*-dinitrobenzene is determined polarographically.

Shabunin and Lavrenchuk (79S) determined microamounts of chlortetracycline in waste water by a method based on the fluorescence of its decomposition products. A 20-ml sample is brought to pH 8.5 to 9 and diluted to 25 ml. Its fluorescence is then compared with that of a similarly prepared standard of approximately the same concentration in a LOF-54 fluorometer. Methyl alcohol, isopropyl alcohol, butyl alcohol, phenols, diethyl ether, dimethyl ketone, carbon tetrachloride, and cyanides do not interfere.

Zamyslova (97S-99S) described methods for determining specific substances in water. These included tetranitromethane, dimethylformamide, and hydroperoxide of isopropylbenzene.

Klinger (47S) noted that a disadvantage of the amperometric method for the determination of collectors in the flotation of pulp and organic substances in waste water due to anodic oxidation and deposition of butylxanthate and xylenol on graphite electrodes can be minimized by mechanical cleaning of electrodes with nylon fiber brushing devices or by removing the oxidized products from electrodes through periodic alternating polarization of electrodes. A concentration of 10^{-4} to $10^{-6}M$ organic materials in pulps can be determine amperometrically with these cleaned electrodes.

DePablo (19S) determined total glycoluril in swimming pool water. The determination includes the addition of EDTA and sodium arsenite solutions followed by a mixed reagent consisting of sodium hydroxide, potassium ferricyanide, and sodium nitroprusside. After 20 minutes, the absorbance is determined at 540 m μ . Time, reagent preparation, and additions are critical. The precision is less than 0.1 mg per liter at the 8 mg glycoluril per liter level and less than 0.2 mg per liter at 26 mg of glycoluril.

Vorob'eva (90S) determined hexadecanol in surface water. The material is extracted in benzene; the extract is made up to 15 ml, dried with 1 gram of sodium sulfate, and a 3-ml aliquot is

mixed with 1 ml of specially prepared benzene solution. After 20 minutes at 65° to 70°, 1 ml of 1*N* sodium hydroxide is added, the mixture shaken 1 to 2 minutes, and the color of the benzene layer after separation and drying with sodium sulfate is compared with that of standards by using a blue filter in the colorimeter. Accuracy with 0.06 to 1 mg of hexadecanol per liter is $\pm 6.4\%$ of the determined amount.

To determine hydrazine in waters containing organic matter, Kalinina (46*S*) added a few crystals of sodium azide to remove nitrite ions from the sample and a few drops of 2*N* hydrochloric acid to neutralize the sample. The sample is then mixed and two 50-ml aliquots are taken for analysis. Iodine is added dropwise to one, and the solution reduced again with 0.1*N* sodium sulfite to remove the hydrazine. After 0.8 ml of 10% *p*-dimethylaminobenzaldehyde in 2*N* hydrochloric acid is added to both aliquots, which are kept for 30 minutes in the dark, the light absorption of the two aliquots is measured by using a blue filter and the first aliquot as a blank.

Lur'e and Nikolaeva (59*S*) described a determination of small amounts of hydroquinone in solutions and waste water. To 5 ml of sample is added 1 ml of acetate buffer, pH = 5.75; 1 ml 0.004*N* ferric chloride containing 1.5 ml of hydrochloric acid per liter; and 0.5 ml of 0.5% 1,10-phenanthroline containing 2 ml of hydrochloric acid per liter. This is diluted to 20 ml with water. The absorption is determined at 508 $m\mu$ against a blank after standing 1 hour. If the water contains resorcinol and pyrocatechol, the sample is treated with lead acetate whose pH is adjusted to 5.75 to 5.85 with 0.1*N* sodium hydroxide 10 minutes later, and then 0.25 ml of freshly prepared 2% sulfite ion is added. The determination is continued as above but the absorbance is measured only after 30 minutes' standing.

Zadera (96*S*) outlined a colorimetric method for the determination of lignin substances in waste waters. To 100 ml of sample, 4 ml of 10% acetic acid are added and the absorbance is determined; 2 ml. of fresh 10% sodium nitrite are then added, and after 20 to 120 minutes the absorbance is determined. The precision of the method is 10%.

Loehr and Higgins (86*S*) reported that the wet extraction of lipids in waste water, sewage sludge, and effluents is more efficient than the Soxhlet method where the filter does not retain all the insoluble lipids of the water. A series of 10 samples can be determined in 4 hours. The wet extraction method can extract motor oil, tristearin, and sodium oleate up to concentrations of 6500, 7400, and 9200 mg per liter, respectively, with the same efficiency as at lower concentrations.

Yazhenskaya (93*S*) reported that

naphthalene in water can be determined by extracting with heptane and measuring the absorbance of the extract at 220 $m\mu$; or air can be passed through a 200-ml sample on a boiling water bath and then through two absorbers containing ethyl alcohol and the absorbance of the ethyl alcohol at 220 $m\mu$ can be compared to that of similar solutions prepared using water containing known amounts of naphthalene.

Hegi, Fischer, and Maerki (40*S*) described a chromatographic method to determine 10 μ g or more of nicotine in water. Details of the procedure are given.

Ryckman, Burbank, and Edgerley (73*S*) outlined advantages of new collection, concentration, and characterization procedures for the evaluation of organic pollutants. Myrick and Ryckman (65*S*) designed and used a technique to obtain significant quantities of organic refractories from a large volume of highly turbid natural water. Sedimentation and diatomite filtration were used as pretreatment processes. Activated carbon adsorption filters in series were used under both basic and acidic conditions to concentrate these dissolved agents.

Yurko and Volkova (94*S*) developed a colorimetric method for the determination of phthalic anhydride in waste water. Hopkins and Head (43*S*) reported that concentrations as low as 0.01 ppm of pyrethins in public water supplies may be extracted with light petroleum ether and determined either spectrophotometrically or by gas-liquid chromatography. The spectrophotometric method may be used quantitatively over a range of 7 to 100 μ g. The gas-liquid chromatography method gives good response down to 0.1 μ g of total pyrethins.

