

BIBLIOGRAPHICAL ABSTRACTS
OF
METHODS FOR ANALYSIS OF SYNTHETIC DETERGENTS

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For SUBCOMMITTEE T-2 of COMMITTEE D-12

FOREWORD

The volume of analytical work with surfactant and syndet compositions continues, with application of either newly developed or refined equipment. Chromatographic procedures of all kinds as applied to these materials have had much attention, as have spectrophotometric examinations. A number of newly published official methods indicate an increase in standardization activities.

The matter of improved subject indexing was brought to my attention by W. B. Smith of Marchon Products Limited, Whitehaven, Cumberland, England. The cumulative subject index which features this edition of the bibliography was prepared utilizing his valuable constructive criticisms. His interest and cooperation are sincerely appreciated.

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BIBLIOGRAPHICAL ABSTRACTS

1933

(10B) R. Krammes and C. Maresh, "Identification of Textile Finishes," *Am. Dyestuff Reporter*, Vol. 42, pp. P317-327 (1933).

Textile extracted with ethanol and the surfactants are in this extract. These identified by treating individual portions with hydrochloric acid and potassium hydroxide. Reaction as to clarity separates as to soap, cationic or sulfonated products. To portions, barium chloride solution is added, and sulfated or sulfonated oils precipitate. Confirmatory test by immersing copper wire in solution attaching them to 45 v "B" battery. A deposit at the positive wire indicates anionic agent, and at the negative wire cationic. Acidification of solution with hydrochloric acid gives better deposit if cationic.

1940

(34A) F. O. W. Meyer, "Permeability and Absorption Properties of Bituminous Coatings," *Allgemeine Oel und Fett Zeitung*, Vol. 37, pp. 165-171, 207-211 (1940); *Chemical Abstracts*, Vol. 35, p. 7578 (1941).

The reactions for the production of various types of sulfated and sulfonated oils, alcohols and complex sulfonates such as amides, amines, condensation products and the kind, are classified in a schematic outline, and methods given for determination and identification of true sulfonic acids in the presence or absence of solvents, soaps and sulfuric esters.

1941

(34Aa) F. M. Archibald and E. L. Baldeschwieler, "Analysis of Petroleum Oil-Soluble Sulfonic Acid Soaps," *Indus-*

trial and Engineering Chemistry, (Analytical Edition), Vol. 13, pp. 608-611 (1941).

Source and types of petroleum sulfonic acid soaps discussed. Evaluated for specific gravity, color, viscosity, water content, salt content, oil content, alkalinity and soap content.

(34B) E. Chambers and G. W. Watt, "The Identification of Sulfonic Acids," *Journal of Organic Chemistry*, Vol. 6, pp. 376-383 (1941).

Use of S-benzylthiuronium chloride in identification of sulfonic acids investigated and the melting points of 34 acids determined. Applied mainly to naphthalene derivatives. Benzene, toluene and xylene sulfonic acid derivatives tested.

(35A) E. H. Huntress and J. S. Autenrieth, "Identification of Organic Compounds. IV.—Chlorosulfonic Acid as a Reagent for Identification of Alkylbenzenes," *Journal, Am. Chemical Soc.*, Vol. 63, pp. 3446-3448 (1941).

Principle involves introduction of one sulfonyl group into the aromatic nucleus by direct treatment with excess chlorosulfonic acid in chloroform followed by conversion of the resultant sulfonyl chlorides into the corresponding sulfonamides. Melting points of many alkylbenzenes given, the identification being extended to 37 aromatic hydrocarbons.

1942

(39A) M. W. Partridge, "Forensic Chemical Examination of Materials Containing Soaps. I.—Detection of Soaps and Soap Stains," *Quarterly Journal of Pharmacy and Pharmacology*, Vol. 15, pp. 21-30 (1942); *Chemical Abstracts*, Vol. 36, p. 4777 (1942).

Common soaps and soap stains on fabrics can be qualitatively compared in ultraviolet light. The iron hydroxamate test for carboxylic acids is a sensitive test for soap. The saturated fatty acids can be characterized as acids containing seven or more carbon atoms by conversion into the corresponding ketones, and formation of their 2,4-dinitrophenylhydrazones. For qualitative tests, soaps can be extracted from fabrics with alcohol. Interfering substances can be removed from this extract with ether.

(42A) I. S. Shupe, "Identification of Some Amine Emulsifiers (For Cosmetics)," *Journal of the Association of Official Agricultural Chemists*, Vol. 25, pp. 227-230 (1942).

Primary and secondary amines identified as their benzenesulfonyl, *p*-toluenesulfonyl or *p*-bromobenzenesulfonyl derivatives, and the melting points of many given.

1944

(50A) E. W. Blank and L. E. Utter, "Qualitative Test for Bicarbonate Ion in Soap Products," *Oil and Soap*, Vol. 21, pp. 27-28 (1944).

The test consists of transferring 25 ml of hypochlorite solution (25 g to 1000 ml) to a 60 ml separatory funnel, adding 25 ml KBr solution (75 g to 1000 ml) and adding 5 ml carbon tetrachloride, then 0.5 to 5 g of sodium bicarbonate sample. The separatory funnel is shaken and the carbon tetrachloride allowed to settle and a brown or pink color noted. The approximate amount of bicarbonate is noted from the depth of color.

(50B) W. Gottschaldt, "Determination of Silicates in Laundering Compounds," *Fette und Seifen*, Vol. 51, p. 432 (1944); *Chemical Abstracts*, Vol. 44, p. 6662 (1950).

Speedy gravimetric determination of silica, by mixing 1.5 to 2.0 g ammonium chloride, 15 ml concentrated hydrochloric acid, heating the mixture 10 min on a steam bath, diluting with 50 ml boiling water, filtering, and igniting the precipi-

tate. In the absence of phosphates, a colorimetric method using the color reaction with ammonium molybdate was suggested.

1945

(60A) C. L. Ogg, W. L. Porter, and C. O. Willits, "Determining the Hydroxyl Content of Certain Organic Compounds; Macro and Semimicro Methods," *Industrial and Engineering Chemistry* (Analytical Edition), Vol. 17, p. 394 (1945).

Modification of macroprocedures for determining hydroxyl content of hydroxylated fatty acids and alcohols using internal indicator. For colored solutions potentiometric method developed. Semimicro procedure described. Acetylating solution of one volume acetic anhydride in three volumes pyridine and hot water hydrolysis used.

1946

(77A) S. R. Palit, "A Direct Volumetric Method for the Analysis of Soap," *Oil and Soap*, Vol. 23, pp. 58-60 (1946).

Mixture of ethylene or propyleneglycol and isopropanol excellent solvent for soap, permitting direct titration with acid, in same solvent mixture to sharp endpoint.

1947

(88A) W. H. Hunt and M. H. Neustadt, "Rapid and Improved Methods of Soap Analysis," *Journal of the American Oil Chemists' Soc.*, Vol. 24, pp. 1-3 (1947).

Described are methods alternative to official procedures, for moisture, matter insoluble in water and in alcohol, total anhydrous soap, chloride, unsaponified and unsaponifiable matter, rosin, and titer test.

(89B) Miroslav Liby, "Detection and Determination of Tylose in Soap and Detergents," *Chemicky obzor*, Vol. 22, pp. 213-215 (1947); *Chemical Abstracts*, Vol. 42, p. 1750 (1948).

Tylose is hydrolyzed by boiling hydro-

chloric acid and the dextrose determined with Fehling solution.

1948

(97A) E. W. Blank, "Semimicroanalysis of Soap. I. Semimicro Determination of the Usual Constituents," *Journal of the American Oil Chemists' Soc.*, Vol. 25, pp. 438-446 (1948).

Procedure for the complete semimicro-analysis of soap described. Methods are analogous with the usual methods of analysis and require very little special apparatus. The results were comparable with those obtained by macro methods.

(106A) H. Keith, "A New and Simple Identification of Tylose," *Deutsche Lebensmittel-Rundschau*, Vol. 44, pp. 232-233 (1948); *Chemical Abstracts*, Vol. 43, p. 2126 (1949).

The material to be tested is boiled with an excess of 10 to 15 per cent sodium hydroxide solution for a short time, and while still hot a solution of copper sulfate is added. In case tylose is present the clear supernatant solution will be colored blue-violet. With small amounts the color appears after short standing.

(107A) P. Leman, "Determination of Ethylenediaminetetraacetic Acid in Cleaners and Preparations used to Remove Lime Deposits," *Annales des falsifications et des fraudes*, Vol. 51, pp. 171-172 (1948); *Chemical Abstracts*, Vol. 53, p. 2000 (1959).

Mix 1 cc of a 10 per cent solution of the preparation with 0.5 cc acetic acid, 0.2 cc of 1 per cent cobalt nitrate solution, and 1 cc hydrogen peroxide of 110 volumes. Heat 15 min at 100C, adjust to 5 cc and compare colorimetrically with a set of standards.

(111A) J. B. Wilson, "Report on Quaternary Ammonium Compounds in Foods," *Journal of the Association of Official Agricultural Chemists*, Vol. 31, pp. 480-484 (1948); *Chemical Abstracts*, Vol. 43, p. 7151 (1949).

Food samples containing varying quantities of lauryldimethylbenzylammonium

chloride were examined by the ferricyanide method.

1949

(111Aa) E. W. Blank and B. Rotolo, "Semimicro Analysis of Soap. III.—Semimicro Determination of Potassium," *Journal of the American Oil Chemists' Soc.*, Vol. 26, pp. 102-103 (1949).

Sodium uranyl zinc acetate was used to precipitate the sodium from a solution of the charred soap mass, and the precipitate weighed and the potassium calculated by difference. Satisfactory agreement between the macro and the micro method was found.

(116A) S. H. Maron, I. N. Ulevitch, and M. E. Elder, "Fatty and Rosin Acids, Soaps, and Their Mixtures. Conductometric and Potentiometric Analysis," *Analytical Chemistry*, Vol. 21, pp. 691-695 (1949).

Utilizing solutions of isopropanol-water as solvent, potentiometric titrations of limited value, and conductometric methods quite suitable. Direct titrations with base for fat and rosin acids, or with acid for soaps. By indirect titration soap and acid content, or soap and alkali contents of soap and soap solutions determined with single titration.

(116B) O. Micaelle and P. Desnuelle, "The Stability of Sulfuric Esters of Hydroxyethylamides (Sulfated Fatty Amides), and the Analysis of Lyes Containing Fatty Amides," *Oléagineux*, Vol. 4, pp. 720-725 (1949); *Chemical Abstracts*, Vol. 44, p. 3725 (1950).

Discussion of degree of hydrolysis of O-SO₃H linkage. Amide linkage does not hydrolyze, but soap content of commercial product is increased up to 3.2 per cent during treatment corresponding to proportion of fatty acid chains bound in ester linkage. Sulfate of hydroxamide slightly more stable than that of dodecyl alcohol.

(116C) W. J. Miller and J. T. R. Andrews, "The Determination of Potassium in Soap and Mixed Caustic Lye," *Journal of the American Oil Chemists' Soc.*, Vol. 26, p. 309 (1949).

A modification of the method for the determination of potassium, as the periodate, and interferences noted.

(116D) W. K. Moseley, "Field Testing for Quaternary Ammonium Compounds," *Milk Plant Monthly*, Vol. 38, No. 7, pp. 76-77 (1949); *Chemical Abstracts*, Vol. 44, p. 2701 (1950).

Procedure involves formation of a red precipitate after the addition of standard eosin indicator to the quaternary solution to be tested. The quantity of standardized anionic solution required to change the color of this mixture from red or pink to white provides a direct measure of the concentration of the quaternary. The preparation of test paper for determining quaternary compounds is also described.

1950

(122A) E. C. Bate-Smith and R. G. Westall, "Chromatographic Behavior and Chemical Structure: Naturally Occurring Phenolic Substances," *Biochimica Biophysica Acta*, Vol. 4, pp. 427-440 (1950); *Chemisches Zentralblatt*, Vol. 121, p. 1801 (1950).

R_f values in butanol-acetic or metacresol-acetic acid were reproducible to ± 0.02 . Dyes used belong to the class of flavones, etc.

(122B) R. Bernstein and M. Haftel, "The Determination of Borax in Soap," *Journal of the American Oil Chemists' Soc.*, Vol. 27, pp. 45-47 (1950).

An accurate method provides for simultaneous removal of soap, silicates, phosphates, and carbonates by precipitation with strontium chloride and use of methyl purple instead of methyl red indicator during adjustment of the solution acidity prior to titration with standard alkali. The procedure is not applicable to synthetic detergents which do not precipitate, as above.

(125A) C. Fuchs, "Titrimetric Soap Analysis," *Fette und Seifen*, Vol. 52, pp. 23-26, 105-108 (1950); *Chemical Abstracts*, Vol. 44, p. 7570 (1950).

Sample of unfilled soap in neutral 80 per cent ethanol titrated with standard acid to phenolphthalein endpoint and

then with standard alcoholic hydrochloric acid to bromophenol blue endpoint. Total fatty acids determined in soaps containing lauric or synthetic fatty acids by splitting with mineral acid, extracting with ether and determined gravimetrically. Mersolates determined by difference.

(129A) S. E. Kay, "Experiments at Mogden on the Evans' Modified Methylene Blue Method for the Estimation of Anionic Detergents in Sewage," *Journal and Proceedings*, Inst. Sewage Purification, (1950), pp. 403-407, 408-415; *Chemical Abstracts*, Vol. 47, p. 4018 (1953).

Evans' (reference (125)) modified method involves measurement of depth of blue color of solution in chloroform at pH. 3.25 and 7.0. Extrapolation of straight line joining these two points to a pH. —2.0 reported to give detergent concentration. Effects of thiocyanate, nitrate and anion-active bodies in urine not removed and Evans' method unsatisfactory.

(141A) E. T. Roe and D. Swern, "Determination of Long-Chain Hydroxamic Acids," *Analytical Chemistry*, Vol. 22, pp. 1160-1162 (1950).

Procedure consists in the hydrolysis to carboxy acid and hydroxylamine hydrochloride with known excess of aqueous alcoholic hydrochloric acid followed by titration of either the excess hydrochloric acid or the hydroxylamine hydrochloride. The former technique gives slightly low results; the latter slightly high. Hydroxylamine hydrochloride cannot be titrated in the presence of fatty acids containing 10 or less carbon atoms. Precision of duplicate determinations usually within 1 per cent.

1951

(164A) E. R. Garrett and R. L. Guile, "Potentiometric Titrations of a Polydicarboxylic Acid: Maleic Acid-Styrene Copolymer," *Journal*, Am. Chemical Soc., Vol. 73, pp. 4533-4535 (1951).

Procedures given for analysis by aque-

ous and anhydrous titrations. For latter used acetone solvent and methanolic sodium hydroxide.

(167B) É. F. Hillenbrand, Jr., W. W. Sutherland, and J. N. Hogsett, "Determination of Residual Glyoxalidine Fungicides on Apples and Cherries," *Analytical Chemistry*, Vol. 23, pp. 626-629 (1951).

Titration of glyoxalidines and hydrolysis products, which are cationic surfactants, with standard solution of anionic agent in two-phase system of chloroform and water using methylene blue indicator. Based on Jones method, (reference (60)).

(174A) R. Munier and M. Macheboeuf, "Partition Microchromatography on Paper of Alkaloids and Various Biological Nitrogenous Bases. V.—Separation of the Nitrogenous Constituents of the Phosphoaminolipides; Choline, Ethanolamine, and Serine," *Bulletin de la société de chimie biologique*, Vol. 33, pp. 862-867 (1951).

Good separation with tail-free spots obtained with mixture of butanol 50, ethylene chlorohydrin 10, concentrated ammonium hydroxide 5, and water 16. Aqueous saturated butanol acidified with acetic acid also suitable.

1952

(189A) R. Castagnou and R. Quilichini, "Color Test for the Dioxyethylenic Groups," *Bulletin de la société de pharmacie de Bordeaux*, Vol. 90, pp. 118-121 (1952); *Chemical Abstracts*, Vol. 46, p. 9018 (1952).

Labat's reaction (heat 0.1 ml of alcoholic solution of sample in 2 ml sulfuric acid with 0.1 ml 5 per cent solution of gallic acid at 100 C) depends on presence of $\text{—O} \cdot \text{CH}_2 \cdot \text{O—}$ grouping. The same green changing to blue formed by this reaction is obtained with polyoxyethylene derivatives and with some samples of ethyl ethers not treated to remove peroxides. Possible oxidation products discussed.

(200A) S. H. Maron, I. N. Ulevitch and M. E. Elder, "Determination of

Soap, Acid, and Alkali in Synthetic Latexes," *Analytical Chemistry*, Vol. 24, pp. 1068-1070 (1952).

Conductometric titrations can be utilized for analysis of GR-S latices, prepared with either fatty or rosin acid soaps. Soap and free acid content determined by individual direct titrations with acid or base, or by use of indirect procedure involving addition of hydrochloric acid followed by addition of base in single titration.

(201A) R. Mitchell and B. B. Clark, "Determination of Quaternary Ammonium Compounds Including Acetylcholine, Tetraethylammonium, and Hexamethonium," *Proceedings of the Society of Experimental Biology and Medicine*, Vol. 81, pp. 105-109 (1952); *Chemical Abstracts*, Vol. 47, p. 1213 (1953).

