use the plus (+) sign to denote degree of acidity although he does use the minus (—) sign for alkalinity. In a table which shows the relationship of different degrees of reaction to the number of bacteria developed (p. 393) he presents under "reaction" the following figures: 40, 35, 30, 25, 20, 15, 10, 5, 0, —5, —10, —15, —20, —25, adding, "Numbers refer to cubic centimeters per liter of normal acid or alkali necessary to change it to phenolphthalein neutral point. Minus (—) means an alkaline solution." The plus (+) sign was apparently not used by Fuller, the figure itself without any sign standing for acidity. While the writer has not definitely ascertained when and by whom the plus (+) sign was first used, it is probable that it was first brought into general use by the *Report of the Committee of Bacteriologists of the American Public Health Association* (2) in 1898. This committee, of which Fuller was a member, made the following recommendation (2, p. 75): "Manner of expressing the degree of reaction of culture media: Since at the time the reaction is first determined culture media are more often acid than alkaline, it is proposed that acid media be designated by the plus sign and alkaline media by the minus sign, *and that the degree of acidity or alkalinity be noted in parts per hundred*" (writer's italics). "The bulk of available evidence from both Europe and America points to a reaction of +1.5 as the optimum degree of reaction for bacterial development in inoculated culture media" (p. 76).

It is quite evident that animal pathologists and bacteriologists in general have substituted for the methods proposed by Fuller those proposed by the Committee of the American Public Health Association of 1898, although they usually cite or designate Fuller's scale, while plant pathologists use Fuller's original recommendations with the exception of adding the plus (+) sign to indicate acidity.

Since +10.0, +15.0 in 1,000 c. c. of the medium correspond to +1.0, +1.5 respectively in 100 c. c. of the medium it is evident that the degree of acidity recommended for plant pathogens corresponds to the acidity recommended for bacteria in general, and it is necessary to know the author's interpretation of Fuller's scale when considering the degree of acidity described or recommended.

H. R. ROSEN
AGRICULTURAL EXPERIMENT STATION
UNIVERSITY OF ARKANSAS

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(2) *Report of a Committee of Bacteriologists to the Committee of the American Public Health Association on the Pollution of Water Supplies.* Jour. Amer. Pub. Health Asso., 23: 56-100. 1898.
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(4) SMITH, ERWIN F. *Bacteria in relation to plant diseases.* Vol. 1. Published by the Carnegie Institution of Washington. 1905.
(5) SMITH, ERWIN F. *An introduction to bacterial diseases of plants.* W. B. Saunders Company, publishers, Philadelphia, 1920.

THE AMERICAN CHEMICAL SOCIETY

(*Continued*)

DIVISION OF RUBBER CHEMISTRY

W. W. Evans, *chairman*
Arnold H. Smith, *secretary*.

Report of committees, executive, physical testing, abstract, chemical analysis and accelerator.

Mineral rubber: C. O. NORTH. The purpose of this paper is to bring out the desirable and undesirable properties of M. R. in order that M. R. makers will appreciate more fully how their product is employed. Changes in stress strain relations, hysteresis losses, permanent set, energy of resilience and abrasion with increase in M. R. ratio to rubber are shown. M. R. is essentially a plastic material. When a stock containing it is stretched, the M. R. flows with the rubber. On release the M. R. flows back with the rubber. The principal evidence of its presence is a slowing up or logging of the return.

The Tetra-hyroxyphenyl derivative of rubber and its tetra-methyl ether. HARRY L. FISHER AND HAROLD GRAY. The tetra-phenoxy derivative of rubber described by Weber (ber. 33, 791) is shown to be the tetra-hydroxyphenyl derivative not only by the method of formation, and by its solubility in aqueous NaOH, but especially by the fact that it can very readily be methylated to the corresponding tetra-methoxyphenyl derivative. The reaction for preparing the hydroxyphenyl compound from rubber tetrabromide and phenol is like a Friedel-Crafts' reaction, being speeded up by the use of anhydrous aluminum chloride, zinc chloride, etc. Other halogen derivatives of rubber such as the dihydrochloride also react with phenol.

