

BIBLIOGRAPHICAL ABSTRACTS

1933-1957

1933

(10A) S. von Pilat, J. Serada, and W. Szankowski, "Mineral Oil Sulfonic Acids," *Petroleum Zeitung*, Vol. 29, No. 3, pp. 1-11 (1933); *Chemical Abstracts*, Vol. 27, p. 2287 (1933).

Sulfonic acids formed in mineral oil refining divided into three classes according to solubility of calcium salts. Calcium salts soluble in water but insoluble in ether differentiated from others by insolubility of their heavy metal salts.

1935

(16A) G. F. Nadeau and L. E. Branchen, "The Quantitative Titration of Amino Acids in Glacial Acetic Acid Solution," *Journal, Am. Chemical Soc.*, Vol. 57, pp. 1363-1365 (1935).

Rapid and precise method for quantitative determination of organic amino acids based on titration of strong acids and bases in acetic acid systems, crystal violet, alpha-naphtholbenzein, and benzoyl auramine. Found to give results in accord with those obtained potentiometrically with chloranil electrode.

(17A) D. M. Smith and W. M. D. Bryant, "Rapid Determination of Hydroxyl by Means of Acetyl Chloride and Pyridine," *Journal, Am. Chemical Soc.*, Vol. 57, pp. 61-65 (1935).

Rapid and precise method for determination of primary or secondary hydroxyl groups in organic compounds based on use of acetyl chloride and pyridine. Under similar conditions the pyridine, acetyl chloride is shown as more reactive than acetic anhydride.

1944

(53A) R. M. MacPherson, "Alkyldimethylbenzylammonium Chloride as a Sanitizing Agent for Eating Utensils," *Canadian Journal Public Health*, Vol. 35, pp. 198-202 (1944); *Chemical Abstracts*, Vol. 38, p. 4359 (1944).

A test kit devised for field determination of the concentration of the chemical in rinse solutions utilized ethylene dichloride or chloroform, bromothymol blue as indicator and endpoint when the aqueous layer remains blue.

1946

(70A) R. Dreywood, "Qualitative Test for Carbohydrate Material," *Analytical Chemistry*, Vol. 18, p. 499 (1946).

Solution of anthrone in concentrated sulfuric acid gives permanent green coloration with carbohydrate material.

1947

(84A) R. W. Eyer, E. D. Klug, and F. Diephuis, "Determination of Degree of Substitution of Sodium Carboxymethyl Cellulose," *Analytical Chemistry*, Vol. 19, pp. 24-27 (1947).

Acid-wash method, conductometric and colorimetric methods developed for research and control purposes for application to purified or unpurified samples with various degrees of substitution. The acid-wash method involves precipitation with methanol acidified with hydrochloric acid or nitric acid, removal of excess acid by methanol wash. The dried acid is dissolved in excess sodium hydroxide and back titrated. The conductometric procedure requires solution of the sodium carboxymethyl cellulose in distilled water containing a small amount of sodium hydroxide of standard solution. The solution is then titrated conductometrically with standard hydrochloric acid solution. The colorimetric method involves dissolution of the sample in sodium hydroxide solution, acidification with sulfuric acid and heating under reflux to produce glycolic acid. The Calkins procedure using 2,7-dihydroxynaphthalene is then used with slight modification.

(89A) E. E. Morse, "Anthrone in Estimating Low Concentrations of Sucrose," *Analytical Chemistry*, Vol. 19, pp. 1012-1013 (1947).

Procedure for estimation of sucrose concentration in the range of 10 to 250 parts per million using 0.05 per cent solution of anthrone in concentrated sulfuric acid. Said to give increasing accuracy and sensitivity over the Molisch alpha-naphthol test.

1948

(104A) G. C. Harris and T. F. Sanderson, "Resin Acids. I. An Improved Method of Isolation of Resin Acids; The Isolation of a New Abietic-Type Acid, Neoabietic Acid," *Journal, Am. Chemical Soc.*, Vol. 70, pp. 334-339 (1948).

Good separation of the diamylamine salt of abietic acid effected in the presence of resin acids.

1949

(113A) M. W. Cucci, "A Review of Quaternaries," *Soap*, Vol. 25, No. 11, pp. 133, 135, 137, 139 (1949).

Review of methods.

(113B) P. N. Degens, Jr., and H. van der Zee, "The Influence of Synthetic Detergents on Sewage Purification," *Chemisch Weekblad*, Vol. 45, pp. 166-170 (1949); *Chemical Abstracts*, Vol. 43, p. 5140 (1949).

Mentions that the methylene blue-chloroform test for anion active detergents cannot be used for sewage because a urine component in ordinary sewage gives a positive reaction.

(120A) F. J. Viles, Jr., and L. Silverman, "Determination of Starch and Cellulose with Anthrone," *Analytical Chemistry*, Vol. 21, pp. 950-953 (1949).

Colorimetric determination of starch and cellulose at wave length of 625 μ using 0.1 per cent solution of anthrone in concentrated sulfuric acid. Various requirements of method are included.

(123A) A. Z. Conner and R. P. Eyler, "Analysis of Sodium Carboxymethyl Cellulose. Copper Salt Precipitation Method for Determining Assay Value and Degree of Substitution," *Analytical Chemistry*, Vol. 22, pp. 1129-1132 (1950).

Copper salt of carboxymethyl cellulose precipitated, weighed and analyzed for copper content iodometrically. From the weight of the precipitate and copper content the assay value, that is, the carboxymethyl cellulose content, and the degree of substitution may be calculated. Method particularly applicable to purified grades.

1951

(155A) F. J. Ball and W. G. Vardell, "Spectrophotometric Analysis of Tall Oil Resin Acids," *Journal, Am. Oil Chemical Soc.*, Vol. 28, pp. 137-141 (1951).

Resin acids isolated from tall oil by cyclohexylamine precipitation method.

(174A) R. Neu, "Analysis of Laundering and Cleaning Compounds.—VI. Qualitative Identification of Phosphates," *Fette u. Seifen*, Vol. 53, pp. 148-149 (1951); *Chemical Abstracts*, Vol. 45, p. 6860 (1951).

Alkylidimethylbenzylammonium chloride gives a precipitate with metaphosphates and tripolyphosphates in acetic acid solution. Only the tripolyphosphate precipitate is soluble in excess quaternary. Pyrophosphates do not react with the quaternary.

(178A) E. P. Samsel and R. A. DeLap, "Colorimetric Determination of Methyl Cellulose with Anthrone," *Analytical Chemistry*, Vol. 23, pp. 1795-1797 (1951).

This method useful for the determination of methyl cellulose mixed with other carbohydrate materials. By combining methoxyl determination, selective solubility (particularly in hot water), and colorimetric determination of carbohydrates by the anthrone method, various mixtures were analyzed successfully. Methyl cellulose determined with an accuracy of approximately ± 5 per cent.

(179) W. H. Simmons, *et al.*, "Examination of Detergent Preparations," *Analyst*, Vol. 76, pp. 279-286 (1951).

Systematic testing for true soap, alkali carbonate, phosphate and silicates, abrasive

or fillers, synthetic organic detergents of sulfated or sulfonated types, sulfated glycerides, propyleneglycol derivatives, quaternaries, methyl- and carboxymethyl cellulose, naphthenic acid soaps.

(182A) M. H. Swann, "Colorimetric Determination of Rosin and Rosin Esters," *Analytical Chemistry*, Vol. 23, pp. 885-888 (1951).

Described for use in paints and varnishes. Qualitative method uses benzene solution treated with acetic anhydride and concentrated sulfuric acid. Red or violet coloration indicates presence. Quantitative colorimetric method for free rosin and rosin soaps described using controlled conditions as for qualitative test, reading on colorimeter. Rosin esters determined by closely controlled variation of the qualitative method.

(195A) J. Glazer and T. D. Smith, "Determination of Sodium Oleate in Dilute Aqueous Solution," *Nature*, Vol. 169, p. 497 (1952).

Modification of the Epton method. Checked using a Langmuir surface balance and both methods in good agreement and accurate to ± 1 per cent.

1952

(202A) J. A. Nevison, "Analysis of Syndets," *Journal, Am. Oil Chemists' Soc.*, Vol. 29, pp. 576-582 (1952).

Review of methods to cover sulfated alcohols and olefins, using hydrolysis and silica gel absorption methods; alkylaryl sulfonates by mass spectrometer, *p*-toluidine hydrochloric acid by volumetric or benzidine hydrochloride methods; fatty acid derivatives; nonionics; packaged detergents; Wurzschnitt elemental analysis method; anthrone method for carboxymethyl cellulose, ultraviolet and infrared methods suggested; and syndet residues by sensitive colorimetric methods.

(207A) A. H. Tilson, W. V. Eisenberg and J. B. Wilson, "Identification of Certain Quaternary Ammonium Compounds, as Reineckates," *Journal, Assn. Official Agricultural Chemists*, Vol. 35, pp. 459-467 (1952).

The reineckate salts of thirteen quaternary compounds prepared and their crystallographic properties determined. These are given in convenient form for identification.

(208A) P. F. Warfield, "Test for Highly Active Methylene Groups," *Analytical Chemistry*, Vol. 24, pp. 890-891 (1952).

Colorimetric test for phenols and naphthols among others. Test consists of adding 10 ml of developer solution (*p*-diethylaminoaniline 4.0 g; sodium carbonate monohydrate 75.0 g; distilled water to make one liter) to about 1 mg of unknown and mixing thoroughly. To this one drop of 10 per cent aqueous sodium ferricyanide solution added. A blue-green color indicates phenols or naphthols and rusty brown a negative reaction.

1953

(222A) C. V. Francis, "Sodium Carboxymethyl Cellulose. Determination of Degree of Substitution of Active Agent," *Analytical Chemistry*, Vol. 25, pp. 941-943 (1953).

Procedure described using uranyl ion as the precipitating agent, forming insoluble uranyl carboxymethyl cellulose. This gravimetric procedure said to be useful on a variety of carboxymethyl cellulose samples and shows minimum recovery of 95 per cent sodium carboxymethyl cellulose, with a precision within ± 0.01 of degree of substitution. This method is particularly useful for routine analytical control with total time of 2 to 4 hr and no special equipment necessary.