General method presented for determination in buffer solutions, nerve homogenates, diluted serum or plasma, and urine of quaternary ammonium compounds in which at least one chain is 4 carbons or longer, or is $\text{C}_6\text{H}_5\text{—CH}_2\text{—}$, or some modification thereof. Method is based on method of Auerbach and uses bromophenol blue as the complexing dye. The lower limit of sensitivity is about 2 gamma per sample. Certain adaptations of the method permit the estimation of these compounds in undiluted serum or plasma, and other modifications make it suitable for determination of acetylcholine and other compounds in concentrations as low as 1 or 2 gamma. Tertiary amines do not interfere.

(202a) R. Neu, "Analysis of Laundering and Cleaning Compounds. IX.—Reaction of Technical (Sodium) Tripoly Phosphate and (Sodium) Hexameta Phosphate with Benzylisothioure Hydrochloride," *Fette und Seifen*, Vol. 54, p. 682 (1952); *Chemical Abstracts*, Vol. 47, p. 12846 (1953).

Benzylisothioure hydrochloride (10 per cent solution in 10 per cent acetic acid) gives precipitate with sodium metaphosphate even in the presence of 50 times the amount of tripolyphosphate. Reaction sensitive to one part in 11,100.

(204A) A. G. Richardson, J. S. Pierce, and E. E. Reid, "N-Aryl-N'-alkyloxamides for the Identification of Primary Alkyl Amines," *Journal, Am. Chemical Soc.*, Vol. 74, pp. 4011-4012 (1952).

Several aryl oxamate esters are useful as reagents for the identification of aliphatic amines. They react readily and selectively with primary aliphatic amines to give oxamides, ArNHCOCONHR . From six reagents and twenty amines 107 derivatives were prepared and characterized. Reagents advantageous over acid chlorides and aryl isocyanates in their indifference to water and alcohols.

(205A) R. Senju, "A New Method for Determining Lignosulfonic Acid in Sulfite Spent Liquor," *Bulletin of the Chemical Society of Japan*, Vol. 25, pp. 131-132 (1952); *Chemical Abstracts*, Vol. 47, p. 10222 (1953).

Liquor treated with polyglycolglucosamine and the excess titrated with polyvinyl alcohol sulfate. Solution becomes turbid through the formation of a precipitate in excess polyglycolglucosamine; at the equivalence point the colloidal solution clears and the precipitate becomes flocculent. Very dilute solutions can be titrated with toluidine blue.

(206A) H. Stüpel, "Analysis and Evaluation of Synthetic Dishwashing, Laundering, and Cleansing Agents," *Seifen-Öle-Fette-Wachse*, Vol. 78, pp. 599-601, 628-629 (1952); Vol. 79, pp. 4-5 (1953); *Chemical Abstracts*, Vol. 47, p. 6681 (1953).

Discussion of tests and per cent active material.

(207a) D. E. Terry, K. R. Eilar, and O. A. Moe, "Fatty Amine Products of High Molecular Weight. Quantitative Titration with Acetic Acid," *Analytical Chemistry*, Vol. 24, pp. 313-314 (1952).

Use of perchloric acid as titrant in glacial acetic acid as solvent with wide variety of fatty amine products including primary, secondary or tertiary fatty amines and their acetates, fatty amino nitriles and compounds containing both primary and secondary amino groups. Colorimet-

ric titration with crystal violet satisfactory for acetate materials.

1953

(220A) K. W. Ellis and N. A. Gibson, "The Micro-Estimation of Cobalt with Triphenylmethylarsonium Thiocyanate," *Analytica Chimica Acta*, Vol. 9, pp. 275-280 (1953).

Used in the estimation of cobalt in the tris(ethylenediamine) cobalt (III) triphosphate determination.

(225A) H. Grunze and E. Thilo, "Condensed Phosphates and Arsenates. XI.—Paper Chromatography of Polyphosphates," *Sitzungsberichte der deutschen Akademie der Wissenschaften zu Berlin (Klasse für mathematik und allgemeine Naturwissenschaften)*, No. 5, 26 pp. (1953) (published 1954); *Chemical Abstracts*, Vol. 49, p. 6028 (1955).

Chromatographic procedures given for identification and separation of higher polyphosphates up to octapolyphosphate. Some procedures given for low molecular weight polyphosphates. For high molecular weight products used solvent of isopropanol 70 cc, water 10 cc, 20 per cent trichloroacetic acid 20 cc, 25 per cent ammonium hydroxide 0.3 cc. A formula relation between the number of condensed polyphosphates and R_f values was established.

(229a) J. E. Jackson, "Determination of Primary Fatty Amines in Amine Mixtures. Potentiometric Titration Technics," *Analytical Chemistry*, Vol. 25, pp. 1764-1765 (1953).

Use of chloroform as solvent, the perchloric acid titration method becomes satisfactory. Details of procedure given.

1954

(237A) A. Badinaud and J. Guiraud, "Mechanism of Auerbach's Reaction Between Dyes and Quaternary Ammonium Compounds," *Travaux de la société de pharmacie de Montpellier*, Vol. 14, No. 3, pp. 119-122 (1954); *Chemical Abstracts*, Vol. 49, p. 8747 (1955).

Most of methods for quaternaries developed on Auerbach's reaction whereby certain acidic dyes react with quaternary

compounds to give product insoluble in water but soluble in certain organic solvents. Shown from shift in absorption spectrum of bromophenol blue when cetyltrimethylammonium bromide added that reaction probably involves complex formation.

(239A) R. T. Blickenstaff, J. R. Schaeffer, and G. G. Kathman, "Semimicro Determination of Hydroxyl Group in Long Chain Alcohols," *Analytical Chemistry*, Vol. 26, pp. 746-748 (1954).

Method based on sulfation of alcohol with chlorosulfonic acid, neutralization of the sulfation mixture, and titration of the sodium alkyl sulfate with cetyltrimethyl ammonium bromide using the Epton method (reference (84)). Interferences are noted, but are relatively few.

(241A) J. Carlos, "Análisis Químico Cuantitativo de los Detergentes Sintéticos Aniónicos," Buenos Aires: *Color (Aitea)*, 8 pp. (1954); *Chemical Abstracts*, Vol. 48, p. 1190 (1954).

(244a) H. Étienne, "Evaluation of Commercial Phosphates," *Industrie chimique belge*, Vol. 19, pp. 1021-1024 (1954); *Journal of Applied Chemistry*, (London), Vol. 5, p. i-385 (1955); *Chemical Abstracts*, Vol. 52, p. 8594 (1958).

Qualitative tests for distinguishing between *ortho*-, *pyro*-, *tripoly*-, *tetrapoly*-, and *polymetaphosphates* are described. Tests include the benzidine color test for *orthophosphates* and a number of specific reactions of phosphates with aqueous $\text{Co}(\text{NH}_3)_6\text{Cl}_3$ in 10 per cent acetic acid. A slight modification of the method for determining total P_2O_5 by alkaline hydrolysis of the precipitated ammonium phosphomolybdate gives greater sensitivity and accuracy. Conditions governing the quantitative precipitation of barium phosphates at controlled pH (1.8-4) have been elucidated and procedures for determining sulfate, chloride ions, combined Na_2O , and alkalinity are given. The two different crystalline forms of *tripolyphosphate* can be distinguished by

the difference in temperature rise of each on hydration.

(244E) F. Fauvarque, "Determination of Tetrasodium Pyrophosphate in Sodium Tripoly Phosphate by X-ray Diffraction," *Industrie chimique*, Vol. 41, pp. 44-45 (1954); *Chemical Abstracts*, Vol. 48, p. 12614 (1954).

Estimation of small quantities of tetrasodium *pyrophosphate* in commercial *tripolyphosphate* depends on fact that addition of known amounts of *pyrophosphate* to sample results in proportional increase in intensity of the tetrasodium band (4.39\AA) without changing appreciably intensity of *tripolyphosphate* (4.57\AA). As little as 0.5 per cent can be determined, precision varies between 0.2 and 0.5 per cent absolute.

(253A) Fr. Jaminet, "Microchromatographic Separation on Paper of Some Ethylene Oxide Polymers," *Journal de pharmacie de Belgique*, Vol. 9, pp. 318-324 (1954); *Chemical Abstracts*, Vol. 49, p. 14266 (1955).

Solvent of butanol, water and glacial acetic acid after decantation by ascending paper chromatography permits separation of components of a mixture of Carbowax 6000, 4000, 1540, and polyethylene glycol 400. Applied to the separation of base components of ointments. R_f values given.

(254D) S. Lindenbaum, T. V. Peters, Jr., and W. Rieman, III, "Analysis of Mixtures of the Condensed Phosphates by Ion-Exchanging Chromatography," *Analytica Chimica Acta*, Vol. 11, pp. 530-537 (1954).

Ion-exchanging chromatography method described for separation and determination of each constituent of mixture of *ortho*-, *pyro*-, *tri*-, *trimeta*- and *tetrameta* phosphate. Mean error of 0.2 per cent of sample. Vanadomolybdate colorimetric method used for assay of eluted materials.

(255A) F. Micheel and H. Schweppe, "Chromatographic Separation of Higher Fatty Acids on Cellulose Acetate Paper," *Angewandte Chemie*, Vol. 66, (5), pp.

136-137 (1954); *Analytical Abstracts*, Vol. 1, 1874 (1954).

Saturated straight-chain fatty acids separated on cellulose acetate paper as the potassium salts of the corresponding hydroxamic acids in ethyl acetate:tetrahydrofuran:water ratio of 0.6:3.5:4.7 by volume and identified by the intense coloration with ferric chloride.

(256B) R. Neu, "Tetraphenyldiboron Oxide. III.—New Tests for Choline, Acetyl Choline and Other Quaternary Ammonium Compounds," *Zeitschrift für analytische Chemie*, Vol. 143, pp. 30-38 (1954); *Chemical Abstracts*, Vol. 48, p. 13545 (1954).

Variety of fairly low molecular weight quaternary compounds in the presence of flavanols give reddish orange precipitates with the complex boron oxide which show characteristic fluorescence under ultraviolet and easily distinguished from the yellow precipitates which flavanol and phenyldiboron oxide alone give. Similar behavior with high molecular weight compounds.

(259a) M. Ohashi, "The Determination of Surface-Active Substances in Sugar Solutions by the Polarographic Method," *Proceedings, Research Society Japan Sugar Refineries' Technologists*, Vol. 3, pp. 55-78 (1954); *Chemical Abstracts*, Vol. 49, p. 6633 (1955).

Determined by height of the polarographic maximum. 0.04 per cent potassium chloride solution used because height of maximum at this concentration stable. Details of application given.

(262B) S. Siggia, "Quantitative Organic Analysis via Functional Groups," pp. 8-21, John Wiley and Sons, Inc., New York, N.Y. (Second Edition) (1954).

Analysis of hydroxyl group whether primary, secondary, phenolic or enolic, by esterification, acetylation, phthalation and in the presence of primary or secondary amines, on adjacent carbon atoms. Other functional group analyses given.

1955

(274C) J. Bollé, P. Ragon, and T. Julig, "Physical Chemical Research on

Pure Polyethylene Oxide Derivatives, Analytical Applications," *Mémoires des Services chimiques de l'état* (Paris), Vol. 40, pp. 333-344 (1955); *Chemical Abstracts*, Vol. 52, p. 5915 (1958).

Molecular refraction and parachors were obtained for a series of fatty alcohols and their one to five mole adducts with ethylene oxide. Rheochors were determined with a MacMichael viscometer. The data were used in combination with mean molecular weights calculated from hydroxyl indexes obtained by the reaction with acetic acid in dry pyridine to determine the composition of the polyethylene oxide derivatives. Equations given for relation to mean molecular weight. Values given for the compounds tested.

(276a) J. Cason and G. A. Gillies, "Absorption and Chromatography of Fatty Acids on Charcoal," *Journal of Organic Chemistry*, Vol. 20, pp. 419-427 (1955).

Static adsorption isotherms for series of saturated, unsaturated and branched chain acids on charcoal determined. Use for separability by solvent displacement chromatography indicated, and equipment suggested.

(277B) W. Davey and J. R. Gwilt, "Analytical Applications of Sodium Lauryl Sulfate. II.—Volumetric Determination of Sulfates," *Journal of Applied Chemistry*, Vol. 5, (9), pp. 474-476 (1955); *Analytical Abstracts*, Vol. 3, p. 659 (1956).

Method of Gwilt (reference (280a)) used in determination of sulfate ion. This ion in sample solution is precipitated as barium sulfate, the excess barium ion then precipitated with sodium lauryl sulfate and the unchanged sodium lauryl sulfate titrated with benzalkonium chloride.

(279A) A. K. Ghosh and K. L. Roy, "Rapid Method for Estimation of Calcium and Magnesium in Foods and Other Phosphate-Bearing Substances," *Naturwissenschaften*, Vol. 42, pp. 644-645 (1955); *Chemical Abstracts*, Vol. 52, p. 2654 (1958).

The Versene titration method for calcium and magnesium was modified for phosphate-containing materials. An aliquot of the acid solution of food ash with 10-15 mg of calcium was neutralized with ammonia, then acidified with one to two drops concentrated hydrochloric acid, 3 cc 30 per cent stannic chloride solution added, 10 ml of 5 per cent NH_4OH solution and water to 50 ml. The mixture was warmed up to hydrolyze the tin salt, boiled for a few minutes and filtered. The filtrate is titrated in the standard Versene manner. If only calcium is present, 10 ml of 10 per cent sodium or potassium hydroxide and some murexide indicator are added prior to titration. For the combined amount of calcium and magnesium, 5 ml ammonium hydroxide-ammonium chloride buffer and Eriochrome Black T indicator are used.

(280a) J. R. Gwilt, "Analytical Applications of Sodium Lauryl Sulfate. I. The Volumetric Determination of Barium," *Journal of Applied Chemistry*, Vol. 5, (9), pp. 471-474 (1955); *Analytical Abstracts*, Vol. 3, p. 659 (1956).

Quantitative interaction with the barium ion to give a precipitate insoluble in water or alcohol. A known excess of sodium lauryl sulfate is added to the solution of the barium ion, precipitate filtered off and the unchanged lauryl sulfate titrated with a quaternary ammonium salt in the presence of methyl yellow indicator and chloroform.

(298C) A. Simon and E. Steger, "Microchemical Identification of Condensed Phosphates," *Naturwissenschaften*, Vol. 22, pp. 604-605 (1955); *Chemical Abstracts*, Vol. 51, p. 17598 (1957).

Typical crystals are formed from various types of phosphates by placing a grain of the unknown phosphate on an object carrier with a drop of dilute acetic acid and saturated hexaaminocobalt (III) solution. These said to be adequate for identification of the species.

(299B) C. R. Szalkowski and W. J. Mader, "Determination of Sodium (Carboxymethyl) Cellulose," *Journal of the*

American Pharmaceutical Association, Vol. 44, pp. 533-535 (1955); *Chemical Abstracts*, Vol. 49, p. 16351 (1955).

Sodium carboxymethylcellulose is determined by precipitating as the copper salt and determining colorimetrically with 2,7-naphthalenediol.

(302A) R. Wickbold, "Advances in the Determination of Anion-Active Substances with *p*-Toluidine Hydrochloride," *Fette, Seifen, Anstrichmittel*, Vol. 57, pp. 164-168 (1955); *Chemical Abstracts*, Vol. 50, p. 4531 (1956).

Optimal pH and choice of solvents for the procedure described.

1956

(308A) H. Barkworth and J. E. Chadburn, "The Effect of Teepol on the Methylene Blue Test," *Dairy Industries*, Vol. 21, pp. 785-786 (1956); *Chemical Abstracts*, Vol. 51, p. 16997 (1957).

Addition of Teepol gives reduction times differing not only from the average, but the differences vary greatly and are at times very large. The differences decrease when the reduction time is $4\frac{1}{2}$ hr or longer.

(309A) R. Bennewitz, "Determination of Ion Types of Capillary-Active Materials by Means of Color Indicators," *Fette, Seifen, Anstrichmittel*, Vol. 58, pp. 832-833 (1956); *Chemical Abstracts*, Vol. 52, p. 5005 (1958).

Color changes accompanying the addition of various indicators to anionic, cationic, and nonionic surfactants were determined and tabulated. Lists the various reagents used for the qualitative identification of the species present, and the color changes which occur with them.

(310A) V. Cerchez and O. Popescu, "Determination of Sulfonic Acids in Presence of Sulfuric Acid. Application to the Analysis of Sulfonic Acids," *Academia republicii populare Romine, Filiala Cluj, Studii si cercetari de chimie*, Vol. 7, No. 1-4, pp. 155-165 (1956); *Chemical Abstracts*, Vol. 52, p. 8851 (1958).

The sulfuric acid and the sulfonic acids are precipitated as slightly soluble barium salts. The difference in weight between the dry and ignited precipitate permits the calculation of barium sulfate corresponding to the sulfuric acid present. The procedure applied to the analysis of sulfonic acids present in different fractions of crude oils gave satisfactory results.

(317A) H. Étienne, "Determination of Ethylene Oxide Condensates (Polyglycolics) Through Precipitation by Means of Phosphotungstic Acid," Second International Congress of Surface Activity, Vol. IV, pp. 76-82 (1956).

The nonionic is precipitated as a phosphotungstic acid complex in a hydrochloric acid solution of barium chloride. The precipitated complex is washed with water, dried, weighed and calcined. Loss on calcination represents the active ingredient in the complex. Free or unreacted fatty alcohols can be determined by petroleum ether extraction of the liquid containing the complex. Much data given on recoveries and showing the small relative error of the method.