Microscopy of rubber fillers. IRENE C. DINER. The principal rubber fillers, namely, barytes, whiting, zinc oxide, lithopone, litharge, red lead, antimony sulphide, iron oxide and gas black, are included in a large chart showing the characteristics which differentiate these microscopically. Among the properties shown are size, color, shape, crystal structure, impurities and optical properties. A basis of a positive method of identification of the fillers is indicated, based upon 17 different measurable physical properties. It is hoped that this method will in time supercede the present lengthy and cumbersome qualitative and quantitative analysis. Besides being shorter it is more exact since it gives the exact state in which the filler is present rather than merely the amount of each element with a good guess as to their association, for example, whether the substance be present pure or impure, hydrated or anhydrous, etc.

The use of the microscope and photomicrographs in the study of inorganic materials used in rubber. BENTON DALES AND W. W. EVANS.

Recent developments in the art of rubber microsectioning. HENRY GREEN. A method for making microsections of rubber has been developed which is free from the various inconveniences associated with the method heretofore employed of freezing the sample with CO_2 and liquid air. The elasticity is destroyed and sufficient rigidity acquired by treating the sample with dilute SC_1 solution. Sections are obtained ½ mm x 4 mm in area, which under a magnification of 1,500 diameters appears, in round numbers, to be a strip 3 ft. wide and 20 ft. long. This is sufficient to show all the characteristics of the sample. The sections can be made exceedingly thin, beautifully transparent and of uniform thickness.

Piperidine-piperidyl-dithiocarbamate as a rubber accelerator in the presence of zinc oxide: G. S. WHITBY AND A. H. SMITH. One part of piperidine-piperidyl-dithiocarbamate in a 90 rubber 10 S, 10 zinc oxide mix cuts the time of vulcanization at 141 degrees C from three hours to less than one minute. At 131 degrees C it cuts the time of vulcanization from seven hours to one minute. It is fully vulcanized in three to four minutes at 115 degrees. When only two parts of S are used the time of vulcanization is one minute at 141 degrees, or two minutes at 131 degrees, or ten minutes at 115 degrees. Stress-strain data are given.

An improved oven for accelerated aging of rubber. C. W. SANDERSON.

General discussion. *Accelerated aging tests.* Led by W. W. Evans, review of article, *Ten years experience with accelerated aging tests,* by W. C. Geer and W. W. Evans.

An apparatus and method for abrasion tests on rubber compounds: J. C. SPROUL AND W. W. EVANS.

The determination of true free sulfur and true coefficient of vulcanization in vulcanized rubber: W. J. KELLY. The application of the method devised for pure gum stocks (J. Ind. Eng., Ch. 12: 875, 1920) to compounded stocks. Free sulfur determined by the 'satured alcohol'' method. For combined sulfur the sample is extracted with alc. KOH and ether HCl, the latter removing any sulfide as H_2S and dissolving accelerators which form water on ether soluble hydrochlorides. In this way considerable sulfur is removed in addition to that extracted by acetone and which had previously been considered as combined with the rubber. In some cases an additional extraction with water is necessary, but the details of this remain to be worked out.

Corrected stress-strain curves for rubber: J. W. SHIELDS. An improved method is explained for drawing stress-strain curves for rubber which takes into account the decrease in cross section of the specimen. Such curves do not have the S shape which is characteristic of uncorrected curves. The corrected curves show the modulus of rubber to be least at the smallest elongation and to increase gradually as the specimen is stretched. The method for determining the true modulus of unstressed rubber is illustrated. The equation for this curve is developed and the values of the equation constants given for several stocks.

The determination of the particle size of pigments. W. W. VOGT. By a turbidimetric method it is possible to determine the capacity of a pigment to extinguish direct light rays. This capacity, called obscuring power, is a direct function of the average particle size of the pigment. The values of O. P. are shown to be consistent with particle size as determined microscopically and furthermore to be in close accord with the practical compounding value of the pigment. Hence, by the determination of the O. P. of an unknown pigment it is possible to predict what its practical compounding value will be in relation to known pigments.