(224A) U. Gallo and S. Casadio, "Non-ionized Surface-Active Agents and Alkaloids," *Bulletin Galencia*, Vol. 16, pp. 98-102 (1953); *Chemical Abstracts*, Vol. 50, p. 10430 (1956).

Reactions of best known nonionics with alkaloidal precipitants studied. Precipitates, suspensions, and turbidity were produced in most cases, except for picric acid, but alkaloids were not detectable in the presence of surfactants.

(229A) G. B. Johnson, "Field Testing of Quaternaries," *Soap*, Vol. 29, No. 7, pp. 114-115, 135 (1953).

Field test based on Auerbach's procedure. Consists of addition to the solution to be tested of chloroform, a tablet of bromophenol blue and sodium carbonate, and the separated chloroform layer matched against standards for quantities. Intensity of color varies with the molecular weight of the quaternary so that separate standard colored glasses must be used for each quaternary tested.

(231A) F. E. Luddy, A. Turner, Jr., and J. T. Scanlan, "Spectrophotometric Determination of Cholesterol and Triterpene Alcohols in Wool Wax," *Analytical Chemistry*, Vol. 25, pp. 1497-1499 (1953).

Method depends upon colorimetric development by the Liebermann-Burchard method by treatment with acetic anhydride and sulfuric acid. In present work the 1,4-dioxane addition reduces the rate of color development and permits operation at room temperature. A spectrophotometer permits use of a single solution for determination of both absorption bands for triterpenes and cholesterol.

(231B) A. R. Martin and A. C. Lloyd, "Simplified Procedure for Water Determination by the Karl Fischer Method.—I. Application to Dry-Cleaning Detergents and Solvents," *Journal, Am. Oil Chemists' Soc.*, Vol. 30, pp. 594-597 (1953).

Karl Fischer method as modified by Seaman further modified for application to dry-

cleaning detergents. Normal propanol substituted for methanol in standardization.

(231C) W. Mohr and A. Wortman, "Determination of the Alkalinity of Cleaning Solutions by Titration," *Proceedings*, 13th. Int. Dairy Congress, (The Hague), Vol. 3, pp. 863-865 (1953); *Chemical Abstracts*, Vol. 47, p. 11762 (1953).

Phenolphthalein suggested as indicator for this does not measure bicarbonates when present.

(236A) A. Stehlik and B. Novarik, "A Method for the Determination of Organically Bound SO_3 in Sulfonated Oils," *Ceskoslovenska kozarstvi*, Vol. 3, pp. 56-57 (1953); *Chemical Abstracts*, Vol. 49, p. 8617 (1955).

Known methods were tested and a separation, ashing and titration technic worked out.

1954

(238A) P. Bert, "Method of Determination of Alkyl Sulfates by Quaternary Ammonium Compounds," 1st World Congress on Surface Active Agents, Vol. I, pp. 185-188 (1954).

Titration method using a known quaternary, eosin in acetone as indicator, buffer solution of citric acid, and the endpoint considered when the solvent becomes colored. Titer determination given and application to several alkyl sulfates made. Reproducibility approximately ± 0.2 per cent.

(240A) F. E. Brauns, J. B. Hlava and H. Seiler, "Determination of Sulfur and Barium in Organic Barium Sulfonates," *Analytical Chemistry*, Vol. 26, pp. 607-608 (1954).

Determination of both sulfur and barium on the same sample accomplished by use of a small cation exchange column. The barium is adsorbed and the sulfonic acid obtained quantitatively in the filtrate. Barium quantitatively eluted with strong hydrochloric acid solution from the upper end of the column by inverting following absorption.

(240B) "Methods of Testing and Analysis of Soaps and Detergents," Canadian Government Specifications Board, Standard 2-GP-11, Amendment 9 Nov., 4, 1954.

Qualitative method for differentiation in ionic types. Use of combined 0.05 per cent uranine and 0.05 per cent methylene blue added to solution of detergent. Distilled water used for comparison. Deeper blue color than standard indicates presence of cationics, various shades of green indicate anionics. Nonionics have no effect on color. Soap presence determined by adding 20 per cent magnesium nitrate solution to detergent solution where curdy precipitate indicates soap.

(242A) C. C. T. Chinnick and P. A. Lincoln, "The Quantitative Analysis of Quaternary Ammonium Compounds," 1st

World Congress on Surface Active Agents, Vol. I, pp. 209-214 (1954).

Review of methods: dichromate; ferricyanide; phosphotungstate and phosphomolybdate; anionic detergent methods; methylene blue; bromophenol blue; adsorption indicator methods. Discuss interference of nonionic surfactants on the analysis. Methods of precipitation for nonionics cannot be used. Use of methylene blue and bromophenol blue useful only under certain outlined conditions. Use of anionic for estimation of quaternary in the presence of nonionic acceptable by controlling the volume of the solution to be titrated, and its sodium chloride content. An assay of both quaternary and nonionics readily accomplished by applying bromophenol blue together with precipitation of total surfactant with phosphotungstic acid.

(242B) F. E. Critchfield and J. B. Johnson, "A Differential, Alkalimetric Determination of Sulfuric Acid-Hydrochloric Acid and Sulfuric Acid-Nitric Acid Mixtures," *Analytical Chemistry*, Vol. 26, pp. 1803-1806 (1954).

These acid mixtures are determined by titration with standard morpholine in acetonitrile medium. The hydrochloric-sulfuric mixtures can be determined potentiometrically and colorimetrically, but the nitric-sulfuric mixtures only potentiometrically. Method easy to perform, and rapid and accurate.

(242C) A. H. Delsemme, "Analysis and Identification of Surface Active Agents by Infrared Spectrophotometry," 1st World Congress on Surface Active Agents, Vol. I, pp. 192-196 (1954).

Useful for determination of impurities, for preliminary qualitative analysis, and for identification of functional types of surfactants for comparison purposes. Useful for differentiation of isomeric structure and for quantitative determination of length of the carbon chain.

(244A) H. Etienne, "Secondary Products of the Sulfonation of Fatty Alcohols and of Alkylbenzenes," 1st World Congress on Surface Active Agents, Vol. I, pp. 215-219 (1954).

Gives two schemes for separation of products into inert or sulfonated fraction, mono- and disulfonates. Used infrared method to compare the separation products of sulfonated alkylbenzene. Presence of sulfones as inerts or partial inerts indicated in this separation.

(244B) H. C. Evans, "Determination of Non-Ionic Detergents in Very Dilute Solutions," 1st World Congress on Surface Active Agents, Vol. I, pp. 202-205 (1954).

Modification of procedure of Haakh, von Candie and Möbus for use at concentrations as low as 1 ppm. Special reagent consists of sulfonation and condensation product of

resorcinol and glucose. Method depends upon precipitation of nonionic and reagent on filter aid and this washed and precipitate finally dissolved in alcohol and the color intensity determined. Good reproducibility indicated. Interference by secondary alkyl sulfates and alkylbenzene sulfonates.

(244C) M. Farvarque, "Proportion of Tetrasodium Pyrophosphate in Triphosphosphate," 1st World Congress on Surface Active Agents, Vol. I, pp. 228-232 (1954).

Quantitative estimation of tetrasodium pyrophosphate in triphosphosphate made by goniometric X-ray method. Two procedures given, one rapid and the other slower but more precise.

(244D) A. Fayaud and S. Rivera, "Chemically Pure Sodium Lauryl Sulfate and a Routine Method of Analysis," 1st World Congress on Surface Active Agents, Vol. I, p. 233 (1954).

A chemically pure sodium lauryl sulfate was prepared by recrystallization. A rapid volumetric method for analysis is given. Consists of hydrolysis with standard hydrochloric acid, use of a blank determination under the same conditions, and titration to phenolphthalein endpoint with standardized sodium hydroxide. Calculation given for sodium lauryl sulfate and lauric sulfate. Free alcohol can be determined by ether extraction.

(251A) E. Heinerth, "Determination of Silicic Acid in Laundering Compositions," *Fette u. Seifen Anstrichmittel*, Vol. 56, p. 595 (1954); *Chemical Abstracts*, Vol. 50, p. 13480 (1956).

The determination can be carried out with reasonable accuracy by heating 2.5 g sample with 5 ml concentrated nitric acid and 2.5 g sodium nitrate until light-colored and homogeneous (about 10 to 20 min), cooling, diluting carefully with 100 ml water, boiling out nitrogen oxides and filtering off the silica.

(252A) J. A. van der Hoeve, "Analysis of Textile Auxiliary Products," *Journal, Soc. Dyers Colorists*, Vol. 70, pp. 145-154 (1954); *Chemical Abstracts*, Vol. 49, p. 7307 (1954).

Analytical scheme for analysis of cation-anion active products and the polyamides which react like polyethylene oxide products. The analytical scheme is based on determination of any organically combined nitrogen, determination of the ionic nature of the compound and the determination of the type of hydrocarbon in it.

(254A) W. Koot, "Qualitative and Quantitative Method for Studying and Determining an Adjuvant Anion or Active Cation," *Ingenieur Chimique*, Vol. 36, No. 195, pp. 12-17 (1954); *Chemical Abstracts*, Vol. 48, p. 13540 (1954).

Use of methylene blue and chloroform as indicator of ion type. Anionic if the chloro-

form is colored deep blue and the water phase only slightly colored. For determination of cation used chloroform and bromophenol blue. Cation active if chloroform colored bright blue-green and water phase colored mauveblue.

(254B) C. Kortland and H. F. Dammers, "Qualitative and Quantitative Analysis of Mixtures of Surface-Active Agents with Special Reference to Synthetic Detergents," 1st World Congress on Surface Active Agents, Vol. I, pp. 197-201 (1954).

See Reference (285). This is the preliminary portion, discussing qualitative analysis, and the separation of the surface active ingredient after qualitative identification.

(254C) E. Kuyper and D. Vader, "The Continuous Determination and Indication of the Active-Ingredient Content of "Tee-pol" Solutions," 1st World Congress on Surface Active Agents, Vol. I, pp. 382-385 (1954).