(317B) H. C. Evans, "Alkyl Sulfates. I. Critical Micelle Concentrations of Sodium Salts," *Journal of the Chemical Society* (London), pp. 579-586 (1956).

Concentration *versus* specific conductivity used to determine critical micelle concentration of *n*-sodium alkyl sulfates of 8 to 19 carbon atoms in length and position of sulfate groups ranges from terminal to medial position. Slopes of plots used to estimate number of gegenions in micelles. Data from these measurements used to identify unbranched chain primary and secondary alkyl sulfates.

(318A) F. Feigl and D. Goldstein, "Identification of Organic Compounds by Spot Tests. III. — Ethylenediaminetetraacetic Acid," *Anais da associação, química do Brasil*, Vol. 11, pp. 135-137 (1952) (published 1956); *Chemical Abstracts*, Vol. 50, p. 15325 (1956).

Property of ethylenediaminetetraacetic acid of preventing precipitation of nickel as dimethylglyoxime or zinc as 8-hydroxy-

quinolate used by drop comparison. To one drop add drop solution to be tested, to other water. To each add drop aqueous ammonia and drop dimethylglyoxime solution. Red color in blank if no sequestering agent present. Similar reaction with zinc 8-hydroxyquinolate.

(326A) I. Garcia and J. Couerbe, "Circular Paper Chromatography Applied to the Identification of Guanidino Derivatives of Biological Origin and Their Enzymic Degradation," *Bulletin de la société de chimie et de biologie*, Vol. 38, pp. 791-799 (1956); *Chemical Abstracts*, Vol. 50, p. 16936 (1956).

Technique for separation and identification of materials including urea and other guanidine derivatives.

(326B) I. Garcia and J. Couerbe, "Partition Chromatography of Quaternary Ammonium Antiseptics," *Chimie analytique*, Vol. 38, pp. 432-434 (1956); *Chemical Abstracts*, Vol. 51, p. 5373 (1957).

Separation of 5 to 15 gamma of long-chain quaternary ammonium ions based on separation by ascending paper chromatography with solvent containing pyridine, benzene, acetic acid, and water, ratios of 80:80:2:8 parts by volume, spraying with 5 per cent sodium carbonate solution, drying, spraying with 0.02 per cent bromophenol blue solution, washing with water to remove excess indicator. Blue spots show where both quaternary ion and dye are present. Various examples of both dyes and quaternaries were used.

(336A) R. H. McDowell, "Detection of Small Quantities of Alginates," International Seaweed Symposium (Second) Trondheim 1955, pp. 131-137 (published 1956); *Chemical Abstracts*, Vol. 52, p. 985 (1958).

Alginates can be precipitated from very dilute solutions by calcium ions and the precipitate made visible by Night Blue. Details of test given. Can be made on one drop of solution under magnification. Application of test to soap and other products, and details of preparation for test given.

(338A) S. Musha, M. Ito, and T. Yanagi, "Electrometric Titration Methods for Determining Surface-Active Agent. I.—Comparison of the Methods," *Bunseki Kagaku*, Vol. 5, pp. 476-480 (1956); *Chemical Abstracts*, Vol. 51, p. 13655 (1957).

Titration of sodium lauryl sulfate with hexadecylpyridinium chloride and *vice versa* were investigated by electrometric methods such as potentiometric, high frequency, turbidimetric, photometric and fluorometric. Turbidimetric titration required a considerable time with reproducibility of 0.2 per cent. Fluorometric titration could be done in 10 min with 0.4 per cent reproducibility. The high-frequency method gave fairly good results if beat frequency type apparatus was used. A potentiometric method was not suitable.

(338B) T. Nakagawa and I. Nakata, "Partition Chromatography of Polyethylene Glycols," *Kôgyô Kagaku Zasshi*, Vol. 59, pp. 710-712 (1956); *Chemical Abstracts*, Vol. 52, p. 4418 (1958).

Polyethylene glycol with different molecular weight was subjected to paper chromatography by use of butanol saturated with water as the developing agent and Dragendorff solution as coloring agent. The R_f values are given for each fraction of the polyethylene glycol. The mechanism was discussed and based on the theory advanced a counter current extraction was tried using water and butanol.

(338C) T. Nakagawa and I. Nakata, "Paper Chromatography of Nonionic Surfactants. I. — R_f and HLB values of Nonionic Surfactants of the Polyoxyethylene Series," *Journal of the Chemical Society of Japan* (Industrial Chemistry Section), Vol. 59, pp. 1154-1156 (1956); *J. Am. Oil Chem. Soc.*, Vol. 34, p. 64 (1958).

Most suitable solvent system was normal butanol-pyridine-water in 5:2:5 parts by volume. Development made at 0°C. Modified Dragendorff reagent used consisting of one volume of a mixture of (0.85 g $\text{Bi}(\text{OH})_3 \cdot \text{NO}_3$, 100 cc glacial acetic acid, and 40 cc water), one volume

of the mixture of 8 g KI and 20 cc water, four volumes of acetic acid, and ten volumes of water. Commercial nonionics examined. Inverse correlation of R_f and HLB values with one exception

(338D) H. Negoro and S. Seno, "Analysis of Surface-Active Agents. III.—Paper-Electrophoretic Separation," *Annual Report of Takamine Laboratory*, Vol. 8, pp. 119-122 (1956); *Analytical Abstracts*, Vol. 5, No. 4219 (1958).

Cationic material behavior studied at 325 to 700 v and 1mA/cm. Studied in various buffers. 0.1 per cent methyl orange used as color reagent. At pH 4 and 7, separated benzalkonium and benzethonium in one hour. Nonionic of ethylene oxide alkylaryl ether type migrates toward cathode at pH 10, rate increasing with increase in degree of polymerization.

(339A) Y. Nemoto and K. Sone, "Simplified Colorimetric Analysis of Anionic Detergents," *Nagoya-shi Kôgyô Kenkyûjo Kenkyû Hôkoku*, No. 13, pp. 81-82 (1956); *Chemical Abstracts*, Vol. 51, p. 15154 (1957).

Edward's method (reference (317)), was modified so as to omit the procedure of filtering the methylene blue-anionic detergent complex. This is an improvement for analyses of the detergent at 10 to 40 mg per liter concentrations.

(341A) J. M. Preston and S. R. Epton, "Volumetric Determination of the Concentration of Solutions of Ionic Detergents and Dyes by the Bubble Pressure Method," First World Congress on Detergents and Surface-Active Products, Vol. I, pp. 310-315 (1956).

Pure anionic and cationic surfactants titrated against one another volumetrically using a bubble-pressure end point. The titration was also done on ionic dyes. All details of the procedure are described.

(344A) A. Sabatini, "Colorimetric Method of Determining EDTA," *Farmaco (Paria)* (Edizione pratica), Vol. 11, pp. 504-511 (1956); *Chemical Abstracts*, Vol. 53, p. 8948 (1959).

To 4 cc of the sample solution properly diluted and adjusted to pH 3.5 to 8 is added 1 cc of 0.1N chromic trichloride

solution and the mixture heated 5 min to 100 C. After quick cooling the extinction is measured spectrophotometrically at 545 m μ against a blank of water treated in the same way. As standard can be used the bisodium salt or the 2-sodium salt of the calcium chelate of EDTA.

(348A) R. L. Shriner, R. C. Fuson, and D. Y. Curtin, "The Systematic Identification of Organic Compounds," pp. 268-270, John Wiley & Sons, Inc., New York, N. Y. (Fourth Edition) (1956.).

Sulfonates give characteristic S-benzylthiuronium derivatives, conversion to sulfonyl chlorides and treatment to give amines, or treatment with *p*-toluidine hydrochloride.

(348B) I. Smith, "Paper Chromatography," *Soap, Perfumery & Cosmetics*, Vol. 29, pp. 421-426 (1956); *Chemical Abstracts*, Vol. 50, p. 9929 (1956).

General review considering R_f values, ascending and descending chromatography, location of substances on chromatograms, multiple dipping, desalting technique and reverse phase methods.

(350A) A. A. Spryskov and S. P. Starikov, "Sulfonation Reaction. XL.—Determination of Benzene - Disulfonic Acids," *Journal of General Chemistry of the U.S.S.R.*, Vol. 26, pp. 2909-2913 (1956); *Chemical Abstracts*, Vol. 52, p. 6067 (1958).

Details of separation by formation of amine salts as 2-naphthylamine or benzi-dine and titrated with sodium hydroxide. Said to be useful in the presence of mono- and trisulfonic acids.

(358A) K. Wilson, "Determination of Active Ingredient in Carboxymethyl Cellulose Preparations," *Svensk Papperstidning*, Vol. 59, pp. 218-222 (1956).

Method described for low viscosity technical grade of sodium carboxymethyl cellulose. Sample is acidified in 1N hydrochloric acid solution then neutralized to phenolphthalein endpoint with slight excess. 80 per cent ethanol is added and stirred until the supernatant liquid becomes clear. Filtered on sintered glass crucible, washed with 95 per cent ethanol and the alcohol displaced with ether. The

crucible is dried to constant weight. Degree of substitution was determined by ashing the residue, dissolution in water and titration to methyl red endpoint and degree of substitution calculated. Any chloride must be corrected for by determination of content in residue. Good reproductibility obtained

1957

(360A) ABCM — SAC Committee, "Recommended Methods for the Analysis of Trade Effluents. Determination of Synthetic Detergents," *Analyst*, Vol. 82, pp. 826-834 (1957).

The extraction of a methylene blue-anionic detergent complex into chloroform is recommended for low concentrations of anionic detergents. Following tentative methods were recommended: High concentrations of anionic detergent by titration with cetylpyridinium bromide and methylene blue indicator or with cetyltrimethylammonium bromide and bromophenol blue indicator; high concentrations of cationic detergent by the reverse of the anionic titration; low concentrations of anionic detergent by reduction of anionic activity; high concentrations of nonionic polyoxyethylene-type detergent by precipitation of a detergent-barium molybdophosphate complex and either spectrophotometric measurement or determination of phosphorus; and separation of anionic and cationic detergents by ion exchange.

(360B) T. Akiyama and M. Harada, "Direct Titration of Sulfate," *Kyoto Yakka Daigaku-Gakuhō*, Vol. 5, pp. 48-50 (1957); *Chemical Abstracts*, Vol. 52, p. 1859 (1958).

Add 45 cc ethanol adjusted to pH 2.8-3.5 by adding 0.25M perchloric acid, 5 drops of 0.2 per cent Alizarin Red S solution to 50 cc test solution containing approximately 39 mg of sulfate ion as sulfuric acid and titrate with 0.1M barium perchlorate solution. The presence of hydrochloric or nitric acids in the solution gives a slightly larger value; the existence of perchloric or hydrobromic acids does not affect the determination.

(360C) T. Akiyama, H. Ichida, and T. Yabuuchi, "Determination of Polyphosphate by Tris (ethylenediamine) cobalt Bromide," *Kyoto Yakka Daigaku Gakuhô*, Vol. 5, pp. 36-38 (1957); *Chemical Abstracts*, Vol. 52, p. 1859 (1958).

Tripolyphosphate is treated with tris-(ethylenediamine) cobalt solution. Compositions of precipitates given, and these filtered, washed with diluted reagent solution, dried at 110 C to constant weight and weighed as their salt of tris(ethylenediamine) cobalt.

(360D) T. Akiyama, M. Fujiwara, and H. Ichida, "Chromatography of Polyphosphates," *Kyoto Yakka Daigaku Gakuhô*, Vol. 5, pp. 39-41 (1957); *Chemical Abstracts*, Vol. 52, p. 1859 (1958).

Ortho-, pyro-, trimeta- and hexameta-phosphate solutions spotted on filter paper and chromatographed with various developing solvents and the phosphate band identified on the chromatogram by spraying with ammonium molybdate solution, heated, acidified benzidine solution added, and heated to develop blue color. Other developing solutions and R_f values given.

(360E) "Copper in Soaps and Soap Products," ASTM Methods D 460 - 58. Compilation of ASTM Standards on Soaps and Other Detergents (1957).

Copper as colored compound with di-ethylthiocarbamate determined photometrically.

(360F) J. Asselineau, "Applications of Chromatography to Fatty Acids," *Chimie analytique*, Vol. 39, pp. 375-383 (1957); *Chemical Abstracts*, Vol. 52, p. 4415 (1958).

Review of the applications of partition and gas-phase chromatography to the isolation of acid fractions from mixtures to the separation of fatty acid mixtures into groups and single acids, and to the identification of fatty acids. 115 references.

(360G) A. Badinaud and A. Boucherle, "Applications of Polarography to the Analysis of Several Polyethylene Glycols

and their Derivatives," *Congrès de la société pharmaceutique de France* (neuvième) Clermont-Ferrand, pp. 147-155 (1957); *Chemical Abstracts*, Vol. 53, p. 17747 (1959).

For each concentration of polyethylene glycol (PEG) tested the relative inhibitory power (P), calculated as $1(b_i/b_o)$, where b_i/b_o is the trace height in the absence of inhibitor and is a function of the log of the PEG concentration. The molecular weight of an unknown is determined by comparison with a known PEG. The PEG esters of fatty acids show an activity much the same as the glycol portion of the PEG. Since these polarographic methods are nonspecific, the effects may be obtained with many surfactants.

(360H) F. Baykut and S. Baykut, "Paper Chromatography of Saturated Aliphatic Monocarboxylic Acids," *Revue de la faculté des sciences de l'université d'Istanbul* (série C22), pp. 267-270 (1957); *Chemical Abstracts*, Vol. 52, p. 4415 (1958).

A simple mathematical relationship was worked out between the R_f value and the number of carbon atoms in the homologous series of the higher aliphatic saturated monocarboxylic acids from C_{10} to C_{22} . The R_f value was the quotient of the distances from the starting point of the substance in question and from the starting point of the solution medium, and depended on the nature of the solution medium and on the temperature. Other details of the method given.

(361A) E. W. Blank, "The Analysis of Alkybenzene Sulfonates," *Proceedings, Chemical Specialties Manufacturers Assn.*, 44th Annual Meeting, pp. 113-117 (1957) (see reference (388)).

(361B) R. A. G. Carrington and H. C. Evans, "Alkyl Sulfates, Part II.—Spectra of the Cyclohexylammonium Salts in the 800 cm^{-1} Region," *Journal of the Chemical Society* (London), pp. 1701-1709 (1957).

Absorption band of 790 cm^{-1} for all compounds having substituent group attached at even-numbered position on hydrocarbon chain, while odd-numbered substitution frequency varied between 837 and 810 cm^{-1} , position not dependent on number of carbon atoms in the chain.

(361C) R. Charonnat, M. Miocque, and J. Yonger, "Detection of Quaternary Ammonium Compounds in Milk," *Annales pharmaceutiques françaises*, Vol. 15, pp. 673-677 (1957); *Chemical Abstracts*, Vol. 52, p. 15770 (1958).

Mix 50 ml milk with 0.5 ml 0.15 per cent helianthin solution, 1 ml sodium hydroxide (density 1.33 diluted 1:1), and 20 ml chloroform, shake, mix 45 ml of the emulsion with 55 g anhydrous copper sulfate, allow part of the chloroform to separate and pour it off. Wash with 25 ml chloroform in portions, dry the combined chloroform solution with sodium sulfate, filter, mix with 0.5 ml 2N hydrochloric acid by shaking and allow to stand. A positive reaction is indicated when the aqueous solution is rose colored. Glassware should not be washed with detergents which could cause false positive result.

(361D) Committee on Analysis of Trade Effluents, "Recommended Methods for the Analysis of Trade Effluents," *Analyst*, Vol. 82, pp. 826-834 (1957).

Methods are given for effluent examination for anionic detergents at low and high concentration; cationic detergents at low or high concentrations; nonionic detergents; and anionic and cationic surfactants in admixture. Methods used: Anionics, low concentrations—Longwell and Maniece, (reference (290)), complex of surfactant and methylene blue in alkaline solution extracted with chloroform and compared colorimetrically with a standard. Anionics, high concentration: Two methods given—A. Epton (reference (84)), for opalescent samples in which cationic materials are used to decompose the chloroform complex of anionic and methylene blue to give water soluble methylene blue, B.—of Barr, Oli-

ver and Stubbings (reference (97)), for clear effluents where anionic is titrated with cetyltrimethylammonium bromide and bromophenol blue soluble complex in chloroform. Cationic surfactants: High concentration; reversal of the anionic method A. For low concentration cationic, reduction in anionic activity determined by adding known quantity of anionic to sample and determining anionic available by Degen's method, reference (219)). Nonionic detergents determined by precipitation of a detergent-barium phosphomolybdate complex from ethanol solution. Precipitate either dissolved in ethylene glycolmonomethyl ether and hydrochloric acid and optical density determined under ultraviolet at 3100 Å or digested with mixture of sulfuric acid and perchloric acids and the phosphorous determined by the molybdenum blue method.

Anionic and cationic detergents in admixture determined (a) by passing solution through cation-exchange Zeo-Karb 225 and cation active material adsorbed, or (b) cation active in excess of anionic by determining anionic directly by one of the methods given or by using Zeo-Karb exchange resin and adding cationic to eluate to give results equal to (a). Estimation of cationic in presence of slight excess or less of anionic by passing solution through the Zeo-Karb and determining anionic alone. Latter three methods developed by H. J. Finch.

