

## BIBLIOGRAPHICAL ABSTRACTS

1888 - 1956

1888

(1) T. H. Norton and J. H. Westenhoff, "XXXIV.—On the Amine Salts of Benzene-Sulfonic Acid," *American Chemical Journal*, Vol. 10, pp. 129–140 (1888).

Analysis in usual manner by oxidation to sulfuric acid.

1921

(2) A. W. C. Menzies and S. L. Wright, "The Application of a Differential Thermometer in Ebullioscopy," *Journal*, Am. Chem. Soc., Vol. 43, pp. 2314–2323 (1921).

Various systems described are benzil in benzene, anthracene in chloroform, and naphthalene in ether or in carbon tetrachloride.

1929

(3) W. Herbig, "Die Ole und Fette in der Textilindustrie," *Wissenschaftlicher Verlag*, Stuttgart, p. 405, 2nd edition (1929).

A method is proposed for the determination of organically combined sulfuric anhydride. The sample is hydrolyzed with hydrochloric acid, the sulfate then being determined in the water layer after extraction of the fat with ether. Inorganic sulfate is determined by washing the sample, dissolved in a solvent, with concentrated sodium chloride solution followed by the determination of alkali sulfate in the wash water.

1930

(4) L. Lematte, G. Boinat, E. Kahane, and M. Kahane, "On the Phospho- and Silicostates of Some Quaternary Bases, Analytical Applications," *Comptes rendus*, (Paris), Vol. 191, pp. 1130–1132 (1930).

Method based upon precipitation of the quaternary complexes followed by ignition and gravimetric determination of the residue.

1931

(5) D. Burton and G. F. Robertshaw, "Analysis of Sulfonated Oils," *Le Cuir technique*, Vol. 20, pp. 243–247, 287–293 (1931); *Chemical Abstracts*, Vol. 26, p. 613 (1932).

Qualitative test for sulfonated oil described. Methods outlined for determining acid index, pH, water, ash content, non-saponifiable content, total alkali, sulfuric anhydride organically combined and neutral sulfuric anhydride, free and combined fatty acids.

(6) W. Schaefer, "Determination of Naphtha Sulfonic Acid Soaps in Mixture of Sulfonic Acid Soaps and Mineral Oil," *Chemische Umschau auf dem Gebiete der Fette, Ole, Wachse and Harze*, Vol. 58, pp. 131–132 (1931); *Chemical Abstracts*, Vol. 25, p. 3861 (1931). Analysis for unsaponified material, fatty acids, and sulfonic acid soaps.

1932

(7) H. Jahn, "Organic  $\text{SO}_3$  in Sulfonated Oils and Related Compounds," *Siefenstetter-Zeitung*, Vol. 59, pp. 45–47 (1932).

Jahn considers the organic  $\text{SO}_3$  in oils as chemically combined. Total  $\text{SO}_3$  is determined by evaporating a 1 to 2 g sample with 2 ml 50 per cent NaOH, followed by ashing. The ash is dissolved in water, acidified with HCl, and precipitated with  $\text{BaCl}_2$ .

1933

(8) R. Hart, "Determination of Sulfuric Anhydride in Sulfonated Oils and Other Products," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 5, p. 413 (1933).

A method is given for the analysis of sulfonated oils, Igepon, Gardinol, etc.

(9) H. Jahn, "The Analysis of Aliphatic Alcohol Sulfonates," *Chemiker-Zeitung*, Vol. 57, pp. 383–384 (1933).

To determine total sulfuric acid in ester combination in an aliphatic alcohol sulfonate, heat 3 to 4 g sample on a water bath for 10 min with 100 ml of a mixture of equal parts of ethyl alcohol and trichlorethylene. Filter, evaporate filtrate, boil with 2:1 HCl, dilute with water, wash with ether, and determine  $\text{SO}_3$  by precipitation as barium sulfate.

(10) K. Lindner, A. Russe and A. Beyer, "The Determination of Fatty Alcohols in Their Sulfonation Products," *Fettchemische Umschau*, Vol. 40, pp. 93–96 (1933); *Chemical Abstracts*, Vol. 27, p. 3838 (1933).

Product refluxed with acid, any "true" sulfonic acid removed, and the fatty alcohol recovered by extraction with petroleum ether.

1934

(11) J. Hannay, "New Detergent and Finishing Agents," *Journal*, Soc. Dyers and Colorists, Vol. 50, pp. 273–282 (1934).

A scheme of analysis of the new agents is described.

(12) G. S. Hartley, "Effect of Long-Chain Salts on Indicators—Valence Type of Indicators and the Protein Error," *Transactions*, Faraday Soc., Vol. 30, pp. 444–450 (1934).

Effects of certain quaternaries on various indicators support theory that when colored ions of indicator are in both forms of like sign with the micelle, colloid probably has little effect. Other conclusions are drawn concerning direction of displacement.

(13) W. Kling and F. Puschel, "Determination of Alkyl Alcohol Sulfonates in Dilute Solution," *Melliand Textilberichte*, Vol. 15, pp. 21–23 (1934).

The alkyl alcohol sulfonate is precipitated with benzidine hydrochloride, filtered, dissolved in hot alcohol to separate it from inorganic sulfate, and the benzidine alkyl sulfate is titrated with NaOH. Results with a solution containing 0.1 to 0.2 g per liter were within  $\pm 5$  per cent.

1935

(14) F. M. Biffen and F. D. Snell, "Analytical Properties of Commercial Sulfated Alcohols," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 7, p. 234 (1935).

Alkyl sulfates may be identified qualitatively by their instability to heat, stability to cold acids, lathering properties when acidified, and hydrolysis on long boiling with acid to liberate  $\text{NaHSO}_4$ .

(15) K. Brass and A. Beyrodt, "Chemical Technical Investigations of Problems on Dyeing and Printing. VIII. Wetting Agents and Their Examination," *Monatschrift für Textil-Industrie*, Trade Issue II, Vol. 50, August, 1935, pp. 53-56.

A detailed analysis is given for a fatty alcohol sulfonate.

(16) K. Lindner, "Determination of 'True' High-Molecular Sulfonic Acids in Textile and Tanning Materials," *Chemiker-Zeitung*, Vol. 59, pp. 388-389 (1935).

Procedures are outlined for determining sulfonic acids in the presence of soaps, sulfate esters, and aliphatic alcohol sulfonates.

(17) C. J. Pedersen, "The Quantitative Estimation of Gardinol and Brilliant Avrol," *American Dyestuff Reporter*, Vol. 24, p. 137 (1935).

Fatty alcohol sulfates react with 1-amino-5-ethoxybenzothiazole to form insoluble salts. Soap does not interfere but sulfonates do. This reaction has been utilized to estimate the fatty alcohol sulfates turbidimetrically by comparison with standards.

(18) E. Swindells, "Analysis of Sulfonated Aliphatic Alcohols and Similar Condensation Products," *Dyer*, Vol. 73, pp. 120-121 (1935).

Herbig's method is useful in the analysis of sulfuric acid esters in which the sulfo group is attached to oxygen. Products in which the sulfo group is attached directly to a carbon atom cannot be split completely with acid. In such cases the undecomposed detergent is extracted with butanol.

1936

(19) A. Bohanes, "A Qualitative Procedure for Testing Detergents with Respect to New Materials Used in Washing and Cleaning," *Chemicky Obzor*, Vol. 11, pp. 155-158 (1936).

The author uses 1 per cent acetic acid, saturated calcium sulfate solution, neutral lead acetate, and barium chloride to identify the detergents as (1) soaps, (2) sulfonated neutral fatty acids, (3) sulfonated fatty alcohols, and (4) Igepons and Lamepons. Additional semi-specific tests are made for nitrogen, sulfur, etc.

(20) Z. E. Zin'kov, Ya. L. Danyushevskii, V. Reinshtein and G. M. Khomyakovskii, "Determination of Sulfonic Acids of Naph-

thalene," *Journal of Applied Chemistry* (USSR), Vol. 9, pp. 1997-2020 (1936); *Chemical Abstracts*, Vol. 31, p. 2552 (1937). Scheme for analysis of various sulfonic acids by separation with specific organic amine compounds and these titrated with sodium hydroxide.

1938

(21) Ralph Hart, "Determination of Sulfur in Surface-Active Agents," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 10, pp. 688-689 (1938).

This is a method based on the extraction of sulfated or sulfonated organic compounds from ether solution by means of concentrated salt solution.

(22) G. S. Hartley and D. F. Runnicles, "The Determination of the Size of Paraffin-Chain Salt Micelles from Diffusion Measurements," *Proceedings*, Royal Soc., Vol. A168, pp. 420-440 (1938).

Bromophenol blue used for titration end-point of anion against cation. Details of method and accuracy are given.

(23) A. Lottermoser and R. Steudel, "The Effect of Surface-Active Anions and Cations on Silver-Halogen Sols and Photographic Emulsions. II. Measurement of the Absorption of Surface-Active Alkyl Pyridine Cations by Silver Iodide," *Kolloid-Zeitung*, Vol. 83, pp. 37-51 (1938); *Chemical Abstracts*, Vol. 32, p. 4409 (1938).

Measurement of absorption by silver iodide sol. Possible use as a quantitative estimate of quaternaries.

(24) H. Rath and G. Schöniger, "A New Test for Eulan New and Eulan NK," *Kleppzig's Textil-Zeitung*, Vol. 41, p. 129 (1938); *Chemical Abstracts*, Vol. 32, p. 3620 (1938).

Extraction of Eulan New from wool by boiling in ammonia solution. Test depends upon green coloration with ammoniacal copper sulfate solution, and blue in its absence. For Eulan NK acetic acid treatment is used followed by starch addition and iodine solution drop by drop. A momentary blue color changing to yellow is positive while the blue color is permanent in a negative reaction.

1939

(25) D. Burton and G. F. Robertshaw, "Sulphated Oils and Allied Products—Their Chemistry and Analysis," A. Harvey, London (1939).

This book contains a number of analytical methods for various fractions of sulfated oils, such as free fatty acids, sulfated compounds, highly sulfated compounds and sulfonic acids, higher alcohols, mineral oil, and other neutral bodies.

(26) H. Gerber and J. Sporleder, "Determination of Sulfate in Highly Sulfonated Organic Substances such as Hydrocarbons, Fatty Acids, etc.," *Textilberichte*, Vol. 20, p. 212 (1939).

Total sulfate (A) is determined as  $\text{BaSO}_4$  by ashing and igniting 3 to 4 g of the product with 1:1  $\text{BaCO}_3$ - $\text{BaO}_2$  mixture, adding the residue to  $\text{H}_2\text{O}$  containing Br, adding 15 to 20 ml of 30 per cent HCl, boiling for 10 min, and filtering off the  $\text{BaSO}_4$ . Sulfate combined with inorganic radicals (B) is determined by extracting 10 g of the product with 100 g of BuOH, dissolving the residue in  $\text{H}_2\text{O}$ , and precipitating the sulfate as  $\text{BaSO}_4$  ( $\text{BaCl}_2 + \text{HCl}$ ). Sulfate combined with organic radicals = A-B. This method is also suitable for the analysis of Turkey-red oil.

(27) R. Griessbach, "The Preparation and Uses of New Exchange Absorbents Especially Resin Bases," *Angewandte Chemie*, Vol. 52, pp. 215-219 (1939).

Suggests the use of exchange resins for absorption of organic acids.

(28) R. Hart, "Determination of Active Ingredients and Fatty Matter in Surface-Active Agents," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 11 pp. 33-34 (1939).

A method is outlined which is suitable for the analysis of true sulfonic compounds and sulfuric acid esters. It consists of extracting the active ingredients with solvents over a concentrated salt solution, evaporating the solvent, heating the residue to constant weight, and determining the loss in weight upon ashing the residue. A procedure is also given for determining fatty matter in sulfuric acid esters, provided the organically combined  $\text{SO}_3$  is known.

(29) W. Kimura and H. Taniguti, "Preparation of Pure Sodium Salts of Higher Alkyl-Sulfates (and Their Analysis)," *Journal, Soc. Chem. Ind., Japan*, Vol. 42, Suppl. binding 89 (1939) *Chemical Abstracts*, Vol. 33, p. 5941 (1939).

Products purified by extraction, and analysis made by acid hydrolysis for varying periods of time depending upon the alkyl chain length.

(30) F. M. Scales and M. Kemp, "The Fundamental Principles of Chlorine Sterilization and a New and Positive Germicide," *Association Bulletin* (Intern. Assoc. Milk Dealers), Vol. 31, pp. 187-208 (1939).

Procedure described for the identification of alkylaryl sulfonate and for estimating quantity present. Based upon blue color formation when a solution of alkylbenzene sulfonate is added to a solution of o-tolidine and sodium hypochlorite.

(31) E. Schmidt, "Analysis of the Products of the Sulfonation of Fatty Alcohols," *Textil*, Vol. 4, pp. 650-653 (1939).

Sulfuric acid esters of a saturated fatty alcohol regenerate the pure alcohol on hydrolysis. Unsaturated alcohol sulfates give a mixture of pure alcohol and glycol because of the addition to the double bond. The

analysis is based on this reaction. The product is boiled with an acid and the mixture extracted with ether or ligroin. The extract is filtered and evaporated to constant weight. If sulfonic acids are present, the procedure is modified somewhat to remove any substances containing the sulfonic acid group from the ligroin or petroleum ether extract.

#### 1940

(32) R. Hart, "Cationic Surface-Active Agents of Trivalent Nitrogen Type," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 12, pp. 400-402 (1940).

A procedure is given for the analysis of products of the fatty acid amidoamine type. Determinations include: the nature of acid used to make the products soluble, nitrogen as alkalinity, total active ingredients or aminoamine, free fatty acids, total fatty acids, condensed amine, and uncondensed amine.

(33) A. J. Krog and C. G. Marshall, "Alkyl-Dimethylbenzylammonium Chloride for Sanitization of Eating Utensils," *American Journal of Public Health*, Vol. 30, pp. 341-348 (1940); *Chemical Abstracts*, Vol. 34, p. 3855 (1940).

Suggests that concentrations easily checked colorimetrically in the field by inspectors.

(34) K. Linsenmeyer, "Qualitative Differentiation of Wetting Agents and Detergents," *Melliand Textilberichte*, Vol. 21, p. 468 (1940).

Eight different classes of surface active agents are established on the basis of reaction with 5 per cent acetic acid, water of 20 deg hardness, and concentrated hydrochloric acid. The classes are: soaps, sulfonated oils (Monopole oil), highly sulfonated oils (Avirol), naphthalene sulfo acids (Nekal), fatty alcohol sulfates (Gardinol), fatty acid condensation products (Igepon), protein fatty acid combination products (Lamepon), and ethylene oxide condensation products (Igepal).

#### 1941

(35) H. Gnam, *Die Losungs und Weichmachungsmittel*, Stuttgart, p. 330 (1941).

This paper describes the quantitative determination of nonionic detergents by precipitation with ammonium cobalt thiocyanate.

(36) G. V. Shirolkar and K. Venkataraman, "Wetting Agents in Textile Processing, Part VIII. Purification and Analysis of Commercial Wetting Agents," *Journal, Indian Chem. Soc.*, Industrial Edition, Vol. 4, pp. 61-71 (1941).

Dioxane and ethyl acetate are especially suitable for extracting wetting agents from mixtures with inorganic salts. Detailed methods are described for determining moisture content, saponification value, nitrogen, sulfur, iodine number, and alkyl sulfate con-

tent. Per cent of alkyl sulfate was determined by the method of Brass and Beyrodt.

(37) Vizern and Guillot, "Analysis of Cleansing and Wetting Agents on the Basis of Sulfonated Fatty Acid Amides," *Annales de chimie analytique et de chimie appliquée*, Vol. 23, pp. 235-237 (1941).

Sulfonated fatty acid amides show a ratio of fatty substance to "active" substance of about 0.8. Active content is determined by extracting a dried sample with ethyl alcohol and evaporating the alcohol. Fatty substance is determined by saponifying a sample with HCl in the presence of NaCl, extracting the fatty acids with chloroform, evaporating off the chloroform, and weighing the residue.

1942

(38) E. Flatow, "Identification and Determination of Quaternary Ammonium Compounds in Antiseptics," *Pharmazeutische Zentralhalle*, Vol. 83, pp. 181-185 (1942); *Chemische Zentralhalle*, 1942, II, pp. 565-566; *Chemical Abstracts*, Vol. 37, p. 4857 (1943).

Qualitative colorimetric reaction. Gives quantitative test based upon dichromate treatment followed by thiosulfate in potassium iodide-sulfuric acid solution.

1942

(39) D. Geltner and W. W. Razim, "The Analysis of Cation-Active Softening Agents," *American Dyestuff Reporter*, Vol. 31, pp. 409-412 (1942).

A general scheme of analysis of commercial textile-softening agents is given. This involves (1) preliminary analysis to determine if the sample is of the cation-active type, (2) ashing, (3) alcohol extraction, (4) volatile matter determination, (5) hydrolysis, (6) isolation and identification of the amine.

(40) J. H. Percy and C. J. Arrowsmith, "Conductimetric Assay of Inorganic Salt in the Presence of Wetting Agents," *Industrial and Engineering Chemistry*, Analytical Edition Vol. 14, pp. 151-153 (1942).