Gavrilescu (31*S*) reported that various experimental data show that glucide substances occur in both fresh and salt water. These substances are a result of algae and phytoplankton. The results verify the validity of the Puettier theory. Ivleva, Semenov, and Datsko (44*S*) used *p*-aminohippuric acid and a spectrophotometer to determine reducing sugars in natural water. The average error is 10 to 15% and the sensitivity of the determination is 20 μ g of sugars. Semenov, Ivleva, and Datsko (78*S*) found that the optimum condition for hydrolyses of reducing sugars in natural waters preparatory to their determination is heating the sample for 12 hours at 100° in 1*N* sulfuric acid.

Sokolov (81*S*) noted that when waste water is acidified to pH of less than 2, an acidic, water-insoluble styromal forms; thus, the formed precipitate is stabilized by a gelatin solution and the styromal is determined nephelometrically.

Carlucci and Silbernagel (14*S*) described a method using carbon-14 uptake

by the diatom *Cyclotella nana* for the bioassay of vitamin B₁₂ in sea water.

Gottauf (38*S*) outlined an improved heat-space technique for gas chromatographic determinations of 2-pentanone, hexanal, isoamyl alcohol, limonene, hexyl acetate, PrSSPr, and Me₃N. The lower limit of detection is $<10^{-8}\%$ with a relative standard deviation of ± 0.05 .

Weiss, Johnson, and Kwon (91*S*) reported that organic components of natural waters can be chromatographed on suitable liquid phase columns (poly *m*-phenylene ether, silicone, polyethylene glycol) with helium as a carrier and at an optimal temperature of the column of 150°. Use of a hydrogen flame detector established that 0.1- μ l samples were sufficient for routine analyses.

Harder and Holden (39*S*) described two methods for the determination of methane gas in ground water.

Ettinger (24*S*) reviewed developments in the detection of trace organic contaminants.

RADIOACTIVITY AND ISOTOPIC ANALYSIS

Loveridge, Gordon, and Weaver (46*T*) determined actinium-227 in effluent by coprecipitation with ferric hydroxide. If algae are present, the precipitate is dissolved in acid and oxidized with nitric acid and perchloric acid before adjusting the acidity to pH 1.6 and extracting the actinium with bis(2-ethylhexyl)phosphoric acid in heptane. The actinium is back extracted into 1.5*N* hydrochloric acid, and the solution passed through an anion exchange column to remove traces of lead, bismuth, and polonium. The eluate containing actinium is evaporated. After drying overnight and allowing any actinium-228 to decay, the residue is dissolved in dilute acid and fed into a reverse phase partition chromatography column to separate actinium from rare earths and from its own daughters. The eluate is evaporated and the residue dissolved in dilute acid and counted. Radiochemical recoveries range from 80 to 95%.

Tyrell, Demetopoulos, and Collinson (72*T*) used a nuclear emulsion to determine the alpha activity of drinking water. Solutions were concentrated by evaporation and adsorbed on 50-micron nuclear emulsions. After suitable exposure and development, the emulsions were scanned for alpha tracks.

Kotlyarov and Khitrov (41*T*) described a 4- π counter for measuring beta activity in sea-going laboratories. The performance of the instrument was tested by measuring strontium-90 and cerium-144 in ocean water and beryllium at the ocean's bottom. Characteristics of the counter include a background of 1.5 to 2.6 cpm and an efficiency of 60 to 80%. Other methods for determining beta activity in water were described by Haberer (29*T*), Yakimov (77*T*), and

Henke (33*T*). The latter two investigators also described methods for measuring alpha and gamma activity. Malvicini (48*T*) reported that total alpha and beta activity can be measured directly and continuously on a flowing stream by a gas-filled proportional counter connected to a recorder. Gamma activity is measured with a scintillation counter.

Crosby and Chatters (17*T*) advocated the use of anion-exchange resins for stripping water of carbon dioxide for carbon-14 determination. Equipment that uses anion exchangers and that can be adapted to pump-site sampling, in-well sampling, or sampling of surface waters was developed. Analytical results, by means of which resin techniques and standard methods are compared, proved to be within the normal errors expected in the carbon-14 method.

Methods for the determination of radioactive cesium in rainwater include one by Fonseca *et al.* (22*T*) that uses ammonium molybdophosphate as an ion exchanger; one by Derecki, Geisler, and Jaworowski (18*T*) that is a modification of the preceding method; one by Benes (9*T*) that counts the beta activity of cesium-137 dipicrylamine; and a method by Johnson *et al.* (36*T*). Other determinations of cesium-137 include one by Shipman and Mueller (65*T*) that uses permanganate as a precipitant; one by Folsom, Pillai, and Finnin (21*T*) that uses the pulse height spectrum of the internal conversion electrons acting upon detectors to determine cesium-137 in sea water; and one by Boni (13*T*) that uses $K_2CoFe(CN)_6$ as the ion exchanger to absorb cesium from neutral, concentrated hydrochloric acid, concentrated nitric acid, and highly salted (sodium nitrate) sodium hydroxide solutions.

In a determination of radiocerium in biological matter and sea water by Hampson (32*T*), cerium-141 and cerium-144 are coprecipitated with iron or titanium (as hydroxides) and are purified by solvent extraction with bis(2-ethylhexyl) hydrogen phosphate in *n*-heptane. Cerium-144 is measured by counting the beta irradiation from the praseodymium-144 daughter; aluminum sheet is used to absorb the beta irradiation from cerium-141.

Hahn and Marsh (30*T*) described a determination of cobalt-60 that involves stripping cobalt from an organic layer and gamma counting it in a deep well-type scintillation counter. For exchangeable cobalt in sea water, Viswanathan *et al.* (74*T*) recommended coprecipitation and determination by activation analysis.

