

## NOTES.

**The Interaction of Tin and Phenol.**—In a short paper<sup>1</sup> the author previously pointed out that phenol very readily attacked tin at temperatures above the boiling point of water. The common practise in certain serum laboratories of setting tin-lined cans containing solid phenol into an autoclave for the purpose of expediting the melting had led to the production of a precipitate when the phenol was diluted with water. There was no evidence of any precipitate in the liquid phenol. The precipitate was heavy, sticky and grayish-white. It was found to be made up largely of inorganic residue although there seemed to be a rather constant quantity of organic matter. The inorganic residue proved to be stannic oxide.<sup>2</sup>

Since reporting the above findings a careful investigation of the formation of the precipitate and the attending phenomena has been made, the results of which are described below.

**Experimental.**—A quantity of chemically pure phenol obtained on the open market in glass containers was redistilled in a still made entirely of glass. The purified phenol gave no residue upon ignition. It was protected from moisture.

A sheet of pure tin-foil 0.025 mm. in thickness, was cut into fine strips. Fifty g. of the tin strips and 200 g. of the pure phenol were placed together in a still made entirely of glass. The chief feature of the still was a double condensing arrangement. The first condenser, one cm. in internal diameter, possessed an outer jacket through which warm water circulated for the condensing of the phenol vapor. The auxiliary condenser, which was connected in series to the phenol receiver, consisted of a glass worm offering large surface and bent into a compact form for immersion into an ice and calcium chloride mixture.

Heat was applied externally through an asbestos mat until the phenol began to boil; then very slow distillation was permitted. After 2 hours the phenol remaining in the still began to get opalescent, but no definite separation of precipitate had taken place. About 30 cc. of a colorless liquid had collected in the secondary condenser. In the receiver to the primary condenser ordinary phenol had condensed and crystallized.

**Examination of Residue.**—The opalescent liquid remaining in the distilling flask was poured from the remaining tin strips into distilled water. A heavy gray precipitate formed immediately. This settled out in a short time leaving a clear liquid above. The precipitate was

<sup>1</sup> Presented at the Kansas City meeting of the A. C. S. in 1917.

<sup>2</sup> A chemist with a company which uses large quantities of phenol said that the experience of his company had corroborated my findings in that they had been using tin still-heads for the distillation of phenol and were required to replace them frequently.

then washed thoroughly many times with hot water, then with hot alcohol and finally with hot water. The washing was continued until ferric chloride produced no color reaction for phenol in the wash water. The precipitate, after drying to constant weight *in vacuo* over sulfuric acid, was subjected to a regular combustion analysis for carbon and hydrogen with the result that less than 0.1% carbon was found.

Tin in the residue was determined by the volumetric method after reduction to the stannous state.

Subs., 0.1820, 0.2135.

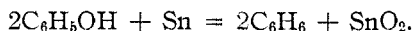
Calc. for  $\text{SnO}_2$ : Sn, 78.77. Found: 78.62, 78.58.

**Examination of the Crystalline Distillate.**—When a few of the crystals were added to a great excess of water no turbidity developed. Neither did it give a residue upon ignition. It therefore, contained none of the tin. A melting-point determination gave  $42.5^\circ$  (corr.), which is the melting point of pure phenol. With sodium hydrochlorite and ammonium chloride a crystal of the distillate developed the typical bluish coloration characteristic of phenol. We are justified in considering the solid distillate to be unchanged phenol.

**Examination of the Liquid Distillate.**—The distillate was a clear water color. It possessed the odor of benzene. Boiling-point determinations gave  $80^\circ$ . Ten cc. of the distillate was nitrated with a mixture of fuming nitric and conc. sulfuric acids under the conditions to furnish mononitrobenzene. Boiling-point determinations on the faintly yellow oil resulting showed  $210^\circ$  (uncorr.) The distillate (liquid) was assumed to be benzene.

**Interaction at Ordinary Temperatures.**—After tin had stood in contact with a sample of pure phenol at a temperature of  $30^\circ$  for 18 months no visible precipitate developed upon the addition of the phenol to an excess of water. Provision was made to keep the phenol dry during the contact with the tin. The latter was completely submerged in the phenol.

**Conclusions.**—The course of the interaction of tin and phenol at temperatures above the boiling point of water is probably as follows,



At low temperatures (ordinary room) the rate of interaction is very slow. The results of the observations and experiments have a significant bearing upon the manufacture and transportation of phenol.

The writer expresses his indebtedness to Dr. C. W. Hobbs, of the Serum Laboratory, at Manhattan, Kansas, for bringing the subject to his attention.

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**The Use of Phosphoric Acid Instead of Sulfuric Acid in Melting-Point Determinations of Organic Solids.**—A sulfuric acid bath in melting-point apparatus possesses the well-known disadvantage of fuming at

temperatures much above 150°. Phosphoric acid has proved to be an excellent substitute, for it does not fume at temperatures as high as 350°.

The preparation of the bath consists in heating orthophosphoric acid to the highest temperature desired, in order to produce the necessary concentration. As phosphoric acid attacks ordinary glass the use of some resistance glass, such as Pyrex, is essential. Even Pyrex, however, is etched, but so slowly that the same container may be employed for several months before its transparency is seriously impaired. On standing, the acid becomes somewhat clouded, owing to absorption of water from the air, but the addition of a further quantity of water with subsequent reheating will relieve this opacity. Any discoloration, moreover, may be destroyed by introducing a few crystals of sodium nitrite.

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**Correction.**—In my article on Friedel and Crafts' reaction in the September, 1920, *JOURNAL*, the following corrections should be made: p. 1873, line 10, read 6-carbethoxy-2-nitrobenzoic acid; p. 1873, line 13, read 2-carbethoxy-3-nitrobenzoic acid.

WALTER A. LAWRENCE.

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### NEW BOOKS.

**Radioaktivitaet.** By STEFAN MEYER AND EGON R. V. SCHWEIDLER. B. C. Teubner, Leipzig, 1916. 540 pages. 87 figures. 23 × 15 cm. Price, about 170 marks.

To scientific men, one of the most unfortunate results of the recent European war was the serious interruption of international intercourse in all branches of science. Although Meyer and v. Schweidler's *Radioaktivitaet* appeared in 1916, it has not been generally available in this country and can hardly be said to be so now, owing to the unfavorable regulations imposed in Germany and Austria upon the export of scientific literature. The interests, however, attaching to this exhaustive treatment of a subject which, with ever increasing force, impresses upon us its far-reaching importance to various branches of science, will justify a belated review.

The rapidity of the development of the subject of radioactivity, in spite of the retarding influence of the war, is clearly demonstrated by the fact that the progress of the past 4 years has opened entirely new fields and has left the treatment of others far behind the present status of development. Even during publication, this difficulty was recognized by the authors and was taken care of, as far as possible, in a number of additions to the text in the form of "Nachtraege."

The authors state that the contributions of Germany and Austria to the science of radioactivity invite a German treatise, although it parallels the standard English and French texts of Rutherford and Mme. Curie.