Sodium sulfate content of solutions containing this salt and wetting agent is determined by comparing conductance of test solution with that of known mixtures of the two.

(41) S. Ram, "Analysis of Washing Powders Containing Sulfates and Sulfonates," *Analyst*, Vol. 67, p. 162 (1942).

The sample is ashed with excess  $\text{Na}_2\text{CO}_3$ , and total sulfate is determined in an aqueous solution of the melt. Another portion of the sample is extracted several times with saturated NaCl solution and alkali sulfate determined in the extract. The difference between the two results gives the sulfur present as alkyl sulfate or as sulfonated oil.

(42) K. E. Schwarz, H. J. Schroder and M. v. Steckelberg, "The Polarographic Determination of Surface-Active Substances in Water," *Zeitschrift für Elektrochemie*, Vol.

48, pp. 6-9 (1942); *Chemical Abstracts*, Vol. 37, p. 489 (1943).

Determination by means of a simple polarographic apparatus with dropping mercury electrode at a constant applied potential. The oxygen dissolved in the water is used as the reducible substance. A definite amount of potassium chloride is added so that at a definite ratio of concentration of oxygen a current maximum exists.

1943

(43) M. E. Auerbach, "Germicidal Quaternary Ammonium Salts in Dilute Solution. A Colorimetric Assay Method," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 492 (1943).

Quaternary ammonium salts are determined by extraction with ethylene dichloride of the colored complex formed with bromophenol or bromthymol blue. The intensity of the color is measured in a colorimeter and compared with that produced by standard solutions.

(44) J. C. Harris, "Colorimetric Determination of Alkyl Benzene Sulfonates," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 254 (1943).

A method is described for the estimation of alkyl benzene sodium sulfonate which is sensitive to one part per million of pure sulfonate. The analysis may be made visually or photometrically and is based upon the formation of a blue color when an alkyl benzene sulfonate is added to a solution of orthotolidine and sodium hypochlorite.

(45) A. Hintermaier and L. C. Kelber, "Determination of the Active Substances in Mersol and Mersol Solutions in the Form of Sodium Mersolate," *Fette und Seifen*, Vol. 50, pp. 413-415 (1943); *Chemical Abstracts*, Vol. 40, p. 2325 (1946).

A by-difference saponification method with, or without, correction for sodium sulfate or sodium sulfite.

(46) L. Palfray and G. Desseigne, "Analysis of Mixtures of Alkyl Sodium Sulfates," *Comptes rendus*, (Paris) Vol. 217, pp. 213-214 (1943).

Refluxing of sample in normal potassium hydroxide in phenyl methyl alcohol and titrated with hydrochloric acid in the presence of phenol red. More accurate and more rapid hydrolysis than using normal sulfuric acid or potassium hydroxide in alcohol.

(47) A. Parisot, "Analyses of Certain Materials Derived from Fats, Wetting Agents, Etc.," *Corps gras, savons*, Vol. 1, pp. 11-14 (1943); *Chemical Abstracts*, Vol. 40, p. 5269 (1946).

Procedures for system of analysis of materials derived from fats and various wetting agents. Several agents analyzed by elemental analysis and then hydrolyzed with identification of the resulting materials.

(48) M. v. Stackelberg and H. Schütz, "Quantitative Estimation of Surface Active Substances by Polarographic Adsorption Analysis," *Kolloid-Zeitschrift*, Vol. 105, pp. 20-26 (1943).

Surface-active substances decrease the maximum height of polarograms by adsorption at the mercury-solution interface. The decrease in height depends on the content of surfactant in solution.

#### 1944

(49) M. E. Auerbach, "Colorimetric Assay of Quaternary Ammonium Salts," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 16, p. 739 (1944).

Method described uses benzene solution of bromophenol blue indicator to overcome difficulty experienced with ethylene dichloride. Extraction made, then centrifuged and read photometrically at 600 m $\mu$ .

(50) D. Berkowitz and R. Bernstein, "Analysis of Soap-Synthetic Detergent Mixtures in Bar Form," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 16, p. 239 (1944).

Precise methods are given for determining soap, fatty matter, alcohol soluble and insoluble matter, and synthetic detergent in soap-synthetic detergent bars. Synthetic detergent is determined as the difference between total alcohol soluble matter and the sum of soap, fatty matter, and alcohol-soluble chlorides.

(51) A. Hintermaier, "Identification of Relatin and Tylose," *Fette und Seifen*, Vol. 51, pp. 367-368 (1944); *Chemical Abstracts*, Vol. 44, p. 6661 (1950).

Color reaction with alpha-naphthol sulfonic acid given only by carbohydrate-like compounds but does not distinguish between them. Microscopic examination under 10% caustic alkali containing methylene blue suggested.

(52) A. Hintermaier, "Determination of Relatin and Tylose in Laundering Compounds," *Fette und Seifen*, Vol. 51, pp. 368-369 (1944); *Chemical Abstracts*, Vol. 44, p. 6662 (1950).

Colorimetric method based upon alpha-naphtholsulfonic acid given in which alcohol-insoluble matter is used in the determination.

(53) J. M. Koch, "Analysis of Petroleum Oil-Soluble Sodium Sulfonates by Adsorption," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 16, pp. 25-28 (1944).

This is a method for the analysis of petroleum-oil-soluble sodium sulfonates by adsorption on a column of Attapulug clay from naphtha solution and the displacement of soap from the clay with methanol.

(53A) F. D. Snell and F. M. Biffen, *Commercial Methods of Analysis*, McGraw-Hill

Co. Inc., New York, N. Y., pp. 367-380 (1944).

Chapter on sulfated oils and related products. Gives qualitative and quantitative methods and these latter are related to sulfonated oils.

#### 1945

(54) A.B. Ashton and N. J. Stead, "Estimation of Wetting-Agent Concentration," *Metallurgia*, Vol. 32, pp. 53-56 (1945).

For control purposes, the concentration of wetting agent in a sulfuric acid-copper sulfate pickling solution is determined by measuring the surface tension by means of the capillary rise method. The method gives good results for concentrations of wetting agent below 0.30 per cent.

(55) G. Desseigne, "Hydrolysis of Fatty Alcohol Sulfates in Weak Acid," *Industries corps gras*, Vol. 1, pp. 136-139 (1945); *Chemical Abstracts*, Vol. 40, p. 4901 (1946).

Rates of hydrolysis at boil given for normal C2 to C12 sodium alkylsulfates. In normal hydrochloric acid the hydrolysis rate varies with chain length, and passes through a minimum at C5. Percentage hydrolysis and other pertinent data given. Presence of sodium sulfate considerably reduced the rate of hydrolysis while sodium chloride has much smaller effect.

(56) A. S. DuBois, "Methods for the Estimation of High Molecular Quaternary Ammonium Compounds," *American Dyestuff Reporter*, Vol. 34, pp. 245-246 (1945).

Methods are discussed for estimating these compounds in solution and on various surfaces.

(57) A. S. DuBois, "Argentimetric Estimation of High-Molecular Quaternary Ammonium Halides," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 17, pp. 745-746 (1945).

Uses a Borowski modification of Fajans' method for silver nitrate titration in 25 to 50 per cent aqueous isopropanol solution. Indicators are eosin and dichlorofluorescein which gave little difference in sample size, but this did affect bichromate titration.

(58) B. DuBose and V. B. Holland, "A Method for Determining Organically-Combined Sulfuric Anhydride in Surface-Active Products," *American Dyestuff Reporter*, Vol. 34, pp. 321-322 (1945).

Consists of extraction in ether, evaporation of ether, oxidation with nitric acid, and finally perchloric acid. The sulfate is precipitated as the barium salt and determined gravimetrically.

(59) A. Hintermaier, "Determination of Soaps and Sulfonates in Detergents," *Fette und Seifen*, Vol. 51, pp. 10-12 (1945).

General analytical methods are described and include: qualitative tests for fat acids, carbonates, and tylose; determination of unsaponifiable and sulfonate in fat acid-free

detergents; and determination of sulfonates in detergents containing fat acids. Shorter procedures are described for factory control tests.

(60) J. H. Jones, "General Colorimetric Method for Determination of Small Quantities of Sulfonated or Sulfated Surface-Active Compounds," *Journal, Assoc. Offic. Agr. Chemists*, Vol. 28, pp. 398-409 (1945).

This is a method based upon the formation of a color when methylene blue is mixed with an anionic detergent. Intensity of color is compared with a set of standards.

(61) J. M. Preston, "Tensimetric Analysis of Surface-Active Electrolytes," *Journal, Soc. Dyers and Colourists*, Vol. 61, pp. 165-166 (1945).

Anionic detergent is determined by titration with cationic agent. At end point a sharp rise occurs in the surface tension of the anionic detergent solution.

(62) F. Putnam and H. Neurath, "Interaction Between Proteins and Synthetic Detergents. II. Electrophoretic Analysis of Serum Albumin-Sodium Dodecyl Sulfate Mixtures," *Journal of Biological Chemistry*, Vol. 159, pp. 195-209 (1945); *Chemical Abstracts*, Vol. 39, p. 4633 (1945).

Distribution between complexes depends upon the detergent: protein weight ratio. Forces involved believed ionic in nature.

(63) S. E. Sheppard and A. L. Geddes, "Amphipathic Character of Proteins and Certain Lyophilic Colloids as Indicated by Absorption Spectra of Dyes," *Journal of Chemical Physics*, Vol. 13, pp. 63-65 (1945).

Suggested analysis of quaternaries by change in color from aqueous to organic solvent solutions.

(64) J. H. Skinkle, "A Systematic Scheme of Identification for Organic Finishing Agents," *American Dyestuff Reporter*, Vol. 35, pp. 449-452 (1945).

Organic finishing agents are extracted from fabrics by successive treatment with the following solvents: carbon tetrachloride, ethyl alcohol, water, dioxane, and dilute hydrochloric acid. The alcohol extract may contain soaps, sulfonated oils, sulfated alcohols, and cationic softeners. Suitable qualitative tests are made to determine which of these is present in the extract.

(65) A. Steigmann, "Experiments with a Cationic Wetting Agent," *Journal, Soc. Chem. Ind.*, Vol. 64, No. 3, p. 88 (1945).

A new technique for the preparation of spot test papers is described and the effect of Sapamine upon the isoelectric point of gelatins mentioned.

(66) P. Van Rysselberghe and J. M. McGee, "Polarographic Reduction Potentials of Quaternary Ammonium Ions," *Journal, Amer. Chem. Soc.*, Vol. 67, pp. 1039-1040 (1945).

Procedure given and the half-wave re-

duction potentials of a number of quaternaries given, but no striking regularity is apparent from the homologous series investigated.

1946

(67) E. W. Blank and A. Troy, "Determination of Borax in Soap and Synthetic Detergents," *Oil and Soap*, Vol. 23, pp. 50-55 (1946).

Based upon the precipitation of soluble silicates, carbonates, and orthophosphates by strontium chloride, whereas strontium metaborate is soluble in excess chloride. After removal of insoluble salts the metaborate is converted to boric acid and this titrated in the presence of mannitol.

(68) F. Brooks, E. D. Peters, and F. Lykken, "Analysis of Oil Soluble Petroleum Sulfonates. Extraction-Adsorption Method," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 18, pp. 544-547 (1946).

An extraction-adsorption method is described for determining mineral oil, sulfonic acid soap, water, carboxylic acid soap, free alkali, and inorganic salts in oil-soluble petroleum sulfonates.

(69) D. Burton and L. F. Byrne, "The Constitution of Sulfated Oils. I. Methods for Determining the  $-\text{COOH}$ ,  $-\text{OSO}_2\text{OH}$ , and  $-\text{SO}_3\text{H}$  Groups and Their Salts," *Journal, International Soc. Leather Trades Chem.*, Vol. 30, pp. 306-315 (1946).

Sulfated oils may contain carboxyl, alkyl sulfate, and sulfonic acid groups, free or partly neutralized with sodium, potassium, ammonium, or combinations. Methods given.

(70) M. L. Corrin, H. B. Kelvens and W. D. Harkin, "The Critical Concentration for the Formation of Micelles as Indicated by the Absorption Spectrum of a Cyanine Dye," *Journal of Chemical Physics*, Vol. 14, pp. 216-217 (1946).

Change in color of certain dyes as a means for analysis. Color change attributed to micelle formation.

(71) A. S. DuBois, "Testing the Quaternary Ammoniums," *Soap*, Vol. 22, No. 11, pp. 125, 127, 129, 131, 139, 141, 143 (1946).

Review listed by: Estimation of cations; bromophenol blue methods; Hartley-Runicles method; Auerbach method; Brooks and Hucker method; Botwright method; methods based on dyes; Amerse method; iodine-iodide methods; ferricyanide; potassium bichromate; estimation of anions; other tests. Seventeen references.

The Amerse method (Botwright) relies on solubilizing an insoluble salt, and disappearance of turbidity as the end-point. The indicator is comprised of N/250 solution of Erythrosin B.

(72) A. S. DuBois and D. D. Dibblee, "The Influence of Surface-Active Cationic Germicides on the Bacterial Population of

Milk," *Journal of Milk Technology*, Vol. 9, pp. 260-268 (1946).

Found the bromophenol blue test buffered at pH 4.6 unsuitable and preferred the Hartley and Runnicles method and modified this for rapid qualitative testing.

(73) E. F. Göbel, "Ionic Agents and Their Identification (Active Agents for the Reduction of Surface Tension)," *Revista Quimica Industrial* (Rio de Janeiro), Vol. 15, No. 173, pp. 16-23 (340-347) (1946); *Chemical Abstracts*, Vol. 41, p. 2593 (1947).

Methylene blue and uranin give coloration such that in dilutions of 1:2000 to 1:5000 coloration changes are utilized to distinguish between acyl amides and alkylaryl sulfonates.

(74) J. P. Harmon, "Wetting-Agent Concentration in Water Solution Determined by Drop-Number Method," *U. S. Bur. Mines, Circular No. 7351* (1946).

The concentration of a known wetting agent can be determined closely enough for control purposes in studies of the wetting of mine dusts by the drop-number method by use of a Traube stalagmometer. The apparatus and procedure are described.

(75) T. H. Harris, "Determination of Quaternary Ammonium Compounds in Fruit Juices," *Journal Assoc. Offic. Agr. Chemists*, Vol. 29, pp. 310-311 (1946).

A detailed method is given for the quantitative extraction and determination of quaternary ammonium compounds in fruit juices.

(76) T. V. Marron and J. Schifferli, "Direct Volumetric Determination of the Organic Sulfonate Content of Synthetic Detergents," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 18, p. 49 (1946).

The organic content is determined in samples of alkyl and alkaryl sulfonates by shaking with an aqueous solution of p-toluidine hydrochloride and extracting with carbon tetrachloride. The carbon tetrachloride takes up an amine sulfonate salt which is titrated in alcohol solution with standard alkali and metacresol purple as indicator. The organic content is calculated from "per cent organic" equivalent of the alkali solution standardized against a reference sample of known organic content.

(77) P. W. Morgan, "Determination of Ethers and Esters of Ethylene Glycol," *Industrial and Engineering Chemistry*, Anal. Edition, Vol. 18, pp. 500-504 (1946).

Oxyethylene content determined quantitatively by modification of the alkoxy method in which hydrogen iodide decomposes the polyglycol structure into ethyl iodide and ethylene, which are collected and determined volumetrically in standard solutions of silver nitrate and bromine respectively.

(78) O. E. Sundberg and G. L. Royer, "Microdetermination of Halogens and Sulfur," *Industrial and Engineering Chemistry*, Anal. Edition, Vol. 18, pp. 719-723 (1946).

Sulfur determination of inorganic alkali sulfonates or compounds containing C, H, O, S, N, alkali metals and halogens obtained by use of modified Grote combustion tube followed by volumetric titration. Oxidation products treated with standardized barium chloride solution using dipotassium rhodizonate indicator.

(79) J. B. Wilson, "Determination of Quaternary Ammonium Compounds in Foods," *Journal, Assoc. Offic. Agr. Chemists*, Vol. 29, p. 311 (1946).

This method is a modification of Auerbach's bromophenol blue-quaternary complex extraction procedure for the determination of quaternary ammonium salts in foods. An alternate procedure is suggested in which the quaternary base is precipitated as the salt of ferricyanide, followed by determination of excess ferricyanide by titration of the iodine liberated from iodides in acid solution.

#### 1947

(80) T. F. Baleskaya, "Identification of Alkylbenzene," *Journal of General Chemistry* (USSR), No. 2, pp. 489-496 (1947).

Analysis of binary mixtures of acyl amino derivatives of secondary butyl-benzene and isobutyl benzene.

(81) A. Brunzell, "Dodecyl Sodium Sulfate," *Svensk Farmaceutisk Tidskrift*, Vol. 51, pp. 101-106 (1947).

Dodecyl sodium sulfate is determined by titrating the acid liberated by acid hydrolysis. If sodium sulfate is present, the benzidine separation method of Kling and Puschel is used.

(82) E. L. Colichman, "Photocolorimetric Method for the Determination of Quaternary Ammonium Salts," *Analytical Chemistry*, Vol. 19, p. 430 (1947).