In a determination of iodine-131, Brutovsky *et al.* (15*T*) extracted the iodine in carbon tetrachloride, precipitated the iodide as silver iodide, and dissolved the precipitate in sodium cyanide.

As little as 10^{-11} curies of iodine-131 per liter can be determined by this method. Fairman and Sedlet (20*T*) determined iodine-131 in milk, water, and other materials by ion exchange on silver chloride.

Kautsky and Schmitt (33*T*) described a determination of iron-55 and iron-59 in sea waters. Iron along with added carrier elements (inactive iron, manganese, zirconium, zinc, cobalt, cerium, and yttrium) was precipitated at pH 10.2 from acidified sea water samples by the addition of sodium hydroxide. Then, through a series of precipitations, iron was separated and fired to the magnetic iron oxide form. As little as 0.45×10^{-12} curies of iron-55 per liter could be determined by radiation measurement of the magnetic iron oxide.

Tamas and Opauszky (71*T*) used the reactions of equilibrium between water and carbon dioxide at room temperature and at 900° C to determine the oxygen-18 content of small water samples. The method is more reproducible at room temperature than at 900° C but its long time of exchange (24 hours) is a disadvantage. A mass spectrometer was used to analyze the isotopes. Vetshtein (73*T*) also used mass spectrometric analysis to determine isotope oxygen composition of water. The method consists of the determination of oxygen evolved from water by an improved persulfate method instead of carbon dioxide used in the old method. The method gives reproducible results with an accuracy of $\pm 0.02\%$.

Hahn and Ross (31*T*) described a method for the determination of radioactive phosphorus in water. Phosphorus is concentrated with aluminum hydroxide, the precipitate dissolved in perchloric acid, and a manganese dioxide scavenging step performed to remove radioniobium. Phosphorus is then separated from other activities by extraction of the phosphomolybdate complex into a butanol-chloroform mixture, back extracted into ammonia and precipitated as magnesium ammonium phosphate, which is weighed and beta-counted.

Rushing (61*T*) applied the coprecipitation of polonium-210 with tellurium by stannous chloride as precipitant to the determination of polonium-210 in water. The doubly precipitated tellurium-210 polonium precipitate is alpha-counted on 0.22-micron membrane filters. A shortcut single precipitation method is also given. The methods have sufficient sensitivity to permit their use in the determination of lead-210 in water by polonium-210 ingrowth.

Determinations of radium in water were reported by three different teams of investigators. Barker and Johnson (5*T*) determined radium by coprecipitating it with barium sulfate and comparing the alpha activity of the precipitate to a standard radium solution.

Polynkova, Titova, and Novikov (56*T*) mixed saturated calcium chloride solution and aqueous solutions containing 0.48 gram radium per liter at various pH's and added sodium carbonate until calcium carbonate began to precipitate. Carbon dioxide is removed by a stream of air, and then activated carbon and sodium carbonate are added to precipitate calcium carbonate and radium. Optimum pH of precipitation for various activated carbon materials is given. In another determination of radium using activated carbon, Drozhzhin, Lazarev, and Nikolaev (19*T*) described a method for determining radium without advance chemical separation. A sample of water is acidified, air bubbled through it to remove any radon present, and then the container is sealed. The sample is allowed to age and the accumulated radon is adsorbed on activated charcoal by bubbling nitrogen through the sample. The radon is finally desorbed from the charcoal and run through an ionization or scintillator counter. The initial radium concentration of the water is then calculated. Radium concentrations ranging from 10^{-13} to 10^{-11} gram per liter can be determined by the method.

Spirin, Belle, and Chimina (67*T*) described a method of measuring radon content in water as well as the decomposition products radium B and radium C that are in equilibrium with radon. The measuring system consisting of 20 Geiger-Mueller counters is able to measure samples of a volume down to 0.5 liter. The sensitivity is 1.3×10^{-10} curies.

Skonieczny (66*T*) reported methods for the determination of radioactive ruthenium and niobium in water and sewage. Iwashima (34*T*) reviewed 230 references on the separation and determination of radioactive ruthenium in natural water.

Basu (7*T*) described a method for the estimation of sulfur-35 in sea water. Sulfur is oxidized by boiling the sample with hydrogen peroxide in ammoniacal solution. Through a series of precipitations purified barium sulfate is formed which is then beta-counted to determine the concentration of sulfur-35. Sensitivity of the method is $0.005 \mu\mu\text{C}$ sulfur-35 per milliliter of sea water.

Several methods of determining strontium-90 in natural waters were reported. In a method for determining strontium in sea water, described by Azhazha (4*T*), strontium is separated from calcium through precipitation with sodium carbonate in the presence of disodium EDTA and nitric acid, which removes the calcium by dissolving calcium nitrate only.

Petit and Kienberger (55*T*) determined strontium-90 in drinking water at levels equal or greater than 5 dpm per ml by adding strontium carrier, evaporating the sample to dryness, and leaching the

residue with 1*N* hydrochloric acid. The leachate was scavenged with ferric hydroxide and strontium oxalate precipitated in solution of ammonium oxalate and ammonium chloride. The precipitate was metathesized to strontium nitrate in concentrated nitric acid, dissolved in water, and reprecipitated. Finally strontium oxalate was precipitated and counted in a proportional counter. An absorption curve was run to correct for yttrium-90 and strontium-90.

Ralkova (58*T*) described a rapid control method for the determination of strontium-90 in drinking water. The method is based on the coprecipitation of trivalent cations, including yttrium-90, with ferric hydroxide. Strontium-90 remains in the filtrate assisted by calcium carrier. According to Aksel'rod (2*T*), the determination of strontium-90 background activity in water basins requires concentration of at least 100 liters of water. Concentrated hydrochloric acid added directly to the water sample aids desorption of strontium-90 from suspended solids and prevents its adsorption on the container walls. To determine strontium-90 in river waters, strontium-90 and calcium must be separated with nitric acid. Before the nitrate separation of strontium and calcium, the trivalent ions of iron and aluminum must be separated by precipitation with ammonium hydroxide.