A known volume of solution containing sodium alkyl sulfate is automatically mixed with a fixed volume of a solution containing an appropriate quantity of a cationic-active material, an indicator, and a buffer of suitable pH, and the optical transmission is recorded. Indicator was bromocresol green, the cation-active agent cetyltrimethylammonium bromide, acetic acid buffer of pH 4.6, and up to 0.05 per cent of sodium alkyl sulfate.

(256A) R. Neu, "Determination of Potassium and Triethanolamine with Sodium Tetraphenyl Borate," *Zeitschrift analytische Chemie*, Vol. 143, pp. 254-257 (1954); *Chemical Abstracts*, Vol. 49, p. 2949 (1955).

To about 1 per cent solution of product at pH 5 to 6 with acetic acid, add about 3 per cent sodium tetraphenyl borate in doubly distilled water. After 90 min filter and wash with acetic acid water pH 5 to 6. This contains both potassium and triethanolamine. Treat the weighed precipitate with perchloric acid, filter and wash with ethanol. Dry and weigh the potassium tetraphenyl borate. Results are about 2 per cent low in both cases because of appreciable solubility of both precipitates.

(259A) C. Paquot, "On the Complexity of the Problems Posed in the Analysis of Commercial Detergent Products," 1st World Congress on Surface Active Agents, Vol. I, pp. 171-174 (1954).

Discussion of the problems involved in qualitative and quantitative analysis. Qualitative analysis for the inorganic constituents is relatively simple except for the polyphosphates which degrade on drying. The analysis of active ingredient is considerably more difficult and methods of Wurtzschmitt and Bergeron have been used. Quantitative analysis for the inorganic portion possible but separation of the phosphates into the several types and in the presence of another

not resolved. Suggested are several techniques for chemical analysis, of active ingredient, as well as by ultraviolet and infrared.

(260A) F. J. Pollak, "Recent Advances in the Use of Sodium Carboxymethyl Cellulose as a Detergent Aid," 1st World Congress on Surface Active Agents, Vol. II, pp. 689-691 (1954).

Widaly method based on dissolution of all inorganic and organic salts in hydrochloric acid-acidified methanol and only silica and carboxymethyl cellulose (CMC) remain in the residue. CMC then determined by titration with bichromate and ferrous ammonium sulfate. Best results obtained by introducing filter aid and centrifugal separation. Method: transfer 1 g of well mixed sample to 250 ml beaker. Add 90 ml methanol and 10 ml 3N hydrochloric acid. Heat to boiling on water bath and simmer gently 15 to 20 min. Cool and transfer to suitable centrifugal cup, washing the beaker with small amount of methanol. Add 1 g kieselguhr (free from organic matter) and stir mixture. Centrifuge at 3500 rpm. Decant methanol and wash residue twice with 50 ml lots of methanol. Decant and dry the residue cautiously at first to avoid spattering, then for 2 hr at 100 C. Transfer residue from cup to 750 ml conical flask, wash cup with 10 ml concentrated sulfuric acid. Maintain temperature of mixture between 80 to 90 C for 30 min. Dilute to 400 to 500 ml with water. Cool thoroughly and add 1 to 2 g potassium iodide. Titrate liberated iodine with N/10 sodium thiosulfate using starch as indicator. Carry out blank omitting only the sample. 1 ml N/10 potassium bichromate equals 0.0008 g sodium carboxymethyl cellulose.

(262A) R. Raemaekers, "The Determination of Tri- and Pyrophosphate in Tripolyphosphate," *Comptes rendus*, 27^e Congres Int. industries Chimiques, Brussels, Vol. 2 (1954); *Industries Chimiques Belges*, Vol. 20, Special No., pp. 603-607 (1955); *Chemical Abstracts*, Vol. 50, p. 14450 (1956).

Determination of tripolyphosphate and pyrophosphate in tripolyphosphate by the Raistrick and the Kuhlmann Co. methods was systematically studied. Coprecipitation of the zinc salts of tripoly and pyrophosphates do not give precipitates of theoretical composition. By application of the two methods to mixtures of pure materials a standard curve is obtained which allows determination of mixtures, with an error of less than 1 per cent, by measuring the zinc oxide content of the mixture. Details of the modifications given.

(263A) J. P. Sislev, P. Desnuelle, and M. Naudet, "A Brief Sketch of the Contribution of l'Iterg to the Analysis of Surface Active Compounds," 1st World Congress on Surface Active Agents, Vol. I, pp. 189-191 (1954).

Discussed briefly are the problems in the analysis of sulfates of fatty ethyleneoxide amides, condensation products of fatty acid peptides, commercial alkylaryl sulfonates and finally evaluation of the properties of surfactants.

(267A) H. Stüpel and A. Y. Segesser, "The Practical Application of the *p*-Toluidine Method for the Analysis of Synthetic Detergents," 1st World Congress on Surface Active Agents, Vol. 1, pp. 175-179 (1954).

Describes the *p*-toluidine method in the quantitative analysis of raw materials; determination of molecular weight; for control analysis; control of raw material; practical application.

(269A) G. T. Walker, "Analytical Aspects of Cold-Wave Lotions. II." *Soap, Perfumery & Cosmetics*, Vol. 27, pp. 920-922 (1954); *Chemical Abstracts*, Vol. 48, p. 14126 (1954).

Review of methods of analysis in these products of wetting agents and other constituents.

(270A) R. Wemelle, L. Bourgeois, and J. Molle, "Method of Analysis and Identification of Fatty Alcohols—Application to the Complex Products of Synthesis," 1st World Congress on Surface Active Agents, Vol. I, pp. 224-227 (1954).

Used for identification are hydroxyl number, index of saponification after acetylation, hydrobromic acid esterification. Gives table of values for oxo alcohols from C_{10} to C_{18} . The fatty alcohols are sulfonated under standard conditions and the products purified then hydrolyzed with hydrochloric acid and the alcohols characterized. A calcium monophthalate of the fatty alcohol is formed and the melting points determined. Table given comparing these values of oxo and natural fatty alcohols.

(270B) R. Wickbold, "On the Application of the *p*-Toluidine Method to the Analysis of Anion-Active Substances," 1st World Congress on Surface Active Agents, Vol. I, pp. 180-184 (1954).

Discusses research on the completeness of precipitation; determination of active ingredient in the presence of builders; and determination of equivalent weight.

(271A) J. P. Wolff, "Determination of Rosin in Soap," 1st World Congress on Surface Active Agents, Vol. I, pp. 220-223 (1954).

Describes volumetric method by selective esterification with methyl alcohol in the presence of catalyst, but incomplete esterification is the chief fault. Colorimetric method based on acetic anhydride reaction described and given are methods used to improve the color stability. Use of ultraviolet for estimation and characterization suggested. Discusses gravimetric method by esterification and solvent extraction. Suggests chromatographic method using

chloroform solution and alumina column and elution with alcohol.

(271B) G. Zuppiroli, "Contribution to the Analysis of Technical Fatty Alkylamides," 1st World Congress on Surface Active Agents, Vol. I, pp. 206-208 (1954).

Fractionation of the reaction product of coconut fatty acids with large excess of diethanolamine by solution in water and extraction with ether. The clear ethereal upper layer contains free fatty acid and the soap formed with diethanolamine. The aqueous layer is treated with hydrochloric acid to form the salt of the amine. Several fractions are separated. Described the structure of compounds obtained by the fractionation.

1955

(271C) T. Akiyama, M. Fujiwara, H. Okamoto and K. Fusaka, "Chemical Analysis of Polyphosphate," *Bulletin, Kyoto College Pharmacy*, No. 3, pp. 19-22 (1955); *Chemical Abstracts*, Vol. 50, p. 4720 (1956).

Quantitative precipitates of zinc pyro and zinc tripolyphosphates are formed from their solution under pH 3.8 to 4.1. Zinc tripolyphosphate is dissolved in ammonium solution and is titrated with standard Versenate solution. Orthophosphate is precipitated as magnesium ammonium phosphate, dissolved in hydrochloric acid and then titrated with standard Versenate solution, adjusting to pH 10.0.

(274A) A. Barber, C. C. Chinnick, and P. A. Lincoln, "The Action of Morpholine on Some Quaternary Ammonium Salts and its Application to Their Analysis," *Journal Applied Chemistry* (London), Vol. 5, pp. 594-599 (1955); *Chemical Abstracts*, Vol. 50, p. 16568 (1956).

Reaction of morpholine with certain quaternary ammonium compounds splits off an alkyl group, giving a tertiary amine and an N-alkylmorpholine. The yields are quantitative. The composition of the quaternary compounds can be ascertained by fractional distillation of the reaction products, and the analysis can be adapted to the determination of single compounds and mixtures.

(274B) H. Bennett, "Recent Advances in the Analytical Chemistry of Silicate Materials," *Transactions, British Ceramic Soc.*, Vol. 54, (6), pp. 319-332 (1955); *Analytical Abstracts*, Vol. 3, 670 (1956).

Review of rapid and direct methods for determination of silica by precipitation of the molybdosilicate with an organic base, for example, 8-hydroxyquinoline or quinoline. Gravimetric time 4 hr, volumetric 2 hr, accuracy ± 0.5 per cent.

(276A) R. L. Caswell, "Report on Quaternary Ammonium Compounds," *Journal, Assn. Official Agricultural Chemists*, Vol. 38, pp. 304-305 (1955).

Recommends ultraviolet spectral method for certain quaternaries most frequently used, and gives characteristic maxima for the Beckman DU spectrophotometer. Ferri-cyanide method used successfully with the exception of some formulations of alkyl-tolylmethyltrimethyl ammonium chloride.

(277A) R. H. Crockett, "The Evaluation of Commercial Alkyl Sulfates," *Journal, Soc. Cosmetic Chemists*, Vol. 6, pp. 124-129 (1955); *Chemical Abstracts*, Vol. 50, p. 7483 (1956).

Methods and procedures for determining hydroxyl value of fatty alcohols, active detergent, free fatty alcohol, inorganic sulfates and chlorides, and water content are given.

(278A) A. J. Dutta and B. K. Mukherji, "Use of Ion-Exchange in the Rapid Estimation of Fatty Acids in Soap," *Indian Soap Journal*, Vol. 20, p. 187 (1955); J. W. Jenkins, *Journal, Am. Oil Chemical Soc.*, Vol. 34, p. a52 (1957).