This procedure is a modification of Auerbach's method. It is based upon the formation of the bromophenol blue-quaternary complex in the presence of a known excess of the dye, followed by measurement of the color intensity without extraction of the complex. The method is less sensitive than Auerbach's but is claimed to be suitable for concentrations greater than 100 ppm.

(83) M. L. Corrin and W. D. Harkins, "Determination of the Critical Concentration for Micelle Formation in Solutions of Colloidal Electrolytes by the Spectral Change of a Dye," *Journal, Am. Chem. Soc.*, Vol. 69, pp. 679-683 (1947).

Change in color or reduction in fluorescence used in quantitative analysis by a titration method.

(84) S. R. Epton, "Method of Analysis of Certain Surface-Active Agents," *Nature*, Vol. 160, pp. 795-796 (1947).

Improved titrimetric method for analysis of anionics such as alkyl sulfates and sulfonates described. Equivalence point observed from change in intensity of methylene blue of hexadecylpyridinium bromide titrating agent. Endpoint not affected by excess inorganic salts or temperature variations. Substances having more than one ionizable group cannot be analyzed by this method.

(85) J. F. Gain and C. A. Lawrence, "A Rapid Method for Estimation of Use-Dilution Concentrations of Quaternary Ammonium Germicides," *Science*, Vol. 106, p. 525 (1947).

Data are presented to show that the method of Hartley and Runnicles can be used to determine the amount of available quaternary ammonium salt in the presence of organic matter.

(86) H. B. Goldstein, "Qualitative Analysis of Textile Processing Agents," *American Dyestuff Reporter*, Vol. 36, pp. 629-640 (1947).

This article is concerned with the qualitative analysis of the more common textile processing agents.

(87) O. B. Hager, E. M. Young, T. L. Flanagan, and H. B. Walker, "Determination of High Molecular Weight Quaternary Ammonium Compounds as the Triiodides," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 19, pp. 885-888 (1947).

Two qualitative and three quantitative methods described. Triiodides rapidly precipitated from aqueous solutions by addition of solution containing iodine and potassium iodide. These colored solids show marked change in color when more than the equivalent amount of iodine is provided. The triiodides can be isolated, dissolved in dilute alcohol and determined colorimetrically or by titration with sodium thiosulfate, but most accurate and precise method was potentiometric titration with a solution of iodine.

(88) L. F. Hoyt, "Qualitative Method for Detecting Surface-Active Agents," *Journal, Am. Oil Chemists' Soc.*, Vol. 24, pp. 54-56 (1947).

Small amounts of anionic, cationic, or nonionic surface-active agents can be detected by their solubilizing action on oil-soluble blue dye (National Brilliant Oil Blue BMA). As little as 0.002 per cent agent can thus be detected. Soap also gives a positive test but can be eliminated by acidification and extraction of the fatty acids released.

(89) J. M. Lambert, "Volumetric Analysis of Colloidal Electrolytes by Turbidity Titra-

tion," *Journal of Colloid Science*, Vol. 2, pp. 479-493 (1947).

The concentration of surface-active colloidal electrolyte in dilute aqueous solutions can be determined by titrating anionic agents with cationic agents (or vice versa) under such conditions that a colloidal precipitate is produced near the equivalence point. The end point of the titration is shown by the maximum turbidity of the solution.

(90) G. E. W. Sexton, "Oils, Fats and Surface-Active Agents," *Annual Reports on Progress in Chemistry* (Chem. Soc., London), Vol. 44, pp. 287-290 (1947); *Chemical Abstracts*, Vol. 43, p. 4595 (1949). Reviews with many references.

(91) C. B. Shaffer and F. H. Critchfield, "Solid Polyethylene Glycols (Carbowax Compounds)," *Analytical Chemistry*, Vol. 19, pp. 32-34 (1947).

A gravimetric and colorimetric method for determination of solid Carbowaxes based upon reaction of polyglycols with the heteropoly acids, silicotungstic, and phosphomolybdic. Details of procedures given.

(92) D. A. Shirreff, "Quantitative Analysis of Igepon T," *American Dyestuff Reporter*, Vol. 36, pp. 313-314 (1947).

The concentration of sodium salts of oleyl methyl tauride (Igepon T) is determined by precipitation with acidified benzidine hydrochloride solution, washing with water to remove acid and with petroleum ether to remove fatty acids (from soap). The washed precipitate is dissolved in aqueous alcohol and titrated with NaOH or evaporated to dryness and weighed. The procedure is also suitable for a number of other synthetic surface-active substances which contain sulfonic acid in the molecule, such as alkyl- and alkyl aryl sulfonates, and sulfated fatty alcohols.

(93) A. Steigmann, "New Analytical Reactions with Anionic, and Cationic Wetting Agents," *Journal, Soc. Chem. Ind. (London)*, Vol. 66, pp. 355-356 (1947); *Chemical Abstracts*, Vol. 42, p. 1525 (1948).

Orthosilicates and metaphosphates of cetyltrimethylammonium bromide or of Sappamine KW 200 per cent absorb dyes and reagents used for spot tests. Alavlon also precipitates mercury, gold, and platinum in slightly acidic solutions of their chlorides.

#### 1948

(94) J. F. Alicino, "Micro-Volumetric Method for Determination of Sulfur in Organic Compounds," *Analytical Chemistry*, Vol. 20, pp. 85-86 (1948).

Usual Pregl method followed with certain modifications. Used dipotassium rhodizonate and titrated with barium chloride. Reproducibility high, sharp endpoint, and no blank necessary.



(95) "Analysis of Soap Containing Synthetic Detergents," *Official and Tentative Methods of the American Oil Chemists' Society*, (1948).

Methods Db1 to Db11 include moisture and volatile matter; alcohol-soluble and alcohol-insoluble matter; free acid or free alkali; water insoluble; total alkalinity of alcohol insoluble; total anhydrous soap; chlorides; saponification value; alkaline silica; fatty matter; rosin.

(96) Association Official Agricultural Chemists, "Changes in Official and Tentative Methods of Analysis," *Journal*, Assn. Off. Agr. Chem., Vol. 31, pp. 105-108 (1948).

Described ferricyanide method for commercial preservatives in bottled beverages, containing fruit juices, beer, table sirup.

(97) T. Barr, J. Oliver, and W. V. Stubbing, "The Determination of Surface-Active Agents in Solution," *Journal*, Soc. Chem. Ind. (London), Vol. 67, pp. 45-48 (1948).

Anionic detergents can be titrated with a standard solution of a pure cationic agent such as cetyltrimethylammonium bromide. The titration is conducted using methylene blue as an indicator in the anionic solution which is layered over chloroform. At the end point, the chloroform layer takes on a blue color. Similarly, cationic agents can be titrated with an anionic solution using bromophenol blue as indicator.

(98) Canadian Government Specifications Board, "Schedule of Methods of Testing and Analysis of Soaps and Detergents," *Specification No. 2-GP-11*, Oct. 23, 1948.

In addition to soap methods listed are the following for synthetic detergents; alcohol insoluble; water insoluble; sugar; chlorides; moisture; sulfates; anhydrous, salt-free synthetic organic detergent; silicates; phosphates; carbonates.

(99) M. W. Cucci, "Practical Field Test for Quaternaries," *Soap*, Vol. 24, No. 8, pp. 129-131 (1948).

Use of chloroform, bromophenol blue, aqueous solution, and titration with quaternary solution in calibrated test tubes.

(100) A. S. DuBois, "Testing the Quaternary Ammoniums—A Review of 1946-1948," *Soap*, Vol. 24, No. 11, pp. 122-125 (1948).

Review by the various methods used; bromophenol blue, methods based on dyes, ferricyanide method, potassium bichromate, turbidity and by test papers. Seventeen references.

(101) S. R. Epton, "New Methods for the Rapid Titrimetric Analysis of Sodium Alkyl Sulfates and Related Compounds," *Transactions*, Faraday Soc., Vol. 44, p. 226 (1948).

Anionic detergents may be determined in solution by titration with cationic surfactant (cetyl pyridinium bromide) using acidified methylene blue as indicator. The titration is

run over a layer of chloroform. At the end point, the intensity of color in both layers is the same.

(102) T. L. Flanagan, Jr., T. J. Drennen, and G. R. Goetchius, "Determining Quaternary Content in Sanitizing Solutions," *Soap and Sanitary Chemicals*, Vol. 24, No. 4, pp. 163, 165 (1948).

A 0.2 per cent solution of a sodium salt of a condensed arylsulfonic acid (Tamol N) containing 0.02 per cent of blue dye (FD&C Brilliant Blue 1) is added dropwise to the sample solution. From the number of drops required to produce maximum turbidity, the concentration of quaternary salt is determined by comparison with a previously prepared table giving the number of drops required by known concentrations. Some quaternary salts do not react.

(103) H. H. Goldthorpe and J. Nixon, "Sewage Treatment. IV. Synthetic Detergents; Inhibition of Precipitation; Determination of Surface-Active Agents," *Journal and Proceedings*, Inst. Sewage Purif., 1948, Part 1, pp. 109-113; *Chemical Abstracts*, Vol. 46, p. 5752 (1952).

Literature of determination reviewed. If frothing of sewage occurs at pH3, upon shaking vigorously, a surface active agent is probably present.

(104) W. J. Harper, P. R. Elliker, and W. K. Moseley, "Quaternary Test: Sensitive Method for Testing Concentration of Quaternary Type Germicides," *Soap and Sanitary Chemicals*, Vol. 24, No. 2, pp. 159, 161, 163 (1948).

The method is based on the formation of a red precipitate upon addition of quaternary ammonium compounds to eosin yellow and subsequent titration to a colorless compound with an anionic agent such as Aerosol O.T. Amounts as low as 10 ppm can be detected even in the presence of 1 per cent of skim milk. A paper strip test based on the above method was also developed.

(105) A. Hintermaier, "The Analysis of Alkylsulfates and Nomenclature Suggestions," *Angewandte Chemie*, Vol. A60, pp. 158-159 (1948).

A general discussion is presented of various methods of analysis.

(106) J. A. van der Hoeve, "Analysis of Textile Auxiliary Products," *Recueil des Travaux Chimiques*, Vol. 67, pp. 649-664 (1948).

Tests are described for the qualitative analysis of washing, wetting, and retarding agents and emulsifiers. The tests are based upon color formation with specific reagents and precipitate formation on mixing cationic and anionic surface-active agents.

(107) J. M. Lambert and W. F. Busse, "Testing of Colloidal Solutions by Dye Solubilization," *Journal of Chemical Physics*, Vol. 16, pp. 847-848 (1948).

Solubilization isotherms suggest that the method of investigation might be suitable for analytical work.

(108) S. S. Marsden and J. W. McBain, "Oriented X-Ray Diffraction Patterns Produced by Hydrated Liquid Crystals," *Journal of Chemical Physics*, Vol. 16, p. 633 (1948); *Chemical Abstracts*, Vol. 42, p. 6597 (1948).

Data developed for several concentrations of sodium dodecyl sulfate.

(109) C. L. Ogg, C. O. Willits and F. J. Cooper, "Volumetric Determination of Small Amounts of Soluble Sulfates," *Analytical Chemistry*, Vol. 20, pp. 83-85 (1948).

Titration with barium chloride solution using dipotassium rhodizonate or tetrahydroquinone. Use of standard filters and continuous following of color change to give true endpoint.

(110) D. A. Shiraeff, "Quantitative Analysis of Synthetic Detergents. Extension of Igepon T Method to Various Agents Containing the Sulfonic Acid Group," *American Dyestuff Reporter*, Vol. 37, pp. 411-414 (1948).

The benzidine hydrochloride precipitation method for Igepon T can be used with some modifications for the analysis of any detergent the benzidine salt of which is insoluble in water, is crystalline, easily filterable, and soluble in a neutral organic solvent.

(111) A. Treffler, "Identification of Cleaner Ingredients," *Soap and Sanitary Chemicals*, Vol. 24, June, 1948, pp. 43-45, 159.

Qualitative and quantitative tests are presented for components of cleaning compounds as follows: water, soap, synthetic detergent, silicates, borax, phosphates, and carbonates.

#### 1949

(112) T. F. Boyd, J. M. MacQueen and I. Stacy, "X-Ray Diffraction Patterns for the Identification of Surface-Active Agents," *Analytical Chemistry*, Vol. 21, pp. 731-732 (1949).

Describe procedure and diffraction data for 19 crystalline surface active agents including alkylbenzene sulfonates, alkyl sulfosuccinates, alkyl sulfates and one nonionic.

(113) F. J. Cahn, "Testing of Solutions (for Quaternary Ammonium Compounds)," *U. S. Patent No. 2,471,861*, May 31, 1949.

The quaternary ammonium content of a solution may be determined by titration with an anionic substance using dichlorofluorescein as indicator. At the end point, the pink color due to the indicator disappears. The addition of an immiscible solvent, such as chloroform or ethylene dichloride, improves the end point.

(114) P. Desnuelle and O. Micaelli, "The Analysis of Sulfated Fat Acid-Condensation Products," *Oléagineux*, Vol. 4, pp. 353-357 (1949).

A method of analysis is given which determines total fat acids, free fat acids, fat acid amides, fat acid esters, sulfated fat acid amides, sulfated fat acid esters, and sulfated fat acids.

(115) R. M. Kelley and E. W. Blank, "Determination of Bicarbonate in Soap Products," *Journal, Am. Oil Chem. Soc.*, Vol. 26, pp. 685-687 (1949).

Method consists of heating sample containing bicarbonate and carbonate to 200 C for half-hour in a dry flask connected to the usual carbon dioxide train. Sodium carbonate is determined in the usual way.

(116) H. P. Lundgren, "Application of Detergents to Protein Fiber Preparation," pp. 317-324, *Advances in Protein Chemistry*, Vol. V, Academic Press, Inc., New York, 1949.

Discussion of protein-detergent complexes, indicating that reaction is on basic groups of the protein. Both anionic and cationic agents are effective and their formation is on essentially quantitative basis. Such complexes are reduced to protein by the extraction of the detergent with 60 to 70 per cent acetone, but complete only in the presence of salt. Limiting proportion of one detergent ion for each 43 equivalents of basic groups present in egg albumin molecule.

(117) J. Oliver and C. Preston, "Estimation of Nonionic Detergents," *Nature*, Vol. 164, pp. 242-243 (1949).

Polyethylene glycol detergents are treated with barium chloride to remove sulfates, and then with hydrochloric and phosphomolybdic acids. The precipitate formed is flocculated, filtered, washed, dried, and weighed. A comparison with known amounts of the same detergent is necessary.

(118) A. Parisot, "Rapid Industrial Analysis of Soaps and Commercial Soap-like Products," *Olearia*, Vol. 3, pp. 13-20 (1949); *Chemical Abstracts*, Vol. 44, p. 2263 (1950).

Method based upon solubility of soaps and sulfonated products in aqueous-alcoholic potassium chloride solution and the solubility of other constituents in petroleum ether. Method given in detail for use with saponaceous creams, beauty creams, dentifrice, liquid soaps, shampoos, etc.

(119) M. Robinet and N. Chevron, "Analysis of Sulfuric acid esters by the Benzidine Method," *Bulletin des sociétés chimiques Belges*, Vol. 58, pp. 324-330 (1949).

The benzidine method of Kling and Puschel gives high results for sulfuric anhydride in sulfuric acid esters. The error is due to the presence of free fatty acids or their benzidine salts in the precipitate of benzidine alkylsulfate. If the precipitate is washed with ethyl ether, the fatty acids are removed and better results obtained.

(120) M. R. J. Salton and A. E. Alexander, "Estimation of Soaps and Ionized Detergents," *Research* (London), Vol. 2, pp. 247-248 (1949).

Pinacyanol bromide is used as an indicator in titrations of anionic with cationic soaps. Method is stated to be rapid and accurate to 1 per cent.

(121) K. G. Weckel, P. Elliker, F. Barber, C. K. Johns, C. Jensen, "Report of the Common Applied Laboratory Methods," *Journal of Milk and Food Technology*, Vol. 12, p. 280 (1949); *Chemical Abstracts*, Vol. 45, p. 1261 (1951).

Tests for quaternary ammonium compounds discussed.

(122) P. W. O. Wijga, "Determination of Surface-Active Materials in Dilute Solutions," *Chemisch Weekblad*, Vol. 45, pp. 477-480 (1949).

A description of some previously published methods is given. A method is also described for determining anion active materials by titration with cationic agent in the presence of Pontamine Fast Red 8 BNL as indicator.

#### 1950

(123) J. Bergeron, R. Derenemesnil, J. Ripert, and G. Monier, "Analysis of Anion Active Derivatives," *Bulletin mensuel d'information ITERG* (Inst. Tech. Etudes et Recherches Corps Gras), Vol. 4, pp. 118-130 (1950).

Twenty-four types of anion-active detergents are classified into three groups. Detailed directions are given for their systematic analysis.

(124) A. L. Draper and W. O. Milligan, "Karl Fischer Titration of Solid Soaps and Detergents," *Texas Journal of Science*, Vol. 2, pp. 209-212 (1950); *Chemical Abstracts*, Vol. 44, p. 9169 (1950).

Comparison by three methods: heating in air at constant temperature to constant weight, vacuum drying at low temperature, and the Karl Fischer method. Latter method concluded as most accurate.