Molt (51*T*) reported that the nitric acid method for determining strontium-90 in water is improved by separating strontium as the nitrate from calcium nitrate. This is accomplished by boiling the nitrate precipitate in anhydrous amyl alcohol in which the soluble calcium nitrate is separated from the insoluble strontium nitrate.

Jeanmaire, Patti, and Bullier (35*T*) and Talvitie and Demint (70*T*) used ion exchange and precipitation techniques to determine strontium-90 in water. Sereda and Bobovinkova (63*T*) suggested a simple method for large-scale observation of the strontium-90 content of fresh water reservoirs. Addition of ammonium carbonate and ammonium hydroxide to the sample precipitate calcium salts. The method permits the precipitation of carbonates, while uranium and potassium remain in the settling basin. Strontium-89, strontium-90, cerium-141, zirconium-95, niobium-95, barium-140, etc. are precipitated with calcium carbonates. Precipitation is followed by the ordinary radiochemical process of dissolution in acid and rare earth separation on iron hydroxide. Yttrium daughter products are precipitated later as yttrium oxalate. Relative error in strontium separation from yttrium is $\pm 5\%$.

A more improved method permits the precipitation of strontium and calcium directly upon separation from other

radioactive impurities and measurement of the beta spectrum of the carbonate precipitate after 2 to 15 days. The accumulated yttrium-90 verifies strontium-90 content. Shipman (64*T*) determined strontium-90 in sea water after concentration by manganese dioxide.

Several methods for determining strontium-90 in water, but along with cesium-137, were reported. Golutvina *et al.* (25*T*) noted that in the electro-dialysis of natural water in a glass electro-dialyzer with a platinum mesh anode and a platinum mesh cathode contained within a cellophane membrane cell concentrating strontium-89, strontium-90, and cesium-137 in the cathode cell was very slow; much faster concentration was obtained when a circular platinum wire anode surrounding the cathode cell was used. Determination of strontium-90 and cesium-137 in rain water was outlined by Govaerts, Guillaume, and Walch (26*T*). A liter of sample, to which strontium nitrate and cesium nitrate carriers have been added, is acidified, filtered, and concentrated to 100 ml. Two 40-ml aliquots are removed. Strontium sulfate is precipitated from one, and cesium cobalt nitrite from the other. The precipitates are counted automatically at 10-minute intervals for 5 hours in a thin window 2π Geiger counter.

Kautsky (37*T*) reviewed work to 1961 on the checking of radioactivity in sea water and described methods that he used for the determination of strontium-90 and cesium-137 in sea water. Strontium was precipitated as carbonate and freed from calcium by nitric acid separation. After 14 days, strontium-90 was determined by separation and measurement of the yttrium-90 formed. After carbonate and potassium cobaltate precipitation of cesium and its subsequent conversion to iodobismuthate, the cesium-137 was determined by measurement of its beta radiation. Patin (54*T*) reviewed 26 references on the determination of strontium-90 and cesium-137 content of sea water.

Aksel'rod and Lyalikov (3*T*) described a photometric determination of thorium in natural waters using Arsenazo III. Details of the procedure are given. The complex is measured at 665 $m\mu$ using a red filter. Extraction of thorium from dilute solutions with gelatin tannate was described by Keshishyan, Andreev, and Danilov (39*T*). Addition of 1% tannin to a solution containing 1,500 μg thorium per liter did not result in precipitation between pH 1 and 12; however, addition of 1% gelatin to the same solution precipitated a gelatin-tannate complex that occluded different amounts of thorium. The maximum amount of thorium precipitated occurred at pH 3 to 4.

Determination of thorium in tap water and sea water was reported by

Akaishi (1*T*). Thorium was precipitated with sodium hydroxide at pH 9 in the presence of calcium and phosphoric acid. The precipitate was dissolved in 1*N* nitric acid and ethyl alcohol and then passed through an anion exchange column to separate calcium, magnesium, and phosphate ions. Thorium was eluted with 8*N* hydrochloric acid and determined by Neothorin spectrophotometry. The method has a sensitivity of 0.5 μg of thorium.

Several investigators reported determinations of tritium in natural waters. Bibron (11*T*) described two methods that use a low-background coincidence counter. In the first method, tritium is concentrated by electrolysis in aqueous form and then introduced into a water-miscible scintillation medium. In the second method, tritium in the sample is converted to benzene, which serves as the scintillation solvent for an organic scintillation medium. Lal and Athavale (44*T*) used a nonelectrolytic enrichment method that permits an accurate determination of tritium/hydrogen ratios exceeding 10^{-17} . A factor of 20 enrichment suffices to measure up to 25 years of "age" levels of tritium in ground waters.

Modes and Koch (50*T*) used a proportional counter to determine the absolute activity of tritiated water. Tritiated water was reduced to $^3\text{H}_2$ in a sealed bomb, and the $^3\text{H}_2$ was carried with propane into a cylindrical counting tube. Specific activity values were 1% higher than those obtained microcalorimetrically and 2% lower than those from a steam ionization chamber. Nishiwaki and Kawai (52*T*) outlined a routine method of monitoring tritium in natural waters. The tritium concentration of samples is enriched electrolytically from a 6-liter volume to a final volume of 5 ml or less. The radioactivity of the enriched tritium is measured in a liquid scintillation counter.