The Dutta Mukherji method depends upon the principle of salt splitting by a laboratory sulfonated coal cation exchange process. Other details of comparative testing between the sulfonated coal product and Dowex 50 resin are given.

(280A) R. Hedin, "Colorimetric Analyses of Silicates," *Berichte deutsches chemische Gesellschaft*, Vol. 32, pp. 385-390 (1955); *Chemical Abstracts*, Vol. 50, p. 14451 (1956).

Suggested tests and standard solutions are given for spectrophotometric analyses of silica and other oxides.

(281A) H. Holness and W. R. Stone, "Separation Within the Homologous Series of *n*-Alkyl Sulfates, *n*-Alkyl Pyridinium and *n*-Alkyl Trimethyl Ammonium Halides by Paper Chromatography," *Nature*, Vol. 176, p. 604 (1955).

Effective when chain length varies by two carbon atoms. For *n*-octyl and *n*-dodecyl sulfates 15 per cent aqueous ethanol was used while 40 per cent aqueous ethanol was effective for separating mixtures of *n*-tetradecyl, hexadecyl, and octadecyl sulfates. Position shown by spraying with aqueous solution of pinacryptol yellow after drying. Under ultraviolet light spots of anionic material fluoresce bright orange on pale yellow background. Minimum amount detectable 1 μ g. Indicated as successful with other sulfonated detergents. Parallel investigation with cationic materials carried out using 30 per cent aqueous ethanol with 5 per cent concentrated hydrochloric acid. Position of spots shown by spraying with aqueous solution of rhodamine BS containing commercially available optical bleach. Sprayed chromatogram exposed to ammonia vapor and examined under ultraviolet light. Spots fluoresce bright vermilion on white background. Sensitivity about 1 μ g.

(284A) T. Kondo, "The Behavior of Dye

Ions in Detergent Solutions.—I. Interactions of Simple pH Indicators with Detergents," *Journal, Chemical Soc. Japan, Pure Chemical Section*, Vol. 76, pp. 1374-1378 (1955); *Chemical Abstracts*, Vol. 50, p. 8286 (1956).

Absorption spectra of the mixtures of dye-detergent studied at 15 ± 2 deg. From the results, the change of light absorption near the critical micelle concentration was discussed. Mixtures studied were methyl orange-dodecylpyridinium bromide, neutral red-sodium dodecyl sulfate.

(290A) X. Mamaliga, "Polarographic Study of the Interfacial Activity of Certain Quaternary Ammonium Salts," *Comunicaciones, Academiei Republicii Populare Române*, Vol. 5, pp. 1583-1591 (1955); *Chemical Abstracts*, Vol. 50, p. 16461 (1956).

Polarographic adsorption study undertaken. Found that compounds containing the octyl radical have nearly identical interfacial activity, while the compounds containing the octyl group are all different. Concluded that the hexadecyl group obliterates the effect of the carboxy and acid-amide functions in the activity of the substances on the polarized interface, while the octyl group does not interfere in this effect.

(290B) H. Negoro and S. Seno, "Analysis of Surface-Active Agents.—II. Color Reaction for Field Test of 1:500 Dilute Hyamine Solution," *Annual Report, Takamine Laboratory*, Vol. 7, pp. 122-125 (1955); *Chemical Abstracts*, Vol. 50, p. 16040 (1956).

Search for suitable indicator for Hyamine determination showed that an indicator composed of methyl orange 0.1, bromophenol blue 0.05, sodium bicarbonate 1, and sodium chloride 5 per cent, pH adjusted to 6.5 to 11.2, was most suitable. When 1 ml of the indicator was added to 5 ml of the Hyamine solution to be tested, and viewed through a transmitted light, red color indicated 1:1000 dilution, yellowish brown indicated 1:500 and green indicated 1:250 dilution.

(291A) R. Neu, "The Differentiation of Long-Chain Cation-Active Compounds from Ampholytes and Amine Salts," *Fette u. Seifen*, Vol. 57, (8), pp. 568-570 (1955); *Analytical Abstracts*, Vol. 3, p. 1428 (1956).

Treatment of long-chain quaternary with 3:5:7:3':4'-pentahydroxy-flavone in methanol and then with freshly prepared methanolic solution of tetraphenyldiboroxide, and an orange precipitate produced and the precipitate is stable to aqueous ammonia solution. Similar precipitate given by long-chain amine salts and ampholyte-active compounds but with these the precipitate is dissolved with 2 ml of 2 N ammonia solution. The reaction can be used to distinguish the two classes of compounds, and short chain quaternaries give no precipitate.

(298A) H. Schwerdtner, "Concentration Determination of Surface-Active Textile

Assistants in Water Solutions," *Textile u. Faserstofftechnik*, Vol. 5, pp. 569-572 (1955). *Journal*, Am. Oil Chemists' Soc., Vol. 33, p. 300 (1956).

Analytical methods reviewed. Authors method by titration with Rose Bengale as indicator described, which is especially applicable to aliphatic anionic substances with 10 to 20 carbon atoms in the chain.

(298B) A. B. Steele and L. D. Berger, Jr., "Characterization of Nonionic Detergents" *Proceedings*, Chemical Specialties Manufacturers Assn., Dec., 1955, pp. 185-187.

See reference 310.

(299A) "Soap and Detergents," Swiss Society for Analytical and Applied Chemistry. Hans Huber, Bern, Switzerland, (1955).

Complete system for analysis of soap and soap containing agents. Methods for soap and fatty acids, alkalies, phosphates, lesser constituents, examination of the alcohol insoluble portion, and bleaching agents. Under synthetic detergents are given methods for qualitative separation, and quantitative methods. "Qualitative" methods include isolation of active ingredient followed by identification of ion type; detection of aromatic ring; specific reaction of naphthalene ring; identification of albumin products; presence of alkali salts; pH value; tests for nitrogen, sulfur, phosphorus and halides. "Quantitative" methods include: Active ingredient by alcohol or ether extraction; hydrolysis methods; active ingredient by *p*-toluidine method; the Kortland and Dammers analytical scheme (Reference (285)).

(301A) H. L. Webster and A. Robertson, "The Rapid Determination of Total Fatty Acid in Unbuilt Soap Products," *Analyst*, Vol. 80, pp. 616-619 (1955).

Excess of standard calcium chloride solution added to weighed sample of soap in solution, and after heating, the calcium soap removed by filtration and excess calcium determined by titration with ethylenediaminetetraacetic acid solution, murexide being used as indicator. Correction of small quantity of alkali carbonate required and applicable to built soap with modification, but with loss of time-saving feature.

(303A) K. Yoshimura and M. Morita, "Invert Soaps.—I. Gravimetric Determination with Phosphotungstic Acid," *Bulletin*, Nat. Hygiene Laboratory, Tokyo (Japan), No. 73, pp. 141-143 (1955); *Chemical Abstracts*, Vol. 50, p. 7483 (1956).

For the determination of benzalkonium chloride and (alkylbenzyl)-trimethylammonium chloride a gravimetric procedure was devised.

(303B) K. Yoshimura and M. Morita, "Invert Soaps.—II. Colorimetric Determination of Benzalkonium Chloride with Phosphomolybdic Acid," *Pharmaceutical Bulletin*

(Japan), Vol. 3, pp. 432-434 (1955); *Chemical Abstracts*, Vol. 50, p. 15104 (1956).

The intensity of the blue color formed by the quaternary and phosphotungstic acid is measured spectrophotometrically between 400 and 1000 $m\mu$ (1956).

(305) T. Akiyama, M. Fujiwara, and H. Ichida, "Behavior of Polyphosphate with Tris(ethylenediamine)cobalt Bromide," *Bulletin*, Kyoto College Pharmacy, No. 4, pp. 21-22 (1956); *Chemical Abstracts*, Vol. 51, p. 4865 (1957).

The precipitation and pH range of polyphosphate with this complex was studied. Details of optimum pH ranges for pyro- and hexameta phosphates given. Tri- and tetrametaphosphates do not form precipitates but become oily.

(306) D. Aoki, Y. Iwayama, and K. Oka, "Colorimetric Determination of Invert Soap," *Yakuzaijaku*, Vol. 16, No. 1, pp. 11-12 (1956); *Chemical Abstracts*, Vol. 51, p. 9185 (1957).

Colorimetric methods for determining benzalkonium chloride and benzethonium chloride developed. Solutions shaken with sodium carbonate solution and chloroform, then with a solution consisting of cobalt chloride hexahydrate:ammonium thiocyanate: water at 24:35:100 for 1 min, filtered, and the extinction determined colorimetrically. Alcohols, glycols or other common solvents do not interfere.

(307) C. W. Ayers, "Estimation of the Higher Fatty Acids C_7-C_{18} ," *Analytica Chimica Acta*, Vol. 15, pp. 77-83 (1956).

Colorimetric methods developed for higher fatty acids by precipitation of the corresponding potassium soaps with either copper or cobalt nitrate at pH 9, solution in chloroform, and measuring the optical density of the solutions at 675 $m\mu$ for copper and 525 $m\mu$ for cobalt. This method applied to the hydrolyzates of esters and resins, and the analysis of cosmetic and textile emulsions.

(308) A. Barber, C. C. T. Chinnick, and P. A. Lincoln, "The Analysis of Mixtures of Surface-Active Quaternary Ammonium Compounds and Polyethylene Oxide Type of Nonionic Surface Active Agents," *Analyst*, Vol. 81, pp. 18-25 (1956).

Method described for overcoming the interference of polyethylene oxide nonionic agents with bromophenol blue method in determining quaternary ammonium compounds. Determination of polyethylene oxide nonionic agent by precipitation with heteropoly acid improved. Removal of quaternary ammonium cations which interfere is possible with ion-exchange resin. Methods and application given.

(309) I. Behr, "Simultaneous Analyses by an Ion-Exchange Method of Phosphorus, Sulfate and Chlorides," *Bulletin*, Research Council Israel, A, Vol. 5, pp. 259-262 (1956); *Analytical Abstracts*, Vol. 4, No. 1812 (1957).

