

posed over methyl alcohol for varying periods of time before immersion in oil, one can always reduce, even if not totally remove, the inevitable distortion due to the injection of considerable quantities of preservative, and be assured that the material comes out of the oil unchanged. Since carbolic acid when warmed, easily and thoroughly mixes with oil, it can be added if desired, but so far I have not observed the least disadvantage from the use of unmixed oil alone.

Material immersed in oil need drain only a few minutes before it can be wrapped or covered and used for dissection. The wrapping quickly takes up the slight amount of adhering oil, and by being impregnated with it, greatly slows the drying out of the material. Except for the slight odor of the oil, bodies so stored are practically odorless, and quite in contrast to those kept in watery solutions, leave practically no evidence of external contact even when handled with bare hands. After being thoroughly impregnated with oil the epidermis resists drying very much better, and the eyelids, nose, lips, digits, ears and genitalia do not require such careful protection during dissection. But above all else the untidiness and soiling, unavoidable especially when vaseline is used, are wholly obviated.

Since any wooden tank can be used as a container, no expensive equipment is required. A galvanized iron lining no doubt will last indefinitely, and cement tanks have not been found too pervious. Exposure to cold can cause no difficulty, and, if introduced accidentally, water can be drawn off easily. The cost of the oil is low, especially when its practical permanence is considered, and since it is not easily ignited until it reaches a temperature of 80° C., underwriters have raised no objections to its use. Lighted matches can be thrown into open tanks without causing an explosion of gases or igniting the oil. Indeed some heating of the latter when contained in an open vessel is necessary before explosion of the liberated gases occurs. Consequently, ordinary care is all that is required to avoid accident when in its presence.

The particular grade of oil which I have used for several years is known as mineral seal oil. It has a slight yellowish tinge, and a specific gravity of 0.85 at room temperature. It has only a slight odor, which is wholly inoffensive, and, in fact, negligible. Since it can be obtained in large quantities and does not need renewal, it is extremely economical, and since it is almost colorless, it can be used to advantage also for preserving smaller specimens and even for museum preparations. It indeed seems to be an unexcelled medium for the storage of anatomical material. That this estimate of it is justified seems to be indicated also by the experience of friends who are using it. It would seem to be particularly advantageous when it is necessary to store material for long periods of time because of an intermittent or insufficient supply, or when, for some reason, it is desired to repeat measurements or to make volumetric determinations, at a later date.

ARTHUR WILLIAM MYER

STANFORD UNIVERSITY

THE AMERICAN CHEMICAL SOCIETY. VI

RUBBER DIVISION

John B. Tuttle, *Chairman*

Arnold H. Smith, *Secretary*

Report of Executive Committee.

Report of Secretary.

Report of Fruit Jar Ring Committee. L. J. PLUMB, chairman.

Report of Committee on Physical Testing. H. E. SIMMONS, *chairman*.

A new method for the determination of sulfur in rubber mixtures: G. D. KRATZ, A. H. FLOWER AND COLE COOLIDGE. Transfer the finely divided samples (0.5 gm.) to an Erlenmeyer, and add 10 c.c. of ZnO + HNO₃ solution (200 gms. ZnO in 1 liter HNO₃). Then add 15 c.c. fuming HNO₃, whirling the flask until the sample is decomposed. When solution of the rubber is complete, add 5 c.c. Br-H₂O and evaporate mixture to a foamy syrup. Cool flask and add a few crystals of KClO₃. Evaporate the mixture to dryness and bake at the highest temperature of a Tirrell burner until all nitrogen compounds are eliminated. When baking is

complete, cool, take up in HCl (1:6), filter and precipitate sulfates in the usual way, especial care being observed in washing BaSO_4 free from chlorides. The method is accurate to 0.1 per cent.

