

## NEW BOOKS

**NORME ITALIANE PER IL CONTROLLO DELLE SOSTANZE TENSIOATTIVE**, I EDIZIONE, edited by Commissione Tecnica Governativa per gli olii minerali, i grassi, i colori, le vernici e i detergenti (Stazione Sperimentale per le Industrie degli olii e dei grassi, Milan, Italy, 1965, price not indicated).

This handbook is the result of four years of work by a group of 30 specialists who are members of the Italian Government Technical Commission for the surface active agents and by the Italian Committee for the study of detergents. It represents the last volume of a series of publications prepared by a Government Technical Commission for the study of mineral oils, fats, dyes, paints and detergents and it is the first Italian handbook covering standardized procedures for the analytical control of surface active agents. The table of contents lists the following topics: analytical procedures for raw materials, inorganic adjuvants and organic adjuvants; analytical procedures for anionic surfactants; analytical procedures for final products such as soaps and household detergents. There are tables covering the specifications of the solvents used, chemical and physical data of fatty alcohols, fatty acids. All together, 117 analytical procedures are described. They are very clearly reported and include a short description of the apparatus used, reagents involved, a.s.o. The looseleaf binding has been adopted by the publisher; this setup is quite unusual for a handbook and will make easier the replacement of the pages containing the procedures which may eventually become obsolete.

This book can be recommended to any chemist involved in both the routine and nonroutine control of the specifications of anionic surface active agents and of the raw materials used in their manufacturing.

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**PAINT TECHNOLOGY MANUALS. 6. PIGMENTS, DYESTUFFS AND LAKES**, edited by C. J. A. Taylor and S. Marks (Reinhold, 340 p., 1966).

This is the sixth volume in a seven-volume series which is being published on behalf of the Oil and Colour Chemists' Association. This volume contains seven chapters, eight color plates and 13 black and white plates. The chapters cover General Introduction, Color, Testing and Evaluation of Pigments, White Pigments and Extenders, Colored Inorganic Pigments,

(Continued on page 101A)

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# ABSTRACTS OF PAPERS 58TH ANNUAL MEETING NEW ORLEANS, LOUISIANA

— 1 —

## ALPHA OLEFIN SULFONATE FORMULATIONS AND PROPERTIES

*E. A. Knaggs, J. A. Yeager, M. Nussbaum and E. J. Buerki*

The recent commercial availability of high-purity alpha olefins in the C<sub>10</sub> to C<sub>20</sub> range, combined with continuous SO<sub>2</sub> falling film processing technology, has made possible the commercialization of alpha olefin sulfonates.

The effect of olefin purity and chain length, and sulfonation processing conditions on derived product properties is reviewed, indicating that a variety of specialties can be tailored, depending on the raw material feedstock.

Alpha olefin sulfonates have been dried on a commercial scale, both in a spray tower and on a drum drier. The resultant beads and flakes are free-flowing and nonhygroscopic.

Emphasis is also placed on liquid detergent formulations both for household light-duty liquid applications, and for various cosmetic specialties including shampoos, bubble baths and skin cleansers. Results of Draize rabbit eye irritation studies, and rabbit skin irritation studies established the alpha olefin sulfonates to be significantly milder than conventional anionics used today.

The extreme mildness properties of these olefin sulfonates combined with their improved biodegradability, and formulation flexibility, underscore the importance of this "third generation" detergent in meeting our ever increasing detergent standards.

— 2 —

## NEW NONIONIC DETERGENTS DERIVED FROM EPOXIDIZED OILS. IV

*K. L. Johnson*

The results of an experiment designed to investigate optimal composition, catalyst level, molar ratio, and cosolvent level for the reaction of methylepoxysearate with polyoxyethylene glycol are presented. The results of this study show that methyl epoxysearate can be readily converted to nonionic wetting agents of technical importance in the most rudimentary processing equipment. The reaction takes place at ambient temperatures, atmospheric pressure, and without a cosolvent. Dry raw materials and adequate mixing are the only stringent requirements.

