

## THE AMERICAN CHEMICAL SOCIETY

DIVISION OF ORGANIC CHEMISTRY

(Continued)

*Symmetrical tribromophenylpropionic acid and its reaction with acetic anhydride:* ROBERT CHAMBERS. Phenylpropionic acid condenses with acetic anhydride to form a phenyl-naphthalene dicarboxylic anhydride. This reaction holds for derivatives of phenylpropionic acid where there is a free ortho hydrogen. It was desired to investigate the action of acetic anhydride on a di-ortho substituted phenyl propionic acid. The above acid was prepared from meta nitro cinnamic acid as follows: reduction with zinc and hydrochloric acid gave meta amido hydrocinnamic acid. Bromination and subsequent diazotization in boiling ethyl alcohol gave 2,4,6 tribromohydrocinnamic acid. Heating to 145° in a sealed tube with bromine gave *aa* dibrom 2,4,6 tribromohydrocinnamic acid. The latter with hot alcoholic potash gave 2,4,6 tribromophenylpropionic acid. With acetic anhydride the above acid does not condense to a phenyl-naphthalene derivatives but forms an anhydride which may be hydrolyzed to the original acid.

*The reactions of alpha anthroquinonesulfonic acids with mercaptans:* E. EMMET REID, COLIN M. MACKALL and G. E. MILLER. Sodium anthroquinone-alpha-monosulfonate and the 1.5 or 1.8 disulfonates react readily with mercaptans in water solution to replace the sulfonic acid group by —SR to give anthraquinone alkyl thio-ethers or dithio-ethers,  $\alpha$ -C<sub>14</sub>H<sub>9</sub>O<sub>2</sub>.SR, 1, 5-C<sub>14</sub>H<sub>9</sub>O<sub>2</sub>(SR)<sub>2</sub> and 1,8-C<sub>14</sub>H<sub>9</sub>O<sub>2</sub>(SR)<sub>2</sub>. The disulfonates may give the intermediate alkyl thio-ether sulfonate.

*The polymers of pinene:* G. B. and C. J. FRANKFORTER and E. R. KRYGER.

*Contribution to our knowledge of the chemistry of calcium carbide:* G. B. FRANKFORTER and A. E. STOPPEL.

*A new lactone from oil of orange:* FRANCIS D. DODGE. Essential oils of citrus species, obtained by expression, on standing generally deposit solids from which certain lactones derived from coumarin have been obtained. In the present communication is described a new lactone of rather unusual properties obtained from the sediment of West Indian oil of orange. It forms colorless needles (m. p. 88–90°) easily soluble in alcohol and ether, slightly so in ligroin. Optical rotation is about —38° in alcohol. On acidifying an alkali-

line solution it yields a crystalline acid (m. p. 151°) from which no crystalline salts could be obtained. It yields no acetyl derivative, and can not be reconverted into the lactone. It is readily oxidized by permanganate. The lactone, like coumarin, yields a crystalline compound with bisulfite. Analysis indicates the empirical formula: C<sub>16</sub>H<sub>18</sub>O<sub>3</sub>.

*The bromination of 2-amino-p-cymene:* ALVIN S. WHEELER and IRA W. SMITHEY. Pure p-cymene, obtained from spruce turpentine, was nitrated and the 2-nitro-p-cymene was reduced with Sn and HCl. The acetyl derivative of 2-amino-p-cymene in CCl<sub>4</sub> solution was boiled with bromine. Bromo derivative, needles, m. 122°; yield 60 per cent. Hydrolysis gave free amine, liquid, b. 169°–170° at 20 mm.,  $d_4^{25}$  1.3012,  $n_D^{20}$  1.5781. HCl salt, plates, m. 206°–210°. HBr salt, plates, m. 205°. Diazobromoaminocymene, canary yellow needles, m. 146°–148° (decomp.). Oxidation of bromo-acetylaminocymene with neutral permanganate gave a toluic acid derivative, m. 213°. Hydrolysis with acid gave the bromoamino acid, needles, m. 151°; HCl salt, plates, m. 190° (decomp.). No bromoamino toluic acid of this description could be found in the literature. The Br atom appears to be in the 3 position.