Takahashi (69*T*) described a measurement of tritium at natural levels in water. The distilled sample and sodium hydroxide are put into an electrolytic cell containing nickel and stainless steel electrodes. After the volume has been reduced to one twelfth of its initial size, the tritium concentration is 8 to 10 times as great as that in the initial volume. Hydrogen is then generated by exposing magnesium chips to the sample vapor at 570° to 600° C and the counting tube filled. Zel'venskii *et al.* (80*T*) showed that the maximum degree of enrichment in tritium and oxygen-18 is obtained under total reflux at an average column temperature of 52.5° C and a pressure of 100 mm of mercury (in the upper section).

Determinations of deuterium and heavy water in natural water were reported by several investigators. Bastick, Baverez, and Castagne (6*T*) sepa-

rated H_2 -HD-D₂ gases formed by the reduction of water-heavy water mixtures with iron by passing them through a chromatographic column packed with aluminum oxide impregnated with ferric chloride as the stationary phase and using helium as the carrier gas at the temperature of liquid nitrogen. The maximum error of determination was ± 0.05 .

Applications of infrared spectrometry to the measurement of deuterium and heavy water were described by Bayly, Stevens, and Thurston (87); Bosselaar (147); Fydelor and Rawson (237); and Knezevic (407). Lazzarini (457) reported good agreement between heavy water concentrations determined by means of a vapor pressure osmometer and calculated values. Gandara, Gispert, and Rojas (247) studied relationship between heavy water concentrations and the variation in density vs. temperature of water.

Techniques for the determination of fallout activities in natural waters were discussed by several investigators. After concentrating radioactive species by electrodeposition, Richter and Wright (597) used a nonselective stripping agent and standard chemical separation procedures to determine thallium, cadmium, ruthenium, silver, cesium, selenium, tantalum, strontium, and cadmium plus ruthenium. Wallace (757) outlined separation and purification procedures for strontium, calcium, barium, lead, polonium, and beryllium, and radiometric methods for strontium-89, strontium-90, cesium-137, cerium-141, cerium-144, barium-140, and the naturally occurring lead-210, polonium-210, and beryllium 7. Yamaoka *et al.* (787) used both the ion exchange method and gamma ray spectrometry to determine radionuclides in rain water and also investigated methods for the determination of small amounts of uranium-237 and uranium-238. Wood and Richards (767) modified and extended a sequential analysis scheme for the radiochemical determination of fallout nuclides in drinking water and rain water. In the same sample they were able to determine manganese-54, yttrium-91, promethium-147, and zirconium-95 as well as the more commonly determined barium, cesium, cerium, and strontium fallout nuclides. Malvicini (477) reviewed the methods of control and measurement of radioactivity in waste waters and presented detailed determinations of strontium and cesium.

Rovinskii (607) derived an equation that permits the calculation of the concentration of a radioactive contaminant in the water of stationary reservoirs at any moment after the introduction of the contaminant. Its validity was verified experimentally in lakes having a surface of 4.5 and 11.3 km². Lai and Goya (437) prepared a compendium of

radiochemical procedures for the following fission-product nuclides: cesium-137, strontium-89, strontium-90, yttrium-91, cerium-141, cerium-144, barium-140, zirconium-95, ruthenium-103, ruthenium-106, molybdenum-99, and iodine-131. All procedures use either solvent extraction, precipitation, or ion exchange chromatography.

Methods for the determination of low levels of radioactivity were presented by several investigators. Kranz and Weise (427) used a proportional counter to measure low levels of radioactivity in river waters. In a continuous measurement of low levels of activity in water, Quirk (577) used a stream of hot air to evaporate a fine spray of water. The resulting solids were collected on a filter, positioned under a detector, and counted. According to Grune *et al.* (277), factors that must be considered in selecting an ion exchange resin for concentrating low-level concentrations of nuclides include ionic radius, activity of ion, valence, hydration of resin and ion, diffusion rate through the resin, flow rate through the column, and temperature. Resin cross-linkage, mesh size, capacity for exchange, and stability are also important.

Grune *et al.* (287) also described a method of wet ashing ion exchange resins by the use of concentrated perchloric, nitric, and sulfuric acids, followed by measurement of activity of the ashed resins. A dry-ashing procedure was studied and found to be less practical than direct dry ashing in a muffle furnace. Elution was considered impractical as a rapid radio-assay technique. Schonfeld, Kibbey, and Davis (627) tested γ -ray spectrum resolution for use in studies of processes concerned with the decontamination of waste water containing only 10^2 to 10^5 $\mu\mu\text{C}$ of radionuclides per liter. Tests with two-multichannel spectrometers and associated data analyses by the method of least squares were made with solutions of cobalt-60, strontium-85, cerium-141, and cerium-144 added as tracers to water containing strontium-90, zirconium-95, niobium-95, ruthenium-106, cesium-134, and cesium-137. Some of the solutions were concentrated by evaporation by factors up to 20 to evaluate this feature for improving accuracy of analyses. For comparison, the solutions were also analyzed by standard radiochemical methods.

Agreement between the two methods was excellent for cobalt-60 and strontium-85 concentrations down to about 500 $\mu\mu\text{C}$ per liter (scatter of ± 5 to $\pm 20\%$). Agreement between the two methods was good for cesium-137 at activities down to 3,000 to 4,000 $\mu\mu\text{C}$ per liter; comparative values from the two methods differed by factors of 5 to 10 for cerium-144, except at activities above 5,000 $\mu\mu\text{C}$ per liter. More dilute solutions were successfully

analyzed only when first concentrated by evaporation. Because of large statistical fluctuations at ruthenium-106 concentrations as high as 1,000 to 2,000 $\mu\mu\text{C}$ per liter, no particular significance can be given to the data for this nuclide.

Stefanov *et al.* (687) removed uranium, thorium, and radium from solution by adsorption on KU-2 resin at pH 4 to 6. Uranium can be separated from thorium and radium by extraction of the resin with 3.6% oxalic acid; thorium can be removed by treatment of the resin with 5% oxalic acid; and thorium and radium can be removed by treatment of the resin with 20% hydrochloric acid or acetic acid.