The solubility of sulfur in rubber: C. S. VENABLE AND C. D. GREEN. Values have been obtained for the solubility of sulfur in rubber at 55°, 75°, 95° C. The procedure used was essentially to pack thin rubber strips in sulfur, maintain at the desired temperature until equilibrium was reached, and then analyze for combined and free sulfur. Various precautions were adopted to insure that equilibrium was reached. It was found that the solubility of sulfur in rubber increases slowly with the vulcanization coefficient, this increase being more rapid as the temperature increases. It was found that when this coefficient was greater than 7 per cent. combined sulfur, it was impossible to obtain solubility values by the method used due to the fact that the rubber becomes almost impermeable to free sulfur. This impermeability probably has more to do with the non-blooming of hard rubber stocks than the increased solubility of free sulfur. By extrapolating the curves obtained, it can be calculated that a stock having 4 per cent. combined sulfur at 140° C will be saturated with about 15 per cent. free sulfur.

Reactions of accelerators during vulcanization. IV. Mechanism of the action of zinc compounds: C. W. BEDFORD AND L. B. SEBRELL. Zinc sulphydrate vulcanizes rubber in the presence of sulfur at ordinary temperatures. Zinc persulfides are stable compounds and vulcanize rubber in heat cures far more rapidly than ordinary sulfur. Zinc salts of organic accelerators form polysulfides without decomposition into zinc sulfide and disulfides. Disulfides are changed to mercaptans by hydrogen sulfide and in the presence of zinc oxide they form zinc salts. (An answer to Bruni, *India Rubber J.*, 62 [1921] 63.)

The action of volatile organic solvents and vulcanizing agents on organic compounding materials and resinous gums: FREDERICK DANNERTH. The purpose of this investigation has been to obtain fundamental data for the industries which use plastic masses. The amount of matter soluble in certain liquids at stated temperatures has been studied. We have also noted: The amount of a given solvent which will mix with any other given solvent at a given temperature; the swelling action of certain solvents on stated organic materials; the action of certain vulcanizing agents on pitches and oils as well as their action on chicle, balata, guttapercha and jelutong. The influence of the product has been studied, first using only organic compounding material and secondly using only the resinous gums.

The preparation and testing of crude rubber: O. DE VRIES.

SECTION OF CELLULOSE CHEMISTRY

Harold Hibbert, *chairman.*
G. J. Esselen, Jr., *secretary.*

Acetolysis of spruce pulp. Preliminary communication: WALTER RUSSELL AND LOUIS E. WISE. Acetolysis of spruce sulfite pulp, when carried out as in the case of acetolysis of cotton, yields appreciable amounts of cellobiose octaacetate. The yield of cellobiose appears to depend on the normal (Alpha) cellulose content of the pulp, rather than on the so-called "total cellulose." The cellobiose reaction furnishes another link in the chain of evidence that the normal cellulose of spruce wood is chemically similar, if not identical with that of cotton.

Studies on cellulose chemistry. III. The constitution of cellulose: HAROLD HIBBERT. The recent work of Karrer and of Freudenberg on octacetylcellobiose confirms the view advanced previously by the writer that the ratio of this product to dextrose pentacetate obtained from the decomposition of cellulose acetate is much higher than corresponds to the pentaglucosidylglucose formula for cellulose put forward by Hess. The results cast considerable doubt on the correctness of his formula, but, on the other hand, are in no way in disagreement with that advocated by the writer. Further evidence in support of these is to be found in the recent paper of Denham on the methylation of cellulose.

IV. The action of HBr on carbohydrates and polysaccharides: HAROLD HIBBERT AND HAROLD S. HILL. Cellobiose gives a yield of 27 per cent. of bromomethylfurfuraldehyde while lactose gives less than 7.0. These two derivatives are structurally identical and there is thus the possibility of utilizing this reaction for the determination of configuration. The mechanism of the reaction is being carefully studied in view of its bearing on the constitution of cellulose.