— 3 —

## THE EFFECT OF UNSATURATION ON DETERGENCY OF TALLOW ALCOHOL SULFATES

*R. S. Klonowski, C. M. Josefson, F. Ozkan and T. W. Findley*

Preparatory to a study of selective hydrogenolysis of animal fats, and to determine the effect of such selectivity on the function of detergents made from tallow alcohols. Soil removal and whiteness retention were measured for 5 concentrations (0.05% to 0.25%), 5 levels of unsaturation (0% to 50%) of tallow alcohol sulfates and ethoxylated (3 moles) tallow alcohol sulfates, with and without builder, at 3 levels of water hardness (0 ppm to 300 ppm), and at 3 temperatures (100F to 180F). Selective experimental design and statistical evaluation of results enabled conclusions to be reached with a minimum of experimental determinations.

Sulfates of tallow alcohol mixtures containing unsaturated alcohols have better soil removal properties than mixtures containing only saturated alcohols. This is especially true for built systems, but less evident with ethoxylated alcohol sulfates. It is most evident at low temperatures or high water hardness.

— 4 —

## ETHER ALCOHOL SULFATES FROM OLEYL ALCOHOL

*J. K. Weil, A. J. Stirton and Eileen B. Leardi*

A study on the effect of oxyethylation, oxypropylation and oxybutylation of tallow alcohols has now been extended to include the unsaturated component.

Distribution constants, the ratio of ether alcohol reactivity to parent alcohol reactivity, have been found to be about 2 and increasing for the ethylene oxide reaction, 0.5 and constant for the propylene oxide reaction, and 0.3 and constant for the butylene oxide reaction. Thus propylene oxide and butylene oxide reactions give higher yields of the important first derivative than the ethylene oxide reaction.

First and second derivatives from each epoxide were separated by distillation. The derivatives, which were 97-99% pure according to GLC, were characterized by freezing point and refractive index.

Sulfation of the unsaturated ether alcohols with dioxane-SO<sub>2</sub> led to high-purity ether alcohol sulfates which were evaluated as detergents. The products were soluble in water and toluene and were effective detergents and lime soap dispersing agents.

Sulfation of the unsaturated ether alcohol sulfates with 1.2 moles of chlorosulfonic acid gave a product with 60% double bond retention. Similar sulfation of oleyl alcohol gave only about 25% retention of the double bond. Alkyl substitution of the ether or additional groups did not change the degree of protection. Ether alcohols sulfated by this method likewise showed improved solubility and favorable properties in combination with saturated ether alcohol sulfates and soap.

— 5 —

## SPRAY DRYING OF HEAVY DUTY DETERGENT FORMULATIONS: ADVANTAGES OF TRIMETAPHOSPHATE IN PLACE OF TRIPOLYPHOSPHATE

*S. J. Silvio and Mario Ballestra*

Plant tests were made to evaluate the economic as well as processing advantages of using sodium trimetaphosphate in place of sodium tripolyphosphate. The Ballestra Spray Tower System containing the

continuous feeding and crutching units was used. The sodium trimetaphosphate was converted to completely hydrated sodium tripolyphosphate continuously within the crutching mixture. The spray tower operations and efficiencies as well as the finished product characteristics were compared when using the trimetaphosphate to produce the finished product and when using the low temperature-rise tripolyphosphate normally employed in this plant. Results showed that the use of trimetaphosphate gave the following advantages: 1) a fully hydrated slurry without the lumps and viscosity problems normally caused by tripolyphosphate; 2) a finished product with a higher percentage of the P<sub>2</sub>O<sub>5</sub> in the desirable form of tripolyphosphate; 3) significantly increased tower capacity; 4) higher finished product moisture content; 5) steadier tower operation resulting in better control of finished product moisture and density.

— 6 —

## PENTANE FROM THERMAL DECOMPOSITION OF LIPOXIDASE-DERIVED PRODUCTS

*C. D. Evans, G. R. List, Ami Dolev, D. G. McConnell  
and R. L. Hofmann*

Thermal decomposition of 13-hydroperoxyoctadeca-9,11-dienoic acid forms pentane predominantly to the exclusion of practically all other short-chain hydrocarbons. Oxidative polymers are simultaneously formed during lipoxidase oxidation of pure linoleic acid, and the polymer fraction also yields pentane to the exclusion of other hydrocarbons under similar thermal conditions. Chromatographic pure fractions of the hydroperoxide and the polymer were separated from the unoxidized linoleic acid in yields of 36 and 27%, respectively. Polymer formation simultaneously with hydroperoxides by lipoxidase is a new concept important to edible oil quality and oil processing. Thermal release of pentane is helpful in interpreting the structure of oxidative dimers and polymers and is in agreement with current findings on the properties of oxidative polymers.