(362A) D. C. Cullum, "Separation of Sarcosine from Methylaminodiacetic Acid," *Analyst*, Vol. 82, pp. 589-591 (1957).

Separation given, which includes adsorption on column of De-Acidite E. Eluant contains sample.

(362B) V. Diaconescu, "Rapid Method for Determination of Lignosulfonic Acids," *Buletinul institutului politehnic din Iasi*, Vol. 3, (1-2), pp. 101-106 (1957); *Analytical Abstracts*, Vol. 6, 1404 (1959).

Dimethyl-1-naphthylamine gives quantitative precipitate with lignosulfonic

acids in an aqueous medium containing not less than 0.057 g hydrochloric acid per ml. External indicator paper treated with sulphanilamide reagent (1.5 g in 50 ml water mixed with 1 per cent sodium nitrate solution). Maximum error about 4 per cent. Determination takes about 3 to 5 min.

(363A) M. Doležil, "Use of Fluorescent Indicators for the Determination of Small Amounts of Surface-Active Compounds. I.—Determination of Organic Sulfates and Sulfonates," *Collection of Czechoslovak Chemical Communications*, Vol. 22, pp. 396-400 (1957); *Chemical Abstracts*, Vol. 51, p. 17604 (1957). (See reference (315)).

(363B) R. Dubrisay, "Application to Chemical Analysis of Some Capillary Phenomena," *Chimie analytique*, Vol. 39, pp. 226-229 (1957); *Chemical Abstracts*, Vol. 51, p. 15336 (1957).

Fatty acids can be concentrated from aqueous suspensions by foams. This method used to study the formation of acids during oxidation of oils and tars.

(363C) H. Étienne, "Determination of Ethylene Oxide Condensates (Polyglycols) by Phosphotungstic Acid Precipitation," *Parfums, cosmétiques, savons*, No. 137, pp. 33-37 (1957); *Chemical Abstracts*, Vol. 51, p. 17615 (1957).

Ethylene oxide condensate determined in the presence of fatty alcohols in shampoos and the like. The product is determined by precipitating in the presence of barium chloride and phosphotungstic acid solutions, separating, weighing, then igniting, and reweighing.

(363D) "Cleaning Compound, Synthetic Detergent (Non-Abrasive)," Federal Specification, Interim, P-C-00431b (GSA-FSS), Revision 1 (Feb. 6, 1957).

For alkylaryl sulfonate type surfactant the active ingredient content is determined by an extraction with carbon tetrachloride of the complex of surfactant with *p*-toluidine hydrochloride and this titrated potentiometrically with sodium hydroxide solution to a pH 10 to 10.2 endpoint. For nonionics, alkanolamides and

others an acetone extract of the dry product is made and this extract calculated as per cent activity.

(363F) "Scouring Compound for Floors and Porcelain Surfaces," Federal Specification, P-S-00311 (VA), Feb. 15, 1957.

Prepare saturated solution of reagent grade benzidine dihydrochloride in distilled water. Place one ml of reagent on a watch glass. Sprinkle in powder. Blue coloration of particles are positive for the presence of bentonite.

(363G) J. Gánczarzyk, "Determination of Lignosulfonic Acids with the Aid of Chlorine Water," *Chemia Analityczna* (Warsaw), Vol. 2, pp. 441-451 (1957); *Chemical Abstracts*, Vol. 52, p. 13252 (1958).

A new colorimetric method for determining lignosulfonic acids in water is based on the color reaction of the sulfonic acid with chlorine water. Details of test given.

(363H) K. Gassner, "Analysis of Condensed Phosphates," *Mikrochimica Acta*, pp. 594-606 (1957); *Chemical Abstracts*, Vol. 51, p. 17598 (1957).

Hydrolysis that occurs during paper chromatographic determination of condensed phosphates avoided by shortening the time of transit and/or working at lower temperatures. Traces of orthophosphate in the presence of polyphosphates determined by reducing the former to molybdenum blue with ascorbic acid at pH4. Many interfering ions can be removed with disodium ethylenediaminetetraacetate. Other details of determination given.

(363J) J. A. Gautier and J. Renaut, "The Analysis of Detergents; New Method Applicable to the Alkali-Metal Salts of Sulfated Long-Chain Primary Aliphatic Alcohols," *Chimie Analytique*, Vol. 39, pp. 189-192 (1957); *Analytical Abstracts*, Vol. 4, No. 4049 (1957).

Proposed volumetric and gravimetric methods based on precipitation of $(\text{ROSO}_3)_2\text{Ni}(\text{NH}_3)_4$ with excess Ni^{2+} in aqueous ammonia solution. Precision ± 1 per cent. Short chain compounds up

to ten carbon atoms not precipitated. Sodium and ammonium salts do not interfere. Dissolve sample (about one millimole) in 25 ml of water by heating. Add 0.2M aqueous NiCl_2 , 5 ml and 10 ml of 10 N ammonia, cool well, dilute to 50 ml with 10N ammonia and mix. After 10 min either filter and titrate an aliquot of filtrate with 0.1 M disodium ethylenediaminetetraacetic acid, or collect precipitate on No. 4 sintered glass crucible, wash free of chloride ion with saturated ammonia solution and dry to constant weight at 35 C.

(363K) H. Goff and F. E. Blachly, "A Conductivity Method for the Determination of Soap in Refined Vegetable Oil," *Journal American Oil Chemists' Soc.*, Vol. 34, pp. 320-323 (1957).

The determination of soap in refined fatty oils by water extraction and analysis for sodium by measurement of the conductivity of the water extract studied.

(364A) J. C. Griffith, "The Quantitative Estimation of a Nonionic Detergent," *Chemistry & Industry* (London), pp. 1041-1042 (1957).

Titration with potassium permanganate to oxidize the hydroxyl end group to the corresponding aldehyde or acid, and potentiometric titration with potassium triiodide were unsuccessful.

The ultraviolet spectrum of Triton X-100 shows peak at 278 $m\mu$ and small inflection at 283 $m\mu$. Peak was reproducible for solutions of the same concentration. Concentrations as low as 0.005 per cent weight to volume determined. Peak attributed to aromatic ring and Lissapol NX could presumably be analyzed similarly.

(366A) W. H. Houff, D. R. Christie, and R. H. Beaumont, "Analysis of Aromatic Sulfonation Reaction Mixtures," *Analytical Chemistry*, Vol. 29, pp. 1866-1868 (1957).

Aromatic sulfonation mixtures containing sulfuric acid and sodium sulfate analyzed by three-step procedure. The sulfonic acid is determined by titration with base of the regenerated sulfonic acid after

precipitation of the sulfate with barium hydroxide, and passage of the filtrate through cation exchange resin. Separate determination of the total acidity and total sulfate permits calculation of both sulfuric acid and sodium sulfate. In alternative method sulfonic acid, total acid and total anion determined. Neither procedure suitable for aryl sulfonates having insoluble barium salt nor where anions other than sulfonate, sulfate or hydroxide are present.

(366B) R. H. Hughes and R. J. Martin, "Determination of Methyl, Methylene, and Alkylbenzene Group Types by Infrared Absorption," *Am. Soc. Testing Mats.*, pp. 127-149, Disc., pp. 149-150 (1957). Issued as separate publication *ASTM STP* No. 224.

Discussion of the findings concerning technique, and a second part concerned with a group-type analysis for the determination of the different types of alkyl-substituted benzenes by using the 10.0 to 15.0 μ portion of the spectrum. A method was developed whereby mono-substituted and *o*-, *m*-, and *p*-disubstituted benzenes can be determined but only with severe restrictions as to over-all sample composition. Tri- and higher substituted benzenes as well as polynuclear aromatic hydrocarbons interfere in this scheme of analysis.

(366C) A. Ito, "Identification of Naphthalenesulfonic Acids by Paper Chromatography," *Kôgô Kagaku Zasshi*, Vol. 60, pp. 1006-1008 (1957); *Chemical Abstracts*, Vol. 53, p. 11110 (1959).

Sodium salts of 1- and 2-naphthalenesulfonic acids and 1,5-, 1,6- 2,6- and 2,7-naphthalenedisulfonic acids fused with potassium hydroxide at 245-250 C, dissolved in water and subjected to paper chromatography by use of 2 per cent ferric chloride solution and diazotized *p*-chloraniline as coloring agents. The R_f values for each compound are given.

(367A) M. Kehren and M. Rösch, "X-Ray Investigations on Water-Free and Water-Containing Alkyl-Polyethylene Oxides. I.—Literature Search. II.—Spe-

cific Investigations," *Fette, Seifen, Anstrichmittel*, Vol. 59, No. 1, pp. 1-8, 80-86 (1957).

Literature on general X-ray investigations with various detergents and their solutions with particular emphasis on nonionic substances, particularly myristyl and stearyl alcohols and their ethylene oxide adducts reviewed. The X-ray spacings for various lengths of the ethylene oxide chains in polyethylene oxide derivatives of the above alcohols discussed for both the solutions and the free materials.

(367B) G. M. Kosolopoff and C. H. Roy, "Behavior of Some Alkylphosphonic Acids in Paper Chromatography and Paper Electrophoresis," *Journal Chemical Society* (London), pp. 3428-3430 (1957).

The R_f values for paper chromatography of RPO_3H_2 with the alkyl chain ranging from methyl to hexyl given for saturated aqueous butanol solvent pair. Migration values given for electrophoretic transfer on paper. Both techniques give locatable spots but no direct correlation of migration rates with structure can be made. Location of the substances made either by alkali-treated bromothymol blue or in the form of the lead salts. Location of 0.01 mg quantities of the acids possible.

(367C) M. Krajčinović and J. Jovanović, "Quantitative Determination of Sodium Carbonate and Sodium Bicarbonate in a Mixture Containing Soap Powders," *Fette, Seifen, Anstrichmittel*, Vol. 59, pp. 753-755 (1957); *Chemical Abstracts*, Vol. 53, p. 18515 (1959).

Method given for the determination of sodium carbonate and sodium bicarbonate in mixtures containing soap powders, water glass, sodium sulfate and sodium borate. The carbonate concentrations are determined by measuring indirectly the loss of carbon dioxide. Water of hydration is determined by the xylene method.

(367D) M. Kurata and S. Inoue, "Separate Determination of Nonionic Surfactant in the Presence of Ionic Agent,"

Yukagaku (J. Japan Oil Chem. Soc.), Vol. 6, pp. 92-96 (1957); *Journal, Am. Oil Chemists' Soc.*, Vol. 36, p. 192 (1959).

Nonionic surfactants such as polyoxyethylene ethers of higher alcohols of HLB 16 to 12. Sodium sperm alcohol sulfates (or sodium alkylbenzene sulfonate) or a cationic oleyl picolinium chloride were added to the 0.07 to 0.3 per cent solutions of nonionic surfactants. To three meqs. of such mixture 8 meq. of ion-exchange resin added at flow rate of 0.14 to 0.22 ml per ml resin per min. Recovery of nonionic in the effluent was 96.8 ± 2.5 per cent.

(367E) M. Kurata and S. Inoue, "Assay of Both Nonionic Surfactants and Quaternary Ammonium Salt in the Mixture. In Combination of Ferricyanide and Epton Methods or Ferricyanide and Bromophenol Blue methods," *Abura Kagaku* (J. Japan Oil Chemists' Soc.), Vol. 6, pp. 96-100 (1957); *Journal, Am. Oil Chemists' Soc.*, Vol. 36, p. 192 (1959).

Mixture of higher alcohol ether of polyoxyethylene glycol (HLB 18) and oleyl picolinium chloride or lauryltrimethyl ammonium chloride could be separately determined by combining known methods.

(367F) G. Lang and K. Pilz, "Volumetric Determination of Sulfate in Solutions of High Chloride Content," *Kali und Steinsalz*, Vol. 2, p. 100 (1957); *Chemical Abstracts*, Vol. 52, p. 170 (1958).

Titration of sulfate ion with barium chloride modified in the presence of large amounts of chloride by the addition of mercuric acetate to remove the chloride ion. The recommended ratio of mercury ion to chloride was 3.5:10. The aliquot to be titrated should contain at least 0.1 millimole of sulfate ion. Alizarin Red S was used as indicator.

(367G) K. D. Ledwoch, "Infrared Analysis of Alkylbenzenes," *Erdöl und Kohle*, Vol. 10, pp. 595-596 (1957); *Chemical Abstracts*, Vol. 52, p. 979 (1958).

Values given for the Bunsen extinction coefficient and the molar extinction coefficient for toluene and for ethyl, butyl, hexyl, octyl, decyl and dodecylbenzene. From measurements of the intensity of the infrared K absorption band can be derived a simple relation between a' and the molar composition of mono- n -alkylbenzenes. The values of epsilon of homologous series are constant except for the first two members. Thus it is possible by measuring the a' and the epsilon, the density and the molecular weight completely to analyze mixtures of alkylbenzenes and paraffin hydrocarbons.

(367H) U. Lichthardt, "Chromatographic Identification of Fatty Acids Produced in the Analysis of Lacquers," *Farbe und Lack*, Vol. 63, pp. 387-393 (1957); *Chemical Abstracts*, Vol. 52, p. 748 (1958).

Test used extensively for identification of the fatty acids obtained in lacquer analysis. A reversed phase technique was used. The paper was treated with undecane saturated with glacial acetic acid and developed with glacial acetic acid saturated with undecane for 14 to 16 hr, the dried slips then immersed in an aqueous cupric acetate solution. The copper salts formed were made visible with $K_4Fe(CN)_6$.

(367J) S. M. Livengood and C. A. Johnson, "Analysis of High Amide Fatty Alkanolamides," *Proceedings, Chemical Specialties Manufacturers' Assn.*, 44th Annual Meeting, pp. 123-125 (1957). Methods described are for unreacted fatty acidity titration; total free amine by titration with perchloric acid in acetic acid; amine ester separated and determined by technique of Kroll and Lennon (reference (335)); Kjeldahl nitrogen; saponification for total ester content.

(371A) A. J. Milun, "Colorimetric Determination of Primary Fatty Amine Acetates and Fatty Amines," *Analytical Chemistry*, Vol. 29, pp. 1502-1504 (1957).

Method based on reaction of primary amine with salicyl aldehyde. A bright

yellow Schiff base is formed and measured at 410 m μ . No interference from unsubstituted amide, N -alkylacetamide or nitrile. Used successfully on technical grade primary fatty amine acetates and primary fatty amines. Confidence limits at 99 per cent are 10.6 per cent.

(371B) L. C. Mitchell, "Separation and Identification of Acids by Paper Chromatography. III.—The Water-Soluble Inorganic Phosphates," *Journal of the Assn. of Official Agricultural Chemists*, Vol. 40, pp. 1038-1050 (1957).

Method described for separating and identifying *ortho*-, *pyro*-, *tripoly*-, *trimeta*-, and *tetrameta*-water-soluble inorganic phosphates, and for identifying components of them. A mobile solvent of butanol, pyridine, acetone, nitric acid and water is used. The chromatogram was developed by spraying with ammonium molybdate, air drying, and lightly overspraying the front and the back with a benzidine solution.

(371C) F. L. Moore, "Long-Chain Amines — Versatile Acid Extractants," *Analytical Chemistry*, Vol. 29, pp. 1160-1162 (1957).

Solutions of long-chain amines are excellent extractants for mineral acids, many organic acids and complex metal acids. Anionic liquid-liquid extraction analogous to anionic exchange resin technique, but liquid-liquid method is fast, inexpensive, and has higher capacities. Tri- (*iso*-octyl) amine also useful, low water solubility and high organic solvent solubility.

(371D) T. Nakagawa and I. Nakata, "Qualitative Analysis of Nonionic Surfactants on Filter Paper," *Journal of the Chemical Society of Japan (Industrial Chemistry Section)*, Vol. 60, pp. 554-556 (1957); *Analytical Abstracts*, Vol. 5, No. 3051 (1958).

Benzene solution of surfactant spotted on filter paper then developed with mixture of n -butanol, pyridine and water (5:2:5) and treated with various reagents: Dragendorff's reagent or cobaltothiocyanate complex (62 g NH_4SCN and

22.8 g $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ in 100 ml. water) for polyoxyethylene groups, iodine-amylose for surfactants with long-chain alkyl group; 0.4 per cent permanganate for those with reductive group; and concentrated sulfuric acid for resins. When combined with periodate reagent test or hydrolysis with methanolic potassium hydroxide followed by neutralization and extraction with benzene, spot test permits eight main nonionic surfactants to be readily differentiated and 31 others by careful measurement of R_f value.

(371E) T. Nakagawa and R. Mune-yuki, "A Convenient Method to Remove Polyethylene Glycol Included in Nonionic Surfactant," *Annual Report Shionogi Research Laboratory*, Vol. 7, pp. 209-210 (1957); *Chemical Abstracts*, Vol. 51, p. 15974 (1957).

The principle of this method is partition chromatography, in which the fixed phase is water saturated with butyl alcohol absorbed on silica gel, and the ascending phase is butyl alcohol saturated with water.

(371F) E. Nebe, "Electrometric Method for the Determination of Water in Soaps and Detergents," *Fette, Seifen, Anstrichmittel*, Vol. 59, pp. 755-757 (1957); *Chemical Abstracts*, Vol. 53, p. 18515 (1959).

An electrometric method is given for the determination of water in soaps and detergents and compared with the Karl Fischer method and several others.