Minarik (497) showed that γ -spectrometry is suitable for the identification of some natural isotopes in surface waters. Yule (797) used a sodium iodide (thallium) detector to find limits of determination of 72 elements for whole blood, urine, milk, tap water, distilled demineralized water, and polyethylene vials. Craft, Erb, and Hill (167) outlined detailed procedures for chemical separation prior to counting of strontium, barium, yttrium, cesium, iodine, total rare earth activity, cerium, ruthenium, zirconium, and niobium.

Blaga, Blaga, and Chifu (127) devised a variant of the droplet method for determining the isotopic composition of water. A micropipet was incorporated into the apparatus so that the drop volume was determined directly. A mechanical control system allowed very fine adjustment of the water level in the micropipet capillary. The micropipet is easily removed and washed under the best conditions. Pascalau, Blaga, and Blaga (537) described an apparatus that automatically records falling time in the isotopic analysis of water by the falling drop method. Benes, Drasil, and Petrlik (107) described a nuclear emulsion for use in the autoradiographic determination of low alpha activities in water.

MISCELLANEOUS

Marten (367) described automatic colorimetric, Kjeldahl digestion, turbidimetric, solvent extraction, and distillation techniques. Using activation analysis, Grimanis *et al.* (237) determined concentrations of silver, gold, arsenic, barium, bromide, chloride, copper, iodide, potassium, manganese, sodium, rhodium, vanadium, and zinc in lakes; Ikeda, Kondo, and Yamashita (287) determined the concentrations of the rare earth elements in hot spring waters; and Wyttenbach (587) determined concentrations of barium, nickel, copper, uranium, rubidium, and cesium in ground-, river-, and mineral waters.

Various investigators reported analytical methods for determination of

alkalinity and pH of water. Herce and Luppi (26U) reviewed methods for determination of alkalinity. Smith and Hood (50U) recommended a sea water buffer, tris(hydroxymethyl)aminomethane, having a dissociation constant of 1.202×10^{-6} , as a replacement for distilled water buffers to eliminate drift in pH measurements. Temperature effects were determined to be ± 0.005 pH units for $\pm 0.17^\circ$ errors. Gauna (22U) reported that the Berl-Lunge-D'Ans method for the acidimetric determination of calcium bicarbonate in presence of sodium bicarbonate in natural waters containing calcium and magnesium chlorides and sulfates is valid only for waters in which other salts of alkaline earth metals are not present.

In analysis of carbonate brines containing carbon dioxide in the range 0.5 to 9.0% by potentiometric titration and by manometric measurements of total carbon dioxide evolved upon acid treatment, Rettig and Jones (43U) found that a plot of the potentiometric data showed inflection points indicating that the carbonate and bicarbonate end points could differ significantly from the pH values of 8.2 and 4.5 used in routine water analysis. Zavodnov (60U) presented equations for use in the calculation of carbon dioxide and pH of underground waters from the carbonate equilibrium. Results obtained by this method compare favorably with experimental ones. According to Barker (4U), modern high-pressure boilers have a phosphate concentration between 10 and 25 ppm, which puts the theoretical equivalence point in the range pH 5.4 to 5.6.

Three synthetic phosphate solutions, containing a known amount of sodium hydroxide were titrated to a pH of 5.4 and also 4.5. The titration to pH 5.4 gave the nominal phosphate concentration whereas the results at pH 4.5 were too high. Wims, Van Haute, and Rand (57U) reported a simple method for calculating adjustments needed to obtain equilibrium of water. The method involves solving two equations given in two unknowns and assumes that the only basic ions present are bicarbonate, carbonate, and hydroxide.

Beisova, Solomin, and Fesenko (5U) compared sodium hydroxide titration of aluminum- and iron-containing waters with potentiometric titrations. The results of the sodium hydroxide titration are 0.3 to 6.2% high compared with the potentiometric titrations, and they resemble more nearly the sum of aluminum plus ferric iron plus hydrogen ions.

Several investigators described apparatus used in the determinations of chemical and physical characteristics of water. Shvaishtein (46U) proposed

an apparatus for measuring sea ice salinity where the precision need not exceed 0.1 to 0.05% with the use of the transistorized salinometer CM-6M. It consists of a high frequency transistorized voltage converter and a measuring scheme consisting of a microamperometer, rectifier, voltage divider, and filter. The detector of the instrument is a plastic vessel with two graphite electrodes in its walls. The measurement is based on the principle of the determination of electrolytic conductance of solutions. Konovalov and Semenov (30U) described an apparatus that reduces the time of extraction of trace elements and permits the use of greater volumes of water. Research and development work in progress on a portable self-contained water quality monitor was discussed by Testerman *et al.* (52U).

Cozzi (14U) designed a flexible, portable water analysis apparatus for laboratory and field use. Provision is made for the determination of the redox potential, pH, temperature, and conductance; for polarometric and amperometric measurements, including the determination of oxygen; and for colorimetric, turbidimetric, fluorometric, and nephelometric measurements. Natural and industrial waters and aqueous solutions of inorganic and organic substances can be analyzed. Mobile laboratories for analysis of water were described by Brondi *et al.* (11U) and by Haesselbarth and Langer (24U).

In geochemical studies, Larionov, Alekseeva, and Rivman (34U) proved the efficiency of using various analytical methods for analysis of ground waters. For waters having a mineral content less than 0.5 gram per liter, they recommended coprecipitation of metals with cadmium sulfide in addition to chemical and spectral analysis of dry residue. For samples having a mineral content greater than 5 grams per liter, they recommended chemical analysis.

Allemand (2U) applied the laser technique to emission spectroscopy. A sample is placed on the stage of a microscope and is brought visually into focus. Then the microscope is used to focus a laser beam onto the sample. The luminous flux from the laser evaporates the sample, and the vapors emitted by the sample pass through an electric field and produce a discharge that excites a luminous emission strong enough for analysis by a spectrograph.