Passage of solution containing PO_4^{3-} , SO_4^{2-} and Cl through a cationic exchanger (Amberlite IR-120) yields corresponding acids. Total acid found by titration with 0.1N caustic soda to + 8.98, the PO_4^{3-} by back-titration with 0.1N hydrochloric acid to pH 4.63, the chloride on separate portion by silver nitrate and the SO_4^{2-} by difference. Blank must be run on distilled water. Mean error in 80 tests 0.7 per cent for PO_4 , 4 per cent for SO_4 and 1 per cent on Cl.

(310) D. Burton and G. F. Robertshaw, "Proposed Revised Official Methods for the Analysis of Oils, Fats, and Soaps," *Journal, Soc. Leather Trades' Chemists*, Vol. 40, pp. 170-177 (1956); *Chemical Abstracts*, Vol. 50, p. 17482 (1956).

Methods revised are sampling, separation of fatty matter from non-fatty matter, unsaponifiable matter, saponification value, acid value, and iodine value.

(311) R. F. Childs and L. M. Parks, "Determination of Hexachlorophene in Liquid Soaps," *Journal, Am. Pharmaceutical Assn.*, Vol. 45, pp. 313-316 (1956); *Chemical Abstracts*, Vol. 50, p. 10430 (1956).

Hexachlorophene determined in liquid soap by a differential spectrophotometric method consisting of the measurement of the absorbance of a diluted sample at 312 μ at pH 8 and pH 3.

(312) D. Cook, "Relative Basicities of Alkyl Benzenes," *Journal Chemical Physics*, Vol. 25, pp. 788-789 (1956).

Relative basicities determined by measuring the shift in the infrared fundamental stretching frequency of hydrochloric acid in complexes of hydrochloric acid with alkyl benzenes. With substituted side chains, the order of basicity is given.

(313) J. Ruiz Cruz, "Methods of Analysis for Synthetic Detergents," *Grasas y Aceites*, Vol. 7, pp. 243-250 (1956); *Journal, Am. Oil Chemists' Soc.*, Vol. 34, p. 148 (1957).

Review of methods more frequently used. Only analyses of active products are considered.

(314) W. Czerwinski, "Determination of 2,6-naphthalenedisulfonic and 2,7-Naphthalenedisulfonic Acids Present in Mixtures," *Chemische Annalen* (Warsaw), Vol. 1, pp. 77-83 (1956); *Chemical Abstracts*, Vol. 51, p. 1777 (1957).

A modification of the benzidine method is used consisting in the double precipitation of the benzidine salt of the 2,6-acid which enables complete separation of the 2,7-acid.

(315) M. Dolezil, "Use of Fluorescent Indicators for the Determination of Small Amounts of Surface-Active Compounds.—I. Determination of Organic Sulfates and Sulfonates," *Chemische Listy*, Vol. 50, pp. 1588-1592 (1956); *Chemical Abstracts*, Vol. 50, p. 16570 (1956).

A titration method is described based on the quantitative reaction between anion-

active and cation-active compounds. Hexadecyltrimethylammonium bromide is used as a volumetric reagent and sodium eosinate as fluorescent indicator in ultraviolet light. Small amounts of electrolytes do not interfere, and larger amounts make the indicator change less conspicuous.

(316) A. B. Dutta, "Volumetric Method for the Assay of Sodium Sulfate," *Indian Journal Pharmacy*, Vol. 18, pp. 168-169 (1956); *Chemical Abstracts*, Vol. 50, p. 12749 (1956).

Millon's base (HNHg_2O) forms an insoluble compound with sodium sulfate in aqueous solution, liberating an equivalent amount of sodium hydroxide. A volumetric method, based on this reaction, is given.

(317) G. P. Edwards and M. E. Ginn, "Conditions in Activated Sludge Process during Frothing," *Industrial and Engineering Chemistry*, Vol. 48, pp. 246-250 (1956).

For analysis used cetyltrimethylammonium bromide (CTAB) two-phase titration method standardized by the Volhard method for bromides. With the purity of the CTAB known the results may be expressed in milliequivalents per liter.

(318) J. D. Fairing and F. R. Short, "Spectrophotometric Determination of Alkyl Benzene Sulfonate," *Analytical Chemistry*, Vol. 28, pp. 1827-1834 (1956).

Method based upon isolation of alkylbenzene sulfonate (ABS) by formation of complex of ABS and 1-methylheptylamine, extraction by chloroform, removal of chloroform and acid hydrolysis, extraction with amine-hexane mixture. An improved methylene blue determination is made spectrophotometrically. Procedure free from interferences and will measure less than 1 ppb in water, and 50 ppb or less in sewage. Full details of procedure given.

(319) A. V. Few and R. H. Ottewill, "A Spectrometric Method for the Determination of Cationic Detergents," *Journal of Colloid Science*, Vol. 11, pp. 34-38 (1956).

Method sensitive to concentrations of about 10^{-5} M. Analysis depends upon formation of complex with anionic dye and quantitative extraction of the complex into an organic phase in which the dye itself is insoluble. Intensity of color in the organic phase then directly proportional to the concentration of the detergent. Satisfactory analyses were obtained with octyltrimethyl-, dodecyltrimethyl-, cetyltrimethylammonium bromide, and dodecylpyridinium bromide. Method applicable at wide pH variations and salt concentrations. Used orange II dye.

(320) F. Franks, "Paper Chromatography with Continuous Change in Solvent Composition.—I. Separation of Fatty Acids.—II. Separation of Surface Active Agents," *Analyst*, Vol. 81, pp. 384-393 (1956).

Apparatus and method described for

separation of members of an homologous series on a short paper strip, avoiding diffusiveness of zones. Method applied to separation of mixtures of long-chain fatty acids obtained from acetone extractions of laundered articles. Applied to surface active agents with considerable success. Separation of commercial detergents into individual fractions indicated for *n*-alkyl sodium sulfates, alkylbenzene sulfonates, secondary-alkyl sulfates. Comparatively large concentrations of surface active agents could be seen as dark red spots when the strips were treated with rhodamine B after development, or as purple spots under ultraviolet. Low concentrations detected only under ultraviolet. For nonionics bromocresol green proved satisfactory.

(321) French Association for Standardization, "Sodium Salts of Sulfuric Esters of Secondary Alcohols," Pr T 73-010, March 1956.

Analyses given are: unsulfonated material by extraction with ethyl ether; active ingredient by ethanol extraction; sodium chloride.

(322) French Association for Standardization, "Sodium Salts of Sulfuric Esters of Amino Alcohols, Derived from Fatty Acids," Pr T 73-011, March 1956.

These commonly called sulfonated fatty amides are analyzed for: moisture by xylene distillation; total organic matter by isopropanol extraction, except in the presence of triethanolamine salts; total fatty acids by extraction of acid hydrolysate; insoluble in isopropanol by evaporation of aqueous residue after extraction.

(323) French Society for Standardization, "Sodium Salts of Sulfonated Alkane Derivatives—Test Methods," Pr T 73-012, March 1956.

Following determinations given: moisture by xylene method; unsulfonated material by ether extraction of aqueous methanol solution; total salts by evaporation of the extracted aqueous methanol solution; sodium chloride by silver nitrate and ammonium thiocyanate titration; sodium sulfate by extraction of active ingredient with amyl alcohol in acid medium; gravimetric barium sulfate; sodium sulfite by iodimetry; active ingredient by difference; total sulfur in the sulfonate by difference; organic chlorine by difference; total sodium by combustion. Indicated is the precision for each step.

(324) French Association for Standardization, "A.—Sodium Alkylaryl Sulfonates," March 1956.

Analyzed for: moisture by xylene distillation; unsulfonated matter by extraction of aqueous isopropanol solution with low-boiling gasoline; rapid method for active ingredient by isopropanol extraction, or more precise method by similar procedure; sodium sulfite by iodimetry; sodium sulfate as barium

sulfate; sodium chloride; total alkalinity.

(325) French Association for Standardization, "A₃—Sodium Salts of Sulfuric Esters of Primary Alcohols," March 1956.

Treatments for paste, liquid and powder products given. Analysis for: moisture by xylene distillation; unsulfonated materials by low-boiling gasoline extraction of aqueous methanolic solution; combined fatty alcohols by acid hydrolysis of unsulfonate-free liquor, extraction with ethyl ether, drying and weighing then treatment with methanol-potassium hydroxide solution, extract with low-boiling gasoline, dry and weigh; combined fatty matter containing sulfur; fatty acid content; total sulfate and mineral sulfate; chloride.

(326) S. Fumasoni, E. Mariani, and G. Torraca, "Electrochromatographic Separation of Cationic Surface Agents," *Chemistry and Industry*, pp. 69-70 (1956); *Journal, Am. Oil Chemists' Soc.*, Vol. 33, p. 432 (1956).

Straight chain alkylpyridinium chlorides separated and identified by electrochromatographic technic. Quantitative applications with a densitometer successfully applied to the determination of laurylpyridinium chloride in commercial products. Precision of 10 per cent in ranges of 100 to 300 μ g claimed.

(327) J. A. Grande and J. Beukenkamp, "Gradient Elution Chromatography of Phosphates," *Analytical Chemistry*, Vol. 28, pp. 1497-1498 (1956).

A mixture of ortho-, pyro-, tri-, trimeta-, and tetrametaphosphates was separated on an anion-exchange column, developed with a buffered potassium chloride solution which was increased from 0.1 *M* to 0.7 *M* during the run. The mean absolute error of the method is 0.5 per cent.

(328) H. L. Greenwald, G. L. Brown, and M. N. Fineman, "Hydrophile-Lipophile Character of Surface Active Agents and Oils by a Water Titration," *Analytical Chemistry*, Vol. 28, pp. 1693-1697 (1956).

Titration by water of a dioxane-benzene solution of an oil or surface active agent to a cloud endpoint can be used to determine a value related to the hydrophile-lipophile balance character. Applied largely to non-ionic agents, but combinations of anionics and nonionics tested.

(329) E. J. Griffith, "Analysis of Phosphorus Compounds. Rapid Hydrolysis of Condensed Phosphates in Volumetric Analyses," *Analytical Chemistry*, Vol. 28, pp. 525-526 (1956).