(371G) R. Neu, "The Determination of Long-Chain, Quaternary Ammonium Compounds," *Fette, Seifen, Anstrichmittel*, Vol. 59, pp. 503-505 (1957); *Chemical Abstracts*, Vol. 52, p. 15929 (1958).

Cation-active halides can be determined quantitatively through the halide ion. A weighed portion of the cationic is dissolved in water in a volumetric flask, and a known excess of 15 per cent sodium perchlorate is added by a pipet with vigorous agitation, the flask is heated in boiling water for 15 min, cooled to 20 C,

made to volume and filtered. An aliquot of the filtrate is titrated with 0.1N silver nitrate, potassium chromate being used as the indicator, to detect the excess of ClO_4^- ion present.

(371H) V. P. Persiantseva and V. A. Shneider, "Colorimetric Determination of 2,6 and 2,7-Naphthalenedisulfonic Acid in Nickel Electrolyte," *Zavodskaya Laboratoriya*, Vol. 23, p. 1298 (1957); *Chemical Abstracts*, Vol. 53, p. 4016 (1959).

Nickel is separated from the electrolyte by a cation-exchange resin. The electrolyte is reduced to dryness, and the dry residue nitrated with a nitration agent. After reaction ceases, the solution is diluted to volume with distilled water, and the sulfonic acids determined colorimetrically in the yellow solution.

(371J) O. Pfrngle, "Paper Chromatography of Condensed Phosphates," *Zeitschrift für analytische Chemie*, Vol. 158, pp. 81-92 (1957); *Chemical Abstracts*, Vol. 52, p. 4411 (1958).

The paper chromatography methods of Grunze and Thilo (reference (225A)) are slightly modified so that as many as eight condensed phosphates plus phosphoric acid are separated, located by spraying with molybdate reagent, and colorimetrically determined in the paper portions.

(372A) R. Raemaekers, "Determination of Tripoly- and Pyrophosphate in Tripolyphosphates," *Chimie analytique*, Vol. 39, pp. 254-259 (1957); *Chemical Abstracts*, Vol. 51, p. 16203 (1957).

Details of change in procedure to reduce interferences (see reference (262A)).

(373A) B. Rocchi and F. Vittorelli, "Spectrophotometric Determination in Ultraviolet Light of Hexadecyltrimethylammonium *p*-Toluenesulfonate when Associated with Interfering Excipients," *Bollettino chimico farmaceutico*, Vol. 96, pp. 198-201 (1957); *Chemical Abstracts*, Vol. 51, p. 13315 (1957).

Determination of hexadecyltrimethylammonium *p*-toluene sulfonate in composition containing polyoxyalkylene stear-

ates or polyoxyalkylenesorbitan stearate, spectrophotometric measurements are made in alcohol solution at 217.5m μ , 221.5m μ , and 224.2 m μ , alcohol being used as the blank. From the corresponding absorption values, the calculation of quantities present is based on a geometric evaluation.

(373B) M. J. Rosen, "Separation of Nonionic Surface-Active Agents from Mixtures with Anionics by Batch Ion Exchange," *Analytical Chemistry*, Vol. 29, pp. 1675-1676 (1957).

Nonionics separated from anionic products in batch ion exchange method using relatively small amount of strong anion exchange resin. The anionic surface active agent-resin mixture is washed free from nonionic with methanol, and recovered in substantially quantitative yield from the filtrate and washings. Inorganic chloride interferes and nonionic removed by drying and washing with acetone, and gravimetrically determined on the residue. Results with fourteen binary mixtures discussed.

(373C) W. E. Rosen, V. P. Toohey, and A. C. Shabica, "Paper Chromatography of Some Quaternary Ammonium Salts," *Journal, Am. Pharmaceutical Assn.*, Vol. 46, pp. 625-626 (1957); *Chemical Abstracts*, Vol. 52, p. 833 (1958).

Generalizations of the paper chromatographic behavior are given for a series of quaternary salts of isoindolines, tetrachloroisoindolines and related systems.

(374A) K. Röth, "Paper Chromatography in the Soap Field," *Fette, Seifen, Anstrichmittel*, Vol. 59, p. 418 (1957); *Chemical Abstracts*, Vol. 52, p. 11447 (1958).

The paper chromatography of the fatty acids from soap by using a paper with cross-sectioned barrier permits a separation of the major components. The eluant mixture was 74 parts glacial acetic acid, 15 parts methanol, and 11 parts water, the sample was added in toluene

solution, and the development was with malachite green.

(374B) B. Sansoni and L. Baumgartner, "Separation of Phosphates by Paper Electrophoresis. V.—High-Potential Paper Electrophoresis of Condensed Phosphates and Metaphosphates," *Zeitschrift für analytische Chemie*, Vol. 158, pp. 241-251 (1957); *Chemical Abstracts*, Vol. 52, p. 3589 (1958).

Mixtures of condensed phosphates and metaphosphates can be separated by high-potential (80 v per cm) paper electrophoresis with a borate buffer (pH 10).

(374C) R. Sargent and W. Rieman, III, "Analysis of Mixtures of Glycols by Ion-Exchange Chromatography," *Analytica Chimica Acta*, Vol. 16, pp. 144-148 (1957); *Analytical Abstracts*, Vol. 4, No. 2232 (1957).

Mixture of diethylene glycol, ethane diol, propane-1:2 diol, isomers of butane-2:3-diol and glycerol separated quantitatively by anion exchange chromatography on two separate columns of Dowex 1-X8, hydrogen form.

(374D) H. Schwab, "Gradient-Elution Theory. Attempted Separation of Synthetic Anionic Detergents by Ion-Exchange Chromatography," *University Microfilms*, Rutgers University, Publication No. 22546, 97 pp. Dissertation Abstracts, Vol. 17, p. 2171 (1957); *Chemical Abstracts*, Vol. 52, p. 1654 (1958).

Initial work, without much in the way of a method developed.

(374E) S. Siggia, "Analysis of Nonionic Surface Active Agents," *Proceedings, Chemical Specialties Manufacturers' Assn.*, 44th Annual Meeting, pp. 126-129 (1957).

Charznsky identification of nonionic as a whole, of hydrophobic portion only, ratio hydrophobe to hydrophil. Discussed various techniques of estimation. Describes separation techniques by extraction, and ion exchange and final determination. Suggested is turbidity measurement for estimation of traces (0 to 1 per cent), of nonionic.

(374F) P. Spacu and Cl. Vasilescu, "A New Gravimetric Method for the Determination of Pyrophosphates," *Analele universității, "C. I. Parbon" Bucuresti* (Seria stiintelor naturii, No. 13, pp. 79-83 (1957); *Chemical Abstracts*, Vol. 52, p. 19723 (1958).

To cold 5 per cent ammoniacal solution add a 1 per cent solution of $\text{Co}(\text{NH}_3)_6(\text{NO}_3)_3$. The precipitate thus formed is allowed to stand $\frac{1}{4}$ hr, is filtered, washed with a 20 per cent ethanol solution containing 40 ml 25 per cent ammonium hydroxide and 40 ml 1 per cent of the cobalt salt given above, to the disappearance nitrate ions and afterwards with ethanol and ether. The precipitate is dried 15 min in vacuum and weighed as $\text{Co}(\text{NH}_3)_6\text{NaP}_2\text{O}_7$.

(374G) A. Spada, D. Coppini, and M. Montorsi, "Determination of Quaternary Ammonium Compounds of Antiseptic Action," *Farmaco* (Pavia) (Edizione scientifica), Vol. 12, pp. 582-585 (1957); *Chemical Abstracts*, Vol. 53, p. 17429 (1959).

Determined by postassium bichromate precipitation, centrifuging of the precipitate, dissolution in sulfuric acid and the extinction read spectrophotometrically against a blank of dilute sulfuric acid. The analytical factor $(\text{mg}/\text{E}^{1\text{cm}})_r$ is given for several quaternary compounds.

(375A) R. C. Stillman, "The Analysis of Alkyl Sulfates," *Proceedings, Chemical Specialties Manufacturers' Assn.*, 44th Annual Meeting, pp. 117-123 (1957).

Simplest analysis is for alcohol soluble (active ingredient), alcohol insoluble, moisture, unsulfated free alcohols. Specific methods detailed for alcohol soluble, alcohol insoluble, ester SO_3 , combined alcohols, free fatty alcohol, moisture, sodium sulfate, cationic titration of combined SO_3 or active ingredient, alkalinity and pH.

(375B) Swiss Society of Analytical and Applied Chemistry, "Savons et Detergents," Hans Huber, Bern (Switzerland),

(1957). French translation of Soaps and Detergents," (reference (299A)), 1957 (*Supplement*).

(375C) S. Takahama and T. Nishida, "Analysis of Surfactants by Ion-Exchange Method. A New Rapid Semi-micro Method for Determination of Anionics in the Presence of Nonionics," Second International Congress of Surface Activity, Vol. IV, pp. 141-147 (1957).

Weighed sample dried, active ingredient extracted with isopropanol and the insoluble filtered off. Filtrate passed through Amberlite IR-120 cation exchange resin and anionics titrated by alkali microvolumetrically. In the case of materials containing both weak and strong acidic groups such as turkey red oil, extraction made with 1:1 mixture 96 per cent ethanol and benzene and the ion exchanged material titrated potentiometrically. Only chlorides of the inorganic solids interfere and this corrected for by titration with silver nitrate. Nonionics, carboxymethyl cellulose, carboxy methyl sulfate and ethylene diamine tetraacetic acid do not interfere. Results compared with the Epton and *p*-toluidine methods, and good agreement found.

(375D) Y. Takashima, "Determination of Orthophosphate, Pyrophosphate, and Triphosphate with Hexamminecobalt (III) Chloride," *Memoirs of the Faculty of Science, Kyushu University* (Series C), Vol. 2, pp. 161-166 (1957); *Chemical Abstracts*, Vol. 52, p. 2660 (1958).

Weighing forms are $\text{Co}(\text{NH}_3)_6\text{PO}_4 \cdot 4\text{H}_2\text{O}$, $\text{Co}(\text{NH}_3)_6\text{HP}_2\text{O}_7$, and $\text{Na}(\text{CO}(\text{NH}_3)_6)_3(\text{P}_3\text{O}_{10})_2$ when 5 to 25 mg of the phosphates are precipitated with $\text{Co}(\text{NH}_3)_6\text{Cl}_3$ and dried at 110 C. Orthophosphates are precipitated at pH 12.3 the others at pH 7.9. Relative error is equal to or less than ± 2.5 per cent in all cases with single components, and not as good for mixtures. Smaller quantities (8 to 50 γ) can also be determined by precipitation with Co^{60} tagged chloride and radio-metric determination. The error is about ± 10 per cent.

(375E) Y. Takayama, "Determination of Anionic and Nonionic Surface-Active Agents in Aqueous Solution," *Journal of the Chemical Society of Japan*, (Industrial Chemistry Section), Vol. 60 pp. 872-874 (1957); *Analytical Abstracts*, Vol. 5, No. 1925 (1958).

Karush and Sonenberg's procedure, (reference (129)), was applied to a mixture of anionic and nonionic surfactant and found satisfactory if weight of nonionic was less than 30 times that of the anionic. Stevenson's method, (reference (266)), was satisfactory in the absence of anionic but subject to interference by substances other than those mentioned by Stevenson.

(375F) Y. Tanaka, "A New Qualitative Test Method for Nonionic Surface Active Agents: Cloud Number Test," Second International Congress of Surface Activity, Vol. IV, pp. 132-140 (1957).

Five to 10 per cent solution of nonionic in ethanol or propionic acid titrated with 2 per cent phenol solution in water at 25 C until clouding occurs. Cloud number is volume of phenol solution in cc's. This method is applicable to larger range of nonionics, and cloud number is characteristic of chemical constitution and has linear relationship to HLB. Much data given and Spans and Tweens used in showing relationship of cloud number to HLB.

(375G) N. Tanaka and R. Tamamushi, "Amperometric Titration of Dodecylpyridinium Bromide with Sodium Dodecyl Sulfate," *Nature*, Vol. 179, pp. 311-312 (1957).

Amperometric titration of dodecylpyridinium bromide can be made with a concentration of 0.1 to 1.0 millimoles of the compound to ± 0.005 millimoles. Solutions are titrated with 2 or 10 mM sodium dodecyl sulfate solution in 0.1 M caustic soda and 0.2 mM polyoxethylene dodecyl ether at a potential of -1.325 v versus a saturated calomel electrode, the dropping mercury electrode being the indicator electrode. Very sharp end points

result from the low solubility of the reaction product.

(375H) J. R. Tate, "The Quantitative Estimation of Nonionic Detergents," *Chemistry & Industry* (London), p. 1324 (1957).

Suggests Brown and Hayes method (reference (276)) involving ammonium cobalthiocyanate to give a blue precipitate which is soluble in chloroform and can be estimated colorimetrically at 620 m μ . Applicable to several types of polyethylene oxide adducts providing the ethylene oxide length is over three and capable of accuracies better than 1 per cent. Suggested for use in estimating amounts of nonionic taken up by wool.

(375J) L. E. Weeks, M. E. Ginn, and C. E. Baker, "Isolation and Determination by Ion-Exchange Technique of Nonionic Surfactants," *Soap*, Vol. 33, No. 8, pp. 47-50, 113, 115 (1957).

The surfactant anion is first removed by adsorption on a basic anion-exchange resin and the cation subsequently removed by passage of the solution through an acidic cation-exchange resin. The final effluent contains the nonionic agent and is evaporated for isolation and determination. A variety of surfactant mixtures was analyzed and the results shown.

(375K) L. E. Weeks, M. E. Ginn, and C. E. Baker, "Isolation and Determination of Nonionic Surfactants by Ion-Exchange Technique," *Proceedings, Chemical Specialties Manufacturers' Assn.*, (May, 1957), pp. 150-155.

Ionic surfactants adsorbed from ethanol solution on cation exchange resins and nonionic portion determined from the effluent, gravimetrically. Method accurate, and reproducible and useful for control purposes.

(376A) K. Winsauer, "Paper-Chromatographic Determination of Hydroxystearic Acids," *Mikrochimica Acta*, (3-4), pp. 480-484 (1957); *Analytical Abstracts*, Vol. 5, No. 1257 (1958).

Determination and separation of di, tetra, and hexahydroxy acids described,

based on reaction with KIO_4 . Chromatograph developed with KI and starch, hydroxy acids appear as white spots on blue background. Separation by mobile phase consisting of mixture of butanol, ethanol, aqueous ammonia and water.

(377) M. N. Zaliopo, "Method for Determination of Sodium and Potassium Soaps in Their Mixtures," *Masloboino-Zhirovaya Promyshlennost*, Vol. 23, No. 9, pp. 27-29 (1957); *Chemical Abstracts*, Vol. 52, p. 3370 (1958).

Method described with examples for determination of potassium and sodium soaps in a toilet-soap stick.

(378) G. Zubieta, "Determination of Active Detergents in Mixtures," *Industria y quimica* (Buenos Aires), Vol. 18, p. 299 (1957); *Chemical Abstracts*, Vol. 52, p. 6818 (1958).

Two methods were developed for quaternary compounds. A sample is acidified with sulfuric acid to a 5 to 15 per cent concentration of the latter, and bromocresol green was added and the mixture extracted with chloroform, the colored solution being read on a colorimeter. The second method comprises colorimetric measurement of the blue solution formed when the detergent is acidified with sulfuric acid and treated with iodine in the presence of starch to oxidize all reducing substances. Zinc, lead, sulfate, hydrogen sulfide, carbon bisulfide and carbohydrates do not interfere.

1958

(379) S. T. Abrams and F. H. Stross, "Thermal Analysis of Some 12-Hydroxystearates," *The Journal of Physical Chemistry*, Vol. 62, pp. 879-880 (1958).

Change of the thermogram of sodium 12-hydroxystearate on repeated analysis indicated that dehydration of the compound occurred to form sodium oleate. Other thermograms with other soaps given.

(380) A. Ady and L. Hunyady, "Determination of Essential Oils in Soaps and Cosmetics," *Fette, Seifen, Anstrich-*

mittel, Vol. 60, pp. 378-379 (1958); *Chemical Abstracts*, Vol. 52, p. 15839 (1958).

Ethanol-water solution of soap containing sodium carbonate extracted with three separate portions of petroleum ether. The extracts were combined, dried and concentrated to given volume, transferred to a wide-mouth bottle precharged with dibutyl phthalate, and dried at $35 \pm 1^\circ\text{C}$. The residue represents neutral fat, unsaponifiable, and essential oils. Repetition of this determination and drying at $105 \pm 2^\circ\text{C}$ removes the essential oils in the residue. The first determination less the second divided by the weight of sample times 100 gives essential oils with an accuracy of ± 7 per cent.

(381) E. M. Aleksandrova and A. S. Kryukova, "Quantitative Method of Determination of the Quaternary Salts of Aminomethylated Polyglycol Esters of Alkylphenols," *Nauchnye Doklady Vyssheĭ Shkoly* (Khimiya i Khimicheskaya Tekhnologiya), No. 3, pp. 510-512 (1958); *Chemical Abstracts*, Vol. 53, p. 984 (1959).

Two new optical methods for the detection of the concentrations of cation-active materials in aqueous solution. In one instance acid blue K is used as the indicator, dilutions of both dye and surfactant treated in standard manner and the optical density being measured at $610 \text{ m}\mu$. This method not sensitive at concentrations of less than 0.03 g per liter. Small concentrations of the surfactant can be measured directly at $276 \text{ m}\mu$ where Beer's law applies.

