Orthodichlorbenzene An Ideal Fat Solvent

Non-corrosive Qualities, Non-inflammability and High Volatility with Steam Among Advantages Listed

By J. EHRLICH

MANY fat and oil technologists in recent years have been converted to the method of isolating fatty substances by means of an organic solvent. The chief advantages, as against the cold or hot pressing processes, are low initial investment and practically quantitative isolation of the original fat from the cake or meal. The most efficient pressing methods will leave 3-10 percent fat in the cake, depending on the material pressed, whereas a solvent process, when properly conducted, will leave a maximum of ½ percent.

The "orthodichlorbenzene" of commerce has many outstanding properties as a fat solvent. A typical commercial quality contains approximately 75 percent orthodichlorbenzene and 25 percent of the para isomer. Traces of trichlorbenzene may be present. The solvent is available in large quantities at a reasonable price as a byproduct of the manufacture of paradichlorbenzene. The material is a clear, practically white, limpid liquid. It shows only a slight solidification at 7° C, and must be cooled appreciably below this point to assume a pasty consistency. The boiling range is approximately 160-200° C. The specific gravity will vary slightly, a type sample being 1.309 at 25/25° C.

Orthodichlorbenzene is volatile and has a penetrating but not unpleasant camphoraceous odor. Bulletin 167 of the U. S. Department of Agriculture shows that paradichlorbenzene vapor is harmless to human beings under ordinary conditions. Orthodichlorbenzene may be expected to show analogous pharmacological properties. It may, nevertheless, be stored under water, with which it is immiscible. The fire hazard is conspicuous by its absence. A free flame may be played with impunity over the surface of the solvent until the flash point (about 67° C) is reached. But even above this temperature the material will not continue to burn if the flame is removed. This compares very favorably with the chlorinated aliphatic hydrocarbons in general use, and like them has the advantage of non-flammability over solvents of the benzol or gasoline type.

Non-corrosive Properties

BUT a quite common difficulty encountered in oil solvent processes employing chlorinated alipahtic hydrocarbons is the formation of muriatic acid due to the hydrolysis of these chlorine compounds by moisture in the fatty material being extracted. This necessitates thorough preliminary drying of the fatty material and also requires expensive acid-resisting installations. On the other hand, orthodichlorbenzene is characterized by its extreme stability towards water under the most drastic conditions.

In one experiment made at the Verona Chemical Co. to show the inertness of orthodichlorbenzene towards water, a mixture of these materials in vapor form was passed during several hours, through a heated steel tube so that the inside temperature reached 180° C. The exit gases on condensing gave unchanged orthodichlorbenzene and water. The latter showed no trace of muriatic acid present. The inner surface of the steel tube remained bright and uncorroded. In another experiment orthodichlorbenzene was boiled with water in a steel vessel for 48 hours under reflux condenser. Here likewise no sign of corrosion was evident, and no muriatic acid was formed. Obviously then, steel construction can safely be used throughout for solvent plant employing orthodichlorbenzene, and the necessity for preliminary drying of fatty material is eliminated. Moreover, orthodichlorbenzene displays a great avidity for fats, and extracts them completely even from substances containing an appreciable amount of free water.

Because of the relatively high boiling point, it is impractical to recover the solvent by heating with indirect steam. This presents no difficulty, since orthodichlorbenzene is volatile with only 1.5 times its weight of direct steam at atmospheric pressure. Due to this extreme volatility with steam, the recovery of the solvent then becomes a simple direct steam distillation to remove it quantitatively from the dissolved fat content. The vapors from the steam still are readily condensed, and then separate quickly and sharply into a lower layer of orthodichlorbenzene (ready for re-use) and an upper one of water, in which the solvent is insoluble. The loss of solvent is negligible.

A new lot of orthodichlorbenzene when purchased may show a small amount of material not volatile with direct steam. On these batches a preliminary steam distillation is required before using in the solvent plant, in order to obviate any danger of contaminating the fat.

The use of orthodichlorbenzene as a fat extraction medium was described and patented several years ago[1]. There has been a recent application of this process to the quantitative assay of fat bearing materials[2].

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[1] J. Ehrlich to Verona Chemical Co., U. S. Patent 1,610,270 of Dec. 14, 1926.

[2] C. P. Harris—This Journal 7 343-4 (1930). Robert Schwarz —This Journal 7 335-6, 347 (1930).

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Soap Analysis Report

Due to unforeseen delays in organization of the Soap Analysis Committee, it was not possible to finish any work in time to present more than a progress report at the Fall Meeting of the American Oil Chemists' Society in Chicago. Plans, however, have been formulated to study the following determinations in soap analysis: unsaponifiable, unsaponified, total fatty acids, total fatty anhydrides, and true soap. Present official methods as outlined some years ago by the joint committee for the American Chemical Society and the American Oil Chemists' Society provide for the use of ethyl ether in these determinations. This solvent introduces difficulties due, first, to the formation of emulsions, second, to its power of dissolving traces of soap, and third, to the fact that, apparently, water solutions of soap hydrolize and yield fatty acids, introducing another error in the analysis and its necessary correction.