The condensation of citral, with certain ketones and the synthesis of some new ionones: HAROLD HIBBERT AND LAURA G. CANNON. The best method for purifying citral is the one developed by Tiemann. Of the condensing agents hitherto employed, sodium ethylate is the most satisfactory, but metallic sodium is equally efficient. Better yields of a purer product have been obtained. The bisulfite method of purification is capable of general application in the purification of pseudo-ionones, giving yields of about 85 per cent. and chemically pure products. New ionones have been synthesised from methyl propyl ketone and acetophenone.

The rôle of celluloses in plant life: R. W. THATCHER. Celluloses are classified according to their chemical composition into three groups: the hemi-celluloses or pseudo-celluloses; the normal celluloses; and the compound celluloses. Hemi-celluloses are amorphous polysaccharides which are probably reserve carbohydrates deposited in the structural, or cell-wall, materials rather than in storage organs. Normal celluloses are amorphous forms of polysaccharides having an empirical formula similar to that of starch; but exhibiting a characteristic fibrous structure instead of the granular structure characteristic of starches. They are true cell-wall, structural material; they can be hydrolyzed by certain bacteria but probably have no nutritive function in higher plants. Compound celluloses are either colloidal complexes or definite chemical compounds of true cellulose with some encrusting material which serves to stiffen and harden the celullar structure and convert it into ''wood.'' They are among the most inert plant compounds and probably have no role other than that of adding strength and stiffness to the stems or other tissues of plants.

The determination of the ''bromine figure'' or ''chlorine factor'' of pulp and the utilization of these quantities in bleaching: ALFRED TINGLE. The extent to which bromine solutions, approximately decinormal, act on cellulose and on unbleached sulphite spruce pulp, has been investigated. Accurate measurements could only be made when the material was brought into solution before treatment with bromine. Under the experimental conditions used, it was found that bromine did not act on cellulose to any appreciable extent in an hour, but that it acted on unbleached sulphite pulp in stages, one of which was completed in 30 minutes. From measurements of this action a quantity was found, constant for each sample of pulp, which bears a definite and simple relation to the chlorine-consumption in bleaching the pulp.

The alleged absorption of aluminum from solutions of aluminum sulphate by cellulose: ALFRED TINGLE. Both neutral and basic solutions of aluminum sulphate were brought in contact under various conditions with cellulose in the form of (*a*) acid-washed filter paper, (*b*) bleached sulphite spruce pulp. By the methods employed, no change in the aluminum content of the solutions could be detected, except when a pulp was used which contained calcium compounds and gave a strongly alkaline reaction to water, with which it was extracted. The deduction is made that absorption does not occur to any appreciable extent, and that the phenomena which have been accounted for by this cause are due to other causes.

The distillation of methoxyl groups in wood distillation products: L. F. HAWLEY AND SUBRAMANYA AIYAR. It has been reported that the treatment of wood with sodium carbonate previous to distillation increases the yield of methyl alcohol. The source of this increase has now been determined. Maple wood containing 6.09 per cent. methoxyl when distilled gave products with percentages of methoxyl as shown in column 1 of the table. On distilling the wood after treatment with 1 per cent. sodium carbonate the methoxyl distribution is as shown in column 2 of the table and the increase of methyl alcohol is seen to come partly from the dissolved tar and partly from the charcoal. The proportion of total methoxyl in the original wood recovered in the distillation product remains about the same.

PERCENTAGE OF METHOXYL BASED ON THE WEIGHT OF THE WOOD DISTILLED

	Original wood.	Wood treated with Na_2CO_3
Charcoal	0.285	0.044
Settled tar	0.517	0.588
Dissolved tar	0.303	0.173
Pyroligneous acid	1.617	1.953
Gas (methane)	1.306	1.468
Total	4.028	4.226

Acetic ether as a solvent for nitro cellulose and cellulose acetate: H. F. WILKIE. In discussing acetic ether as a solvent for nitro cellulose and cellulose acetate a review is made of the subject of cellulose esters solvents in general. The production of high grade anhydrous ethyl acetates or acetic ether as the pure product is designated and results obtained in experimenting with it as a solvent of cellulose esters point to the strong possibility of it taking the place of amyl acetate. It can be procured in large quantities and the ultimate supply is unlimited. Data is given to support the following claims: It is neutral in reaction and remains so on long exposure to air, light or moisture, and is non-hydroscopic. Acetic ether is a powerful solvent of nitro-cellulose and a good solvent for cellulose acetate. It works well in combination with the other well known solvents and non-solvents, and is a pure substance which evaporates without residue and at a uniform rate, giving rise thereby to homogeneous films. By a very important property it possesses of forming various constant boiling mixtures (especially one with water) more volatile than itself it combines with the advantages of its low boiling point the ability to act as the high boiling solvents in overcoming the evil effects of water absorption.