Atomic absorption analysis of natural and industrial waters is being used more widely. Elements that have been determined include: lithium, sodium, magnesium, potassium, calcium, manganese, iron, cobalt, nickel, copper, zinc, rubidium, strontium, and barium—Billings and Harriss (7U), Wheat

(56U), West and Herrin (55U), Fabricand *et al.* (19U), Kristiansen (32U), Platte and Marcy (41U), Fishman (20U), Fishman and Downs (21U), Slavin (49U), and Strasheim and Kruger (51U).

Correction to standard conditions of color measurements of natural water were discussed by Singley, Harris, and Maulding (47U). They adopted pH 3, a representative value of finished waters, as a reference standard and devised a nomograph that allows ready determination of water color at the standard pH 8.3. Using a quartz spectrophotometer in the wavelength range 335 to 800 m μ , Petrov (40U) compared the platinum-cobalt and imitation scales for the determination of color in natural water. The imitation scale gave spectral curves similar to that of the platinum-cobalt scale but with a characteristic bend in the range 400 to 500 m μ and a nearly constant transmission coefficient between 425 and 465 m μ . Curves of the imitation scale reached their limiting value at 540 to 600 m μ , whereas curves of the platinum-cobalt scale reached the limit at 480 to 580 m μ . A combination in aqueous solution of ferric chloride and cobalt chloride gave a spectral curve closer to that of Ladoga Lake water than either of the two scales examined.

Determinations of specific conductance and dissolved-solids content of water received the attention of several investigators. Duran-Lopez (15U) devised a nomogram for the rapid determination of specific conductance and dissolved-solids content. Shimanova and Ponomarev (45U) developed and tested an apparatus for determining the salt composition of sea water by measuring the electrical conductance. Accuracy was 0.005 ppt for ocean water and 0.01 to 0.02% for sea water. Using an inductively coupled conductance indicator, Reeburgh (42U) measured the absolute specific conductance of solutions prepared from a sample of Red Sea water. The conductance was measured at 2° temperature intervals over the range 1 to 35° C and at 1% chlorinity intervals from 16 to 22‰. The precision of individual measurements was 0.001 mmho per cm in conductance, 0.002% in chlorinity, and 0.001° in temperature.

A new type of plastic hydrometer for the rapid determination of total salts in ground water in the field is described by Yao, Chu, and Yu (59U). The accuracy is approximately 0.2 gram per liter. The effect of photosynthesis and respiration on the electrical conductance of sea water was studied by Park and Curl (39U). They found a 0.06% decrease in conductance after 65 hours of illumination and a 0.03% increase after a 49-hour dark period as a result of photosynthesis and respira-

tion of the diatom *Skeletonema costatum* in sea water enriched with nutrients. There were also changes in pH, total chlorophyll, and oxygen concentration.

Roennefahrt (44U) discussed the theoretical basis of redox-potential measurement and cited examples of applications in the determination of metals and other ions in effluents.

Techniques for sampling of water were discussed by several investigators. Veshchezerov (54U) reported that a piston nonwash sampler can be used to collect water samples for the determination of carbon dioxide and hydrogen sulfide provided that the component to be determined can be bound chemically and converted to a stable compound at the moment of sampling. An appropriate reagent of barium hydroxide and cadmium acetate is poured into the sampler under the movable piston before letting it down into the borehole. The reagent is loaded with a special buret with a valve; the sample is poured from the sampler with a special funnel. This sampling method can be used for the determination of ferrous iron, ferric iron, and other unstable and volatile components.

Cherry (13U) described a portable sampler for collecting water samples from specific zones in uncased or screened wells. The sampler consists of two inflatable boots, one mounted above the submersible pump and the other below it. The sampler is portable and easily repositioned without removing it from the well. Auxiliary instruments for measuring temperature, specific conductance, or other chemical or physical characteristics of the ground water can be inserted between the boots.

Edeline and Heuze (17U) described two tools for collecting hydrocarbons from surface films. The simplest consists of loose-weave filter paper disks placed flat on the surface to remove the hydrocarbon film. The second consists of a copper cylinder 7 cm high and 17.5 cm in diameter, a handle, and lateral orifice of 5 mm. The inside of the cylinder is lined with chromatographic paper and is wetted with distilled water. The sampler is submerged to within 1 cm of the top edge of the paper. One or two drops of concentrated commercial detergent are added at the center of the intercepted circle. When the hydrocarbon film has receded to a 1- or 2-cm annulus at the periphery of the cylinder, the sampler is slowly withdrawn.

Brusilovskii and Kanakina (12U) suggested that differences in the cation-anion sum of ordinary methods of analyzing high-acid waters are due to specific features of the composition of these waters where the hydrogen ion concentration is comparable to the concentration of other components. They present a detailed discussion of calculations that attempt to eliminate

the difference in the cation-anion sum. Dyrssen (16U) used the methods of Gran I and Gran II to determine equivalence points in potentiometric titrations of sea water. In an analysis of waste water, Borchert (9U) used rapid colorimetric and volumetric methods for the quantitative determination of hexavalent chromium, oxidizable cyanides, and chloride in aqueous plant effluents. Experiments by Belyaev (6U) with sea water showed that the values closest to the time concentration of trace heavy metals can be obtained from the frequency curve of the probability "d" of distribution. Use of statistical processing permits one to decrease the error to a few per cent and simultaneously to decrease considerably the number of repeated analyses.

Suspended matter in water was the subject of several papers. Electron micrographs enabled Ishii and Ishikawa (29U) to identify certain constituents of suspended solids in sea water. Eden (18U) summarized a progress report of an analytical panel charged with a comprehensive study of the measurement of turbidity in water. Results obtained by eight laboratories using their own methods to analyze a prepared sample indicated enormous variability between laboratories.