*New derivatives of 2, 3, 8-tribromo-5-hydroxy-1, 4-naphthoquinone:* ALVIN S. WHEELER and T. M. ANDREWS. Action of NaOH on the tribromoquinone (A) gave the 2, 3-dibromo-5, 8-dihydroxy-1, 4-naphthoquinone (B), which, reduced with Zn and H<sub>2</sub>SO<sub>4</sub>, gave 2, 3-dibromo-1, 4, 5, 8-tetrahydroxynaphthalene, greenish needles, m. 164°–166°. Tetracetyl derivative, yellow needles, m. 149°–150°. Acetyl derivative of B, yellow prisms, m. 197°. Methyl ether of A, yellowish red plates, m. 209°–210°. Ethyl ether of A, yellow needles, m. 134°–136°. Aniline derivative of A (Br No. 8 replaced), purplish chip-like crystals, m. 235°. A is converted by Zn and H<sub>2</sub>SO<sub>4</sub> into the trihydroxy derivative, yellowish needles, m. 106°–107°; triacetyl derivative, colorless prisms, m. 220°. Br. No. 8 in A is replaced by Cl with HCl and alcohol, golden bronze plates, m. 152°; acetyl derivative, yellow prisms, m. 160°. Ketone reagents on A do not give well defined products.

*The bromination of 2-amino-p-cymene:* ALVIN S. WHEELER and I. W. SMITHEY. (By title.)

*The production of furfural by the action of superheated water on aqueous corn cob extract:* F. B. LAForge. (By title.)

## DIVISION OF AGRICULTURAL AND FOOD CHEMISTRY

C. E. Coates, *chairman*.T. J. Bryan, *secretary*.

*Suggestions for more rapid and exact methods of analyses for the cheese factory:* S. K. ROBINSON.

*Some problems of the pure food manufacturer:* H. A. NOYES. The preserver of fruit and fruit products has two important problems to solve: (1) The obtaining of a high quality and uniform fruit supply each year, and (2) the processing of fruit in such a way that its natural properties are retained. The pure food manufacturer's inability to accomplish these two things up to the present has allowed the artificial flavor and color manufacturer to develop his products and befuddle the taste of the average consumer. The plans and organization of one large concern, to study the production of quality fruits and to apply research methods to its business, were given.

*Variations in the Concord grape during ripening:* H. A. NOYES, H. T. KING, J. H. MARTSOLF. Variations in the Concord grape during ripening that are of interest to the juice and jam manufacturer were investigated. Results show a gradual increase in sugar content, a decrease in total acids and irregularities in tannin and coloring matter. Weather conditions were an important factor affecting sugar content, warm days and cool nights seeming to be the optimum condition for developing sugar.

*The absorption of copper from the soil by potato plants:* F. C. COOK. Insoluble copper compounds present in a Bordeaux spray containing an excess of lime and present in Pickering spray containing no excess of lime, also a solution of sulfate of copper, were added to the soil near the roots of potato plants in equal strengths and amounts at various intervals during the growing season. Samples of vines, tubers and soil were taken for analyses at frequent intervals. The leaves of the plants grown in the soil receiving the insoluble copper, *i.e.*, the sprays, held the largest part of the copper, the roots but little and the stems an intermediate amount. The tubers contained but traces of copper. Where the soil was treated with the copper sulfate solution the roots were injured and the normal metabolism of the vines interfered with. The tubers from these vines were small and the vines stunted. In these plants the roots held more copper than the leaves. The soluble copper sulfate added directly to the soil caused injury to the plants while the insoluble

copper compounds of the sprays did not. The extra lime of the Bordeaux spray did not reduce the amount of copper absorbed by the plants compared with the results on the Pickering plants. Where the sprays and copper sulfate solution were added to the soil directly practically the same amounts of copper were recovered from the soil samples. Samples of soil from sprayed potato fields showed but minute amounts of copper.

*Pickering Bordeaux sprays:* F. C. COOK.

*Analysis of the Jerusalem artichoke:* A. T. SHOHL. The Jerusalem artichoke, *Helianthus tuberosus*, gives the analysis:

	Water Nx6.25	Fat	Carb.	Fiber	Ash
Per cent...	79.0	3.1	.2	16.5	.8 1.1

The wastage by peeling is 31 per cent., the hydrogen ion concentration (colorimetric) pH 5.0. The carbohydrate is inulin, as determined by polariscope and quantitated after inversion by Benedict's solution. Experiments show it is utilized, and not excreted by diabetics. The nitrogen is largely extracted with boiling water. This non-protein fraction represents 71.5 per cent. of total nitrogen. Of the water soluble non-protein nitrogen 26 per cent. is free amino acid nitrogen and 12 per cent. ammonia nitrogen.

*Measuring soil toxicity, acidity and basicity:* R. H. CARR.

*What puts the "pop" in pop corn?* R. H. CARR. (Lantern.)

*The rate of oxidation of lime-sulfur:* C. A. PETERS and A. L. PRINCE. The rate of oxidation of lime-sulfur is largely independent of the concentration and also of the temperature up to about 80°, when the rate is increased in all but the very dilute solutions.