(125) H. C. Evans, "Determination of Anionic Synthetic Detergents in Sewage," *Journal, Soc. Chem. Ind.* (London), Vol. 69, Suppl. No. 2, S76-80 (1950); *Chemical Abstracts*, Vol. 46, p. 4708 (1952).

Concentration of alkyl or aryl sulfates or sulfonates can be determined by measuring light absorption at 6520 Å of the chloroform extract of the methylene blue salts at pH 3.25 and 0.7, plotting concentration against pH units and extrapolating the curve and reading concentration indicated at the intersection at pH 2.0. No interference by aluminum sulfate, ferric chloride, soap, CNS, nitrate, chloride, or inorganic ions and compounds from urine.

(126) J. A. Gilby and H. W. Hodgson, "A Scheme of Analysis for Commercial Detergents. I," *Manufacturing Chemist*, Vol. 21, pp. 371-376 (1950).

A review is presented of the principal methods of analysis for anionic, nonionic, and cationic detergents. A modified Linsensmeyer method was developed by which the active ingredient of a detergent could be classified as soap, sulfonated oil, protein fatty acid condensation product, ethylene oxide condensation product, alkyl sulfate, alkyl sulfate monoglyceride, fatty acid ester of sulfoacetamide, alkyl aryl polyether sulfonate, and alkyl aryl sulfonate.

(127) J. A. Gilby and H. W. Hodgson, "A Scheme of Analysis for Commercial Detergents. II," *Manufacturing Chemist*, Vol. 21, pp. 423-426 (1950).

Methods given in part I are expanded to include specific procedures for both qualitative and quantitative tests for various types of detergents.

(128) A. Hintermaier, "Determination of Active Ingredients in Laundering Compounds," *Fette und Seifen*, Vol. 52, pp. 689-693 (1950).

Methods are described for determining mono- and disulfonates as well as lauryl pyridinium compounds in laundry compounds.

(129) F. Karush and M. Sonenberg, "Long Chain Alkyl Sulfates. Colorimetric Determination of Dilute Solutions," *Analytical Chemistry*, Vol. 22, pp. 175-177 (1950).

Rosaniline or pararosaniline forms a colored complex with alcohol sulfate. The complex is extracted with a mixture of chloroform and ethyl acetate and its absorption determined colorimetrically. Solutions as dilute as  $5 \times 10^{-6}$  molar can be analyzed.

(130) H. B. Klevens, "Analysis of Colloidal Electrolytes by Dye Titration," *Analytical Chemistry*, Vol. 22, pp. 1141-1144 (1950).

Soap and anionic synthetic detergents may be determined by titration with a cationic dyestuff solution, since the color or fluorescence intensity changes markedly in the region of critical micelle concentration. Similarly, cationic agents may be titrated with anionic dyes. The methods are suitable for mixtures of the agents with hydrocarbons, electrolytes, etc.

(131) C. A. Lawrence, *Surface-Active Quaternary Ammonium Germicides*, pp. 40-48, Academic Press Inc., New York City, (1950).

Review of assay methods in considerable detail, with a good bibliography. Describes P. G. Bartlett method in which standardization with bichromate is used, and a turbidimetric method is followed by clarifying the solution with nonionic detergent.

(132) W. F. Lester and R. D. Raybould, "Sewage Analysis. I. Determination of Anion-Active Detergents in Sewage and Effluents. II. Analysis of Sewage Sludge," *Journal and Proceedings, Inst. Sewage Purif.*, 1950, pp. 392-398, 398-402; *Chemical Abstracts*, Vol. 47, p. 4018 (1953).

The single extraction method not specific for Teepol. Inaccuracies introduced by the presence of sewage organic matter, the thiocyanate and nitrate ions, but these corrected for. Two extractions at low pH appear to eliminate the effect of sewage sludge, but not the two ions mentioned.

(133) W. J. Miller, "A Colorimetric Method for Water-Soluble Silicates in Detergents," *Journal, Am. Oil Chemists' Soc.*, Vol. 27, pp. 348-350 (1950).

Water soluble silica determined rapidly and accurately by measuring the color produced after development of the silicomolybdic acid complex. Citric acid used to eliminate interference from phosphate builders. Semi-quantitative estimation between limits may be carried out even more rapidly by using picric acid standards and visual comparator.

(134) J. R. Munger, R. W. Nippler and R. S. Ingols, "Volumetric Determination of Sulfate Ion," *Analytical Chemistry*, Vol. 22, pp. 1455-1456 (1950).

Volumetric titration with standardized barium chloride solution with colorimetric endpoint using Eriochromeschwartz T indicator in the presence of standard disodium dihydrogen ethylenediamine tetraacetate solution.

(135) R. Neu, "Differentiation of Cellulose Ethers and Cellulose Ether Glycolic Acids," *Fette und Seifen*, Vol. 52, p. 23 (1950); *Chemical Abstracts*, Vol. 44, p. 7531 (1950).

Zephrol (dimethylalkylbenzylammonium chloride) solution yields white precipitate with glycolic acids but cellulose ethers do not react. Reaction very sensitive and alkali salts do not interfere.

(136) R. Neu, "Analysis of Washing and Cleansing Agents. III. Determination of Cellulose Ethers and Cellulose Glycolic Acid," *Seifen-Ole-Fette-Wachse*, Vol. 76, pp. 65-68 (1950); *Chemical Abstracts*, Vol. 44, p. 6173 (1950).

Hydrolysis of products to materials to reduce Fehling solution and the cuprous oxide assayed. Useful in the presence of soda, silica, sodium orthophosphate, and calcium carbonate.

(137) R. Neu, "Analysis of Washing and Cleaning Agents. V. Identification and Analysis of Laundering Raw Materials Using Organic Base Exchangers," *Fette und Seifen*, Vol. 52, pp. 349-352 (1950); *Chemical Abstracts*, Vol. 44, p. 11190 (1950).

Organic ion-exchange resin (Wolfatit-K) can remove alkali cations from synthetic de-

tergents in exchange for hydrogen and the acid solution then titrated to give acid numbers and equivalent weights.

(138) J. C. L. Resuggan, "Evaluation of Detergents," *Milk Industry*, Vol. 31, No. 1, pp. 62-66, No. 2, pp. 67-71 (1950); *Chemical Abstracts*, Vol. 46, p. 7342 (1952).

Methods briefly discussed for the chemical determination of constituents of detergents.

(139) G. Reutenauer, "The Industry of Detergents in Germany," *Bulletin mensuel d'information ITERG* (Inst. Tech. Etudes et Recherches Corps Gras), Vol. 4, pp. 61-74 (1950).

Methods are given for the analysis of sulfated alcohols. Analysis of Mersol, Mersolat, Nekal, and Emulphor is described.

(140) G. Reutenauer, "Analysis of Sulfated Alkyl Aryls. I," *Bulletin mensuel d'information ITERG*, Vol. 4, pp. 197-199 (1950).

Determinations of moisture in alkyl aryl detergents were made by four methods: (1) azeotropic distillation with benzene, (2) azeotropic distillation with toluene, (3) drying at 95 C, and (4) drying under infrared radiation. Methods (1) and (2) are recommended as sufficiently exact and rapid.

II. *Ibid.*, pp. 199-204.

Nonsulfonated hydrocarbon was determined by extraction with petroleum ether (bp 37 to 75 C). Some products are appreciably soluble in petroleum ether. Sulfonated matter was determined by three different methods. (1) treatment with benzedine or p-toluidine and isolation of the addition products thus formed; (2) solution in ethyl or isopropyl alcohol, extraction of nonsulfonated hydrocarbon with petroleum ether, and evaporation of an aliquot part of the alcoholic solution; (3) liberation of the sulfonic acid with HCl and two consecutive extractions with benzene and amyl alcohol. Method (2) is the most satisfactory.

(141) G. Reutenauer, "Determination of Inorganic Matter in Technical Alkyl Aryl Sulfonates," *Bulletin mensuel d'information ITERG*, Vol. 4, pp. 255-257 (1950).

Sodium carbonate, bicarbonate, and sulfate were determined in synthetic detergents by standard methods (titration with HCl for the carbonates, precipitation with BaCl<sub>2</sub> for the sulfate). Reliable results were obtained.

(142) P. Schain, "Single-Solution Detergent-Method for Determining Butter-fat in Milk," *Proceedings*, 43rd Ann. Meeting Milk Ind. Foundation, Lab. Sect., 1950, pp. 12-17; *Chemical Abstracts*, Vol. 46, p. 5739 (1952).

Materials entering into the reagent consist of tetradecyldesoxypolyethylene glycol, dioctyl sodium phosphate, Oil Red O, a fat soluble dye, and methanol. Method said to be simpler than existing methods.

(143) N. M. Schuck and W. H. Koester, "Adaptation of the Volumetric-Evolution Method for Carbonates in Soaps and Synthetic Detergents," *Journal, Am. Oil Chem. Soc.*, Vol. 27, pp. 321-323 (1950).

Modifications make possible adaptation of the method to alkylbenzene sulfonates or similar types of detergents which cannot be hydrolyzed. Alcohol instead of aqueous solutions used to eliminate foaming. Electric mantle or carbon dioxide-free stream of steam used to prevent "bumping."

(144) D. A. Shiraeff, "Basis for the Analysis of Quaternary Ammonium Germicides and Detergents of Ethylene Oxide Condensation Type, and Mixtures Thereof," *Proceedings, Chem. Specialties Mfrs. Assoc.*, June, 1950, pp. 117-122.

A method is presented for the analysis of quaternary ammonium germicides. The method is based on the fact that soap, alkyl aryl sulfonate, and other anionic materials form a turbidity with the cationic germicide. When Igepal (nonionic detergent) is present, a different method is used.

(145) K. Swanston and R. C. Palmer, "The Estimation of Anionic Detergents in Solution by Hartley Titrations," *Journal, Soc. Dyers and Colourists*, Vol. 66, pp. 630-632 (1950).

Anionic detergents are determined by titration with cationic agent under a variety of conditions. The procedure is claimed to be at least as accurate as other published methods but more rapid and convenient.

(146) A. Taylor, "Primary Fatty Alcohol Sulfates," *Pharmaceutical Journal*, Vol. 165, pp. 284-285 (1950); *Chemical Abstracts*, Vol. 45, p. 3122 (1951).

A review with a discussion of analytical methods used by manufacturers.

(147) "Determination of Carboxymethyl-cellulose," *TGA Method No. 42*, Toilet Goods Assoc., Inc., New York City (March 1, 1950). This made by isolation of the copper salt, and the degree of substitution ascertained by weighing and drying the precipitate followed by analysis for copper. Purity calculated from degree of substitution.

(148) I. Vavrch, "Determination of Surface-Active Substances in Refined Sugar. Classification by the Polarographic Method," *Analytical Chemistry*, Vol. 22, pp. 930-932 (1950).

Determined by the effect of height of the oxygen maximum in 0.002 molar potassium sulfate.

(149) G. R. Wallin, "Colorimetric Method for Determining a Surface-Active Agent," *Analytical Chemistry*, Vol. 22, pp. 616-617 (1950).

In acid solution, basic fuchsin reacts with sulfated and sulfonated wetting agents to

produce a magenta-colored complex. The complexes are extracted with chloroform and their color intensities determined photometrically.

(150) E. Weber, "Identification of Tylose in Washing Compounds and Identification of Surface-Active Substances," *Fette und Seifen*, Vol. 52, pp. 477-478 (1950); *Chemical Abstracts*, Vol. 45, p. 2241 (1951).

Wurzschmitt's salicyl aldehyde test for surface active agents also used on tylose and other polysaccharides. Colors given under daylight, ultraviolet, and chloroform extract for a number of agents.

(151) B. Wurzschmitt, "Systematic and Qualitative Examination of Substances with Capillary Activity," *Zeitschrift fur analytische Chemie*, Vol. 130, pp. 105-185 (1950).

Procedures are given for identifying the inorganic constituents of detergent products, such as alkali, carbonate, phosphate, or sulfate. A complete analytical scheme is described for classifying the organic detergent. Emphasis is placed on precipitation reactions with various dyes. Details of an apparatus for determining the saponifiability of the detergent are presented.

(152) B. Wurzschmitt, "The Balance Sheet of Oxygen, Sulfur, Nitrogen, and Alkali—An Important Aid in Detergent Analysis," *Chemiker-Zeitung*, Vol. 74, pp. 16-20 (1950).

The original dried product is analyzed for C, H, O, N, S, halogen, and alkali. These plus additional tests for functional groups and a molecular weight determination serve to identify the detergent. Examples are given in detail for the analysis of condensates of ethylene oxide and fatty alcohols, fatty acid with protein-degradation products, sulfonated compounds, fatty acid amides, and amine compounds.

(153) B. Wurzschmitt, "Detection of Capillary-Active Substances in Washing and Cleaning Materials," *Angewandte Chemie*, Vol. 62, p. 40 (1950).

The detergent is extracted from the compound with methyl alcohol. The extract is tested as follows: aluminum acetate precipitates anion-active substances, silicotungstic acid precipitates nonionics. Nekals, alkyl naphthols, and alkyl naphthalenes, after reaction with  $H_2SO_4$  or  $HNO_3$ , fluoresce under ultraviolet light.

(154) B. Wurzschmitt, *Systemik und Qualitative Untersuchung Capillarkativer Substanzen*, Berlin; Springer-Verlag, 81 pages, (1950).

1951

(155) H. A. Affsprung, N. A. Barnes and H. A. Potratz, "Spectrophotometric Determination of Cobalt as Tetraphenylarsonium Cobaltothiocyanate," *Analytical Chemistry*, Vol. 23, pp. 1680-1683 (1951).

Separation of cobalt from aqueous solutions of inorganic materials by chloroform extraction of the tetraphenylarsonium cobaltothiocyanate.

(156) J. Balthazar, "Qualitative and Quantitative Analysis of Synthetic Detergents," *Ingenieur chimiste*, Vol. 32, No. 182, pp. 169-196; Vol. 33, No. 183, pp. 3-16 (1951).

Qualitative tests are described for determining whether a detergent is anionic, cationic, or nonionic. Both qualitative and quantitative tests are described in detail for determining the character and composition of the detergent. Sample analyses are shown and a table is given outlining the various procedures for identification of the detergents.

(157) H. C. Black, "Determination of Sodium Carboxymethyl Cellulose in Detergent Mixtures by the Anthrone Method," *Analytical Chemistry*, Vol. 23, pp. 1792-1795 (1951).

Colorimetric method described in which a spectrophotometric determination is made at 625 m $\mu$  and comparison made with a standardized curve. Interfering substances are other carbohydrates, carbohydrate derivatives, certain polyoxyethylene derivatives of fatty acids, and phenols.

(158) R. L. Caswell, "Report on Quaternary Ammonium Compounds," *Journal, Am. Off. Agr. Chem.*, Vol. 34, pp. 675-677 (1951).

Study of bromophenol blue in alkaline solution. Considered practical for products containing about 0.1 per cent quaternary.

(159) E. L. Colichman, "Surface and Interfacial Tension Titrations of Long Chain Quaternary Salts in Bromphenol and Bromthymol Blue Solutions," *Journal, Am. Chem. Soc.*, Vol. 73, pp. 1795-1798 (1951).

Quantitative method described. Results show micellar nature of the quaternary-dye "ion pair" compound as formed in aqueous solutions. Relation to polarography and determinations of critical micelle concentration noted.

(160) J. W. Compton and L. M. Liggett, "The Determination of Moisture in Sodium Alkarylsulfonates," *Journal, Am. Oil Chemists' Soc.*, Vol. 28, pp. 81-84 (1951).

The Karl Fischer method was most satisfactory for the moisture determination. Dessication in an oven or over sulfuric acid did not remove all the moisture, and azeotropic distillation methods were not recommended because of operational difficulties and insufficient accuracy.

(161) A. H. Delsemme, "Infrared Analysis for Functional Groups in Surface-Active Compounds," *Mededelingen van de Vlaamse Chemische Vereniging*, Vol. 13, pp. 152-158 (1951).

Infrared techniques are described.

(162) T. M. Doscher, G. M. Myers and

D. C. Atkins, "The Behavior of Nonionic Surface Active Agents in Salt Solutions," *Journal of Colloid Science*, Vol. 6, pp. 223-235 (1951).

Possible utilization of salting out effect of sodium chloride and other salts of alkali metals with nonionic agents.

(163) "Soap and Soap-Products (Including Synthetic Detergents); Methods of Sampling and Testing," *Federal Specification No. P-S-536b*, Jan. 4, 1951.

Analyses and sections are: Sampling 510.1; preparation of sample 510.2; qualitative test for synthetic detergent 510.3; moisture and volatile matter 520.1; free alkali or acid 530.1; anhydrous, salt-free soda soap 560.1; alcohol soluble matter 570.1; water insoluble 580.1; alkaline salts 590.1; silica 600.1; fatty matter 610.1; chlorides in alcohol-soluble 620.1; rosin 630.1; starch 640.1; synthetic detergent 650.1; neutral inorganic salts 660.1.

(164) W. A. Fessler, "Measurement of the Adsorption of Anion-Active Detergents by Materials Commonly Washed," *Am. Soc. for Testing Materials, Papers on Evaluation of Soaps and Detergents, Special Technical Publication*, No. 115, pp. 9-12 (1951).