The extraction of rubber goods: S. W. EPSTEIN AND B. L. GONYO. A report is made of experimental work on the rubber extraction of rubber goods, with tables showing results obtained. The observations and conclusions derived from this work were as follows: (1) Extraction for 8 hours with acetone followed by 4 hours extraction with chloroform does not remove all soluble material from some rubber compounds. (2) After a rubber sample has been extracted with acetone it was found: (a) that chloroform in every case extracted slightly more material than carbon bisulphide; (b) that constant boiling mixtures such as 55 per cent. carbon bisulphide—45 per cent. acetone and 68 per cent. chloroform—32 per cent. acetone extracted from many cheap compounds considerably more material than either chloroform or carbon bisulphide; (c) that usually about 0.1 per cent. of sulphur is present in the extract, whether it is obtained by the use of chloroform, carbon bisulphide, or the mixtures under consideration. (3) The constant boiling mixture of 68 per cent. chloroform and 32 per cent. acetone exhibits a marked ability to dissolve vulcanized rubber, as contrasted to the mixture of 35 per cent. carbon bisulphide 45 per cent. acetone which hardly exhibits this ability at all. (4) It is recommended that the constant boiling mixture 55 per cent. carbon bisulphide and 45 per cent. acetone be used in place of acetone and chloroform to extract rubber samples since: (A) It eliminates one extraction with the necessary weightings. (B) Extraction is complete in 8 hours while the acetone and chloroform extractions require a total of 12 hours. (C) The extraction of free sulphur is complete. (D) A rubber analysis in which the mixed solvent is used, is more accurate than that in which acetone and chloroform are used, because (I.) Little or no rubber is dissolved by this mixture, as compared to chloroform which will in some cases dissolve considerable quantities. (II.) The extraction of cheap rubber compounds is more complete, since the extracts obtained are greater than the sum of the acetone and chloroform extracts.

The theory of balloon fabric protection: JOHN B. TUTTLE.

The expansion of rubber compounds: C. W. SANDERSON. A new apparatus was designed to

measure the expansion of rubber compounds during vulcanization. Values of the coefficient were determined for different classes of compounds and found to be between 2.3×10^{-4} and 3.8×10^{-4} . A study of the relation between the expansion and the increase in specific gravity was made and the conclusion drawn that the increase in specific gravity during vulcanization is due to the pressure exerted on the rubber. Other experiments were made to determine the applicability of the measurements to commercial practise.

Volume increase of compounded rubber under strain: H. F. SCHIPPEL. The addition of pigment to rubber changes it from a substance which has constant volume under strain, to one which undergoes comparatively large volume increases. The amount of this increase depends upon three factors: (1) the extent of strain, (2) the volume proportion of pigment, (3) the average particle size. The larger sized pigments cause greater volume increases, with the notable exception of zinc oxide, which classifies itself under this test with the finer pigments. Prolonged mixing on the mill has only a slight reducing effect upon the volume increase. This general property of rubber compounds throws light upon their physical condition under strain.

The determination of cellulose in rubber goods: S. W. EPSTEIN AND R. L. MOORE. After a discussion of the value of a procedure for determining cellulose in rubber goods and consideration of the literature on the subject, the proposed method is discussed. Sample is digested with cresol at 160° – 185° C. for 4 hours to dissolve the rubber. Filtration is facilitated by addition of a 200 c.c. of petroleum ether. After washing with benzol, 10 per cent. solution of hydrochloric acid, water and acetone, the material is dried and weighed. It is then acetylated by heating for 30 minutes at 75° C. in a mixture of 15 c.c. of acetic anhydride and 0.5 c.c. of concentrated sulphuric acid. This is filtered on a weighted Gooch, washed with 90 per cent. acetic acid and then with acetone and dried and weighed. Loss in weight is recorded as cellulose. Under Results of Analysis are given a number of test compounds, containing varying amounts of cellulose in the presence of various combinations of compounding ingredients. The results by method given indicate its gratifying accuracy. A number of alkali reclaims are given along with cellulose content as obtained by method proposed. It is shown how the percentage of cotton can be

obtained in the presence of leather. Leather, wood, jute and cork are considered. These are broken down by cresol at 185° C. For this reason it is suggested that rubber be digested in cresol for 16 hours at 120° C. when the presence of these is suspected. At this temperature the basic substance of these materials is retained intact. Acetylation gives:

- 95 per cent. of the wood,
- 90 per cent. of the jute,
- 70 per cent. of the leather,
- 21 per cent. of the cork.

An approximation of the amount of cork present is obtained by treating the residue after acetylation with 2 per cent. solution of sodium hydroxide and strong bromine water, in order to remove the unacetylated cork. The loss in weight represents 70 per cent. of the total amount of cork present. It was found impossible to determine each of these ingredients separately, and therefore it was decided to determine them collectively by the acetylation method, and to test for the presence of each by means of proper stains and microscopic examination.

The variability of crude rubber: JOHN B. TUTTLE.

Symposium on the action of accelerators during vulcanization: J. H. SCOTT.

The action of certain organic accelerators in the vulcanization of rubber: G. D. KRATZ, A. H. FLOWER AND COLE COOLIDGE. The activity of the following substances in accelerating the vulcanization of a mixture of 92½ parts rubber and 7½ parts sulfur was investigated: aniline, urea, thio-urea, mono- and di-phenyl-thio-urea, mono-, di- and tri-phenyl-guanidine, the formaldehyde condensation products of aniline and p-phenylene-diamine, and other substances, including those which produce negative acceleration. Comparisons were made on the above mixture; sulfur coefficients are given. Certain substances were found to decompose into simpler substances containing an active nitrogen group which is responsible for the acceleration effected. Molecularly equivalent quantities of substances which contain the same active nitrogen group in their primary nucleus produced the same accelerating activity. Certain nitrogen groups probably function as sulfur carriers with a temporary change from three to five in the valency of the nitrogen.