— 7 —

## EDIBLE OIL QUALITY AS MEASURED BY THERMAL RELEASE OF PENTANE

*C. D. Evans, G. R. List, R. L. Hofmann and Helen A. Moser*

Fatty hydroperoxides produce normal hydrocarbons as part of their thermal decomposition products. The extent of hydrocarbon formation can be measured and associated with the quality and potential stability of an oil. Edible oils, high in linoleic acid, develop 13-hydroperoxy-9,12-octadecadienoic acid as one product of autoxidation. Since this particular hydroperoxide isomer yields pentane on thermal decomposition, the amount of pentane released has been correlated with the flavor of soybean and cottonseed oils and with the peroxide values of the respective oils. The amount of pentane released has an inverse linear relationship to flavor and a direct linear relationship to the peroxide values.

Edible oils exposed to light have a different relationship between flavor score and thermally derived pentane than do the same, but autoxidized, oils. Curves showing the relationship between flavor and peroxide value to pentane for autoxidized and light-struck soybean and cottonseed oils have been plotted. Application and limitations of this analytical method for flavor and quality evaluation will be described.

— 8 —

## SIMPLE PILOT-PLANT BATCH PROCESSES FOR ELIMINATION OF THE HALPHEN-TEST RESPONSE OF COTTONSEED OILS IN CONJUNCTION WITH DEODORIZATION

*P. H. Eaves, H. P. Dupuy, L. L. Holzenthal, E. T. Rayner  
and L. E. Brown*

Cottonseed oils contain cyclopropanoid fatty acids, chiefly malvalic, in amounts ranging from about 0.3 to 0.7%. Even trace quantities of cyclopropanoids are qualitatively detected by the Halphen test, while they may be determined quantitatively by HBr titration. As unusual biological effects have been ascribed to the cyclopropanoids, research has been carried out to develop methods of treating cottonseed oils to render them Halphen-negative and thus eliminate the biological effects.

Based on a laboratory procedure, two simple but effective pilot plant processes utilizing a pilot-plant size conventional deodorizer were developed for the production of Halphen-negative cottonseed oils.

Refined and bleached winterized cottonseed oil containing about 0.53% cyclopropanoids (calculated as malvalic acid) was used as starting material. In one process from 0.5 to 1.5 equivalents (based on cyclopropanoid content in the oil) of distilled cottonseed acids were mixed with the oil; then the mixture was charged to the deodorizer and heated to 450F under about 17 in. Hg vacuum while being agitated vigorously with a small steam sparge. When tests showed the oil to be Halphen negative, the excess free fatty acid was removed and the oil deodorized at 29.9 in. Hg vacuum by sparging with 3% steam. The time required for rendering the oil Halphen-negative (after reaching 450F) ranged from 5 min for 1.5 equivalents of free fatty acid to 75 min for 0.5 equivalents.

In a second process the addition of free fatty acids was omitted, and the small amount of cottonseed acids required for reaction with the cyclopropanoids was generated in situ by heating the oil at 450-455F under about 17 in. Hg vacuum while sparging lightly with 1.5% steam. When the oil tested as Halphen-negative, it was deodorized under high vacuum in the conventional manner. About 1.5 hr of heating at 450-455F were required to render the oil Halphen-negative by this procedure.

— 9 —

## PILOT-PLANT SELECTIVE HYDROGENATION OF SOYBEAN OIL: ACTIVATION AND EVALUATION OF COPPER-CONTAINING CATALYSTS

*K. J. Moulton, D. J. Moore and E. E. Beal*

The linolenate content in soybean oil has been reduced to less than 1% under pilot-plant conditions without increasing the saturates by hydrogenation with an active copper-chromite catalyst.

When used for the selective hydrogenation of soybean oil, several commercial catalysts containing copper had less than optimum activity. Their activity was improved by heating the catalysts at an elevated temperature. During the correlation of hydrogenation data on catalyst activity with oil quality, differential thermal analysis proved a useful

(Continued on page 102A)

(Continued from page 99A)

Organic Dyes and Pigments and The Color House. There is a table of contents and an index.