A procedure is given for measuring adsorption of anionic detergent on fabrics. Detergent concentration in solution is measured before and after adsorption by means of titration with cetyl pyridinium chloride and bromphenol blue indicator.

(165) G. Gauthier and L. Mazau, "Analysis of Synthetic Detergents," *Annales pharmaceutiques francaises*, Vol. 9, pp. 678-690 (1951).

Methods are given for the analysis of sulfate esters and alkyl aryl sulfonates.

(166) H. Haakh, D. v. Candie, and W. Mobus, "Testing for Polyglycol Ethers of the Leonil O Type in Woolen Clothes," *Melliand Textilberichte*, Vol. 32, pp. 699-701 (1951).

Nonionic polyethylene glycol ethers form a red-brown precipitate with a synthetic tannin prepared by condensing resorcinol with glucose in the presence of sulfuric acid. These ethers may also be extracted quantitatively from aqueous solutions with butyl alcohol.

(167) B. Hargitay, "The Effect of Surface-Active Agents on Barger's Micro-method for the Determination of Mole Concentrations," *Experientia*, Vol. 7, pp. 214-215 (1951); *Chemical Abstracts*, Vol. 46, p. 319 (1952).

Use of droplet size and adsorption at the surface separating the bubble and the connecting film. A lower concentration is produced at this surface and a consequent streaming towards the droplet of higher concentration occurs, but with surface active agents the reverse occurs.

(167A) J. A. Hart and E. W. Lee, "A New Color Reaction of Cationic Soaps and Its Application to Studies of Their Interactions with Cellulose Fibers," *Tappi*, Vol. 34, pp. 77-79 (1951).

The color of an alkaline earth metal lake dispersion of Eriochrome Azurol B is sensitive to the presence of cationic soaps. The color change by the cationic agent is measured in a photometer. The method is claimed to be sensitive to one part in ten million of cationic agent.

(168) B. Kaistrick, F. J. Harris and E. J. Lowe, "The Analysis of Commercial Sodium Triphosphate," *Analyst*, Vol. 76, pp. 230-235 (1951).

Pyro- and triphosphates precipitated together as their zinc salts and the ignited precipitate analyzed for zinc by the oxime method. Trimeta and orthophosphates combined are determined as orthophosphate in the filtrate and from a colorimetric orthophosphate determination on a separate sample proportions of each are calculated. Amount of metaphosphate determined by the benzidine method and removed, when necessary, by a modification of it.

(169) R. V. MacAllister and R. K. Lisk, "Polyoxyethylene Stearate. Colorimetric Determination in Dilute Solutions," *Analytical Chemistry*, Vol. 23, pp. 609-610 (1951).

Method for food products depends upon formation of complex between polyoxyethylene stearate and amylose fraction of potato starch. The amylose, involved in the complex formation is free to form an amylose-iodine complex which is determined colorimetrically. Reproducibility  $\pm 0.010$  per cent in solutions as dilute as 0.05 per cent. Application to analysis of other compounds which form complexes with amylose suggested.

(170) H. Machamer, "Determination of Carbon Dioxide in Laundering Compositions by the Rauscher Method," *Fette und Seifen*, Vol. 53, pp. 150-151 (1951); *Chemical Abstracts*, Vol. 45, p. 9900 (1951).

Rauscher's apparatus can be used for control purposes in laundering compositions if 43 per cent sulfuric acid is used instead of hydrochloric acid. Errors with hydrochloric acid appear to be due to the presence of surface-active agents which increase the solubility of carbon dioxide in hydrochloric acid, but the effect is not so pronounced in sulfuric.

(171) M. Marconi, "A Systematic Process for the Analysis of Detergent Products," *Chimica (Milan)*, Vol. 6, pp. 251-254 (1951).

This is a review with some modifications of a previously published method.

(172) R. Matalon, M. R. J. Salton, and M. Cohen, "Molecular Interaction and Its Relation to the Formation of Sodium Dodecyl

Sulfate-Cetyltrimethylammonium Bromide Complex," *Nature*, Vol. 167, p. 241 (1951).

If the reagents are not well purified, traces of polar compounds such as long-chain alcohols will interfere with precipitation methods of determining anionic detergents by cationic and the reverse. Relatively high concentrations of  $C_8$  and  $C_7$  alcohols are needed, but  $C_8$  and  $C_9$  will inhibit the complex formation in equimolar concentration. The alcohol-sodium dodecyl sulfate complex competes with the sodium dodecyl sulfate cetyltrimethylammonium bromide complex. The results corroborate previous work postulating a 1:1 association of octyl alcohol and sodium dodecyl sulfate.

(173) D. D. Miller and P. R. Elliker, "A Rapid Method of Testing for Quaternary Ammonium Compounds in Milk," *Journal of Dairy Science*, Vol. 34, pp. 273-278 (1951).

Quaternary ammonium compounds may be determined in milk by extraction with tetrachloroethane-acetone mixture containing eosin indicator. The extract is titrated with a standard solution of anionic agent. Five to 100 ppm of cationic compound may thus be detected.

(174) S. A. Miller, B. Bann, and A. P. Ponsford, "Extraction of the Active Agent from Detergent Mixtures," *Journal of Applied Chemistry*, Vol. 1, pp. 523-524 (1951).

The active agent in dilute detergent solutions may be separated quantitatively from builders by adding enough sodium carbonate to give a concentration of 4 per cent, extracting with n-butanol, and evaporating the extract in vacuo. The method is applicable to anionic, nonionic, and cationic detergents.

(175) S. H. Newburger, "A Qualitative Test for Tween in Cosmetics," *Journal, Assoc. Offic. Agr. Chemists*, Vol. 34, pp. 109-113 (1951).

The test involves alkaline hydrolysis of the sample, acidification, extraction with benzene, filtration of the extracted aqueous solution, and addition of silicotungstic or phosphomolybdic acid. Formation of a precipitate is a positive test and is characteristic for polyoxyethylene compounds.

(176) J. Pien, J. M. Desirant, and Mme. Rochelle, "Determination of Quaternary Ammonium Bases in Milk and Water," *Annales des falsifications et des fraudes*, Vol. 44, pp. 290-297 (1951).

The sample is treated with a succession of reagents, finally with bromphenol blue, and extracted with chloroform. If the chloroform layer is colorless, quaternary ammonium bases are absent: If the chloroform is colored, more indicator is added and an aliquot of the chloroform layer is taken for colorimetric assay. The method is sensitive to 5 ppm.

(177) J. C. L. Resuggan, "Quaternary Ammonium Germicides," *Chemical Products*, Vol. 14, pp. 311-314 (1951); *Chemical Abstracts*, Vol. 46, p. 684 (1952).

Methods of testing outlined.

(178) G. Reutenauer, "Analysis of Detergents Containing Alkyl Aryls," *Bulletin mensuel d'information ITERG* (Inst. Tech. Etudes et Recherches Corps Gras), Vol. 4, pp. 557-559 (1951).

Methods are given for the determination of water, phosphates, sulfonated matter, and carboxymethyl cellulose in detergent products.

(180) Society of Public Analysts, Analytical Methods Committee, "Examination of Detergent Preparations," *Analyst*, Vol. 76, pp. 279-286 (1951).

Qualitative tests for the presence of the true soap; alkali carbonates, phosphates, silicates, fillers; sulfonated or sulfated organic detergents; sulfated glycerides; polyethylene glycol derivatives, quaternaries; methyl and carboxymethyl cellulose; naphthenic acid soaps. Procedures given.

Quantitative methods given are total organic matter; soap-detergent mixtures; anionic detergents by Epton method; Schindler separation of sulfated oil; sulfated bodies by hydrolysis; sulfonated bodies by toluidine method; nonionic agents by phosphomolybdic acid; quaternaries by reversal of the methylene blue method; naphthenic acid soap by separation of copper salt in benzene in excess copper sulfate; neutral and alkaline electrolyte.

(181) H. Stüpel and A. v. Segesser, "Titration of Anion-Active Colloidal Electrolytes with p-Toluidine," *Helvetica Chimica Acta*, Vol. 34, pp. 1362-1368 (1951).

A method is described for determining anion-active detergents. A detergent complex is formed with p-toluidine hydrochloride which is extracted with an organic solvent and titrated with standard base. An empirically determined factor is required, depending on the molecular weight and number of solvatable groups.

(182) H. Stüpel and H. v. Segesser, "Determination of Anion-Active Colloid Electrolytes with p-Toluidine," *Fette und Seifen*, Vol. 53, pp. 260-264, 327-332 (1951); *Chemical Abstracts*, Vol. 45, p. 9899 (1951).

By using carbon tetrachloride to separate the phases, the p-toluidine titration method can be applied to the determination of alkylaryl sulfonates, alkyl sulfonates, and secondary alkyl sulfates. Factor must be determined for each compound by titrating with compound of known composition. Ether must be used as solvent in cases of fatty alcohol sulfates and fatty acid condensates.

(183) A. S. Weatherburn, "A Modified Method for the Determination of Anionic Surface Active Compounds," *Journal, Am. Oil Chemists' Soc.*, Vol. 28, pp. 233-235 (1951).

Epton method (titration with cationic agent) was modified by calculation of a blank to be added to the observed titration.

(184) J. B. Wilson, "Quaternary Ammonium Compounds," *Journal, Assoc. Off. Agr. Chemists*, Vol. 34, pp. 343-344 (1951).

A test for purity and a method for purification of bromphenol blue are described.

(185) B. Wurzschnitt, "Evaluation of Synthetic Detergents from the Points of View of Producer and Consumer," *Fette und Seifen*, Vol. 53, pp. 209-213 (1951); *Chemical Abstracts*, Vol. 45, p. 9899 (1951).

Various types of synthetic detergents and methods for their analysis are discussed.

1952

(186) "Analysis of Petroleum Sulfonates," ASTM D 855-52T, *ASTM Standards*, Part 5, pp. 313-320 (1952).

Procedures for determination of mineral oil, sodium sulfonates, sodium carboxylate, inorganic salts, water, basicity and acidity, average molecular weight, and specific gravity of crude and refined oil-soluble sodium petroleum sulfonates. Scheme of analysis given in detail.

(187) E. Benk, "Properties and Identification of Polyethylene Glycols," *Fette und Seifen*, Vol. 54, pp. 85-86 (1952); *Chemical Abstracts*, Vol. 46, p. 7343 (1952).

A review.

(188) E. G. Brown, "Determination of Long-Chain Alkyl Pyridinium Halides," *Analyst*, Vol. 77, p. 211 (1952).

In Feigl's spot test for ternary ring bases, sample added to alkyl halide to form quaternary compound which reacts with sodium 1,2-naphthaquinone-4-sulfonate in the presence of sodium hydroxide to give red, violet, or green compound. Test can be applied to determination of small quantity of cation-active compounds of alkyl pyridine halide type.

(189) H. Campbell and D. F. Muggleton, "Estimation of Bis-Quaternary Ammonium Compounds," *Chemistry Industry*, p. 1244 (1952).

Ammonium reineckate method not considered satisfactory as neither specific nor sensitive. Suggests paper chromatography. Chromatogram developed using butanol/morpholine/water solvent. After development paper is dried at 105°C and compound converted to insoluble salt using phosphomolybdic acid. Amount of bis-quaternary proportional to quantity of molybdate blue formed in 2 to 20 micrograms of drug. Also useful for quantities as low as 0.5 micrograms by paper electrophoresis.



(190) A. L. Clingman and D. A. Sutton, "Separation of Benzene-Carboxylic Acids by Paper Chromatography," *Fuel*, Vol. 31, pp. 259-260 (1952); *Chemical Abstracts*, Vol. 48, p. 4372 (1954).

These acids developed with aqueous alcoholic ammonia and finally the dried paper is sprayed with a bromophenol blue indicator. The ammonium salts appear as blue spots on a yellow background. Not possible to separate components having  $R_f$  value differing by more than 0.05. Other solutions for development described.

(191) D. B. Conklin, "A Single-Reagent Field Test for Quaternary Ammonium Solutions," *Journal of Milk and Food Technology*, Vol. 15, pp. 27-28 (1952); *Chemical Abstracts*, Vol. 46, p. 5481 (1952).

Field test using bromophenol blue accurately buffered on the acid side. Blue color of complex dominant if 200 ppm or more of quaternary present. If less than 200 ppm present, various shades of green are produced.

(192) A. Darbey, "Colorimetric Determination of the Sodium Salts of Ethylenediaminetetraacetic Acid," *Analytical Chemistry*, Vol. 24, pp. 373-378 (1952).

Ethylenediaminetetraacetic acid metals are displaced with nickel and the excess nickel precipitated with dimethylglyoxime. The sequestered nickel is liberated at pH 1 to 1.5 and determined colorimetrically at pH 3.5 to 4.0 with potassium dithiooxalate. Maximum absorbance at 508 to 510 m $\mu$ .

(193) G. R. Edwards, W. E. Ewers, and W. W. Mansfield, "Determination of Sodium Cetyl Sulfate in its Solution in Water," *Analyst*, Vol. 77, pp. 205-207 (1952).

The detergent compound is precipitated with an acid methylene blue solution, filtered, and washed on a column packed with ground quartz. The precipitate is dissolved in 70 per cent ethyl alcohol and determined colorimetrically.

(194) H. Etienne, "The Determination of Small Amounts of Surface-Active Agents," *Bulletin*, centre belge étude et document. eaux (Liege), No. 16, pp. 128-130 (1952); *Chemical Abstracts*, Vol. 47, p. 12846 (1953).

(195) H. Etienne, "Determination of Alkyl-Aryl Sulfonates and Alkyl Sulfates," *Industrie chimique belge*, Vol. 17, pp. 373-377 (1952); *Chemical Abstracts*, Vol. 46, p. 10051 (1952).

Method based upon hydrolysis with acid, followed by extraction with ethyl ether. The dried extract is titrated to a methyl orange endpoint with standard alkali for total acidity, followed by a bichromate endpoint to standard silver nitrate solution, to give acidity due to the hydrochloric acid used in hydrolysis.

(196) J. C. Harris and F. R. Short, "Detection of Commercial Sodium Dodecylbenzenesulfonate in Canned Foods," *Food Technology*, Vol. 6, pp. 275-278 (1952).

Used the Barr, et. al., and the Jones methods for estimation in food juices and pulp. The Jones method was more sensitive, but the other, coupled with alcoholic extraction was more readily used.

(197) N. G. Heatley and E. J. Page, "Some Experiments on the Estimation of Nonionic Detergents of the Polyglycol Type in Sewage," *Water and Sanitary Engineer*, Vol. 3, pp. 46-49 (1952); *Chemical Abstracts*, Vol. 48, p. 2296 (1954).

Surface agent extracted with ether. Aqueous alcohol solution treated with 1.0 ml 5 N hydrochloric acid and 0.5 ml of 10 per cent barium chloride and 2.0 ml 1 per cent phosphomolybdic acid in 0.5 N hydrochloric acid. Boiled, settled overnight, precipitate centrifuged, washed with ammonium chloride, taken up in 5 ml of mixture containing 1 volume 5 N hydrochloric acid and 4 volumes methyl Cellosolve. Solution centrifuged and absorption measured against plain acid methyl Cellosolve by ultraviolet spectrograph at 310 m $\mu$ . Blank run. Reduction to molybdenum blue might be used as alternate procedure.

(198) D. Jouslin, "Analysis of Alkyl Aryl Sulfonates," *Chimie analytique*, Vol. 34, pp. 34-36 (1952); *Chemical Abstracts*, Vol. 47, p. 3185 (1954).

The benzene sodium sulfonate reacts with fused sodium hydroxide to form the phenol, which with bromination gives a tribromide which is titrated with sodium thiosulfate to a starch endpoint. A blank of reagents used is also made for comparison calculations.

(199) W. Kopaczewski, "Electrocapillary Analysis of Detergents," *Chimie et industrie*, Vol. 67, pp. 761-766 (1952).

A chromatographic analytical method applied to surface active agents.

(200) G. R. Lewis and L. K. Herndon, "The Determination of Surface-Active Agents," *Sewage and Industrial Wastes*, Vol. 24, pp. 1456-1465 (1952); *Chemical Abstracts*, Vol. 47, p. 2516 (1953).

Two phase dye-transfer titration methods depending upon difference in solubility of the dye-surface-active complexes in water and organic solvent. Acid dyes such as Pontamine Fast Red 8BNL or Brilliant Blue FCF preferred.

(201) R. L. Mayhew and R. C. Hyatt, "The Effect of Mole Ratio Distribution on the Physical Properties of a Polyoxyethylated Alkylphenol," *Journal, Am. Oil Chemists' Soc.*, Vol. 29, pp. 357-362 (1952).

Gives cloud point data for molecularly-distilled alkylphenol. Also provides refractive indices versus mole ratio of ethylene oxide to alkylphenol.

(202) R. Neu, "Analysis of Laundering and Cleaning Compounds. VII. Differentiation of Polyphosphates and Metaphosphates by Means of Long-Chain Quaternary Ammonium Compounds," *Fette und Seifen*, Vol. 54, pp. 397-399 (1952); *Chemical Abstracts*, Vol. 47, p. 894 (1953).