Reactions of accelerators during vulcanization: C. W. BEDFORD AND WINFIELD SCOTT.

The effect of organic accelerators on the vulcanization coefficient: A brief discussion of the already published work on the relationship of the mechanical properties of vulcanized rubber to the chemical composition, and a description of experiments with certain powerful accelerators which demonstrates that under certain conditions good mechanical properties can be obtained in rubber with a very low degree of chemical combination of sulphur.

The effect of compounding ingredients on the physical properties of rubber: C. OLIN NORTH.

Some methods of testing the hardness of vulcanized rubber: H. P. GURNEY.

Symposium on the testing of pigments. Led by GEO. OENSLAGER. Contributions from M. M. Harrison and M. M. Kahn.

The manufacture and use of crimson antimony: J. M. BIERER.

Laboratory aprons: C. P. FOX.

The value of a library to the rubber laboratory: H. E. SIMMONS.

Research on zinc products for the rubber industry: P. R. CROLL AND I. R. RUBY.

DYE SECTION

Charles L. Reese, *Chairman*

R. Norris Shreve, *Secretary*

Introductory remarks: CHARLES L. REESE.

Present condition of German dyestuff plants: T. W. SILL.

Review of the dye situation: J. MERRITT MATTHEWS.

The progress of the American dye industry as shown by the census of the Tariff Commission: GRINNELL JONES.

Photosensitizing Dyes: E. Q. ADAMS.

The color laboratory of the Bureau of Chemistry: H. D. GIBBS.

Alkali fusions: H. D. GIBBS AND MAX PHILLIPS.

The system: naphthalene-phthalic anhydride: K. P. MONROE.

The melting point of pure phthalic-anhydride. The system: phthalic anhydride-phthalic acid: K. P. MONROE.

Benzene sulphonic acids. (I.), benzene disulphonic acid from benzene mono sulphonic acid: C. E. SEMAN.

Notes on testing dyed goods: W. F. EDWARDS. During the war period established prejudice made

a condition that favored placing the blame for defects in dyed goods on the dyer and the American Dye Manufacturers. It has been an easy way for uninformed persons dealing in dyed finished goods to avoid responsibility for defects in the goods which they handle. It is necessary to show these dealers in dyed goods that there are ways of practical testing within their reach that are comparatively safe in showing the color quality of the goods they handle. This would be also great value to the dyer and dye manufacturer as the tests could be made by disinterested laboratories. These tests should be simple and reliable and as fast as possible standardized. The textile testing department of the United States Conditioning & Testing Co. has been making some investigations along this line and have done enough to show that it is quite practical to place the color quality of dyed finished goods on as sound a basis of specification as now obtains for steel and other alloys. In the case of steel the specifications are in terms of chemical analysis, micro-structure and physical tests. In the case of dyed finished textiles the specifications for color quality are in terms of fastness in some form or other and hue, saturation and brilliancy and are determined by empirical tests under controlled conditions. These are details of standardization of these empirical tests that are important to consider from the point of view of the manufacturer of dyes, the dyer and the dealer in dyes and dyed finished goods. Team work by all will lead to results that will insure the future of the American dye industry.

The quality of American dyestuffs: R. S. LUNT. After a review of pre-war dyestuff conditions the author spoke of the early attempts to produce intermediates and dyestuffs in America. The first dyestuffs produced for large tonnage though few in variety. Indigo, only commenced in 1917, now supplies the entire demand. The study and production of alizarines and vat dyes required time but these colors are now almost ready to put on the market. Azo colors, forming the bulk of the production, form a varied line. The Chemical Foundation has provided access to German owned American patents. Present dyestuff industry now comprises 215 concerns employing 26,000 hands, of whom 2,300 are chemists. The great need of the dyestuff chemist is pure standardized intermediates, 141 are now being made. Most manufacturing problems are due to lack of experience. Standardization must rest on a basis of commercial

products rather than on chemical purity. The present productions are satisfactory but there is a need of press cooperation and favorable legislation. The American chemists are now in control of the situation.

Application of dyes: E. W. PIERCE. The author first shows the earliest methods of coloring fabrics by printing and dyeing, tracing the progress of the art to the present high state of development. It is shown that dyes must be more than mere colors; they must have definite characteristics and be practical in their applications. Many possible dyestuffs are little used for lack of proper methods of application. The uses and possibilities of any dye determine its value more than any other factor. The importance of technical service is shown in its relations to both the dye maker and the consumer, also the future development of the industry, dependent on the verdict of the technical department, which will determine which new dyes shall be introduced and which ones are not capable of practical development.