Two samples, one a practically pure tallow soap powder, and the other the same soap containing rosin, were prepared by drying and grinding by Mr. Peterson and mailed to the members. On these samples, the members were asked to determine the unsaponifiable and the unsaponified by the official method, correcting for soap in the final residue, and also for fatty acids by titrating in the cold with alcohol, phenolphthalein and caustic. The same tests were then to be made, according to the FAC method for unsaponifiable using petroleum ether, and making the same corrections if necessary. Of course in determining the unsaponified, it was only necessary to dissolve 5 grams of the soap in 50% alcohol and extract with petroleum ether. However, after these had been extracted with petroleum ether, the soap solutions were boiled to free from alcohol, dissolved in water and re-extracted with ethyl ether as in the preceding method making the same corrections as before.

Due to unforeseen delays, as explained above, we have not yet finished this work. Mr. Sheely and Mr. Church have submitted complete reports as well as the Chairman. The others we hope to receive soon. However, the following general conclusions seem to be indicated already: the difference in the results obtained by the two solvents (after proper corrections are applied) seem immaterial: there is no comparison between the speed and ease of handling the tests, as the petroleum results can be obtained complete in a short time, while due to the emulsions formed, the necessity of making corrections, etc., the ethyl ether method takes several days: further, due to the fact that the soap is dissolved in water for ethyl ether extractions hydrolysis is caused to such an extent that the extractions with ethyl ether gave large residues of extractable matter, which, however, were practically all fatty acids. Of course, we can not render a complete report until all the other collaborators finish their work.

We also propose to study the total fatty acids determinations, the determination of fatty anhydrides, and the measurement of true soap as sometimes the results obtained by calculations from the total fatty acids do not check the results obtained by determining the soap by titration of the acids with caustic and weighing the soap. If any member of the Society or anyone else, has any suggestions along these or other lines for the soap committee to work out, we would be very glad to receive them.

H. P. Trevithick, *Chairman*

Glycerin Analysis Report

The following report of the Glycerin Analysis Committee of The American Oil Chemists' Society was presentel at the Fourth Annual Fall Meeting at Chicago, November 13.

THE report of the Soap Section at New Orleans last May directed attention to the fact that the accepted analysis of the A.O.C.S. Standard Crude Glycerin accounts for only about 98.5% with 1.5% unaccounted for. The analyses used to make up this total were those for true glycerol content by the acetin method, organic residue, ash and moisture. This discrepancy was confirmed in the laboratory of Mr. A. K. Church by an analysis on a sample of Standard C. P. Glycerin certified to by the British Executive Committee in which the acetin result was about 1.5% lower than the apparent glycerin content by the specific gravity or bichromate method. In view of the excellent agreement between cooperative analyses by the acetin method, these discrepancies are very disturbing.

While the problem is chiefly of an academic nature, a letter of inquiry sent out by your chairman evoked a surprisingly large number of replies indicating not only a keen interest but a willingness to work. It was felt that any further cooperative work should take the form of analyses by the bichromate, acetin and specific gravity methods on several commercial distilled glycerins of C.P. or U.S.P. grade and, if possible, on a few highly purified glycerols. A number of laboratories have volunteered to determine moisture on these cooperative samples but, as was expected, few care to undertake the task of preparing a highly purified glycerol. One such attempt has been made, however, and a set of four samples is ready for distribution to fourteen laboratories which have agreed to work along the lines outlined above. We are also in correspondence with the British Executive Committee, from whom we hope to obtain sufficient Standard C. P. Glycerin for our use as a cooperative sample to be distributed later. The committee will be very grateful for any suggestions or advice which will aid in the solution of the problem before us.

J. T. R. ANDREWS, *Chairman.*

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The stockholders of the Will & Baumer Candle Company, Syracuse, have voted approval to an increase in the company's authorized common stock 150,000 shares from 100,000 shares. It was stated that it has no intention to issue the additional shares now.

New Books

HYDROGENATION OF ORGANIC SUBSTANCES: By Carleton Ellis, 8vo, 1014 pp. New York, D. Van Nostrand Co., Inc., $15.00.

CARLETON ELLIS, whose previous work on the hydrogenation of oils has been for many years the standard reference book on the subject, has now completed the preparation of this new and enlarged edition which carries the subject into the fields of general organic chemicals and fuels, including coal and petroleum oils, while at the same time recording all the advances in the hydrogenation of fatty oils since the publication of the original volume. The method of treating the subject is all-inclusive, every process and type of equipment, as well as every patent and every published investigation being recorded.

The study of the subject of hydrogenation is developed in the text in a logical, progressive manner. Preliminary consideration is given to the theory and mechanism of catalysis, catalytic poisons, and the many varieties of catalysts, metallic and non-metallic, that have been proposed. The general applications of hydrogenation in organic chemistry, with particular reference to hydrogenation of carbocylic compounds, are next considered. Nuclear hydrogenations and those at nitrogen linkages are discussed fully, followed by a thorough and complete discussion of the hydrogenation of fatty oils, including a resumé of the developed uses for hydrogenated oils and fats, edible and technical.

The reader is led progressively through the hydrogenation of naphthalene, anthracene, phenanthrene and their derivatives to the hydrogenation of coals and related hydrocarbons and that of mineral oils. The reduction of carbon monoxide by hydrogenation methods and the synthesis of methanol are carefully reported and the final chapters of the book describe the various practical methods for the generation of hydrogen.

This is truly a monumental work, and all chemists who have any interest whatever in organic synthesis or in fatty oils, or in fuels of any kind, will want to include it in their libraries.

All producers and manufacturers of oils, soaps and allied products will also find Ellis' volume of incalculable value.