High molecular weight alkyl-dimethylbenzylammonium chloride does not react with tripolyphosphate but gives precipitate with metaphosphates, particularly in very dilute solutions.

(203) C. W. Pifer and E. G. Wollish, "Potentiometric Titration of Salts of Organic Bases in Acetic Acid," *Analytical Chemistry*, Vol. 24, pp. 300-306 (1952).

Direct titration of salts of organic bases and halide acids when mercuric acetate is added to a solution of the sample in glacial acetic acid prior to titration with perchloric acid in dioxane. Titration potentiometric or visual using crystal violet as indicator. Suggested as suitable to obviate tedious extractions and colorimetric procedures.

(204) J. Renard, "Qualitative and Quantitative Reactions of Hexadecyltrimethylammonium Bromide," *Journal de pharmacie de Belgique*, Vol. 7, pp. 403-408 (1952); *Chemical Abstracts*, Vol. 47, p. 8967 (1953).

Commercial product, Cetavlon, containing 80 per cent active and 20 per cent sodium bromide separated in pure form by extraction with ether-petroleum ether 1:1. Color reactions reviewed. Can be determined colorimetrically by bromophenol blue. Can also be determined titrimetrically by potassium bichromate, determining excess chromate iodometrically. Precipitated complex said to be 7 Cetavlon: 3 potassium bichromate.

(205) G. R. F. Rose and C. H. Bayley, "Determination of Dodecyl-dimethylbenzylammonium Naphthenate in Textile Materials," Natl. Research Council Can., *NRC Bulletin*, No. 2875, 8 pp. (1952); *Chemical Abstracts*, Vol. 47, p. 6662 (1953).

Method used was the eosin yellowish or tetrabromofluorescein test for quaternary compounds. Tetrachloroethane solvent used, and Aerosol used for titration purposes.

(206) H. Stüpel, "Determination of the Active Contents of Soap and Detergent Formulations," *Chemiker-Zeitung*, Vol. 76, pp. 252-256 (1952); *Chemical Abstracts*, Vol. 46, p. 8396 (1952).

A review with 61 references.

(207) H. Terayama, "Methods of Colloid Titration (A New Titration between Polymer Ions)," *Journal of Polymer Science*, Vol. 8, pp. 243-253 (1952).

Direct titration for colorimetric change using metachromatic dyes such as toluidine blue, brilliant cresyl blue, etc. Indirect titration method also described where mate-

rials are weakly acid, weakly basic, or amphoteric. Describes indirect titration of carboxymethylcellulose, alginates, proteins.

(208) "Determination of Monostearate (Ethylene Glycol)," *TGA Method No. 58*, Toilet Goods Assoc. Inc., New York City, (Oct. 6, 1952).

Depends upon saponification with potassium hydroxide, oxidation with periodic acid, titration with sodium thiosulfate and potassium iodide solution using starch indicator.

(209) R. Wickbold, "Newer Methods for the Analysis of Alkaryl Sulfonates and Fatty Alcohol Sulfates," *Fette und Seifen*, Vol. 54, pp. 394-397 (1952); *Chemical Abstracts*, Vol. 47, p. 892 (1953).

The p-toluidine titration method supplemented by weighing the precipitate formed, which can then be used for a fast equivalent weight determination. Sodium sulfate can be determined by titration with lead nitrate solution.

(210) Widaly, "The Determination of Surface-Active Agent in Factory Control," *Seifen-Ole-Fette-Wachse*, Vol. 78, pp. 143-145 (1952); *Chemical Abstracts*, Vol. 47, p. 6680 (1953).

Thymolsulfonephthalein is used as indicator in lowering the pH of the samples containing alkaline materials. The p-toluidine precipitation method using carbon tetrachloride is then used.

(211) J. B. Wilson, "Determination of Quaternary Ammonium Compounds as Reineckates," *Journal, Assoc. Agr. Chemists*, Vol. 35, pp. 455-458 (1952).

Gravimetric method using ammonium reineckate.

#### 1953

(212) H. Ballezo, "Titration of Acids and Bases in Non-Aqueous Solution. II," *Mitteilungen des Chemischen Forschungs Institutes der Wirtschaft Österreichs*, Vol. 7, pp. 126-131 (1953); *Analytical Abstracts*, Vol. 1, No. 956 (1954).

Alkyl- and arylamines, amino acids, amino-alcohols, cyclic nitrogen bases, alkylene-oxides, sulphonamides and other organic and inorganic acids titrated potentiometrically in glacial acetic acid, conductimetrically in thionyl chloride and by means of indicators. Macro- and micro- titrations given.

(213) H. M. Bregoff, E. Roberts and C. C. Delwiche, "Paper Chromatography of Quaternary Ammonium Bases and Related Compounds," *Journal of Biological Chemistry*, Vol. 205, pp. 565-574 (1953); *Analytical Abstracts*, Vol. 1, No. 737 (1954).

Method for identification in biological materials described. Various solvent systems described and the modified Dregendorff solution ( $\text{KBiI}_4$ ) most generally applicable reagent. Acetone solutions of alka-

loid reineckates suitable for chromatography.

(214) P. Cazaux, J. Canellas, J. Bailenger and J. Troadec, "Polarographic Classification of Detergent Surface-Active Agents," *Bulletin, Soc. Pharm. Bordeaux*, Vol. 91, pp. 38-40 (1953); *Chemical Abstracts*, Vol. 48, p. 389 (1954).

A formula used to determine a constant of adsorption of the detergent affecting the maximum oxygen concentration on the mercury drop. Evaluation of a number of detergents given.

(215) D. Coppini and R. Camerini, "Colorimetric Determination of Carbowax and its Behavior with Alkaloid Reagents," *Bollettino chimico farmaceutico*, Vol. 92, pp. 363-367 (1953); *Chemical Abstracts*, Vol. 48, p. 3853 (1954).

Lists reagents which precipitate Carbowax 1500 and 4000. Gives colorimetric method using ferrocyanic acid precipitation and method.

(216) F. H. Cozzi, "Chromatography—Its Use in the Quantitative Analysis of Detergent and Soap Ingredients," *Bibliographic Series*, No. 29, Code No. PB 111210, Technical Library, Quartermaster Research and Development Laboratories, Philadelphia, June 1953.

Eighty-three abstracts, covering 24 pages, and the literature from 1942 to June 1953. Concerned largely with methodology and method improvements and has a few references to soaps and fatty acids.

(217) A. Darbey, "Colorimetric Determination of Ethylenediaminetetraacetic Acid in Soaps and Detergents," *Soap*, Vol. 29, No. 8, pp. 81, 83, 85, 87, 153 (1953).

Ethylenediaminetetraacetic acid metals are displaced with nickel and the excess nickel precipitated with dimethylglyoxime. The sequestered nickel is liberated at pH 1 to 1.5 and determined colorimetrically at pH 3.5 to 4.0 with potassium dithiooxylate. Maximum adsorption at 508 to 510 m $\mu$ . Interference by phosphate removed by precipitating as tricalcium phosphate and the copper precipitated with 5,7-dibromohydroxyquinoline.

(218) P. N. Degens, Jr., H. C. Evans, J. D. Kommer and P. A. Winsor, "Determination of Sulfate and Sulfonate Anion-Active Detergents in Sewage," *Journal Applied Chemistry (London)*, Vol. 3, pp. 54-61 (1953).

Small quantities of anionic agents determined quickly by a modification of the Jones method. Interfering substances usually made harmless by choice of the volume ratios of aqueous and organic phases and by the number of extractions. The CNS-ion requires special treatment, and two methods are given. Sulfate and sulfonate detergents

estimated separately by hydrolysis followed by estimation of residual detergent.

(219) P. N. Degens, H. van der Zee and J. D. Kommer, "Anionic Syndets in Amsterdam Sewage," *Sewage and Industrial Wastes*, Vol. 25, pp. 20-25 (1953); *Chemical Abstracts*, Vol. 47, p. 4529 (1953).

Analysis based on methylene blue-detergent complex through an extraction with chloroform. Sulfate anionic detergent obtained by difference between it and total anion active agent.

(220) J. N. Duperray, "Determination and Separation of Sulfonated Alkoxyaryls and Alkoxy Sulfates," *Bulletin mensuel d'information ITERG*, Vol. 7, pp. 325-328 (1953); *Chemical Abstracts* Vol. 47, p. 12126 (1953).

Purity determined titrimetrically using aqueous solutions of agents colored by tetrabromophenol-sulfonaphthalein and chloroform using suitable cationic agent. Separation of sulfate and sulfonate carried out by hydrolysis, followed by petroleum ether extraction for the sulfonate.

(221) J. C. Espector, "Quantitative Chemical Analysis of the Anionic Synthetic Detergents," *Color "AITEA"* (Argentina), Vol. 1, No. 8, pp. 39-42, 47-50, 53, (1953); *Chemical Abstracts*, Vol. 48, p. 7919 (1954).

Suggests new method for analysis of alkylaryl sulfonic acids. Based upon treatment with strong acid with boiling to separate free sulfonic acid, and this measured volumetrically.

(222) S. D. Faust, "Syndets in Sewage can be Measured," *Water and Sewage Works*, Vol. 100, pp. 242-243 (1953); *Chemical Abstracts*, Vol. 47, p. 10781 (1953).

Concentration in sewage determined by methylene blue method. Applicable to alkyl sulfates, alkyl sulfonates, alkylaryl sulfonates, amine salt of alkylaryl sulfonate, alkylaryl polyether sulfate, and alkylaryl polyether sulfonate.

(223) T. E. Furlong and P. R. Elliker, "An Improved Method of Determining Concentrations of Quaternary Ammonium Compounds in Aqueous Solutions in Milk," *Journal Dairy Science*, Vol. 36, pp. 225-234 (1953); *Chemical Abstracts*, Vol. 47, p. 6567 (1953).

Titration with standard anionic agent with eosin indicator. For milk the quaternary must be extracted.

(224) U. Gallo, "Chromatography of Some Synthetic Nonionic Surface-Active Agents," *Bollettino chimico farmaceutico*, Vol. 92, pp. 332-338 (1953); *Chemical Abstracts*, Vol. 48, p. 1712 (1954).

Surface active agents have high  $R_f$  value. Postonal, an ethylene oxide polymer, showed more definite  $R_f$  values than other compounds. Small variation in fatty acids of

Tween 61 causes large differences in readings. Neutral solvents containing butyl alcohol give zero  $R_f$  values; butyl alcohol and acetic acid medium; and benzene and ethyl alcohol give high values. Triton X-100 behaves like Tween.

(225) B. E. Gordon and R. S. Urner, "Determination of Sulfite and Sulfate (as Sodium Sulfate) in Sodium Petroleum Sulfonates—An Amperometric Titration," *Analytical Chemistry*, Vol. 25, pp. 897-901 (1953).

The inorganic salt content of petroleum sulfonates can be determined by amperometric titration. Lead nitrate is used as the titrant. This is said to be more rapid than the ASTM procedure.

(226) L. S. Harrow and K. S. Heine, "The Separation and Determination of Sulfonated Naphthalene Intermediates in Certifiable Coal-Tar Colors," *Journal, Assoc. Off. Agric. Chem.*, Vol. 36, pp. 936-943 (1953); *Analytical Abstracts*, Vol. 1, No. 994 (1954).

Procedure for separation from dyes of sulfonated naphthalene intermediates by column chromatography and subsequent spectrophotometric determination described.

(227) H. O. Hettche, "A Recorder for the Determination of Surface-Active Substances in Water and Sewage," *Vom Wasser*, Vol. 20, pp. 137-147 (1953); *Chemical Abstracts*, Vol. 49, p. 5733 (1955).

Degree of pollution in water supplies can be continuously measured by changes in surface activity. A ring tensiometer incorporated into an automatic sampling and recording device can measure surface activity every three minutes. The apparatus detected 0.007 ppm of a household detergent.

(228) L. F. Hoyt and J. E. Walker, "Analysis of Hard-Water Soap; the Direct Determination of Active Sulfonate in Mixtures of Soap and Nacconol RB 85," *ASTM Bulletin*, No. 192, pp. 56-57 (1953).

Active organic sulfonate or sulfate components of the synthetic detergent in soaps react quantitatively with a suitable cationic, forming an inert and insoluble complex. Bromophenol blue used as indicator for the two-phase system of water and chloroform. The complex migrates to the chloroform layer. For accurate work the cationic is standardized against the same sulfonate used in the soap.

(229) H. Iwasenko, "Dichlorofluorescein as an Indicator in the Determination of Anionic Detergents," *Journal, Assoc. Off. Agr. Chemists*, Vol. 36, pp. 1165-1169 (1953); *Chemical Abstracts*, Vol. 48, p. 14257 (1954).

Titration of the anionic detergent with a standard quaternary ammonium chloride solution, by using dichlorofluorescein as indicator.

(229A) J. K. N. Jones and J. B. Pridham, "Colorimetric Estimation of Sugars using

Benzidine," *Nature*, Vol. 172, p. 161 (1953).

Orange-yellow color measured photometrically after treatment of sugar solution with benzidine solution in glacial acetic acid. Relation between concentration and absorption linear for many sugars including glucose, glucose derivatives, sucrose, maltose, and mannose. Mild nature of the reagents and wide range of sugars estimated are two main advantages of method.

(230) C. Kortland and H. F. Dammers, "The Determination of the Types of Surface-Active Compounds with Special Reference to Synthetic Detergents," *Chemisch Weekblad*, Vol. 49, pp. 341-348 (1953); *Chemical Abstracts*, Vol. 47, p. 9035 (1954).

Identification discussed, and a mixture containing various types is analyzed.

(230A) S. Kume, T. Yamamoto, K. Otozai and S. Fukushima, "Analysis of High-Molar Substances by Chromatography. I. Paper Chromatography of Polyethylene Oxides," *Bulletin, Chem. Soc. (Japan)*, Vol. 26, pp. 93-96 (1953); *Chemical Abstracts*, Vol. 47, p. 12130 (1953).

Used fluorescein as absorption indicator for a simple method for determining average molecular weight of polyethylene oxides in range of 1000 to 5000. Limiting amount and concentration of determination is  $0.5\mu$  of polyethylene oxide per micromol.

(231) T. Lewandowski, "Anionic Detergents," *Soap*, Vol. 29, No. 4, pp. 49-52 (1953).

Back-titration of cationic bromophenol blue combination with anionic solutions used to estimate the amount of anionic agent in the solutions. Color reactions dependent upon molar rather than weight relationship between anionic and cationic detergent, and is intended for field use.

(232) S. D. Nogere, "Determination of Organic Sulfur Groups. XIII. Sulfonic Acids and Alkyl Sulfates," *Organic Analysis*, Vol. I, pp. 381-385, Interscience Publishers, Inc., New York City (1953).

Gives the benzidine salt and titrimetric methods.

(233) P. Sadtler, "Analysis of Synthetic Detergents by Infrared Absorption Technique," *ASTM Bulletin*, No. 190, pp. 51-53 (1953).

These methods definitely identify the type of compound and give clues on the exact identification of the compound.

(234) N. Schönfeldt, "Determination of Ethylene Oxide Products in Aqueous Solutions or Dispersions," *Nature*, Vol. 172, p. 820 (1953).

A definite volume of ethylene oxide condensation solution is precipitated with a known excess of potassium ferrocyanide which is back titrated with zinc sulfate. Approximately 0.7, 1.0 and 1.5 moles of potassium ferrocyanide are necessary for

the precipitation of nonyl phenol condensed with 6, 9, and 12 moles of ethylene oxide.

(235) Simon-Dorlet, "Analytical Characteristics of Some Synthetic Curarising Agents," *Journal de Pharmacie de Belgique*, Vol. 8, pp. 146-155 (1953); *Analytical Abstracts*, Vol. 1, No. 1053 (1954).

Quantitative method depends upon treatment with sodium chloride in sulfuric acid solution. Solution mixed with iodine solution, the precipitate filtered off and the filtrate titrated with sodium thiosulfate.

(236) F. D. Snell and C. T. Snell, *Sodium Carboxymethylcellulose. Colorimetric Methods of Analysis*, Vol. III, p. 467, D. Van Nostrand Co., Inc., New York City (1953).

Method of estimation by anthrone or 2,7-dihydroxynaphthalene.

(237) S. Weiner, "Determination of Quaternary Salts Employing Colorimetric Measurement of the Deviation from the Equivalence Point," *Chemist Analyst*, Vol. 42, pp. 9-13 (1953); *Chemical Abstracts*, Vol. 47, p. 4249 (1953).

In cases where reactions cannot readily be followed because of precipitation or color, good results are obtained by titrating close to equivalence point and then estimating the difference by colorimetric measurement of solution. Picric acid was used as titrating solution and the precipitate was separated by filtration before optical measurement.

#### 1954

(238) C. W. Ballard, J. Isaacs, and P. G. W. Scott, "The Photometric Determination of Quaternary Ammonium Salts and of Certain Amines by Compound Formation with Indicators. I. Quaternary Ammonium Salts," *Journal of Pharmacy and Pharmacology*, Vol. 6, pp. 971-984 (1954); *Chemical Abstracts*, Vol. 49, p. 7189 (1955).

Determination of quaternary ammonium compounds by titration with anionic agent by measuring change in color intensity of an indicator not applicable to compounds of molecular weight lower than that of cetrimide. Gives method, using bromophenol blue and chloroform.