Requirements of a brief, critical monograph on the chemistry of cellulose: LOUIS E. WISE. The article outlines the type of material and data that should be incorporated into a monograph on cellulose. Many of the older terms that still appear in the cellulose literature should be deleted or redefined. Proper weight should be given to carefully selected fundamental data and to the work of critical investigators. All hypotheses dealing with the constitution of cellulose or its derivatives should be subjected to close scrutiny, and those based on good experimental data should receive emphasis. Recent work on the colloidal properties of cellulose should be included. The monograph besides being critical should be suggestively written so as to stimulate research. A brief volume with a good bibliography appears to be more desirable than a series of monographs.

SECTION OF PETROLEUM CHEMISTRY

T. G. Delbridge, *chairman.*
W. A. Gruse, *secretary.*

An unusual type of casinghead gasoline: C. E. COATES AND B. Y. TIMS. The Terrebonne gas field, which is situated about twenty miles below Houma, Louisiana, gives gas and a pressure of about 1,200 pounds per square inch. This gas is piped to Houma and supplies the town and vicinity with fuel. The pipes are provided with drips which fill up rather frequently and are pumped out. As the condensate did not seem to be like ordinary gasoline either in odor or boiling point a sample was investigated with the following results: Boiling began at 195° C. The fractions first obtained were refractionated and eventually obtained with fairly constant boiling points. The lowest fraction had a formula $C_{12}H_{22}$. The higher fraction seemed to belong to the same series. The condensate contained no members of the parafine series at all, but probably is made up of dicyclopentyl and its homologues.

Some observations on the polymerization of amylene: THOS. MIDGLEY, JR., AND G. W. HANK. The action of dilute and concentrated sulfuric acid and of heat on the polymerization of amylene has been studied in some detail, and a chart of polymerisation reactions is presented. It is suggested that di- and tri-amylene have cyclic structures. The subject is of interest in connection with the gumming of cracked gasolines.

The iodine and bromine values of petroleum products: E. M. JOHANSEN. A number of petroleum products were examined, the results being recorded in numerous tables. It was shown that the total iodine or bromine values do not correspond to the relative unsaturation of petroleum products, as only a part of the absorbed halogen is combined by addition. As the Hanus solution does not permit the separate determination of this part, solutions of bromine or bromine and iodine in carbon tetrachloride were used, by aid of which the desired addition values were obtained. The influence of varying experimental conditions upon the reactions were studied. It appears that the absorption of bromine is less affected by the variations than that of iodine. A bibliography of the literature on the subject was compiled.

Specific heats and heats of vaporization of motor fuels: ROBERT E. WILSON AND D. P. BARNARD, 4TH. The authors present the results of a series of observations on the total sensible heats of completely vaporized motor fuels. These, combined with critically compiled data from the literature on heats of vaporization of motor fuels, make it possible to draw accurate total heat curves over the whole range of temperatures up to 500° C. and derive fairly accurate values for the specific heats of the hydrocarbon vapors. Combinations of this with vapor pressure data make it possible to determine just how hot the air or the fuel must be preheated in order to completely vaporize the motor fuel in a carburetor.

Further observations on the value of the R. E. test: C. H. OSMOND AND T. G. DELBRIDGE. Data are given showing that the R. E. test distinguishes between some oils which give the same results by other emulsification tests. Criticisms of the method as published in the A. S. T. M. Proceedings for 1920 have led to minor changes in the method and particularly in the interpretation of the readings. Further precautions are given, also explanations of some of the procedure. Modification of the test for application to oils of very low viscosity is also given.