A series of experiments indicated that the apparent reasons for variability between laboratories was inherent in the range of instruments, methods, and operators and could not be eliminated by complete standardization. Black and Hannah (8U) used the Brice-Phoenix Absolute Light Scattering Photometer and the Photovolt Lumetron, Model 450, with a nephelometry attachment to measure the light-scattering properties of 17 processed waters. Both instruments were calibrated with a standard fuller's earth suspension. Intensities of scattered light per unit volume of water were 80 to 400 times greater at 15° from the incident beam than at 90°. Because of the different relative scattering intensities from the samples at any given angle, the absolute photometers with high-angular resolution will not give turbidities in close agreement with those measured by the photocell instruments, which integrate the light scattered over a wide angle. With the absolute photometer, relative changes in turbidity equivalent to 0.001 turbidity unit can be measured in low turbidity waters.

Baalsrud and Henriksen (3U) noted that suspended matter in water can be determined by light absorption and light scattering methods. The physical properties of settling, particle size, and filtering, and the chemical properties are determined through inorganic and organic methods of analysis. No one method provides a complete

description of the suspended matter. Tettamanzi (53U) described an apparatus that reduces the time of a suspended solids in water determination from approximately 8 to 2 hours. Hannah and Dwyer (25U) used a Perkin-Elmer Model 521 with single and triple reflection ATR accessories to obtain infrared spectra of colloidal trace contaminants in water.

Several investigators used the spectrograph to determine trace elements in water. Aidarov and Gribanova (1U) used the ISP-22 and KS-55 spectrographs to determine concentrations of lithium, rubidium, cesium, thallium, strontium, and boron in salts, brines, and silts. Using a direct-reading spectrochemical procedure and the rotating-disk, high-voltage spark technique, Kopp and Kroner (31U) determined the following minor elements: silver, aluminum, arsenic, boron, barium, beryllium, cadmium, cobalt, copper, iron, molybdenum, manganese, nickel, phosphorus, lead, strontium, vanadium, and zinc. By using background as the internal standard, concentrations in the order of 0.01 to 100 mg per liter in the processed sample are determined in a matter of minutes by converting the counts from a sequential readout system on a drum calculator. Recoveries of the elements from known solutions ranged from 80 to 113% at the 90% confidence level.

Marti and Herrero (37U) reported the spectrochemical determination of 38 elements in the concentration range 0.001 to 1% in mineral waters. Morris (38U) described the use of gallium oxide in spectrographic procedures. With gallium oxide as a carrier, beryllium oxide was excited by a dc arc. A gas mixture of 80% argon and 20% oxygen was used to determine iron, chromium, nickel, manganese, lead, magnesium, copper, calcium, silicon, and aluminum to 1 ppm. Impurities in distilled and deionized water were determined with gallium oxide as a collector. Zyrin *et al.* (61U) recommended a spectrographic analysis in which the concentration is determined by darkening of analytical lines on a photographic spectrum. Concentrations of approximately 30 trace elements in soils, plants, and water can be determined by this analysis. The sensitivity of the method is generally between 5 and 10 mg per kg of material except for zinc for which the sensitivity is much lower.

By means of spectrochemical analysis, Boswell and Brooks (10U) determined the organic aqueous distributions (%) extraction) for extractions at 25° with cyclohexanone and cyclohexanol. The two solvents were presaturated with the same *M* of hydrochloric acid that the aqueous phase had. The average coefficients of variation for several elements were given. Cyclohexanone is

more efficient but less selective than cyclohexanol. A simple technique for *in situ* extraction of many trace elements in sea water was described by Lal, Arnold, and Somayajulu (33U). The method is based on ferric hydroxide adsorption. A matrix of finely dispersed ferric hydroxide on a natural sponge or fibrous jute framework is towed through the sea. Silicon, titanium, aluminum, and beryllium (present in sea water in concentrations of 10^{-4} to 10^{-9} grams per liter) were extracted from the equivalent of some hundreds of tons of coastal waters. The radioactive nuclides beryllium-7 and silicon-32 were measured in the extract.

Hueckstedt (27U) obtained a sensitive redox indicator from the following reagents: (a) 10 ml commercial 0.025M ferriin solution diluted to 100 ml. with water and (b) 3 ml commercial 0.1N cerium (IV) sulfate solution mixed with 30 ml 25% sulfuric acid diluted to 100 ml with water. These solutions can be stored for at least 2 months, and exclusion of atmospheric oxygen was found to be unnecessary. For the determination of traces of reducing compounds, 0.5 ml of each solution was accurately measured into a white dish, mixed with water, and the test solution added slowly. The end point was marked by the appearance of a bluish red color that turned pure red after standing 30 to 60 seconds.

In studies of light-dependent quality changes in stored water samples, Slack and Fisher (48U) found no significant difference among samples after dark-light storage in polyethylene bottles of three degrees of transparency. After exposure to alternate light and dark periods, the pH ranged from 9.27 to 10.17. In most samples the carbonate concentration ranged from 7 to 13 ppm, and free carbon dioxide and nitrate were not detected. During the period of dark storage, pH decreased 2 units to the range 7.15 to 8.02, and free carbon dioxide and bicarbonate concentrations increased greatly. Nitrate and sulfate increased in all samples. Phosphate concentration was greater in the illuminated samples than in the continuously dark samples, but the magnesium concentration was greater in the continuously dark samples.

Margara (35U) described a simple apparatus for the evaporation of several liters of dilute mineral waters for the determination of the residue and of the principal cations. Standard methods for total hardness, dry residue, alkali metals, sulfate, and chloride were outlined. Bicarbonate was determined by acidimetric titration of the calcium, sodium, and potassium carbonates and of the $x\text{MgCO}_3 \cdot y\text{Mg}(\text{OH})_2 \cdot z\text{H}_2\text{O}$ formed during evaporation of the sample. The ratio x/y does not affect the determination of bicarbonate.

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