(239) J. Blandin and R. Desalme, "New Chromatographic Method for Identification of Synthetic Detergents," *Bulletin mensuel d'information ITERG* 8, pp. 69-73 (1954); *Chemical Abstracts*, Vol. 48, p. 6146 (1954).

Chromatograms produced on strip filter paper by dividing them in aqueous solution containing together fluorescein, fuchsin, methylene blue, and blue for silk 6B. These show characteristic color changes according to detergents added to the solutions. Succession of shades with 20 different detergents tabulated.

(240) S. J. L. Bonting and B. S. Aussen, "The Effect of Surface-Active Compounds

on the Polarographic Waves of Oxygen and Zinc and the Electrocapillary Curve of Mercury," *Recueil travaux chimique*, Vol. 73, pp. 455-461 (1954); *Chemical Abstracts*, Vol. 48, p. 10450 (1954).

Effect of half-wave potentials of oxygen and zinc similar to the effect of these surface active agents on the surface tension. Electrocapillarity shifted towards more positive potential by some agents, and the reverse by others which are named. Desorption indicated by small additional waves at an indicated voltage.

(241) E. D. Carkhuff and W. F. Boyd, "Volumetric Determination of Quaternary Ammonium Compounds and Certain Tertiary Amines," *Journal, Am. Pharm. Assoc.*, Vol. 43, pp. 240-241 (1954); *Chemical Abstracts*, Vol. 48, p. 7259 (1954).

Use of certain anionic agents used with dimethylaminoazobenzene as indicator, in presence of dilute sulfuric acid and chloroform, a wide variety of pharmaceutical preparations such as tablets, ointments, candy-base medication and liquids containing quaternary ammonium compounds can be assayed. Concentrations as low as 1 to 4000 can easily be determined.

(242) C. C. T. Chinnick and P. A. Lincoln, "Quantitative Field Tests for Quaternary Ammonium Compounds," *Laboratory Practice*, Vol. 3, pp. 364-366 (1954); *Analytical Abstracts*, Vol. 1, No. 3035 (1954).

Methylene blue displaced from its chloroform soluble salt of alkylsulfate by a quaternary compound. Method standardized for each compound. Quaternary ammonium salts of bromophenol blue soluble in ethylene dichloride but the sodium salt is not. Method used by titrating alkyl anionic complex with quaternary solution until a colorless organic solvent layer becomes deep blue. This method said to be nearly stoichiometric.

(243) K. S. G. Doss and S. L. Gupta, "Effect of Some Surface-Active Agents on the Capacity of the Dropping Mercury Electrode," *Bulletin, Central Electrochem. Research Inst., Karaikudi*, Vol. 1, No. 2, pp. 9-14 (1954); *Chemical Abstracts*, Vol. 48, p. 11957 (1954).

The effect of these agents on the dropping mercury electrode suggested a promising method for evaluating these agents. Capacity peaks caused by sudden desorption of the agents from the mercury were found in the neighborhood of electrocapillary zero.

(244) G. P. Edwards and M. E. Ginn, "Determination of Synthetic Detergents in Sewage," *Sewage and Industrial Wastes*, Vol. 26, pp. 945-953 (1954); *Chemical Abstracts*, Vol. 49, p. 1997 (1955).

The two-phase titration method of Barr was used to determine anionic detergents

in sewage, and modified to produce a rapid, accurate method. Interference by soap and protein eliminated by adjustment of the pH to 7.0 to 7.5 during the reaction. The use of ethylenediaminetetraacetate avoids errors caused by calcium and magnesium ions. Hexane used as solvent.

(245) J. Fogh, P. O. H. Rasmussen, and K. Skadhauge, "Colorimetric Method for Quantitative Microdetermination of Quaternary Ammonium Compounds—Losses of Quaternary Ammonium Compounds Caused by Glass Adsorption and Concentration in the Foam," *Analytical Chemistry*, Vol. 26, pp. 392–395 (1954). Cetylpyridinium chloride changes the color of bromocresol purple from violet to blue at pH 8.2. Spectrophotometric determination made at 620 m $\mu$ , and calcium, magnesium, ferrous and ferric iron, and copper do not interfere in concentrations up to 1 per cent. Temperature is not critical. Adsorption on scratched glass surfaces may amount to 70 per cent. This overcome by coating with Plexiglas. Foam may also cause concentration in upper layer, and cause error.

(246) U. Gallo, "Analysis of Nonionic Surface Agents," *Bollettino chimico farmaceutico*, Vol. 93, pp. 160–164 (1954); *Chemical Abstracts*, Vol. 48, p. 10475 (1954).

Polyhydroxyethylene compounds give a yellow precipitate with solution of  $(\text{Bi}((\text{SCN}))\text{K}_3$  in excess of reagent. In paper chromatography various compounds give high  $R_f$  values in the same range. As developer Dragendorff's reagent in alcohol is used. Compounds not absorbed on alumina and this method may be used to remove other substances.

(247) T. Green, R. P. Harker and F. O. Howitt, "Use of Adsorption Columns in the Analysis of Emulsions Stabilized with Nonionic Detergents," *Nature*, Vol. 174, p. 659 (1954).

Emulsions separated quantitatively by adsorption in a column containing a mixture of Zeokarb 225 and De-Acidite FF and elution, first with 50 per cent aqueous ethyl alcohol and then trichloroethane. Animal charcoal adsorbs detergent and it cannot be eluted.

(248) S. L. Gupta, "Behavior of Surface-Active Substances at the Dropping Mercury Electrode at Different Frequencies of the Applied Field," *Proceedings, Indian Acad. Sci.*, Vol. 39A, pp. 282–289 (1954); *Chemical Abstracts*, Vol. 48, p. 13743 (1954).

Depressing effect on the capacity of the dropping mercury electrode resulting from adsorbed films of isobutyl alcohol and sodium dodecyl-p-toluidine-2-sulfonate decreased with increasing frequency of the applied field.

(249) W. J. Halstead and B. Chaiken, "Chemical Analysis and Sources of Air-

Entraining Admixtures for Concrete," *Public Roads*, Vol. 27, No. 12, pp. 268–278 (1954); *Chemical Abstracts*, Vol. 48, p. 3658 (1954).

A qualitative and quantitative scheme for analysis is given.

(250) P. D. Haney, et al., "Characteristics and Effects of Synthetic Detergents," *Journal, Am. Water Works Assn.*, Vol. 46, No. 8, pp. 751–774 (1954).

Task group E5.15 report. Contains section on analytical methods for anionic, cationic and nonionic agents. Arrangements being made to develop suitable analytical reference standard.

(251) R. P. Harker, J. M. Heaps, and J. L. Horner, "Use of Adsorption Columns in the Analysis of Soap and Detergent-Stabilized Emulsions," *Nature*, Vol. 173, pp. 634–635 (1954).

Columns of ZeoKarb 225 mixed with animal charcoal remove all organic matter from soap solutions and emulsions. Subsequent elution with the proper solvents gives the separate components. Fatty acids removed with ethanol, lanolin with trichloroethylene and sodium cetyl sulfate with ethylene glycol.

(252) J. A. van der Hoeve, "Analysis of Textile Auxiliary Products," *Journal, Soc. Dyers Colourists*, Vol. 70, pp. 145–154, (1954); *Chemical Abstracts*, Vol. 48, p. 7307 (1954).

Cation-anion active and polyamide (not protein) products behave in many respects like polyethyleneglycol products. Groups studied were phosphonium compounds, alkylamidoalkyl phosphates, alkyl-naphthalene phosphonates, alkylamidophosphates, and alkylamido alkane carboxylates. Analytical scheme based on determination of organically combined nitrogen, determination of the ionic nature of the compound, and determination of the type of hydrocarbon group in it.

(253) R. House and J. L. Darragh, "Analysis of Synthetic Detergent Compositions," *Analytical Chemistry*, Vol. 26, pp. 1492–1497 (1954).

Comparison of cetyl pyridinium bromide titration and alcohol extraction and p-toluidine hydrochloride methods for alkylbenzene sulfonate. Sulfate and sulfonate determined together by hydrolyzing the sulfate overnight and titrating with cetyl pyridinium bromide.

Multiple extracts of the hydrolyzed solution will yield the alkylbenzene with alkyl groups less than 4 carbons in length. Inorganic sulfate by potassium rhodizonate titration method. Oil content by extraction with petroleum ether or isopentane from 70 per cent ethanol solution.

(254) J. V. Karabinos, G. E. Kapella, and G. E. Bartels, "Classifying Detergents," *Soap*, Vol. 30, No. 6, pp. 41–43 (1954).

List 14 types of anionic and nonionic detergents. Qualitative test used for differentiation. Analysis made on the active basis by extraction with 95 per cent ethanol. Ceric nitrate gives deepening in color as positive for hydroxyl groups, and this specific for nonionic polyethylene oxide detergents as well as sorbitan esters and alkanolamides. Aluminum chloride-chloroform test is positive when it gives discoloration of precipitate of chloroform solution. List of color changes as related to specific types of products. Bromine water addition gives indications of various agents. Other reagents are potassium permanganate, alcoholic potassium hydroxide, and acetic acid-sulfuric acid. Summary table lists reactions to tests and positive or negative results with individual types. Test indicated as useful for qualitative identification. Comparison with table and known detergents recommended.

(255) F. J. Loomer, "Colorimetric Determination of Anionic Detergents," *Analytica Chimica Acta*, Vol. 10, pp. 147-150 (1954); *Chemical Abstracts*, Vol. 48, p. 6146 (1954).

Many types may be determined colorimetrically by means of their action on a protein-bromocresol purple complex. Within certain limits the amount of free dye liberated from the complex is proportional to the amount of detergent added. Accuracy about 4 per cent is not as good as a method based on rosaniline, but the protein-complex is not restricted to long chain alkyl sulfates, and can be applied to many more anionics than the rosaniline method.

(256) H. Negoro and H. Sakurai, "Analysis of Surface-Active Agents. I. Determination of Benzethonium Chloride," *Annual Report, Takamine Lab.*, Vol. 6, pp. 89-97 (1954); *Chemical Abstracts*, Vol. 49, p. 16473 (1955).

Bromophenol blue used in microcolorimetry and semimicrotitration techniques. Titration with alkaline bromophenol blue as the indicator by the sodium lauryl sulfate method enhanced sensitivity.

(257) R. Neu and P. Hagedorn, "Analysis of Washing and Cleaning Compositions. XI. Reaction of Alkylated Bisaminoethylamino-acetic Acids with Polyphosphates of Varying Degrees of Condensation," *Fette und Seifen Anstrichmittel*, Vol. 56, pp. 298-302 (1954); *Chemical Abstracts*, Vol. 49, p. 16495 (1955). Degree of turbidity given by octyl, dioctyl, and dodecyl derivatives of bis (aminoethyl) glycine hydrochloride with seven types of polyphosphates described. Use of this reaction for analytical purposes suggested.

(258) F. Neuwald and K. Adams, "Identification and Incompatibilities of Polyethylene Glycols," *Deutsche Apotheker-Zeitung*,

Vol. 94, p. 1258 (1954); *Chemical Abstracts*, Vol. 49, p. 8566 (1955).

Polyethylene glycols are incompatible with cignolin. They can be identified by heating with concentrated sulfuric acid and identifying the dioxane found as the mercury complex with aqueous mercuric chloride.

(259) H. Nogami, K. Sekiguchi, and F. Nakagawa, "Application of a Surface-Active Agent to the Silver Titration," *Journal, Pharm. Soc., Japan*, Vol. 74, pp. 1402-1403 (1954); *Chemical Abstracts*, Vol. 49, p. 4440 (1955).

Effect of Tween 80 on halide solutions in the silver ion titration with an adsorption indicator was studied. The agent prevented the precipitation of silver halides during the titration and the solution remained colloidal and clear. The color change of the indicator was sharp and clear and the endpoint observed more easily. Presence of electrolytes such as sodium sulfate failed to reduce the accuracy of the titration.

(260) K. Peter, "Thymol Blue as a Reagent for Anionic Surfactants," *Fette und Seifen Anstrichmittel*, Vol. 56, pp. 997-1001 (1954); *Journal, Am. Oil Chem. Soc.*, Vol. 33, p. 139 (1956).

Anionic surfactants give a definite red-violet color with thymol blue; 5 ml neutral surfactant solution is added to 5 ml 0.005 normal hydrochloric acid containing 3 drops of 0.1 per cent thymol blue.

(261) J. Pomeranz "Determination of Chloride in Alcohol-Soluble Matter in Detergents," *Chemist Analyst*, Vol. 43, pp. 89-90 (1954); *Chemical Abstracts*, Vol. 49, p. 2100 (1955).

Chloride removed by anion-exchange resin such as Dowex-2, then eluted with 400 ml of normal sodium hydroxide or more if necessary. Chlorine then determined with silver nitrate by the Volhard method.

(262) R. Powell and C. G. Taylor, "Chloroform Extraction of Ferrous-Bipyridine and o-Phenanthroline Complexes with the Aid of Alkyl Sulfates and Sulfonates," *Chemistry and Industry*, 1954, pp. 726-727; *Chemical Abstracts*, Vol. 48, p. 11978 (1954).

Any long chain sulfate or sulfonate will increase the rate of extraction of the 2,2'-bipyridine complex of ferrous, and ferrous-o-phenanthroline complex into chloroform. The method can also be used for determination of alkyl sulfates and sulfonates and for solvent extraction of other water-soluble cationic complexes, especially those with an ethylenediamine group in the ligand.

(263) Mme. Simon-Dorlet, "Methods of Assay for some Quaternary Ammonium Substances," *Journal de pharmacie de Belgique*, Vol. 9, pp. 106-111 (1954); *Chemical Abstracts*, Vol. 49, p. 11954 (1955).

Method uses a reagent comprised of di-

picrylamine treated with magnesium oxide, and the surface active agent with this gives an orange-yellow color which is photometrically read at 430 m $\mu$ .

(264) F. D. Snell and C. T. Snell, *Quaternary Ammonium Compounds. Colorimetric Methods of Analysis*, Vol. II, pp. 63-68, D. Van Nostrand Co. Inc., New York City (1954).

Describes bromophenol blue method for fruit juices, beverages, beer, table syrup, eggs, milk, serum or plasma and urine. Also gives erichrome Azurol B. Meth. A triiodide method is described for use in absence of starch, and on textiles. Potassium triiodide applied to suspected surface, the triiodide extracted with ethanol and colorimetrically determined. A method by oxidation with permanganate also given.

(265) F. D. Snell and C. T. Snell, *Long-Chain Alkyl Sulfates. Colorimetric Methods of Analysis*, Vol. II, pp. 353-355, D. Van Nostrand Co. Inc., New York City (1954).

Gives procedures by rosaniline hydrochloride separated by chloroform extraction; hypochlorite and o-tolidine; and turbidimetrically using as precipitant 1-amino-5-ethoxybenzothiazole hydrochloride.

(266) D. G. Stevenson, "The Absorptometric Determination of a Non-ionic Detergent," *Analyst*, Vol. 79, pp. 504-507 (1954).

Two procedures given which involve precipitation by phosphomolybdic acid and subsequent absorptometric determination of the precipitate. Both methods will detect 10 micrograms of detergent in 10 ml. solution; one method quicker but less accurate and sensitive than the other. Inorganic salts do not interfere. Alkyl sulfate, alkyl-aryl sulfonate, and carboxymethylcellulose cause high results as they appear to be associated with nonionic agent when latter is precipitated.

(267) H. Stüpel, *Synthetische Wasch- und Reinigungsmittel*, pp. 459-479, Konradin-Verlag Robt. Kohlhammer, Stuttgart (1954).

Analysis subdivided by product, builders, and syndet. Methods can be qualitative or quantitative. Reviews general or specific methods for both. Gives in detail van der Hoeve's qualitative method according to 15 groupings. Details Linsenmeier's scheme which is shorter and based upon specific groupings.

Wurzschmitt's classification is based upon elemental analysis. Mentions infrared, ultra violet, and x-ray methods.

Gives a table for qualitative examination of phosphates.

Reviews methods for carboxymethylcellulose.

(268) H. M. Tenney and F. E. Sturgis, "Separability of Hydrocarbons by Elution

Chromatography," *Analytical Chemistry*, Vol. 28, pp. 946-953 (1954).

Various adsorbents used in separating alkanes, alkylbenzenes, and others, and the eluates with n-heptane examined by infrared.

(269) N. W. Tschoegl, "Analysis of Water-Soluble Synthetic Soaps," *Reviews of Pure and Applied Chemistry* (Australia), Vol. 4, pp. 171-206 (1954).

Broad review of 141 references. Subdivides article according to quantitative analysis of anionics by difference methods, cation exchange methods, extraction, barium methods, ashing, acid titration, amine methods, o-toluidine, tensimetric, polarographic, critical micelle, dyestuff-antagonist, soap antagonist.

Quantitative methods for cationics subdivided as to argentometric, salting-out, Kjeldahl, perchloric acid, turbidimetric, inorganic reactants, dyestuff-antagonist. Quantitative analysis of nonionic agents briefly discussed under similar system as above when applicable.

Identification of water-soluble products through class tests, group tests, preliminary tests, additional tests.