(382) R. Alquier, "Colorimetric Determination of Monoethanolamine," *France et ses parfums*, Vol. 1, No. 5, pp. 30-32 (1958); *Chemical Abstracts*, Vol. 53, p. 6540 (1959).

To determine monoethanolamine in triethanolamine, weigh 0.20 to 0.25 g triethanolamine into a test tube, add 1.8 ml distilled water, 1 ml 5 per cent sodium bicarbonate, 1 ml 2 per cent sodium nitroprusside, and 1 ml acetone, and mix.

After 15 min compare color produced with that of a series of tubes containing known amounts of standard monoethanolamine in the reaction mixture.

(383) "Sampling and Chemical Analysis of Alkylbenzene Sulfonates," ASTM Tentative Methods D 1568-58T, Compilation of ASTM Standards on Soaps and Other Detergents (1958).

Procedures for sampling, moisture by distillation or Karl Fischer, neutral oil, pH.

(384) "Testing Detergent Alkylate," ASTM Tentative Methods D 1569-58T, Compilation of ASTM Standards on Soaps and Other Detergents (1958).

Listing of ASTM methods for flash point, water or moisture, specific gravity, color, doctor test, kinematic or Saybolt viscosity, aniline point, Kauri-butanol, refractive index, bromine number, sediment.

(385) "Sampling and Chemical Analysis of Fatty Alkyl Sulfates," ASTM Tentative Methods D 1570-58T, Compilation of ASTM Standards on Soaps and Other Detergents (1958).

Procedures include sample preparation, moisture by distillation, pH, alkalinity, alcohol-soluble matter, alcohol-insoluble matter, unsulfated, combined alcohols, ester sulfur trioxide, sodium sulfate.

(386) E. Angelescu and Y. Davidescu, "Analytical Method for the Determination of Cresols based on Active Capillary Substances," *Academia republicii populare Române* (Studii si cercetări chimice), Vol. 6, pp. 213-231 (1958); *Chemical Abstracts*, Vol. 53, p. 8945 (1959).

A rapid analytical method for the determination of the three isomers in cresol in aqueous solution, by titration with sodium hydroxide, using surface tension as indicator. Near a concentration of 1.2 per cent the slope of the molar axis reaches a constant value which is different for the three isomers. Other details given.

(387) G. G. Berg, "Ascending Chromatography of Polyphosphates," *Analyti-*

cal Chemistry, Vol. 30, pp. 213-216 (1958).

Variables involved in chromatographic analysis of polyphosphates surveyed in terms of their effect on R_f values. Variables including selection and concentration of components of solvent composition, of atmosphere and condition of the paper. Isopropyl alcohol suitable only for lower polymers. Data on chromatographic identification of higher polymers by other solvents obtained.

(388) E. W. Blank, "Analysis of Alkylbenzene Sulfonates," *Soap*, Vol. 34, No. 1, pp. 41-44, 107 (1958).

Describes methods of sampling. Determination of alcohol soluble; determination of molecular weight by benzidine hydrochloride precipitation, the alkylbenzene sulfonate by cetyltrimethyl ammonium bromide titration of the active ingredient; determination of neutral oil by *n*-pentane extraction; water by Karl Fischer method.

(389) W. B. Chess and D. N. Bernhart, "Determination of Small Amounts of Pyrophosphate in Soluble Orthophosphates," *Analytical Chemistry*, Vol. 30, pp. 111-112 (1958).

Pyrophosphate present in less than 1 per cent amounts in orthophosphate determined colorimetrically. The complexing action of pyrophosphate on iron is measured by the 1,10-phenanthroline colorimetric method for iron. Results within 0.04 per cent of actual content below 1 per cent in the mixture. Interference by higher condensed phosphates because of their complexing action. Ca and Mg ions render method useless as their complexes with pyrophosphate are more stable than the iron-pyrophosphate complex.

(390) M. Chiba, "Separation of Non-ionic Surface-Active Agents from Organic Insecticide (Dieldrin) Emulsion and Identification," *Kagaku to Sōsa*, Vol. 11, No. 4, pp. 7-15 (1958); *Chemical Abstracts*, Vol. 53, p. 7498 (1959).

Several kinds of mixed nonionic surfactants in insecticide emulsions were sep-

arated in their pure state by use of silica or alumina column chromatography by using elution liquids such as *n*-hexane, chloroform, cyclohexanone, and acetone. The separated surfactants were identified by a color reaction and by infrared spectrophotometry.

(391) L. E. Cohen, "Turbidimetric Determination of Glassy Phosphates Employing *o*-Tolidine," *Chemist Analyst*, Vol. 47, pp. 65-67 (1958).

Sample dissolved in water-acetic acid mixture, filtered if necessary, and an aliquot containing 1 to 12 mg used. Added are 5 ml glacial acetic acid, then 5 ml of *o*-tolidine reagent (0.5 g *o*-tolidine dissolved in 4.8 ml glacial acetic acid and diluted to 100 ml with 10 per cent mannitol solution), and the turbidity measured at 425 m μ within 15 min. The glassy phosphate content is read from a standard curve. Ortho-, pyro-, tripoly-, and trimetaphosphates, sulfate, chloride, nitrate, borate, metasilicate, and nonionic surfactants said not to interfere. Anionic surfactants react with *o*-tolidine and must be removed from sample. Oxidizing agents also cause blue coloration in interference and must either be removed or destroyed prior to analysis. Reaction involves one mole of *o*-tolidine with two atoms of P in the long-chain condensed phosphate.

(392) A. Davidsohn "The Sulfonation of Alkyl Benzenes. A Routine Test for Unulfonated Matter," *Soap, Perfumery & Cosmetics*, Vol. 31, p. 392 (1958); *Chemical Abstracts*, Vol. 53, p. 8667 (1959).

A 200 g sample with 10 to 15 per cent water added is centrifuged and 20 g of the upper layer in 100 ml water neutralized with sodium hydroxide to slight pink of phenolphthalein and made up to 180 g with water. If 30 ml of this solution mixed with 30 ml of alcohol shows no turbidity, the sample contained less than 2 per cent unulfonated material. With samples sulfonated with sulfur trioxide, a 20 g sample is used. It is diluted to 190 g heated to 80 C, adjusted

to pH 8 to 9, and 30 ml of this tested as above.

(394) H. Étienne, "Determination in Water of Small Amounts of Surface-Active Polyglycols," *Bulletin du centre belge d'étude et de documentation des eaux (Liège)*, No. 40, pp. 159-166 (1958); *Chemical Abstracts*, Vol. 53, p. 1603 (1959).

The method consists of the concentration of one to two liters of water to 100 ml, precipitation of the phosphomolybdic acid complex in the presence of calcium ion and Celite, treatment of the petroleum ether washed precipitate with hydrogen iodide to form the ethyl iodide derivative of the ethylene oxide residues, and the determination of iodine as HIO₃ by the Viebock procedure. Details of the procedure are given.

(395) M. Z. Finkel'shtein, I. M. Timokhin, and Kh. U. Mukhamedov, "The Quantitative Determination of the Sodium Salt of Carboxymethylcellulose," *Izvestiya Vysshikh Uchebnykh Zavedenii, Neft i Gaz*, No. 12, pp. 45-50 (1958); *Chemical Abstracts*, Vol. 53, p. 16809 (1959).

Solutions of sodium carboxymethylcellulose (NaCMC) are acidified to pH 5.0, the precipitation completed with 1 per cent solution of aluminum nitrate, and heated on a water bath to 50 to 55 C. This is followed by filtration, washing and drying. The precipitate is converted from the aluminum to the sodium equivalent. Results are from 1.82 to 5.67 per cent low owing to incomplete precipitation of the aluminum salt. This was overcome by the addition of ethanol to 20 per cent of the volume of the solution. An alternative procedure is to precipitate the CMC as the copper salt with a known portion of copper sulfate solution, the precipitate rejected, and the excess copper determined iodometrically.

(396) C. M. Gardner, C. H. Hale, E. A. Setzkorn, and W. C. Woelfel, "Determination of Combining Weight of Sulfonates," *Analytical Chemistry*, Vol. 30, pp. 1712-1713 (1958).

Purified sulfonates analyzed by burning in stream of oxygen at high temperature (induction furnace) to decompose any compound of sulfur and liberate as sulfur dioxide, then titrated iodometrically. Comparison of sulfated ash, *p*-toluidine and induction furnace burning methods show latter very reliable and rapid.

(397) Mrs. Gatineau, "Analysis of Surfactants by Infrared and Ultraviolet Spectrophotometry," *Revue française des corps gras*, Vol. 5, pp. 254-264 (1958); *Chemical Abstracts*, Vol. 52, p. 14199 (1958).

A bibliographical review of fifteen papers published during 1950 to 1953.

(398) L. Gildenberg and E. W. Blank, "Error in the Sampling of Soap and Detergent Bars for Moisture Determination," *Journal of the Am. Oil Chemists' Soc.*, Vol. 35, pp. 102-103 (1958).

Moisture content in the corners of bars is lower than in samples from the center. For correct moisture, either the whole bar or a symmetrical portion should be taken for analysis instead of slices from the interior of the bar.

(399) G. A. Harlow and G. E. A. Wyld, "Resolution of Acid Mixtures in Nonaqueous Solvents. Potentiometric Titration of Dibasic Acids with Quaternary Ammonium Titrants," *Analytical Chemistry*, Vol. 30, pp. 69-72 (1958).

Solvents are isopropanol, methanol, ethanol, ethyl ether and pyridine. Titrant, prepared in anhydrous isopropanol as well as in water, is tetra-*n*-butylammonium hydroxide. Mixtures of acids of different charge types not readily resolved in water, are resolved in nonaqueous solvents. Sulfuric, oxalic and succinic acids are examples given.

(400) H. Holness and W. R. Stone, "The Separation of Quaternary Halides by Paper Chromatography," *Analyst*, Vol. 83, pp. 71-75 (1958).

Separation of homologous series of quaternary germicides by paper chromatography. New spray reagent permits detection of 0.6 g of quaternary base. Materials examined were saturated *n*-al-

kyltrimethylammonium halides, *n*-alkylbenzyltrimethylammonium halides and *n*-alkylpyridinium halides with alkyl chain lengths of 12 to 18 carbon atoms. Indicator spray solution is freshly prepared before use by mixing 5 ml 0.2 per cent *w/v* aqueous solution rhodamine BS with 10 ml 0.2 per cent *w/v* aqueous solution of Tinopal WG, adding 40 ml 0.880 specific gravity ammonia solution and diluting with water to 100 ml. Time of run about 7 hr.

(401) C. A. Huffins, "Determination of a Nonionic Detergent in Soap Mixtures," *Soap, Perfumery & Cosmetics*, Vol. 31, pp. 369-370 (1958); *Chemical Abstracts*, Vol. 52, p. 15100 (1958).

The method is based on the solubility of the nonionic compound in cold dioxane. Details are given for drying of sample, extraction, filtration, precipitation of the phosphomolybdate complex, and drying of the filtered precipitate. The quantity of active ingredient is taken from a reference graph prepared from known weights of the particular surfactant.

(402) J. Jerkovits, "Quantitative Volumetric Determination of Fatty Alcohol in Fatty Alcohol Sulfates by the *p*-Toluidine Method," *Fette, Seifen, Anstrichmittel*, Vol. 60, No. 1, pp. 44-46 (1958).

Unulfonated matter in fatty alcohol sulfate determined by extracting acidified aqueous alcohol solution with light petrol, removing solvent and weighing dried residue. Determination of fatty alcohol by removing ethanol from the aqueous phase by heating to 105°C and organic sulfate precipitated with *p*-toluidine hydrochloride. Complex formed extracted with ether and solution added to ethanol neutralized to cresol red, and titrated with 0.1N sodium hydroxide. *p*-Toluidine hydrochloride extracted at same time determined argentimetrically with potassium chromate indicator. If molecular weight fatty alcohol unknown, this determined by drying aliquot of the *p*-toluidine complex solution and weighing residue (see reference (209)).

(403) J. Kelly and H. L. Greenwald, "Chromatographic Separation of a Non-ionic Polyether Surfactant," *The Journal of Physical Chemistry*, Vol. 62, pp. 1096-1098 (1958).

A mixed *p*-tert, tert-octylphenoxy-polyoxyethylene-ethanol with 9.7 ethylene oxide units per phenol was separated into its components by chromatography. Silicic acid columns were used with mixed chloroform-acetone eluants. The shape of the elution curve suggested that the resolution was sufficient for the isolation of very pure species. Agreement with the expected distribution of species from the ethylene oxide plus phenol condensation reaction was satisfactory.

(404) W. J. Kirsten, A. Berggren, and K. Nilsson, "Potentiometric Titration of Some Organic and Inorganic Bases with Sodium Tetraphenylborate," *Analytical Chemistry*, Vol. 30, pp. 237-240 (1958).

Quaternary ammonium salts, amine hydrohalides, potassium nitrate, and other inorganic nitrates and sulfates were quickly and accurately determined by potentiometric titration with sodium tetraphenylborate solution, using glass-calomel-silver electrode pairs. As the iodide ion interfered in the determination it was removed by an exchange against acetate ion in an ion exchange column, prior to the titration. Recoveries of most compounds determined by this method ranged between 99 to 101 per cent.

(405) R. Kliffmüller, "Detection of the Polyoxethylene Compounds (Softeners) in Bread," *Deutsche Lebensmittel-Rundschau*, Vol. 54, pp. 59-61 (1958); *Chemical Abstracts*, Vol. 53, p. 9500 (1959).

The method of Jaminet (reference (253A)), modified by using the solvent mixture butanol 50, 25 per cent ammonium hydroxide diluted 1:10 25, and ethanol 12 ml used. This modification increases the R_f values of polyglycol-400-monostearate, Myrj 52, and Tween

60. Breads containing 0.5 to 2 per cent were analyzed.

(406) J. M. A. Lenihan, H. Smith, and J. G. Chalmers, "Arsenic in Detergents," *Nature*, Vol. 181, pp. 1463-1464 (1958).

Arsenic found in detergents to extent of 0.5 to 74 ppm and approximate amounts of arsenic adsorbed on hair. Method of analysis of M. D. Thomas and T. R. Collier, *Journal of Industrial Hygiene and Toxicology*, Vol. 27, p. 201 (1945).

(407) G. R. Lewis, "Determination of Synthetic Surface-Active Agents and Their Effects in Industrial Wastes," *University Microfilms*, Publication No. 24503, 200 pp.; Dissertation Abstract, Vol. 18, pp. 982-983 (1958).

Two phase titration in range 4 to 40 ppm of sodium tetradecyl benzene sulfonate with cetyltrimethylammonium bromide as cationic standard and Pontamine Fast Red 8 BNL as dye-transfer indicators. Average deviation of ± 2 ppm. Other suitable indicators were Brilliant Blue FCG, methylene blue, Safranin Y.

(408) J. E. Lovelock and T. Nash, "A Color Reaction for Nonionic and Amphoteric Soaps," *Nature*, Vol. 181, pp. 1263-1264 (1958).

Ability of certain nonionic and amphoteric surfactants to discharge the color of acetic acid solutions of fluorescein or dichlorofluorescein suggested as basis of possible method of analysis. Based on association of dye molecules with certain types of micelles and low water solubility. Solvents discharge color in inverse ratio to dielectric constant.

(409) I. Lysyj and J. E. Zaremba, "Rapid Quantitative Determination of Sulfur in Organic Compounds," *Analytical Chemistry*, Vol. 30, pp. 428-430 (1958).

Method utilized combustion in flask flushed with oxygen and containing solution of hydrogen peroxide. The solution is boiled to remove excess hydrogen peroxide and oxygen and the sulfuric acid formed is titrated with standard alkali.

Two procedures are given, the second for samples containing organic sulfur, nitrogen, halides, phosphorus or metals. Time analysis about 25 min. Precision ± 1 per cent.

(410) W. F. Maass, E. Buchspiess-Paulentz, and F. Stinsky, "Analysis of Petroleum Sulfonates," *Seifen-Öle Fette-Wachse*, Vol. 54, pp. 575-578 (1958); *Chemical Abstracts*, Vol. 53, p. 133 (1959).

Review of German and U. S. standard analytical methods.

(411) E. Mück, D. Kokes, and F. Langmaier, "Chromatography of the Sulfonated Products of Phenol-Formaldehyde Condensation. I. — Paper Chromatography of Phenosulfonic Acids," *Chemické listy*, Vol. 52, pp. 746-748 (1958); *Chemical Abstracts*, Vol. 52, p. 13295 (1958).

Synthetic tanning materials were chromatographed in the systems isopropanol-ammonium hydroxide and isoamyl alcohol-ethanol-ammonium hydroxide and detected with 1 per cent alcohol in solution of ferric chloride or diazonium salts, or by heating the spots to 180 C. Ultraviolet spectra were charted, and a chromatogram shown. R_f values on Whatman No. 3 paper given.

(412) J. E. Murphy and W. C. Schweimer, "Infrared Analysis of Emulsion Polishes," *Analytical Chemistry*, Vol. 30, pp. 116-126 (1958).

Rapid qualitative determination of wax, resin, polymer and emulsifier in emulsion polishes. Spectra of common components of polishes and of group of amine emulsifiers and their hydrochloride derivatives compiled. Best to separate components then determine infrared spectrum.