Time necessary for the hydrolysis of condensed phosphates to orthophosphate has been reduced from eight to less than 1 hr. To a 0.5 g sample, add 50 ml water, 10 ml hydrochloric acid and 1 g potassium chloride. Evaporate to dryness, dilute to 50 ml, and titrate.

(330) R. L. Hahn and R. Luckhaus, "An Excellent Reagent for the Colorimetric

Determination of Phosphate and Arsenate," *Zeitschrift analytische Chemie*, Vol. 149, pp. 172-177 (1956); *Chemical Abstracts*, Vol. 50, p. 7650 (1956).

A reagent of sodium molybdate and hydrazine sulfate is made. After the blue liquid has thoroughly cooled, it is diluted to volume. The solution containing a known low amount of phosphorus is then treated with a portion of the reagent, let stand, and after a half hour the solution is blue, and is measured at 640 m μ .

(331) M. H. Hashmi and C. F. Cullis, "Paper Chromatography of Fatty Acids and Halogenated Organic Acids," *Analytica Chimica Acta*, Vol. 14, pp. 336-338 (1956).

A number of the lower fatty acids separated on Whatman No. 4 filter paper with a developing solvent of 70 per cent propanol and 30 per cent concentrated aqueous ammonia. The R_f values are given. Acids located on paper by spraying with British Drug Houses, Ltd. Universal Indicator adjusted to pH 9.5 to give red spots on a blue background.

(332) J. W. Jenkins, "Determination of Soaps by Ion-Exchange Resins," *Journal, Am. Oil Chemists' Soc.*, Vol. 33, pp. 225-226 (1956).

Procedure given for preparing Dowex 50 resin, and for use on column. The passage of the soap through the column was followed by elution with ethanol. The effluent was titrated with standard caustic, and agreed closely with the theoretical amount of soap initially used. Directions given for use with commercial soaps.

(333) E. Karl-Kroupa, "Use of Paper Chromatography for Differential Analysis of Phosphate Mixtures," *Analytical Chemistry*, Vol. 28, pp. 1091-1097 (1956).

Several procedures given for differential analysis of condensed phosphates employing special solvents. After ascending development with the two solvents, the sheets are sprayed with a color developing reagent, and the blue zones representing the various salts is developed by irradiation under ultraviolet light. Qualitative evaluation can be made by comparison with standards, or semi-quantitative estimation can be made by eluting the spots with ammonia and water and sulfuric acid and water used in series. The phosphates are hydrolyzed and the phosphorus determined colorimetrically by the molybdenum blue reaction. The method can be applied to commercial phosphates, to built detergents without separating the active ingredient which does not interfere, and to surface waters. Detergent additives do not interfere.

(334) I. Kawashiro and H. Takeuchi, "Detection and Determination of Nonionic Surface-Active Agents in Orange Juice," *Eisei Shikenjo Hôkoku* (Tokyo), No. 74,

pp. 233-237 (1956); *Chemical Abstracts*, Vol. 51, p. 8320 (1957).

Limits of detection of polyoxyethylene 400, 1500, and Tween 20, and 80 studied with iodine solution, phosphomolybdic acid and barium chloride mixture, and the color reaction on adding *p*-hydroxybiphenyl to the precipitate formed by the addition of the phosphomolybdic-barium chloride reagent. Sensitivity given for each.

(335) H. Kroll and W. J. Lennon, "Lauric Acid-Diethanolamine Condensation Products," *Drug & Cosmetic Industry*, Vol. 79, pp. 186-187, 272-277 (1956).

An analytical procedure suitable for determining the major components of amide condensates is outlined with typical compositions tabulated.

Scheme consists of the following: (1) Free total amine, perchloric acid titration in glacial acetic acid, (2) Free fatty acid, titration in isopropanol with standard alkali, (3) Amine esters, solution in diethyl ether and this washed with 10 per cent sodium chloride. Evaporated ether and titrated in glacial acetic acid with perchloric acid, (4) Free diethanolamine, 1 minus 3, (5) Total fatty acid, hydrolysis with 6 N hydrochloric acid, extraction with ether, determination of fatty acids by titration, (6) Total amine, aqueous layer of 5 concentrated to dryness and diethanolamine hydrochloride determined in residue by potentiometric titration, (7) Combined fatty acid, 5 minus 2, (8) Combined diethanolamine, 6 minus 4, (9) Ratio combined amine to combined fatty acid, 8 divided by 7, (10) Dihydrogen ethyl lauramide and amide ester, 9 plus correction for combined fatty acid for amine ester, see 3.

(336) P. A. Lincoln and C. C. T. Chinnick, "Determination of Quaternary Ammonium Compounds as Phosphotungstates," *Analyst*, Vol. 81, pp. 100-104 (1956).

Precipitation of quaternary compounds quantitatively by phosphotungstic acid while precipitation as the ferrocyanide, ferricyanide, dichromate, and reineckate are not always stoichiometric particularly with cations of low equivalent weight.

Method depends upon precipitation and weighing of the ignited product. Possible to calculate amount of quaternary cation and also ionic weight of the quaternary salt. Data given with a variety of compounds to demonstrate value of method.

(337) W. A. Moore and R. A. Kolbeson, "Determination of Anionic Detergents in Surface Waters and Sewage with Methyl Green," *Analytical Chemistry*, Vol. 28, pp. 161-164 (1956).

Methyl green method devised to eliminate or lower interferences encountered in the methylene blue procedure. Interferences from thiocyanate, nitrate, and nitrite ions reduced greatly. Methyl green procedure

not affected by high concentrations of sulfate, chloride, sulfite, and phosphate. Recoveries of alkyl sulfate added to river and lake waters as well as to domestic sewage more consistent and more accurate with the methyl green procedure.

(338) P. Mukerjee, "Use of Ionic Dyes in the Analysis of Ionic Surfactants and Other Ionic Organic Compounds," *Analytical Chemistry*, Vol. 28, pp. 870-873 (1956).

Theory presented for a partition technic for the analysis of all classes of ionic surfactants and similar organic compounds. The dissolved sample is transferred to a volumetric flask, dye and acid or alkali added, plus an immiscible organic solvent and the whole shaken thoroughly. The absorbance of the organic layer is measured in a 1-cm cell in a Beckman Model DU spectrophotometer against the pure organic liquid as a standard. Use a wave length at the band maximum for the particular dye and solvent employed.

(339) Y. Nemoto, "Interaction Between Nonionic Surface-Active Agents and Dyes.—I. Color Change of Cyanine 5R by Nonionic Surfactants," *Nagoya Zasshi Kogyo Kenkyujo Hokoku*, No. 13, pp. 33-36 (1956); *Chemical Abstracts*, Vol. 51, p. 6337 (1957).

The color change of this apparently occurs below the critical micelle concentration of the lauryl alcohol ethylene oxide surfactant, but is dependent upon the concentration of the dye. The color change is also caused by addition of an anionic detergent. The original color is restored by heating and this process is reversible. This is suggested as a means for determination of nonionic surfactants.

(340) S. D. Nogare, "Qualitative and Semiquantitative Tests for Amine Hydrochlorides on Paper Chromatograms," *Analytical Chemistry*, Vol. 28, p. 903 (1956).

Previously prepared papers sprayed with silver nitrate solution, and black metallic silver is deposited on the spots where the amines have been developed, when exposed to ultraviolet. Semiquantitative determination accomplished by cutting out the spots from the paper, using the silver spot tests as guides, and the chlorides determined quantitatively. Blanks used as comparisons, and 88 per cent recovery claimed.

(341) T. V. Peters, Jr., and W. Riemann, III, "Analysis of Mixtures of the Condensed Phosphates by Ion-Exchange Chromatography.—II. Mixtures of Ortho-, Pyro-, Tri-, Tetra-, Trimeta-, and Tetrameta-Phosphates and Graham's Salt," *Analytica Chimica Acta*, Vol. 14, pp. 131-135 (1956).

Fractions of eluate from Dowex-1 column by means of various concentrations of potassium chloride buffered at pH 5.00 contain ortho-, pyro-, triphosphate and a mixture of tetra and tetrameta phosphates and trimeta-phosphate. Similar elution with eluants buffered at pH 9.00 yields eluate fractions containing mixture of ortho-, pyro-,

tri- and tetraphosphates, tetrametaphosphate and trimetaphosphate. Graham's salt retained on the column until treated with hydrochloric acid.

(342) R. Reiss, "A New Method for the Determination of Small Amounts of Quaternary Ammonium Compounds," *Arzneimittel-Forschung*, Vol. 6, pp. 77-79 (1956); *Chemical Abstracts*, Vol. 50, p. 897c (1956).

Method for assay consists in transforming them into the iodide in alkaline or acid medium, extraction with chloroform, oxidation of the iodide to free iodine with sodium nitrite, and colorimetric determination of the iodine. In the presence of alkaloids which are also transformed into iodides, extraction from alkaline medium effects a quantitative separation.

(343) A. A. Rosen, F. M. Middleton, and N. W. Taylor, "Identification of Anionic Synthetic Detergents in Foams and Surface Water," *Journal, Am. Water Works Assn.*, Vol. 48, pp. 1321-1330 (1956); *Chemical Abstracts*, Vol. 51, p. 2206 (1957).

Infrared spectroscopy applied to the detection of anionic surfactants in water and foams. Surfactants in water adsorbed on activated carbon but recovery was low, but sufficient for infrared identification. For isolation of surfactant without carbon sorption cetyltrimethylammonium bromide was added to aqueous surfactant to equivalence point using bromophenol blue indicator, extracted with chloroform and identified by infrared. Best results obtained with alkylbenzene sulfonate. Method not quantitative.

(344) M. J. Rosen and G. C. Goldfinger, "Detection of Surface-Active Alkylaryl Sulfonates by Alkaline Fusion and Formation of an Azo Dye," *Analytical Chemistry*, Vol. 28, pp. 1979-1982 (1956).

Surface active agents containing alkylaryl sulfonate group determined by the purple red or orange color produced when the phenol obtained from their fusion with potassium hydroxide reacts with diazotized dianisidine. Compounds containing nitro or halo substituents on the benzene ring give false negatives.