Like the preceding volumes, this is basically a textbook to be used by students, teachers and others who wish to learn about the Paint and Coatings Industry. The book is written for the beginner and, thus, is of little value to those experienced in Coatings. As a reference book, this volume would have some value, but is not completely up-to-date. For example, there is very little mention of some newer pigments such as: chloride process titanium dioxide, quinaeridones and rust inhibitive pigments other than red lead.

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INSOLUBLE MONOLAYERS AT LIQUID-GAS INTERFACES, by G. L. Gaines, Jr. (Interscience Publishers, Division of John Wiley & Sons, Inc., New York, London and Sydney, p. xiv + 386, 1966, \$14).

THIS IS THE FIRST of a projected series of *Interscience Monographs on Physical Chemistry* edited by I. Prigogine who provides a brief editorial introduction.

After the author's preface the book is divided into nine chapters, each, except for the very brief first chapter, being subdivided into III to VII sections. Indexing, while not extensive, is helpfully divided into three parts for authors, subjects and film compounds.

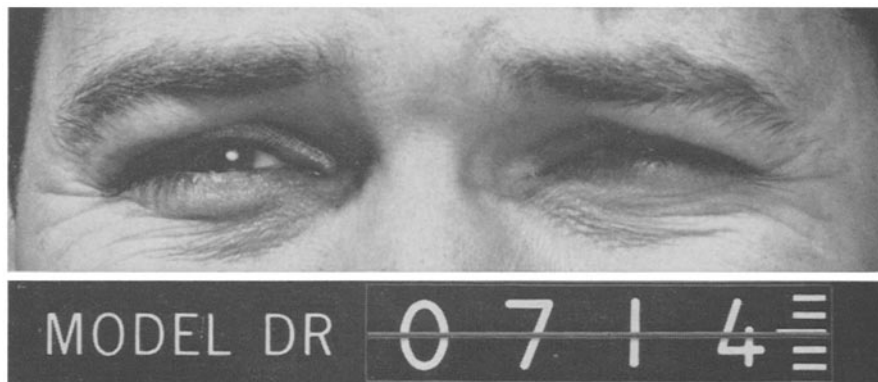
Following Chapter 1, a delightful "Historical Introduction" starting pertinently with Pliny, proceeding to Benjamin Franklin then Young, Gibbs, Langmuir and the rest, the book proceeds to Chapter 2 on "Properties of Liquid Surfaces" with a description of surface phenomena and an all too brief but lucid statement of the thermodynamics involved. Chapter 3, one hundred pages of very excellent description of "Experimental Methods" is replete with helpful details and precautions that characterize the book. Some comment on methods for basic surface tension measurements would have rounded the picture. Chapter 4 is also outstanding on "Properties of Monolayer Films" from a rather general point of view, and Chapter 5 almost equally so on "Properties of Various [individual] Substances in Monomolecular Films." Two short chapters, 6 on "Mixed Monolayers" and 8 on "Transfer of Monolayers to Solids · Multilayers," are separated by 7, a careful analysis of the important topic of "Reactions in Monolayers." A final brief Chapter 9 on "Scientific and Technological Applications" touches many areas but adds no great amount of substance.

References at the end of each chapter are extensive, but not overwhelming, with a careful effort to guide the reader to basic papers and the better review articles.

It is hard to see how a much better book could have been written to cover the area of the author's intent. "It is intended primarily for the research worker who wishes to study monomolecular films either for their own sake or to gain understanding of natural phenomena which involve interfaces and oriented molecules." Among all the books on surface chemistry none is quite like it and few tell their story nearly so well.

Every chemical library concerned with surface films and every specialist in surface films should possess the book, even at its substantial price. It is not that the book will tell the reader all he may wish to know. The author sticks to his last and does not much depart from films at air-liquid interfaces, although he does, indeed, make the proper connections to general theory and liquid-liquid interfaces as the situation indicates. Also, from the nature of the subject, the reader will meet disappointments, since, as the author's scholarly cautionings reveal

(Continued on page 103A)



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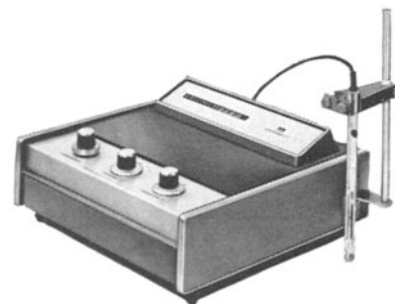
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