(413) G. Nebbia, "Reaction of Quaternary Ammonium Surface-Active Agents and Methyl Orange," *La Chimica e l'Industria (Milan)*, Vol. 40, pp. 638-640 (1958); *Chemical Abstracts*, Vol. 53, p. 3739 (1959).

Evidence that the reaction between methyl orange and tetradecylbenzylidi-

methylammonium chloride is a molar one, and not a complex; by separation of the salt formed, and analysis.

(414) S. H. Newburger, "The Analysis of Shampoos," *Journal of the Association of Official Agricultural Chemists*, Vol. 41, pp. 664-668 (1958); *Chemical Abstracts*, Vol. 53, p. 2542 (1959).

An ion-exchange resin method is proposed for shampoos containing an alkyl sulfate and a fatty acid-alkanolamine condensate as the active ingredients. An analysis scheme is given by chart. Soap can also be determined. Suggested that procedure probably applicable to emulsions containing sulfated and sulfonated esters.

(415) S. Okano and A. Matsubara, "Improvement of Methods Used in Practical Pharmacy. IX. — Colorimetric Determination of Nonionic Surface-Active Agents with Iodine and Starch," *Yakuzai-gaku*, Vol. 18, pp. 33-36 (1958); *Chemical Abstracts*, Vol. 53, p. 6534 (1959).

Application of the MacAllister and Lisk method (reference (169)) to the estimation of the concentration of solutions as dilute as 0.05 per cent with a reproducibility of ± 0.01 per cent.

(416) C. H. Orr and J. E. Callen, "Separation of Polyunsaturated Fatty Acid Methyl Esters by Gas Chromatography," *Journal Am. Chemical Soc.*, Vol. 80, p. 249 (1958).

Report made of the complete separation of the methyl esters of palmitic, palmitoleic, stearic, oleic, linoleic, linolenic, arachidic, and erucic acids by use of a column consisting of 30 per cent Reoplex 400 plasticizer on Celite 545 at 240 C. The esters in each carbon-number class emerge from the column in times which increase with the number of double bonds in the compound.

(417) M. Pernarowski and L. G. Chat-ten, "The Identification and Quantitative Analysis of Certain Quaternary Ammonium Compounds with Ultraviolet Absorption Spectra," *Journal, Am. Pharmaceutical Assn.*, Vol. 47, pp. 211-214

(1958); *Chemical Abstracts*, Vol. 52, p. 9845 (1958).

The determination of quaternary ammonium compounds by ultraviolet absorption spectroscopy described and the ultraviolet spectra for several commonly used cationic surfactants given.

(418) M. Pesez, "Identification of Some Quaternary Ammonium Compounds," *Annales pharmaceutiques françaises*, Vol. 16, p. 441 (1958); *Chemical Abstracts*, Vol. 53, p. 4656 (1959).

Filter paper is submerged in a solution of methylene blue, dried, and then submerged for 1 hr in a solution of mercuric iodide (20 per cent in 10 per cent potassium iodide solution), washed, and dried in the dark. The paper does not yield color to buffered solutions at pH 8, but after addition of an alkyldimethylbenzylammonium salt the solution is rapidly colored blue.

(419) S. Pinchas and P. Avinur, "Determination of Toluenesulfonic Acids in the Presence of an Excess of Sulfuric Acid," *Analytical Chemistry*, Vol. 30, pp. 2022-2023 (1958).

Mixtures of the three isomeric toluenesulfonic acids obtained from toluene and sulfuric acid at room temperature are determined spectrophotometrically in the presence of a large excess of sulfuric acid on the basis of their absorption. The absorptivities of these are given.

(420) F. W. Roberts and G. R. Lawson, "Some Determinations of the Synthetic-Detergent Content of Sewage Sludge," *Water and Waste Treatment Journal*, Vol. 7, pp. 14-17 (1958); *Chemical Abstracts*, Vol. 52, p. 20797 (1958).

Method given in which centrifuged dried, ground sludge is extracted with methanol. The extract may then be subjected to ordinary methods for anionic detergent estimation. Details of surfactant content in sludges given.

(421) M. Roussos, H. Passedouet, and J. Cariou, "Determination of Nonionic Ethenoxylated Surfactants," *Revue française des corps gras*, Vol. 5, pp. 13-24

(1958); *Chemical Abstracts*, Vol. 52, p. 7740 (1958).

The volumetric method of Schönfeldt (reference (234)) based on the reaction between nonionic ethenoxylated products and potassium ferrocyanide was applied to the analysis of several products, with the modification that the excess of ferrocyanide, measured by precipitation with zinc sulfate, was determined potentiometrically instead of titrating it with diphenylamine as indicator. The quantity of ferrocyanide fixed by the first members of polyoxy ethylene glycols is insignificant but increases proportionally with increasing molecular weight.

(422) R. Sargent and W. Rieman, III, "Salting-Out Chromatography. III. Aliphatic and Polyglycol Ethers, Carboxylic Acids," *Analytica Chimica Acta*, Vol. 18, pp. 197-203 (1958).

Previous work demonstrated that analysis of complex mixtures of alcohols, polyglycols and amines facilitated by salting-out chromatography in which process water-soluble nonelectrolyte separated by elution process through column of ion-exchange resin with a concentrated salt solution as eluant. Same process successful in separation of aliphatic and polyglycol ethers. Quantitative analysis accomplished by the chromatographic procedure followed by oxidation with dichromate. Less successful with fatty acids.

(423) S. Sass, J. J. Kaufman, A. A. Cardenas, and J. J. Martin, "Colorimetric Estimation of Tertiary and Quaternary Amines," *Analytical Chemistry*, Vol. 30, pp. 529-531 (1958).

Total amine determined by dissolving the sample in toluene and pipeting a 2-ml aliquot representing 20 to 70 gamma of amine into a test tube. One ml of *cis*-aconitic anhydride reagent was added and allowed to stand 15 min. Add 5 ml toluene, allow to stand 15 min and measure the color spectrophotometrically at 500 m μ . Free amine is determined by dissolving sample in toluene and pipeting a 3-ml aliquot representing 100-800 gam-

ma of amine into a test tube. One ml of chloranil in toluene is added and all heated on a boiling water bath for 15 min. Cool 5 min and measure the color spectrophotometrically at 610 m μ .

(424) R. Schrepfer and H. Egle, "Paper-Chromatographic Identification of Monofatty Acid Esters of Sorbitan, Polyoxyethylene, and Polyoxyethylene-sorbitan," *Zeitschrift für Lebensmittel-Untersuchung und Forschung*, Vol. 107, pp. 510-512 (1958); *Chemical Abstracts*, Vol. 52, p. 16632 (1958).

The procedure was designed for analysis of baking improvers. The esters are extracted with chloroform, saponified and acidified, fatty acids removed with benzene and the residue neutralized with 0.5N sodium hydroxide to pH 7. Interfering cations are removed by ion-exchange and anions with another ion exchanger. Chromatographing was with butanol saturated with water and Pb-(OAc)₄ reagent as developer. The R_f values of the polyhydroxy moieties released from the esters are given.

(425) A. M. Schwartz, J. W. Perry, and J. Berch, "Surface Active Agents and Detergents," Vol. 2, Chapter 14, pp. 323-367 (1958).

Covered are review of identification procedures, classification, qualitative analysis, quantitative determination of individual surfactants such as soaps, noncarboxylic anionics, cationics, ampholytic, nonionic and others, quantitative analysis of mixtures of surfactants, pH and moisture content, analysis of surfactant auxiliaries. Spectral analysis and control methods are given. Behavior of surfactant intermediates with urea, surfactants with proteins.

(426) S. Siggia, "Recent Advances in the Analysis of Soaps and Synthetic Detergents," *Journal of the American Oil Chemists' Soc.*, Vol. 35, pp. 643-648 (1958).

Review of qualitative and quantitative schemes of analysis. Nonionic products reacted with hydrobromic or hydrochloric acids and the split hydrophobe identified by infrared comparison. Traces of

nonionics determined by utilization of potassium iodomercurate which forms turbidity and this compared with prepared standards visually, colorimetrically or nephelometrically, detecting 1 to 10 ppm.

(427) S. Siggia, "Analysis of Nonionic Surfactants," *Soap*, Vol. 34, No. 3, pp. 51-53, 133 (1958).

Methods for determination of nonionic detergents from those for traces to methods for determining high concentrations are reviewed.

(428) S. Siggia, A. C. Starke, Jr., J. J. Garis, Jr., and C. R. Stahl, "Determination of Oxyalkylene Groups in Glycols and Glycol and Polyglycol Ethers and Esters," *Analytical Chemistry*, Vol. 30, pp. 115-116 (1958).

Procedure involves use of hydriodic acid to react with oxyalkylene group, thus forming one molecule of 1,2-diiodo alkane for each group. These diiodo compounds are unstable and split off one molecule of iodine to form corresponding alkylene. Iodine liberated is titrated with standard thiosulfate.

Polyglycol amines, the results correspond to the total oxyethylene groups on the molecule minus one, signifying HI attack on ether linkages, not the CN links.

(429) W. G. Spangler, D. E. Hawes, Jr., and J. A. Kish, "Application of Ion-Exchange Chromatography to the Analysis of Commercial Triphosphate," *ASTM Bulletin*, No. 228, Feb., pp. 61-65 (1958).

Two methods described, one relatively rapid control procedure, and the other a referee method. In the control method only *ortho* and *pyro* phosphates are determined by elution, while for the referee method *ortho*, *pyro* and triphosphate are determined directly by elution. Rapid method consists of 16 fractions separated and analyzed colorimetrically.

Referee method provides 30 fractions which are analyzed separately.

Time for control method about 2½ hr and 5 hr for the referee method. Precision of simultaneous duplicates and

over all respectively; ± 0.66 and ± 1.04 ; Tripolyphosphate ± 0.98 and ± 1.22 per cent (95 per cent limits).

(430) W. I. Stephen, "Analytical Chemistry," *Manufacturing Chemist*, Vol. 29, pp. 158-161 (1958); *Chemical Abstracts*, Vol. 52, p. 12649 (1958).

Annual review of methods including those for detergents.

(431) W. I. Stephen, "Analytical Chemistry," *Manufacturing Chemist*, Vol. 29, pp. 297-300 (1958); *Chemical Abstracts*, Vol. 52, p. 16691 (1958).

Annual review of methods, including those for detergents.

(432) W. I. Stephen, "Analytical Chemistry," *Manufacturing Chemist*, Vol. 29, pp. 526-528 (1958); *Chemical Abstracts*, Vol. 53, p. 4011 (1959).

Review.

(433) C. B. Stuffs, "The Determination of a Nonionic Detergent in Soap Mixtures," *Soap, Perfumery & Cosmetics*, pp. 369-370 (April 1958).

Preliminary test showed that precipitation of the calcium, magnesium and barium soaps followed by filtration and determination of nonionic in the filtrate gave very low results and alcohol extract of the soap followed by ashing and calculation of ash gave inaccurate results. The present method was based on dioxane extract of the detergent mixture (dioxane may explode on evaporation to dryness!) to remove the nonionic surfactant followed by the formation of a phosphomolybdic complex gravimetrically determined. Percentage of surfactant in product determined from a graph prepared for the specific nonionic and its complex.

(434) R. Suffis and D. E. Dean, "Analysis of Chlorophenolics in Soaps and Soap-based Preparations by an Ion-exchange Technique," *Proceedings of the Scientific Section of the Toilet Goods Assn.*, No. 30 pp. 13-14 (1958); *Chemical Abstracts*, Vol. 53, p. 5709 (1959). An ion-exchange procedure for the determination of chlorophenolics in soap

preparations is described, and typical analytical results given.

(435) H. Susi, N. H. Koenig, W. E. Parker and D. Swern, "Infrared Identification of Some Sulfur Derivatives of Long-Chain Fatty Acids," *Analytical Chemistry*, Vol. 30, pp. 443-446 (1958).

Infrared spectra of fatty acid derivatives containing sulfide, sulfoxide and sulfone groups studied.

(436) S. Suzuki, K. Ishihara, Y. Takeuchi, and O. Mitsuma, "Infrared Analysis of Tripolyphosphate," *Journal of the Osaka Institute of Science and Technology* (Kinki Univ.), Parts 1, 4, 9-19 (1958); *Chemical Abstracts*, Vol. 52, p. 19685 (1958).

Infrared spectra of commercial samples were compared with those of pure tripolyphosphate hexahydrate, two modifications of tripolyphosphate, tetrasodium pyrophosphate, sodium hexametaphosphate and orthophosphate. Each sample was ground to particles of 5μ diameter, ground with paraffin and placed between sodium chloride plates. The analysis was confirmed by titration potentiometrically, by treatment with cation exchanger, followed by titration of the free acids with sodium hydroxide.

(437) W. B. Swann, R. J. Zahner, and O. I. Milner, "Improved Saponification Number Determination by Use of Ion Exchange," *Analytical Chemistry*, Vol. 30, pp. 1830-1833 (1958).

Presence of weak acid salts may buffer and obscure endpoint in usual method. Procedure devised in which saponified material in nonaqueous medium passed through cation exchanger. Excess caustic converted to water and salts of organic acids to free acids, titrated potentiometrically. Procedure simplifies endpoint determination and precautionary measures to prevent absorption of carbon dioxide.

(438) K. Szmids, T. Zak, and Zb. Kwiatkowski, "Rapid Quantitative Methods for the Determination of Brightness, Levelers, and Antipitting Agents in Nickel Electroplating Baths," *Transactions of the Institute of Metal Finishing*

(Technical Papers), Vol. 36, pp. 17-21 (1958); *Chemical Abstracts*, Vol. 53, p. 11049 (1959).

Organic brighteners such as *p*-toluene-sulfonamide and wetting agents such as sulfonated isopropylphenylphthalene can be determined quantitatively from their influence on the polarographic maximum of oxygen. When both are present, the wetting agent can be determined separately by the maximum bubble-pressure method for surface tension.

(439) J. C. Vaughn, *et al.*, "Determination of Synthetic Detergent Content of Raw-Water Supplies," *Journal of the American Water Works Assn.*, Vol. 50, pp. 1343-1352 (1958).

Review of methylene blue and infrared procedures for alkylbenzene sulfonates, discussion of interferences and their possible mechanism. Provides detailed procedures for the methylene blue and infrared analyses.

(440) E. Vioque, A. Vioque, and M. del Pilar de la Maza, "Microphotometric Evaluation of Chromatographic Spots. Application to Mixtures of Fatty Acids," *Grasa y aceites (Seville, Spain)*. Vol. 9, pp. 168-175 (1958); *Chemical Abstracts*, Vol. 53, p. 7855 (1959).

Mixtures of fatty acids were separated and determined quantitatively by using paper chromatography and a photographic method. Benzene solutions of the acids were used. An aliphatic hydrocarbon was used as the fixed phase and a 9:10 mixture by volume of acetic acid and water as the mobile phase. The chromatogram was photographed on 35 mm film and the spot density recorded with a densitometer. Good determinations of C_{12} to C_{18} acids were obtained.

(441) P. Voogt, "Application of Ion Exchangers in Detergent Analysis," *Revue des travaux chimiques des Pays Bas*, Vol. 77, pp. 889-901 (1958); *Chemical Abstracts*, Vol. 53, p. 5709 (1959).

Procedures are described for separating mixtures of sodium salts of alkylsulfuric and sulfonic acids, fatty acids, and non-ionics. Three fractions strongly acidic, weakly acidic, and nonionic respectively

are obtained by passing an ethanol solution through the acidic form of Dowex-50 (8 per cent cross-linked), the acetate form of Dowex-1 (2 per cent cross-linked) and the basic form of Dowex-2 (8 per cent cross-linked). The acids are determined by potentiometric titration, and the nonionics are determined gravimetrically.

(442) L. E. Weeks, J. T. Lewis, and M. E. Ginn, "A New Titrimetric Analysis for Ethylene Oxide Condensates," *Journal of the American Oil Chemists' Soc.*, Vol. 35, pp. 149-152 (1958).

Determination of molar ratio of ethylene oxide for adducts with fatty alcohols and alkyl phenols. Method comprises titration with water to a turbidity endpoint of a solution of the adduct dissolved in dimethylformamide with benzene for fatty alcohol adducts, or with increased benzene for alkylphenol adducts. Dimethylformamide test data claimed to compare favorably with hydriodic acid cleavage procedure.

(443) J. B. Wilson, "(Determination of) Quaternary Ammonium Compounds (QAC)," *Journal of the Assn. of Official Agricultural Chemists*, Vol. 41, pp. 581-582 (1958); *Chemical Abstracts*, Vol. 53, p. 2536 (1959).

The use of QAC by manufacturers on wound dressings and swabs for cleansing eyes, ears, noses and throats of young children leaving residues which might cause injury necessitated the development of a method including identification of the compound by its reineckate.

(444) D. C. Wimer, "Potentiometric Determination of Amides in Acetic Anhydride," *Analytical Chemistry*, Vol. 30, pp. 77-80 (1958).

Perchloric acid exhibits increased acidic behavior in the presence of acetic anhydride permitting titration of amides as bases. Perchloric acid in acetic acid or dioxane used as titrant. Endpoints readily determined with a modified calomel-glass electrode couple. Method rapid, reproducible and convenient. Gives ex-

amples of aliphatic amides, among others, such as *n*-dodecylamide.

1959

(445) M. Aoki and Y. Iwayama, "Determination of Ionic Surface-Active Agents with Dyes I.—Erythrosin and Eosin Methods," *Yakugaku Zasshi*, Vol. 79, pp. 522-526 (1959); *Chemical Abstracts*, Vol. 53, p. 16562 (1959).