(345) E. M. Sallee, J. D. Fairing, *et al*, "Determination of Trace Amounts of Alkyl Benzenesulfonates in Water," *Analytical Chemistry*, Vol. 28, pp. 1822-1826 (1956).

Method depends upon concentration of alkylbenzene sulfonate (ABS) by absorption on activated carbon, desorption, removal of interfering materials by several purification steps, and final quantitative estimation by infrared spectrophotometry. The steps of the method are (1) adsorption of ABS on activated carbon (Nuchar C-190), (2) desorption with alkaline benzene-methanol (95 per cent effective), (3) acid hydrolysis, (4) petroleum ether wash, (5) amine extraction as a complex of 1-methylheptylamine, (6) infrared determination of carbon tetra-

chloride or carbon disulfide solution of complex at 9.6 and 9.9 μ . All details of method are given.

(346) B. Saville, "Some Novel Analytical Applications of Reactions Depending Upon the Positive Halogen Character in Alkane-Sulphonyl Halides," *Chemistry & Industry*, p. 660 (1956).

Thiols, alkylthiocyanates, alkylthiosulfates and others can be converted to corresponding alkane sulfonyl halides by aqueous halogen treatment. These halides reacted with cyanide ion to form cyanogen bromide which can be determined by Aldridge's method (Von Braun reaction) between pyridine and cyanogen bromide to produce an intermediate capable of condensing with benzidine to form a red dyestuff and this measured colorimetrically. Simple procedure is to add one volume of bromine water (saturated, diluted 10 times with water), to an aqueous solution containing 2×10^{-2} equivalents thiogen per ml, then add one volume 2 per cent phenol in water containing 5 per cent potassium bromide. Mix well and after 2 min add one volume 0.5 per cent sodium cyanide and 5 volumes Aldridge's reagent. Intensity of red colored solution read using green filter. Only interfering substances are cyanide and thiocyanate ions in the thiogen solution. Blank test indicated.

(347) E. J. Serfass and R. F. Muraca, "Analytical Determination of Trace Constituents in Metal Finishing Effluents.—XII. The Determination of Orthophosphates in Effluents," *Plating*, Vol. 43, pp. 356-358 (1956).

The method is the standard APHA modified to eliminate fluorine, arsenic, silica, and highly colored substances. The sample is fumed with nitric acid and sulfuric, reduced with potassium iodide, and repeatedly evaporated with hydrochloric acid. Insoluble substances are filtered off, ammonium phosphomolybdate is precipitated, then redissolved and reprecipitated, reduced to molybdenum blue, and measured.

(348) E. J. Serfass and R. F. Muraca, "Analytical Determination of Trace Constituents in Metal Finishing Effluents.—XIII. The Volumetric Determination of Sulfate in Effluents," *Plating*, Vol. 43, pp. 500-501 (1956).

The sample is precipitated with hydroxyl ion and silver ion to remove interfering ions. Fluoride ion removed by evaporation with hydrochloric acid, and organics are oxidized with hydrogen peroxide. The sulfate is then titrated with barium chloride by using tetrahydroxy quinone indicator. The method is useful in the 5 to 50 part per million range.

(349) W. T. Smith, Jr., W. F. Wagner, and J. M. Patterson, "Volumetric and Gravitric Analytical Methods for Organic Com-

pounds," *Analytical Chemistry*, Vol. 28, pp. 706-714 (1956).

Determination of quaternaries briefly described, particularly with reference to bromophenol blue indicator and reaction with cetyltrimethylammonium bromide in complex form.

(350) G. Spencer and V. Nield, "Chromatography of Naphthalene-sulfonic Acids," *Chemistry & Industry*, p. 922 (1956).

Ascending paper chromatography was used with a conventional solvent (*tert*-butanol-butanol-water, 40:30:30) to obtain an excellent separation of the mono- di- and trisulfonic acids. The zones were located with a quartz ultraviolet lamp. The zones could be eluted and absorbancy determined at a selected wavelength, thus giving a possible rapid, simple, and accurate method for the analyses of naphthalenesulfonic acids.

(351) A. B. Steele and L. D. Berger, Jr., "Characterization of Nonionic Detergents," *Soap*, Vol. 32, No. 2, pp. 48-50 (1956).

Characterization as to hydrophobic-hydrophilic balance and chemical nature of the hydrophobe by relating "cloud point" (temperature at which nonionic agent separates from dilute aqueous solutions as second liquid phase) with apparent density.

Values plotted fall into characteristic bands which serve to differentiate on the basis of the hydrophobe. Two nonionics having the same cloud point but different densities are recognized as having different hydrophobic components. Figure given illustrating tall oil esters, polyoxyalkylphenols, thioethers, and alcohol ethers.

(352) W. I. Stephen, "Analytical Chemistry," *Manufacturing Chemist*, Vol. 27, pp. 24-25, 29 (1956).

Short review of analytical developments in detergents.

(353) W. I. Stephen, "Analytical Chemistry," *Manufacturing Chemist*, Vol. 27, pp. 229-232 (1956).

Short discussion and review of latest developments in the analysis of detergents.

(354) Y. Takayama and N. Kanaki, "Determination of Nonionic Surface-Active Agents by Iodine-Starch Coloration," *Journal, Chemical Soc. (Japan), Industrial Chemistry Sect.*, Vol. 59, pp. 661-663 (1956); *Journal, Am. Oil Chemists' Soc.*, Vol. 34, p. 52 (1957).

Principle that amylose combined with polyoxyethylene type nonionics and residual amylose reacts with iodine. Absorption at 372 μ preferred to 590 μ . Coexistence of anionic and cationic agents inhibits determination.

(355) Y. Takayama and N. Kanaki, "Determination of Anionic and Cationic Surface-Active Agents by Iodine-Starch Coloration," *Journal Chemical Soc. (Japan), Industrial Chemistry Section*, Vol. 59, pp.

663-665 (1956); *Journal, Am. Oil Chemists' Soc.*, Vol. 34, p. 52 (1957).

Anionic agent broke combination of nonionic with amylose to set amylose free, which gave starch iodine coloration. Thus anionic determined by colorimetry of freed amylose at 610 $m\mu$. The cationic agent (cetylpyridinium chloride) combined with the hydroxyl of starch and the residual free starch reacted with iodine. Cationic agents determined by measurement of the yellow coloration at 372 $m\mu$.

(356) F. A. Vandenheuvel and D. R. Vatcher, "Partition Chromatography of Aliphatic Acids—Quantitative Resolution of Normal Chain Even Acids from C_{12} to C_{24} ," *Analytical Chemistry*, Vol. 28, pp. 838-845 (1956).

Saturated, normal-chain, even numbered acids separated and determined quantitatively on a dichloromethylsilane treated silicic acid chromatographic column with 2,2,4-trimethylpentane as stationary phase and aqueous methanol as eluting solvent. Device for automatically and continuously changing the composition of the eluting solvent and a semi-automatic, motor-driven microburet are described. Method applicable to the determination of the relative proportions of each carbon series represented in the fat sample and to the relative proportion of saturated to unsaturated acids in each carbon series.

(357) E. Wänninen, "Complexometric Determination of Sulfate Ion with Diethylenetriaminepentaacetic Acid," *Suomen Kemistilehti*, Vol. 29B, pp. 184-188 (1956); *Chemical Abstracts*, Vol. 51, p. 4878 (1957).

Sulfate is determined with 0.1 per cent error by adding excess standard barium salt solution and titrating the excess barium at pH 10 with standard solution of the trisodium salt of pentaacetic acid in the presence of the magnesium complex and Eriochrome Black T. Precipitated barium sulfate should be filtered off or its solubility decreased by addition of 15 volume per cent ethanol.

(357A) H. J. Weiser, Jr., "Determination of Triphosphate in Commercial Triphosphate and Detergents Built with Triphosphate," *Analytical Chemistry*, Vol. 28, pp. 477-481 (1956).

Tris(ethylenediamine) cobalt (III) ion has been shown as precipitant for triphosphate ion in acid solution. Tendency for pyrophosphate to coprecipitate reported, but compensation made by calibration curve. Precision and reproducibility good. Two hour elapsed time for analysis. Method applicable to commercial triphosphate and triphosphate built anionic detergents. Appreciable amounts of phosphate built glass interfere, but method gives warning when such interference takes place. Methods also recommended for determination of ortho-,

trimeta-, and total phosphate. Method free from interference from other phosphates, sulfate, and silicate good.

(358) J. B. Wilson, "Quaternary Ammonium Compounds. Detection of Residual Quaternary Ammonium Compounds on Shell Eggs," *Journal, Assn. Official Agricultural Chemists*, Vol. 39, pp. 659-662 (1956).

Detected by washing the egg with water, extracting the water with ethylene dichloride, using bromophenol blue and sodium carbonate solutions. A blue color in the lower layer indicates the presence of the quaternary. The minimum detectable amount is 4 γ . Difficult to completely extract the quaternary from the egg, but it does not penetrate through the shell.

(359) O. Yoda, "The Electrical Conductance of Surfactant Solutions.—III. Conductometric Titration of Cationic Surfactants," *Nippon Kagaku Zasshi*, Vol. 77, pp. 1087-1089 (1956); *Chemical Abstracts*, Vol. 51, p. 7805 (1957).

Confirmed that electrical conductivity shows a distinct minimum when the hydrochloride salts of hexadecylamine and of octadecylamine, dodecyltrimethylbenzylammonium chloride, and polyoxyethylene dodecyl ether are titrated with silver dodecyl sulfate. This phenomenon can be utilized as a means of quantitative analysis.

(360) K. Yoshimura and M. Morita, "Invert Soap.—III. Gravimetric and Colorimetric Estimation of Benzethonium Chloride," *Eisei Shikenjo Hôkoku*, No. 74, pp. 77-80 (1956); *Chemical Abstracts*, Vol. 51, p. 8457 (1957).

Details for gravimetric procedure given, using phosphotungstic acid and weighing the precipitate. For 25 mg of sample the range of error was ± 1 per cent. The colorimetric procedure utilized phosphomolybdic acid and transfer of the precipitate to a volumetric container, made up to volume with acetone, aged, and extinction coefficient measured at 730 $m\mu$. Error range with 0.6 mg is ± 5 per cent.