Foreign dye patents: ROBERT E. ROSE. The difficulty of ascertaining the method of making dyes, even those covered by patents, is much greater than is appreciated, a fact which has never been given publicity. The paper explains the reason for this and shows that overcoming this difficulty is one of the most important tasks before the dye chemists of the country.

Some stones in the foundation of a great national industry: THOMAS H. NORTON. The effort to build up an American coal-tar dyestuff industry, fully equipped to meet the rivalry of the German industry, is outlined. The chief factor in favor of the latter is its enormous capital of accumulated experience and perfect organization. Details are given of the principles and methods employed by the du Pont Company in seeking to establish color works on American soil, equal in extent and variety of product, to any of the giant works on the Rhine. Emphasis is laid upon the work of the "Intelligence Division" which furnishes prompt, accurate and full information on any subject arising, to the numerous divisions engaged in operation or research.

The explosibility and inflammability of dyes: BURR HUMISTON, W. S. CALCOTT AND E. C. LATHROP. A presentation of the factors which cause decomposition of dyes during drying grinding and mixing with special emphasis on explosion and fire risk. Decomposition is due to temperature effects, especially dangerous in exo-

thermic decompositions. The laboratory methods used are designed to deal with decompositions in both solid and dust air phases. Preliminary results are promising finding application in plant practise, insuring more uniform quality in the dyes produced.

Some problems in the identification of dyes: E. F. HITCH AND I. E. KNAPP. It is pointed out that before the American dyestuff manufacturers can develop new colors, they must be able to duplicate the staple foreign dyes, especially the more recent ones, and those which are unclassified. In order to do this it will be necessary to identify these dyes, and in many instances to determine their chemical constitution. The first class of problems that are likely to be met includes (1) the identification of two or more dyestuffs, the composition of one of which is known; (2) the determination of the chemical constitution of an unknown dyestuff; and (3) the separation and identification of the component of a mixture of dyestuffs. The problems in class two deal with the identification of dyestuffs on the fiber. The methods which have been proposed for the solution of these problems are reviewed. In conclusion, a plea is made for closer cooperation between the universities and the dyestuff industry. Several ways are shown in which such cooperation might be effected.

Indicators and their industrial application: H. A. LUBS. The most recent and useful developments in the field of indicators are largely due to need for a series of brilliant and sensitive compounds for the colorimetric determination of H^+ ion concentration of biological fluids. This necessity has given rise to the study of the ranges, and of the salt, protein and other errors of a large number of compounds, as well as to the synthesis of new indicators. The sulfophthalein series of indicators are brilliant compounds and cover a wide range of H^+ ion concentration. These compounds are superior in a number of respects to indicators in general use at the present time and their application in a number of industrial operations would be highly advantageous. The lack of reliability in the case of test papers of litmus and phenolphthalein is pointed out and the use of sulfophthaleins is suggested. Examples of certain procedures in the preparation of dyes and intermediates in which indicators can be of assistance are given.

Vat dyes: M. L. CROSSLEY.

Gentian violet and its selective bactericidal action: M. L. CROSSLEY.

The importance of intensive and original research in the development of the dye industry in America: M. L. CROSSLEY.

Logwood in its relation to the silk industry: EMIL LESSER AND DAVID WALLACE.

Some engineering aspects in the manufacture of dyes: CLARENCE K. SIMON.

Observations on the estimation of the strength of dyes: W. H. WATKINS.

Application of physical chemistry research on dyes: E. K. STRACHAN.

Crystallographic identification of five isocyanines: EDGAR T. WHERRY. Five isomeric or closely related isocyanine dyes have been prepared in the Color Investigation Laboratory of the Bureau of Chemistry by Dr. E. Q. Adams, and crystallized from alcohol. The crystals prove to show brilliant color phenomena, and especially the rare effect known as reflection-pleochroism, the reflection of light of different colors in different crystallographic directions. Models of these crystals have been prepared (and were exhibited at the meeting). It is ordinarily not practicable for any one not specifically trained in crystallography to carry out measurements of interfacial angles of random crystals, because it is a matter of great difficulty to orient given crystals correctly. The fact that the crystals of these dyes have definite colors associated with definite crystallographic directions makes such orientation comparatively easy, and which dye is represented in a given sample can be rapidly and certainly ascertained by a few simple observations of angles, far more readily than by any known chemical method.

The dye situation in the United States and England: T. FRUSHER. CHARLES L. PARSONS, Secretary

(To be continued)

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