*A color test for "remade" milk:* OSCAR L. EVENSON.

*Effect of aging on lecithin-phosphoric acid determination of egg noodles:* R. C. HUMMELL. The results of these experiments indicate that aging does have considerable effect on the lecithin-phosphoric acid determination in egg noodles. By the end of six months this value had decreased to less than two thirds of the original value and in eighteen months had decreased to one half or less than one half of the original amount. Inasmuch as considerable time may elapse from the time at which egg noodles are manufactured until they reach the consumer, it would seem to be quite in

error to judge the egg content by the value obtained in the aforesaid determination.

*Peanut by-products:* J. B. REED.

*Some factors governing the crystallization of lactose in ice cream:* HARPER F. ZOLLER and OWEN E. WILLIAMS. A curve is presented as a result of experimental evidence which serves to separate those mixes which will produce sandiness from those which will not, and is based upon the relationship existing between the protein-serum solids concentration and the concentration of lactose within the mix. It is erroneous to calculate the concentration of lactose on the water basis since the total water in the mix is not available to the lactose because of the competition of the other solids. The effect of the proteins within the mix is not to repress the crystallization of lactose, but they act oppositely in increasing concentration. Because of its slow rate of crystallization lactose hydrate is subject to much supercooling and oversaturation. Protein has very little effect upon its rate of growth. The solubility of lactose hydrate according to the best of experimental deductions is 11.15 per cent. at 0° C. In an ice cream mix containing 10 per cent. fat, 14 per cent. of cane sugar and 65 per cent. of water, the above value for lactose reduces to about 8.9 per cent. calculated on the water basis.

*A rotating thermocouple and cold junction designed for temperature studies in horizontal power ice cream machines:* HARPER F. ZOLLER. A sensitive and experimental thermocouple is described and illustrated which has been designed for the purpose of accurately measuring the temperature of the ice cream mix within the freezer when the latter is rotating at full speed. By maintaining an ice-water cold junction affixed to the shaft of the freezer along with the thermocouple junctions the small temperature differences within the freezer can be measured with an accuracy of .02° C. This latter is also made possible by the use of a five junction copper-constantan thermocouple (of fine wire for small temperature lag effect) and a potentiometric setup embracing a galvanometer of low internal resistance with a potentiometer of microvolt capacity. The unique feature of the instrument is the method of conducting the small e.m.f. from the rapidly rotating shaft to the potentiometer without frictional thermoelectric effects. The instrument has been in regular service for a number of months, has given no trouble, and has measured the rapidly fluctuating temperatures within the mix simply and accurately.

*Cases of supercooling during the freezing of ice cream mixes:* HARPER F. ZOLLER and OWEN E. WILLIAMS. By the use of the rotating thermocouple we have examined the point of separation of ice in a variety of mixes. The measurements were made in a commercial ice cream machine of the Miller type with a capacity of five gallons. The freezing point lowering of the mix was not in harmony with the calculated value, but showed a high supercooling in the mix even in the presence of the swiftly moving beaters and scrapers. The addition of fine particles of substances to promote the formation of crystal nuclei prevented the supercooling of the mix and consequently the freezing was done in a shorter time, and the product was smoother. Both fat and gelatin seem to reduce supercooling in the average mixes. When sand is added to an ice cream mix containing 10 per cent. fat and 0.5 per cent. of gelatin ice begins to separate at only a slightly higher temperature when the brine is at 10° F. during the freezing process. If the brine is much lower there is a greater difference in the supercooling effect when no sand is present in the above mix. When mixes are frozen which have been made from evaporated milk containing lactose crystal nuclei and they have not been destroyed by pasteurization, or other means, no supercooling occurs. A great deal of importance is attached to the degree of supercooling and its influence upon the texture of the ice cream as it comes from the freezer.

*Black discoloration in canned sweet potatoes:* EDW. F. KOHMAN. The black discoloration which occurs in canned sweet potatoes begins in the bottom of the can where there is usually a semi-liquid starch paste which affords close contact with the can. Eventually it may penetrate the entire content of the can. The black formation is due to the combination of iron dissolved from the can with a tannin-like substance in the potatoes. This is localized to a considerable extent just beneath the peel. But as there is also some throughout the potato and especially about the center no change in present methods of peeling would be of advantage. Tannins do not form black compounds with iron unless the latter is in its highest state of oxidation. As air is essential to bring it into this condition, the necessity of tight seams in canned sweet potatoes is emphasized.

CHARLES L. PARSONS,  
*Secretary*