(270) A. S. Weatherburn, "Determination of the Ionic Type of Synthetic Surface-Active Compounds," *Canadian Textile Journal*, Vol. 71, No. 16, pp. 45-46 (1954); *Chemical Abstracts*, Vol. 48, p. 13351 (1954).

A two-phase titration using methylene blue in water and an anionic complex of methylene blue in chloroform, adjusted to equal color depth in both phases, shows deepening of color in water if cationic agent is added, and lightening of water with an anionic agent. Soap acts as a nonionic agent under these conditions, but is distinguished by acidification.

(271) J. B. Wilson, "Quaternary Ammonium Compounds in Milk," *Journal, Assoc. Agr. Chemists*, Vol. 37, pp. 374-379 (1954); *Chemical Abstracts*, Vol. 48, p. 8979 (1954).

Furlong and Elliker method modified for greater sensitivity by increasing sample size. Collaborative study disclosed presence of 1-3 ppm quaternary ammonium compound with no false positive tests. Recovery from milk at levels of 10 to 76 ppm ranged from 68 to 116 per cent with average of 87 per cent.

1955

(272) "Chemical Analysis of Sulfonated and Sulfated Oils," ASTM D 500-55, *ASTM Standards on Soaps and Other Detergents*, pp. 103-120 (Sept. 1955).

Methods for moisture, organically combined sulfuric anhydride, total desulfated fatty matter, total active ingredient, unsaponifiable non-volatile matter, inorganic



salts, total alkalinity, total ammonia, acidity, water-immiscible organic solvents volatile with steam.

(273) "Chemical Analysis of Soaps Containing Synthetic Detergents," ASTM D 820-46, *ASTM Standards on Soaps and Other Detergents*, pp. 71-78 (Sept. 1955).

Methods for moisture and volatile matter, free alkali or free acid, anhydrous salt-free soda soap, alcohol soluble, water insoluble, total alkalinity of alcohol insoluble, sodium silicate, fatty matter, chlorides in alcohol soluble rosin, synthetic detergent by difference, neutral inorganic salt.

(274) A. T. Ballun, G. E. Bartels and J. V. Karabinos, "Comparative Analyses of Polyethenoxy Tallates," *Transactions, Illinois State Acad. Sci.*, Vol. 47, pp. 81-85 (1955); *Chemical Abstracts*, Vol. 49, p. 16475 (1955).

Analyzed by determination of number of ethenoxy units by saponification equivalent, carbon and hydrogen content, iodine number, and by calculation from weights of starting materials. Tabulated data.

(275) E. G. Brown, "Analysis of Polyethylene Glycols and Their Derivatives," *Manufacturing Chemist.*, Vol. 26, pp. 441-442 (1955); *Chemical Abstracts*, Vol. 50, p. 2372 (1956).

The Wurzschnitt method for the qualitative analysis of polyethylene glycols and their derivatives is discussed as well as gravimetric, colorimetric, and volumetric procedures for the determination of these compounds.

(276) E. G. Brown and T. J. Hayes, "The Absorptiometric Determination of Polyethyleneglycol Mono-Oleate," *Analyst*, Vol. 80, pp. 755-767 (1955).

Procedure depends upon formation under controlled conditions of a blue complex between the compound and ammonium cobalthiocyanate, this complex being extracted with chloroform. Measurement of the optical density is made either at 318.5  $m\mu$  or 620  $m\mu$  and given concentration of polyethyleneglycol mono-oleate and the procedure applicable to concentrations of 0 up to one gram per liter. Quaternary compounds giving positive reaction contain a long-chain alkyl group. For polyethyleneglycol monoesters only compounds prepared from original polyethyleneglycols of average molecular weight greater than 300 give the reaction. Effect of terminal grouping is important as a distearate does not react whereas the monostearate is positive.

(277) D. E. C. Corbridge and E. J. Lowe, "Quantitative Infrared Analysis of Condensed Phosphates," *Analytical Chemistry*, Vol. 27, pp. 1383-1387 (1955).

Quantitative determination of condensed phosphates in the form of dry powders. Potassium bromide pressed disc technique

demonstrated with a number of mixtures. Method compares favorably with X-ray and chemical methods as to speed and accuracy.

(278) B. F. Davis, K. E. Wattman, and H. C. Speel, "Maintaining Nonionic Concentration," *Soap*, Vol. 31, No. 12, pp. 73, 75, 77, 79 (1955).

Technique of Karabinos of titrating non-ionic surface active agents with phenolic solution demonstrated as useful for control of plant operations. Method requires development of a standard curve for each compound.

(279) F. Franks, "Partition Chromatography of Synthetic Detergents," *Nature*, Vol. 176, pp. 693-694 (1955).

Alkylaryl sulfonates, secondary alkyl sulfates, quaternary ammonium and pyridine salts can be tested in this manner. Method of ascending and circular chromatography used. Paper impregnated with mobile phase of long-chain fatty alcohol in 1 per cent ethanol. The chromatogram is developed for 10 to 24 hr and paper dried. Immersion in a solution of cupric acetate and sprayed with dilute solution of rhodamine 6GB. After drying the detergent spots were crimson on a pink background in daylight and dark purple on a yellow background under ultraviolet.  $R_f$  values were determined and are given for alkyl sulfates. Other details and data are given for other compounds.

(280) T. Green, R. P. Harker, and F. O. Howitt, "Use of Adsorption Columns in the Analysis of Oil-in Water Emulsions," *Analyst*, Vol. 80, pp. 470-475 (1955).

Method described contains many precautions and depends upon the adsorption of the constituents of the emulsion on an ion-exchange resin and charcoal with subsequent elution of the oil, grease, and detergent constituents with suitable eluents. An apparatus containing 6 adsorption columns and a communal vacuum line was constructed.

(281) C. G. Herrera, "Synthetic Detergents, Their Evaluation and Analysis," *Grasas y aceites* (Seville, Spain), Vol. 6, pp. 144-149 (1955); *Chemical Abstracts*, Vol. 50, p. 2193 (1956).

A review with 54 references.

(282) T. Ino, T. Kondo, K. Meguro, and O. Yoda, "Mutual Action between Surface-Active Substances and Fluorescent Dyes," *Journal, Chem. Soc. Japan, Pure Chem. Sect.* 76, pp. 220-222 (1955); *Chemical Abstracts*, Vol. 49, p. 13671 (1955).

Mutual titration of anionic and cationic agents was conveniently carried out with use of fluorescent indicator. Such indicators were eosin and rhodamine 6GCP.

(283) J. V. Karabinos, J. J. Hazdra, and G. E. Kapella, "Nonionic Detergents," *Soap*, Vol. 31, No. 4, pp. 49-50, 114 (1955).

Method based upon mixed melting point

of the sample and camphor under specified conditions. The number of ethylene oxide units in the detergents is calculated from the mixed melting points, and the weights of materials used.

(284) J. V. Karabinos, "Detergent Solubility," *Soap*, Vol. 31, No. 6, pp. 50-51 (1955).

Titration of nonionic polyoxyethylene ethers and esters with aqueous phenol studied to determine relations between hydrophobic and hydrophilic chain lengths. Method useful in control analysis of ethenoxy chain length where the hydrophobic group is known, but not useful for the determination of ethenoxy chain length of unknown detergents.

(285) C. Kortland and H. F. Dammers, "Qualitative and Quantitative Analysis of Mixtures of Surface-Active Agents with Special Reference to Synthetic Detergents," *Journal, Am. Oil Chemists' Soc.*, Vol. 32, pp. 58-64 (1955).

Qualitative tests are recommended for anionic, cationic, and nonionic agents and are for nitrogen, naphthalene rings, 1-ring aromatic compounds, secondary alcohols, and pyridinium compounds. Gives scheme by extraction methods and further analyses for detergent components.

(286) L. E. Kuentzel, "Calcium Carbonate as an Internal Standard for Quantitative Infrared Analysis," *Analytical Chemistry*, Vol. 27, p. 301 (1955).

An infrared spectrophotometric method for determination of alkylbenzene sulfonate and sodium toluene sulfonate with calcium carbonate as an internal standard discussed.

(287) M. Kurata, "Analysis of Nonionic Detergents," *Journal, Japan Oil Chemists' Soc.*, Vol. 4, pp. 293-298 (1955); *Journal, Am. Oil Chem. Soc.*, Vol. 33, p. 139 (1956).

A review with 25 references.

(288) A. Lassieur, "Analysis of Alcohol Sulfates and of Alkylarylsulfonates," *Chimie analytique*, Vol. 37, pp. 39-44 (1955); *Chemical Abstracts*, Vol. 49, p. 7269 (1955).

Preliminary paper to an extended study of analysis of surface-active agents.

(289) A. M. Lawson, L. A. Jones, and O. T. Aepli, "Volumetric Determination of Soluble Silicates in Detergents," *Analytical Chemistry*, Vol. 27, pp. 1810-1811 (1955).

Rapid volumetric method for determination of soluble silica in alkaline detergents. Sodium oxide can also be determined on the same sample. Modifications necessary for the determination of soluble silica in the presence of carbonates, phosphates and wetting agents. Results are in agreement with gravimetric values within  $\pm 0.05$  per cent of silica. Method depends upon conversion of the soluble silica to the form of  $\text{Na}_2\text{SiF}_6$  which is attained by differential titration

using methyl red and methyl red-xylene cyanol FF indicators.

(290) J. Longwell, W. D. Maniece, "Determination of Anionic Detergents in Sewage, Sewage Effluents, and River Waters," *Analyst*, Vol. 80, pp. 167-171 (1955).

Use of methylene blue, chloroform separation. The fully separated solvent solution is measured photometrically using calibration curves for quantitative estimation.

(291) L. E. Netherton, A. R. Wreath, and D. N. Bernhart, "Determination of Soluble Ortho-, Pyro- and Triphosphates in the Presence of Each Other," *Analytical Chemistry*, Vol. 27, pp. 860-861 (1955).

Rapid, simple, and accurate procedure for analysis of mixtures containing water-soluble phosphates developed, based upon selective alkaline hydrolysis of triphosphate combined with modified colorimetric procedure for determining orthophosphate.

(292) V. W. Reid, T. Alston, and B. W. Young, "Qualitative Analysis of Surface-Active Agents," *Analyst*, Vol. 80, pp. 682-689 (1955).

Considerable simplification claimed if an ultraviolet spectrogram covering 2100-3500 Å. is prepared. Four distinct groups are anionic compounds containing no nitrogen, nonionic compounds containing no nitrogen, anionic compounds containing nitrogen, and cationic compounds containing nitrogen. About 100 commercial compounds have been examined and the classification agreed with data in the literature.

(293) M. J. Rosen, "Detection of Surface-Active Phenol Ethers with Sulfuric Acid and Formaldehyde," *Analytical Chemistry*, Vol. 27, pp. 111-114 (1955).

Test consists of the reaction of formaldehyde with an aromatic nucleus in the presence of sulfuric acid to form a carbonium ion, which polymerizes to a colored complex. A positive reaction is obtained only with compounds in which the aromatic nucleus is not deactivated by substituents such as chlorine, sulfonic radical, nitrogen dioxide radical, carboxy group, and a  $\text{CH}_2\text{N}(\text{R})_3$  radical. Compounds containing benzenoid nuclei give red complexes, while condensed polynuclear aromatic compounds give green, blue, or purple complexes.

(294) M. J. Rosen, "Detection of 2-Hydroxyethylamines by Pyrolysis with Sodium Chloroacetate," *Analytical Chemistry*, Vol. 27, pp. 114-116 (1955).

This group present in several classes of surfactants, such as diethanolamine-fatty acid condensates of the Ninol type, (2-hydroxyethyl) imidazolines, and various 2-hydroxyethylated long chain amines, can be detected qualitatively by quaternizing the group with sodium chloroacetate then pyrolyzing the compositions to yield acetaldehyde which reacts with a solution of

sodium nitroprusside containing diethanolamine to form a blue color. This is a reversal of the Simon test used to detect secondary amines.

(295) M. J. Rosen, "Detection of Surface-Active Agents Containing Polyoxyethylene or Polyoxypropylene Groups by Pyrolysis with Phosphoric Acid," *Analytical Chemistry*, Vol. 27, pp. 787-790 (1955).

Gentle heating of dry material containing polyoxyethylene or polyoxypropylene groups with phosphoric acid yields MeCHO or EtCHO which form blue or orange colors when passed into an aqueous sodium nitroprusside and diethanolamine solution. Glycerides yield acrolein which interferes. Other functional groups do not interfere.

(296) N. A. Rudnev, "Use of Surface-Active Substances for Preventing Coprecipitation with Sulfide Precipitates," *Zhurnal Analiticheskoi Khimii*, Vol. 10, pp. 217-221 (1955); *Chemical Abstracts*, Vol. 50, p. 109 (1956).

The action of a number of basic dyes, protective colloids, and aldehydes in preventing coprecipitation of tellurium with arsenic trisulfide was studied.

(297) N. Schönfeldt, "A Method for the Quantitative Determination of Ethylene Oxide Adducts in their Aqueous Solutions or Dispersions," *Journal, Am. Oil Chemists' Soc.*, Vol. 32, pp. 77-79 (1955).

Quantitative method consists of precipitation of the ethylene oxide adduct in hydrochloric acid solution of known quantity of ferrocyanic acid, filtration and titration with zinc sulfate, and consumption of ferrocyanic acid determined. Reaction takes place on ethylene oxide chain and to precipitate adduct of 6 units of ethylene oxide requires approximately one mole of ferrocyanic acid.

(298) N. Schönfeldt, "A Colorimetric Method for the Determination of Ethylene Oxide Adducts," *Kolloid-Zeitschrift*, Vol. 142, p. 164 (1955); *Chemical Abstracts*, Vol. 50, p. 3954 (1956).

An azo dye is dissolved in an aqueous solution or dispersion of the adduct. The solution is overlaid with an organic solvent, such as xylene or a paraffin hydrocarbon. The adduct is then precipitated by addition from a buret of a solution of silicotungstic acid; the precipitate rises along with the dye to the liquid interphase, and the end point is taken at decolorization of the aqueous layer.

(299) H. Stüpel and A. v. Segesser, "Possible Applications of the p-Toluidine Method for the Analysis of Synthetic Detergents," *Fette und Seifen Anstrichmittel*, Vol. 57, pp. 344-346 (1955).

Procedure given for rapid estimation. Carbon tetrachloride extraction of the precipitated complex takes up alkyl and alkyl-

aryl sulfonates, etc. The ether extract required for alcohol sulfates—lower molecular weight sulfates do not react quantitatively. Fatty acid condensation products containing nitrogen, such as amides or amines can also be separated from the water phase with ether without emulsions. Determination of molecular weight for usage with lower alkyl sulfates for estimation of factor to be applied.

(300) A. Szerat, "Determination of the Propionyl Index According to Pesez. Application of the Assay of Polyethylene Glycols," *Annales pharmaceutiques francaises*, Vol. 13, pp. 516-519 (1955); *Chemical Abstracts*, Vol. 50, p. 2924 (1956).

The hydroxyl groups are acylated with propionic anhydride in the presence of p-toluene sulfonic acid. The unreacted anhydride is treated with a known amount of aniline in benzene, the excess of which is determined with perchloric acid with crystal violet.

(301) E. Vecchi, G. Semeraro, Th. Hoffmann, and J. Büchi, "Quaternary Ammonium Compounds. Polarographic Measurement of their Activity with Relation to their Bactericidal Potency," *Pharmaceutica Acta Helveticae*, pp. 228-250 (1955); *Chemical Abstracts*, Vol. 50, p. 1493 (1956).

Discussion of details of method.

(302) F. T. Weiss, A. E. O'Donnell, R. J. Shreve, and E. D. Peters, "Comprehensive Analysis of Sodium Alkylarylsulfonate Detergents," *Analytical Chemistry*, Vol. 27, pp. 198-205 (1955).

ASTM method D 855-46T modified to permit analysis of sodium alkylarylsulfonate materials. Method with certain precautions can be used with detergents containing inorganic or organic builders. Also applicable to sodium alkyl sulfates provided the residues are maintained in a basic condition prior to drying.

(303) W. S. Wise and N. O. Schmidt, "Amperometric Determination of Ethylenediaminetetraacetic Acid with Zinc Ions," *Analytical Chemistry*, Vol. 27, pp. 1469-1471 (1955).

Solution of ethylenediaminetetraacetate chelates titrated amperometrically with zinc sulfate solution at low pH.

(304) H. A. Zutraen and L. Ter Minasian-Saraga, "Color Change of Bromophenol Blue in the Presence of Quaternary Ammonium Salts," *Comptes rendus (Paris)*, Vol. 240, pp. 869-871 (1955); *Chemical Abstracts*, Vol. 7977 (1955).

Shift in absorption maximum of bromophenol blue from 592 m $\mu$  to 605 m $\mu$  with increasing concentration of certain cationic detergents is not connected with critical concentration for micelle formation, as these appear at a higher concentration.

1956

(305) 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," *Analysis*, 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.

(306) G. P. Edwards and M. E. Cinn, "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.

(307) 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  $10^{-5}$  molar. 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 dodecylpyridine bromide. Method applicable at wide pH variations and salt concentrations. Used orange II dye.

(308) 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.

(309) 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.

(310) 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.

(311) H. J. Weiser, "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.