Alkyl sulfate and alkylaryl sulfonate titrated with cationic surfactant in buffer solution of pH 4.5, in the presence of chloroform and with erythrosin as indicator. The error was equal to, or less than 0.2 per cent. Also applicable to invert soaps and to long-chain aliphatic amines.

(446) M. Aoki and Y. Iwayama, "Determination of Ionic Surface-Active Agents with Dyes. II. Toluidine Blue Methad," *Yakugaku Zasshi*, Vol. 79, pp. 526-530 (1959); *Chemical Abstracts*, Vol. 53, p. 16562 (1959).

Toluidine blue found best indicator in a two-phase titration with anionic surfactant as titrant. The solution was adjusted to pH 8, in the presence of chloroform and change in the chloroform layer from crimson to blue indicates the end-point. An error of ± 0.2 per cent or less was found.

(447) A. W. Ashbrook, "Determination of Small Amounts of Triisooctylamine in Aqueous Solution," *Analyst*, Vol. 84, pp. 177-179 (1959).

Rapid spectrophotometric method for small amounts of tri-isooctylamine by the formation of complex with cobalt thiocyanate. Satisfactory for determination of up to 5 mg amine with standard deviation of ± 0.00012 per cent.

(448) M. Babcock, D. E. Terry, and A. J. Milun, "The Analysis of High Molecular Weight Quaternary Ammonium Chlorides," *Journal of the American Oil Chemists' Soc.*, Vol. 36, pp. 93-94 (1959).

Analysis for minor constituents. Amine hydrochloride determined by titration with standard alkali to phenolphthalein endpoint; if free alkali present titrated

with a standard acid. Sodium chloride titrated with standard silver nitrate after ashing. Total chlorine titration after correction for amine hydrochloride and NaCl determines quaternary ammonium chloride. Free amine titrated with standard acid to bromocresol green endpoint. Details given for procedures.

(449) D. N. Bernhart and W. B. Chess, "Quantitative Evaluation of Paper Chromatograms of Condensed Phosphate Mixtures," *Analytical Chemistry*, Vol. 31, pp. 1026-1028 (1959).

Clear and rapid separation of phosphates by modified acid solvent. Quantitative evaluation of chromatograms are based on hydrolysis and color development of each component, followed by measurement of resulting color intensities of paper with a densitometer.

(450) R. A. Brown, D. J. Skahan, V. A. Cirillo, and F. W. Milpolder, "High Mass Spectrometry. Propylene Polymer, Alkylated Benzene, and Wax Analysis," *Analytical Chemistry*, Vol. 31, pp. 1531-1538 (1959).

Quantitative methods using high mass spectrometer for these materials. For alkylbenzenes highly purified mono $C_{9,12,15,18}$ side chain benzenes prepared and a didodecylbenzene fraction as well. These were used to calibrate the equipment. Shown are instrumental conditions, volume sensitivity coefficients, and sigma peaks. Matrix solution used with digital computer.

Analytical data reproducibility good enough to warrant repeating analyses to 0.01 per cent and reproducible over several years ± 0.2 per cent. Each hydrocarbon type accurate within ± 1 per cent.

(451) W. B. Chess and D. N. Bernhart, "Determination of Traces of Pyrophosphate in Orthophosphates," *Analytical Chemistry*, Vol. 31, p. 1116 (1959).

Ion-exchange chromatography is proposed as the best method for the determination of pyrophosphates, quantities below 0.2 weight per cent $Na_2 H_2 P_2 O_7$

in orthophosphates. Details of the ion-exchange and elution given.

(452) D. C. Cullum and D. B. Thomas, "The Determination of Barium and Sulfate with an E. E. L. Flame Photometer," *Analyst*, Vol. 84, pp. 113-116 (1959).

Sulfate ion in detergent powder is determined by flame photometry. The surfactant is removed by dissolution of the product, extraction in the acid condition with ether, and the aqueous portion made to volume, a portion treated with barium chloride and centrifuged at high speed. The precipitate is washed, and the washing discarded. The precipitate is suspended in starch solution, and the suspension read in an E. E. L. flame photometer with either a Wratten No. 65A or 77 filter. Barium in the range up to 1000 ppm can be determined with the same filters. The anions present in the solution affect the barium determination greatly.

(453) M. Doležil and J. Bulandr, "Use of Fluorescent Indicators for the Determination of Small Amounts of Surface-Active Compounds. II.—Determination of Organic Quaternary Salts and Bases," *Collection of Czechoslovak Chemical Communications*, Vol. 24, pp. 70-73 (1959); *Chemical Abstracts*, Vol. 53, p. 6878 (1959). (see reference (363)).

(454) W. Fong, "Nonionic Detergents in Raw-Wool Scouring Including Studies of Waste Clarification," *American Dye-stuff Reporter*, Vol. 48, pp. P45-50 (1959).

Analysis in scouring water before and after treatment of dirt and grease carrying bound surfactant. Clarification by ultra centrifuging.

Interfering substances prevented estimation by ultraviolet and precipitation of other substances, presumably protein, with the phosphomolybdic precipitation method. Method used was that of Brown and Hayes (reference (276)), the cobaltothiocyanate procedure in which the blue complex was extracted with chloroform and the extract measured absorptiometri-

cally at 620 $m\mu$ at which peak minimum interference occurred.

(455) M. E. Ginn and C. L. Church, "New Columnar and Mixed-Bed Ion Exchange Methods for Surfactant Analysis and Purification," *Analytical Chemistry*, Vol. 31, pp. 551-555 (1959).

Separation of anionic surfactant by ion-exchange resin from the nonionic surfactant. Anionic portion eluted, recovered, and determined gravimetrically, as is nonionic portion. Applied to surfactants not cleaved by acid or base. Accuracy of both methods ± 5 per cent.

(456) H. D. Graham and R. McL. Whitney, "Colorimetric Determination of Sodium Lauryl Sulfate," *Analytical Chemistry*, Vol. 31, pp. 1673-1676 (1959).

Determination of as little as 4 ppm through measurement of the colored product by action of sodium lauryl sulfate upon haloquinone of *o*-tolidine in a buffered system. When buffered at pH 3 purple haloquinoid complex is formed. Interferences of alkylbenzene sulfonate and ester sulfates noted. Other oxidizing agents do not interfere unless combined effect exceeds 7.5 ppm available chlorine, (see references (30) and (44)).

(457) E. Heinerth, "Paper Chromatography of Condensed Phosphates," *Zeitschrift für analytische Chemie*, Vol. 166, pp. 37-39 (1959); *Chemical Abstracts*, Vol. 53, p. 16795 (1959).

After separation by paper chromatography of the condensed phosphates and location by spraying with MoO_4 —solution, and a reducing agent, the spots are cut out, the paper destroyed with perchloric acid, and phosphates hydrolyzed and orthophosphoric acid determined colorimetrically. The sample put on the paper should contain 150-200 γ P_2O_5 .

(457A) H. Holness and W. R. Stone, "A Systematic Scheme of Semi-Micro Qualitative Analysis for Anionic Surface-Active Agents: An Addendum," *Analyst*, Vol. 84, p. 392 (1959).

An additional 9th test by hydrolysis

with syrupy phosphoric acid, and separation by extraction (see reference (366)).

(458) Y. Iwayama, "New Colorimetric Determination of Higher Fatty Acids," *Yakugaku Zasshi*, Vol. 79, pp. 552-554 (1959); *Chemical Abstracts*, Vol. 53, p. 14819 (1959).

Ayer's method for colorimetric determination of fatty acids was examined and a fundamentally new method established. This is based on the fact that the copper salt of a fatty acid dissolves in chloroform in the presence of triethanolamine to show a stable blue color. This can be used for simple and rapid determination of saturated and unsaturated fatty acids of the C_{10-22} series. The method can be applied to the determination of free and conjugated fatty acid in fatty oils and nonionic surfactants.

(459) J. W. Jenkins and K. O. Kellenbach, "Identification of Anionic Surface Active Agents by Infrared Absorption of the Barium Salts," *Analytical Chemistry*, Vol. 31, pp. 1056-1059 (1959).

Barium salts of organic sulfate and sulfonate detergents of types commonly used in commercial products, and infrared curves used for identification. Absorption data given. Molecular weight of organic sample estimated by ashing to yield barium sulfate.

(460) R. G. Jensen and M. E. Morgan, "Estimation of the Monoglyceride Content of Milk," *Journal of Dairy Science*, Vol. 42, pp. 232-239 (1959); *Chemical Abstracts*, Vol. 53, p. 9502 (1959).

Existing methods for the estimation of 1-monoglycerides were modified so that the formaldehyde produced by periodic acid oxidation is measured colorimetrically by reaction with chromotropic acid. Isomerization with perchloric acid was used to convert 2-monoglycerides to 1-monoglycerides.

(461) R. M. Kelley, E. W. Blank, W. E. Thompson, and R. Fine, "The Determination of Alkyl Aryl Sulfonates by Ultraviolet Absorption," *ASTM Bulletin*, No. 237, April, pp. 70-73 (1959).

Procedure by measurement of absorp-

tion at 224 $m\mu$ and spectrophotometric method invalid when interference from materials showing strong 220 to 230 $m\mu$ absorption, such as benzene, toluene and xylene sulfonates. Good correlation between this method and alternate.

(462) J. D. Knight and R. House, "Analysis of Surfactant Mixtures. I.," *Journal of the American Oil Chemists' Soc.*, Vol. 36, pp. 195-200 (1959).

Determination of hydrophobic portion of surfactant molecule by decomposition with 93 per cent phosphoric acid. Aromatic sulfonates, straight chain alkyl sulfates, fatty amides and fatty acid esters gave good yields of starting materials. Dioctyl sulfosuccinate gave mixture of octyl alcohols and olefins; ethylene oxide condensates of lauryl alcohol, tridecyl alcohol and tertiary dodecyl mercaptan gave olefins; diisobutyl phenol-ethylene oxide adducts gave olefins, conjugated olefins and alcohols by rupture of the aromatic ring. Alpha-sulfo fatty acids or alkane sulfonates did not give recoverable oils.

(463) R. H. Kolloff, "Analysis of Commercial Sodium Tripolyphosphate by Reverse Flow Ion Exchange Chromatography," *ASTM Bulletin* No. 237, April, pp. 74-80 (1959).

Commercial tripolyphosphate fractionated by means of pressurized continuous-gradient, reverse flow elution. Quantitative determination of eluted species made by improved molybdenum blue colorimetric procedure. A single fraction is collected and analyzed for each species. Procedure twice as rapid as paper chromatography and two to three times as fast as most rapid ion-exchange procedures. Improved precision claimed over paper chromatography.

(464) E. Q. Laws and W. Hancock, "Some Alkyl-Aryl Sodium Sulfonates as Standard Substances in Detergent Chemistry," *Nature*, Vol. 183, pp. 1473-1474 (1959).

Pure straight chain alkylbenzene sulfonates and alphasdimethyldecyl benzene sulfonates were prepared. Use of methy-

lene blue test, and measurement of colored chloroform extracts show that the optical density times molecular weight was approximately a constant for the members of the series from *n*-octylbenzene upward. Branched chain and commercial dodecylbenzene sulfonate gave values slightly less than theory.

Straight chain alkylaryl sulfonates gave good paper chromatography values using alcohol/water and rhodamine 6B (0.05 per cent *w/v*). Branched chain compounds gave unsatisfactory values.

(465) W. E. Link, H. M. Hickman, and R. A. Morrisette, "Gas-liquid Chromatography of Fatty Derivatives. II. —Analysis of Fatty Alcohol Mixtures by Gas-Liquid Chromatography," *Journal of the American Oil Chemists' Soc.*, Vol. 36, pp. 300-303 (1959).

Acetates of the alcohols derived from sperm oil, and the alcohols derived from soybean and linseed oils by lithium aluminum hydride reduction of the methyl esters of the acids were analyzed according to the method previously described. The method was first tested on known alcohol mixtures, and corresponded in results with the corresponding acids present in the original oils.

(466) M. Romáňuk, "Identification of Fatty Acids C_8 - C_{11} by Paper Chromatography," *Collection of Czechoslovak Chemical Communications*, Vol. 24, pp. 2064-2065 (1959); *Chemical Abstracts*, Vol. 53, p. 15871 (1959).

Descending paper chromatography was used with a 2-ethyl-1-hexanol-water-ammonia solvent system to obtain a successful separation of caproic, heptylic, caprylic, pelargonic, capric and undecylic acids, and the R_f values given. Detection was carried out by spraying the dried chromatograms with chlorophenol red.

(467) G. Russell and E. Whittaker, "The Equivalent Weights of Wetting Agents," *Analyst*, Vol. 84, pp. 253-254 (1959).

Both anionic and cationic wetting agents made to yield equivalent of titratable acid by passage through cation-exchange

resin (Zeo-Karb 225). Proposed method more rapid. Discrepancy found with a potassium salt of Teepol 530. Average difference between proposed and other methods is ± 1.12 per cent.

(468) C. D. Schmulbach, "Quantitative Determination of Traces of Pyrophosphate in Orthophosphates," *Analytical Chemistry*, Vol. 31, p. 116 (1959).

Best for quantities less than 1 per cent by paper chromatography (Karl-Kroupa, reference (333)) by using large number of overloaded sample spots. Aluminum interference removed by precipitation with 8-quinolol and extraction with chloroform. Also use of ion-exchange chromatography for large sample weight and high sensitivity.

(469) J. G. Slack, "The Determination of Anionic Detergents in River Water and Sewage Effluents," *Analyst*, Vol. 84, p. 193 (1959).

Increase in speed of determination of Longwell and Maniece (reference (290)) by single extraction with chloroform. Data show good agreement for surfactant alone, or in sewage or river water.

(470) M. J. Smith, "Analysis of Phosphate Mixtures by Paper Chromatography," *Analytical Chemistry*, Vol. 31, pp. 1023-1025 (1959).

An ascending solvent technique at 4°C for critical appraisal of distribution theories for phosphate mixtures and glasses. Phosphate estimated by the molybdenum blue procedure.

(471) W. B. Smith, "The Analysis of Synthetic Detergents," *Analyst*, Vol. 84, pp. 77-89 (1959).

Review of general analysis based on classification comprising hydrophobic group, linking group and hydrophil group covering all types of surfactants. Qualitative tests for hydrophil, hydrophobe portion. Separation by extractions with light petroleum, ethyl ether or alcohols. Colorimetric determination, anionic-cationic titrations, endpoint determination, and blank corrections and stoi-

chiometry covered. Nonionic analysis detailed by precipitation and cloudpoint methods. Inorganic constituents discussed.

(472) D. Ramaswamy, P. V. Rajalakshmi, and Y. Nayudamma, "Nonaqueous Titration Technique in Leather Research. V.—Analysis of Sulfated Oils," *Bulletin of the Central Leather Research Institute Madras* (India), Vol. 5, pp. 255-268 (1959); *Chemical Abstracts*, Vol. 53, p. 12721. (1959).

Sulfated oils used in fat liquoring are neutralized to pH not less than 5 and contain sodium salts of fatty acids, alkyl sulfates, alkyl sulfonates, sodium sulfate and fatty acids. Separation of first three and the sulfate from the fatty acid by extracting with isopropanol (90 per cent). The soap was determined in an aliquot of the extract by diluting with ethylene glycol-butanol (1:1) and titrating potentiometrically with 0.1N perchloric acid in dioxane. The first three are determined together by treating an aliquot of the extract with a saturated solution of mercuric acetate in acetic acid at 80 C to convert the salts to acetates and titrating with 0.1N perchloric acid in dioxane. Determine the first four by the same procedure applied to the original oil. The alkyl sulfate is determined by treating an aliquot of the isopropanol extract with 0.5N hydrochloric acid and water and evaporating nearly to dryness to hydrolyze. The residue is taken up in ethylene glycol-acetone (3:2) and titrated potentiometrically with standard sodium hydroxide in ethylene glycol-butanol (1:1). Two inflection points are obtained, the first corresponding to neutralization of excess hydrochloric acid and the difference between the two representing the sodium bisulfate formed by the hydrolysis. The fatty acid is determined by titration of the original oil with 0.2N sodium hydroxide in ethylene glycol-butanol. The quantities of each is determined by difference, and the determined values. These procedures were verified in some details by analyses of known

materials, and the procedures in general claimed to be less laborious than the usual methods.

(473) C. J. Turi, "Analytical Studies on Surfactants. I.—Polyethylene Glycols," *Rendiconti istituto superiore di sanità*, Vol. 22, pp. 174-188 (1959); *Chemical Abstracts*, Vol. 53, p. 18516 (1959).

Labat's reaction as adapted by Castagnou and Quilichini (reference (189A)), or Gallo (reference (246)), was positive only with polyethylene glycols of low molecular weight (less than 600). After preheating 2 to 3 min to 200 C to depolymerize them, the test is positive for glycols of high molecular weight. Distillation of the polyethylene glycol yielded AcH, which gives a red ring with the Labat test, reduces Fehling solution, and forms indigo; it also yielded glycols of lower molecular weight which give the typical blue-green ring in Labat's reaction. Polypropylene glycols also underwent pyrolysis yielding propionic aldehyde (red ring). These results correspond to those obtained by Rosen (reference (293)), but are thought to be free of interference by acrolein or other aldehydes formed from sources other than polyethylene glycols by the pyrolytic action of phosphoric acid.