1957

(361) R. J. Bertolacini and J. E. Barney, II, "Colorimetric Determination of Sulfate with Barium Chloranilate," *Analytical Chemistry*, Vol. 29, pp. 281-283 (1957).

Colorimetric determination based on reaction of solid barium chloranilate with sulfate ion at pH 4 in 50 per cent ethanol solution to liberate highly chlorinated acid-chloranilate ion, absorption peak at 530 $m\mu$. Sensitivity about 2 ppm with precision and accuracy about 1 per cent. Interfering cations removed by ion exchange resins, and phosphate, bicarbonate, oxalate, chloride and nitrate do not interfere.

(362) D. C. Cullum, "The Determination of Sodium Laurate in Sodium N-Lauroyl-

sarcosinate," *Analyst*, Vol. 82, pp. 120-122 (1957).

Use of partition chromatography of the corresponding acids on kieselguhr impregnated with alkaline buffer, either being used as moving phase. Lauric acid is titrated with standard alkali.

(363) M. Dolezil and J. Bulandr, "Use of Fluorescent Indicators for the Determination of Small Amounts of Surface-Active Compounds.—II. Determination of Organic Quaternary Salts and Bases," *Chemické Listy*, Vol. 51, pp. 255-258 (1957); *Chemical Abstracts*, Vol. 51, p. 6425 (1957).

Determination of quaternary salts, bases, and amines with 0.01 *M* sodium lauryl sulfate. Use of eosin as an indicator in ultraviolet light enables sharp determination of equivalency and avoids the troublesome and nonprecise extraction procedure. Determination of the salts are independent of pH, but the amines must be titrated in acidic medium, pH 3.5 to 4.5.

(364) E. J. Griffith, "Thermogravimetric Analysis of Complex Mixtures of Hydrates," *Analytical Chemistry*, Vol. 29, pp. 198-202 (1957).

Chevenard thermobalance used to determine water present in each phase of complex mixtures of anhydrous salts and hydrates containing as many as six phases. Method depends upon proper rate of heating to give selective decomposition of phase with highest dissociation pressure occurring. With decomposition of phase with highest dissociation pressure, the substance with second highest begins to decompose, etc. Water present in each phase determined with average error of about 1 per cent. Hydrates analyzed were various tetrasodium pyrophosphate, sodium tripolyphosphate, soda ash, tetra borate, potassium carbonate, magnesium sulfate and cobalt chloride. Examples of pyrolysis curves given.

(365) J. C. Harris, "Analytical Test Methods for the Inorganic Portion of the Detergent Products," *Journal, Am. Oil Chemists' Soc.*, Vol. 34, pp. 199-203 (1957).

Review. Discussed under headings of physical examination, integrated analytical schemes, analysis of individual components. Describes current society activities in this field.

(366) H. Holness and W. R. Stone, "A Systematic Scheme of Semi-Micro Qualitative Analysis for Anionic Surface-Active Agents," *Analyst*, Vol. 82, pp. 166-176 (1957).

Scheme covers 21 types of anionic surfactants. Has not been tested for mixtures of these, but appears applicable. Tests are made on extracted active ingredient. Extraction first with 1-1 mixture of light petroleum ether and diethyl ether to remove inactive fatty material, fatty acids, perfumes, etc. Subsequently extracted with tertiary

butyl alcohol after making alkaline with sodium carbonate solution and saturation with sodium chloride. Tertiary butyl alcohol is said to be excellent because of low boiling point, and easy removal of traces of alcohol by addition of small quantity of *n*-hexane.

Sections consist of extraction of active material, type of surfactant present, materials containing nitrogen in the anion, those not containing nitrogen in the anion, and other specific tests for alkylphenylphenol sulfonates, esters of glycerol, ester groups, mono esters, amide linkage stability to 2 *N* acetic acid (difference between sulfonated castor oil and acyl isethionates), Guerbet test for benzene in the nucleus, benzene or naphthalene in the nucleus, differentiation between alkylidiphenyl sulfonates and alkylbenzene and alkyltoluene sulfonates, and oil and water-soluble petroleum sulfonates.

(367) R. K. Keerthuis and J. G. Kepler, "Gas-Chromatographic Analysis of Higher Fatty Acids; up to and Including Cerotic Acid," *Nature*, Vol. 179, pp. 731-732 (1957).

Higher fatty acids analyzed by means of their methyl esters. Details of carrier, immobile phases and technic described. A newly designed Katharometer capable of withstanding temperatures up to 350 C indicated as suitable for analysis of these esters.

(368) S. K. Love and L. L. Thatcher, "Water Analysis," *Analytical Chemistry*, Vol. 29, Part II, pp. 722-734 (1957).

A review of analytical methods for water analysis with sections on phosphates and silica, including synthetic detergents.

(369) J. D. Malkemus and J. D. Swan, "Analysis of Polyethylene Glycol Esters," *Journal, Am. Oil Chemists' Soc.*, Vol. 34, pp. 342-344 (1957).

Useful with fatty acid monoesters of polyethylene glycol and with fatty acid-ethylene oxide reaction products. Procedure consists of triple extraction at 95 to 100 C with saturated sodium chloride isolation followed by removal of water at reduced pressure and filtration through sintered glass filter. The dried clear product is analyzed for saponification and hydroxyl numbers. Original product consists of unreacted polyethylene glycol, mono- and di-ester and calculations given for estimation.

(370) R. A. Meiklejohn, R. J. Meyer, S. M. Bronovic, H. A. Schuette, and V. W. Meloch, "Characterization of Long-Chain Fatty Acids by Infrared Spectra," *Analytical Chemistry*, Vol. 29, pp. 329-334 (1957).

Use of potassium bromide disk technic to avoid melting of crystallized fatty acids and results in sharper and more reproducible spectra.

(371) O. B. Michelsen, "Photometric Determination of Phosphorus as Molybdovanadophosphoric Acid," *Analytical Chemistry*, Vol. 29, pp. 60-62 (1957).

Phosphate concentrations as low as 0.1 γ

P per ml can be determined by the molybdo-vanadophosphoric acid colorimetric method if very dilute reagents are used to reduce background absorption.

Measurements made at an absorption maximum at 315 m μ . Accuracy within ± 3 per cent when determining 0.1 γ P per ml, and better than ± 1 per cent at a concentration of 1 γ P per ml.

(372) O. T. Quimby and H. W. McCune, "Precipitation of Zinc Phosphates from Solutions of Sodium Ortho-, Pyro-, and Triphosphates," *Analytical Chemistry*, Vol. 29, pp. 248-253 (1957).

Radio-tagged phosphates used in evaluating zinc precipitate method as by Bell. Triphosphate interferes with pyro precipitation and results in both species being significantly in error. Possible to modify method for rapid determination of pyro in commercial triphosphate. pH change brings about nearly complete precipitation of ortho-, pyro-, and triphosphates but gives no promise of providing trustworthy indirect method for any one of the three species in their mixtures.

(373) J. Renault and J. A. Gautier, "A New Procedure for the Determination of Long-Chain Alkali Alkyl Sulfates Derived from Primary Alcohols," *Bulletin, Société Chimique* (France), pp. 208-211 (1957); *Chemical Abstracts*, Vol. 51, p. 7241 (1957).

Alkali alkyl sulfates are precipitated quantitatively with 0.2 *M* nickel chloride in 4 *N* ammonium hydroxide as $(\text{ROSO}_3)_2\text{Ni}(\text{NH}_3)_4$, if *R* is equal to or greater than $\text{C}_{10}\text{H}_{21}$. In the volumetric method the excess Ni^{+2} is titrated with 0.1 *M* ethylenediaminetetraacetic acid, with murexide indicator. In the gravimetric method, the precipitate is collected in a fritted-glass crucible, washed with a saturated solution of ethylenediamine tetraacetic acid in 10 *N* ammonium hydroxide, and dried at 35 C. Precision is 1 to 3 per cent. Sodium sulfate does not interfere, but sodium chloride, ammonium ion and calcium ion interfere slightly.

(374) D. U. Ross and E. W. Blank, "Error in the Determination of Active Ingredient in Detergent Products," *Journal, Am. Oil Chemists' Soc.*, Vol. 34, p. 70 (1957).

Partial solubility of sodium carbonate, sodium bicarbonate, sodium tetraborate and

sodium chloride in methyl and ethyl alcohols cause error, even though sodium chloride is corrected for. Comparison made by the alcohol soluble method, 1:1 acetone-ethyl ether soluble, and ultraviolet absorption. The acetone ether method agrees most closely with the ultraviolet method. Best procedure to continue as for alcohol soluble then to treat the alcohol soluble portion with the 1:1 acetone ethyl ether mixture. Addition of mixture direct to the detergent product not recommended because of limited solubility of active ingredient in the mixture. Notes that method was designed for soap-synthetic mixtures, and soap is insoluble in the 1:1 solvent mixture and useful in separating soap from surface active agents when both are present.

(375) W. G. Spangler, "Analytical Test Methods for Organic Detergents," *Journal, Am. Oil Chemists' Soc.*, Vol. 34, pp. 191-199 (1957).

Review of test methods. Discussion of sampling, general analysis, separation of mixtures and instrumental methods. Anionics are discussed under the headings of acid titrations, amine complexes, barium precipitate, action with cationic dyes or surfactants, nonionics are discussed under headings of physical and chemical methods, also discussed are methods for cationics. 110 References.

(376) H. J. Weiser, Jr., "The Determination of Pyrophosphate in Commercial Triphosphate," *Journal, Am. Oil Chemists' Soc.*, Vol. 34, pp. 124-127 (1957).

Modification of elution procedure of Peters and Rieman and use of an acid eluant for separation of ortho- and pyrophosphate. For determination of phosphate used the Martin and Doty colorimetric method. The chromatographic apparatus is described and the preparation of column given. Four separate eluents used to separate the ortho-, pyro-, triphosphate and trimetaphosphate (also tetrameta) fractions. Eluates are hydrolyzed and colorimetrically determined. Accuracy determined on known samples was good and precision or reproducibility given also was good. Method can be used on syndets for triphosphate determination if the active ingredient is removed by alcohol separation.