The inversion of phases in oil-water emulsions: LEON W. PARSONS. A broad survey has been made of the literature regarding emulsions and a brief discussion is given of the main factors influencing the properties and general behavior of mineral oil-water emulsions. An experimental investigation of these emulsions, with special reference to such as are likely to occur in practice, and to methods of breaking them, has been made. The phenomenon of "inversion of phases" has been studied and data regarding the influence of the following factors on the nature and stability of various emulsions has been obtained: (*a*) Nature of oil used; (*b*) Nature of emulsifying agent; (*c*) Type of emulsion obtained; (*d*) Inversion point and its relation to stability of emulsions; (*e*) Industrial applications of above results to technical emulsions.

Emulsions with finely divided solids: T. R. BRIGGS. The chief function of an emulsifying agent is the formation of a viscous film at the dineric interface. Lowering of the surface tension is not essential and presumably plays no part when finely divided solids serve as emulsifying agents. Oil in water and water in oil emulsions may be produced with finely divided solids; in the first type the solid must be wetted more strongly by water than by oil, but in the second type it must be wetted more strongly by oil. Certain factors influencing the relative wetting are considered. It is also shown that solids tending to form the first type of emulsion exert an "antagonistic" effect on solids tending to form the second type of emulsion, the effect being entirely analogous to Clowe's antagonistic action between soaps of sodium and calcium.

Emulsifying agents in oil field emulsions: J. L. SHERRICK. The presence of some third component, usually a colloid, to serve as an emulsifying agent is recognized by most workers as a necessary condition for stable emulsion formation. Experimental results indicate that earthy matter, carrying adsorbed asphalt, asphaltenes, etc., and present in the oil as a hydrophobe colloid, functions as the emulsifying agent in oil field emulsions. This conclusion is in line with Richardson's work on Trinidad asphalt (*J. Phys. Chem.*, 19, 245-6. 1915). Addition of certain organic solvents decreases the adsorption of asphalt on the earthy material and renders these emulsions unstable. As the adsorbed asphalt is removed the earthy matter ceases to be a hydrophobe colloid such as is necessary for the stabilization of a water-in-oil emulsion.

A short discussion on the formation of crude oil and water emulsions: A. W. MCCOY.

Common Characteristics of crude petroleum emulsions: E. E. AYRES, JR. This paper deals with crude petroleum emulsions as they occur industrially. It is pointed out that these emulsions are comparatively uniform in properties. The subsidence produced by gravity and by centrifugal force is discussed and the two methods compared for crude petroleum emulsions. The coalescence produced by contact under the influence of gravity and high centrifugal force is in the same degree. Three methods for producing coalescence are discussed—heat, filtration and colloid reactions. Heat is often incorrectly applied. Properly used, heat will greatly assist resolution. The filtration method described is based on the principle of contact between water globules and a medium more readily wetted by water than by oil. The method of "colloid reactions" is based on the opposition of hydrophile to the hydrophobe colloid. One may be neutralized by the other so as to yield a zero stability. The formation of reagents to accomplish such reactions is discussed.

Recovering petroleum from emulsions by chemical treatment: RALPH R. MATTHEWS AND PHILIP A. CROSBY. Satisfactory results are shown which were obtained by treating tank bottoms (emulsified petroleum) with a chemical known as Tret-O-Lite, which consists principally of sodium oleate. An aqueous solution when thoroughly mixed with the emulsion at 100-120° F. causes it to separate and results in the recovery of pipe line oil. A later development in the work has resulted in a liquid compound produced by sulphonating oleic acid and then making alkaline with caustic soda. This seems more effective, and methods and results of commercial applications to crude oil emulsions are shown. Experimental work is now going on relative to producing and using an oil soluble compound which will be more effective than the above and more easily handled.

Oil field practice in handling crude oil emulsions: SIDNEY BORN. The latest developments in handling crude oil emulsions are described in some detail. Five different methods are discussed: The steaming plant, chemical treatment, the Cottrell process, the centrifugal method and the topping plant. The types of plants and process most suitable for different fields and conditions will be of interest to the practical operator. A new method developed by the writer is described for handling large amounts of B. S.

CHARLES L. PARSONS,
Secretary