

the wax to insure a perfect seal, and the tube *D* is fused off at the constriction. The contact wire, though held rather firmly by the cork *F*, may now be adjusted by pushing it up or down, and with very little danger of allowing air to gain access to the mercury.

In order that the mercury thread shall not be parted, if through accidental overheating it should be pushed past the contact point, the wire should be sandpapered to a fine thread. Then by properly kinking the wire, the point can be kept centered on the mercury meniscus.

The regulation of an air chamber provides a severer test for the sensibility of a thermoregulator than does the regulation of a water bath. The air bath, for which the regulator described above was designed, will be described in a later publication, as will an account of the temperature records. The regulator has kept the temperature constant to within $0^{\circ}.002$, as read on a Beckmann thermometer, for periods of 8 hours. The daily variations have been considerably larger, but the most satisfactory feature has been the fact that, except for an occasional adjustment of the wire, the regulator has required no attention during three months continuous and uninterrupted use.

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AN ANOMALOUS REACTION OF RESORCINOL.

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It is stated in the literature that resorcinol may be titrated by means of caustic alkali, with Poirrier's blue, C_4B , as indicator, and that resorcinol, under these circumstances, acts as a dibasic acid.¹

As the result of many experiments I have been unable to verify this statement, although phenol and salicylic acid may be titrated readily with caustic alkalies, both being monobasic to the indicator.

If an aqueous resorcinol solution, of 0.1 to 0.5 molar concentration, be treated with one molecular proportion of caustic alkali of normal strength, it soon changes through yellow and greenish yellow shades of increasing depth to a deep brown color. Sodium carbonate effects the same change of color at a slower rate.

If such a resorcinol solution be titrated with standard caustic alkali as rapidly as possible, in the presence of sufficient of the indicator solution (1 : 500), the neutrality point will be reached when between 1 and $1\frac{1}{2}$ molecular proportions of the alkali have been added. The amount of alkali necessary for the neutral color change will vary, for the same concentration of the resorcinol solution, with the strength of the caustic

¹ Engel, *Compt. rend.*, 102, 214-17 (1886); *Chem. News*, 53, 87-88 (1886); also *Bull. soc. chim.*, 45, 321-28 (1886); Allen, *Com. Org. Anal.*, 4th Ed., 3, 337; Cohn, "Indicators and Test-Papers," 2nd. Ed., p. 136.

alkali used and more especially with the time consumed in reaching the end point, which, at best, is neither sharp nor distinct owing to the color changes before referred to.

The reason for this variation will be apparent from the following paragraph:

If after reaching the end point the neutralized solution be allowed to stand for ten to fifteen minutes, *all of the alkali which has been added can be titrated back with a mineral acid.* In other words, it would appear that the resorcinol which before titration had an acid reaction toward Poirrier's blue, is, upon being set free by means of a strong acid, without *any effect* upon the indicator.

As far as I have been able to find from the literature, resorcinol should be expected to act like a diatomic phenol, forming with alkali two series of salts, $\text{HO.C}_6\text{H}_4.\text{OM}$ and $\text{C}_6\text{H}_4(\text{OM})_2$, and the sodium salts corresponding to the above types have been described.¹

I have found, further, that it is not necessary to add so much caustic alkali in order to change the nature of the resorcinol in respect to this property. Even in the presence of one-tenth molecular proportion of caustic alkali, allowing to stand at room temperature for 3 hours, the solution was found to be alkaline upon adding the indicator, and the whole of the alkali which had been added could be titrated back before the blue color of the indicator, denoting acidity, was restored.

These experiments would seem to point to the conclusion that, at least in aqueous solution, the alkali salts of resorcinol are either not formed, or if formed that they are very unstable, and that the alkali induces a reaction whereby the resorcinol is completely converted into another compound of non-phenolic nature, possibly a tautomeric substance.

With this idea in view, an attempt was made to isolate such a product, with negative results, however, as will appear from the following:

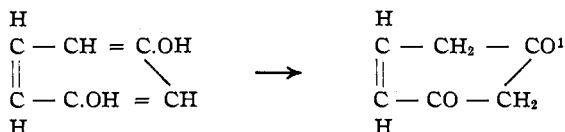
One-tenth molecular proportion of caustic alkali was allowed to act upon a 0.1 molar resorcinol solution for 3 hours, after which it was treated repeatedly in a separator with ether until nothing more could be dissolved out of the alkaline solution. The ether was then evaporated off and the product was recrystallized from hot benzene. The crystals so obtained had the same melting point, and responded to the same chemical tests as the original resorcinol, including acidity to Poirrier's blue. In another similar experiment petroleum ether, in which resorcinol is insoluble, was substituted for the ether, with the result that nothing was extracted from the alkaline solution.

In still another case, where, after the addition of one molecular proportion of caustic alkali and allowing to stand for about half an hour, double the quantity of a mineral acid was added, the effect of the alkali

¹ Forcrand, *Ann. chim. Phys.*, [6] 30, 69 (1893).

upon the resorcinol was not reversed on long standing or by boiling the acid solution for a considerable time. The substance extracted by ether from this acid solution, and in another case from the neutralized solution, responded to all the tests for, and was identified as resorcinol after recrystallization from benzene.

It has been known for a long time that diatomic (and triatomic) phenols with hydroxyl groups in the 1 : 3 position under certain conditions react to form derivatives which must possess a constitution tautomeric to that ascribed ordinarily to the phenol; but I am not aware that there has previously been any experimental data adduced which would indicate that the phenol itself, in alkaline aqueous solution, was capable of undergoing rapidly and completely tautomeric change. In the case of resorcinol the changes in constitution are possibly in accordance with those shown in the following formulas:



From the foregoing it appears to be impossible to titrate an aqueous solution of resorcinol by means of alkali in the presence of Poirrier's blue, despite the statements of the literature cited to the contrary; and as far as I know there is no other indicator toward which this substance shows marked acidic function.

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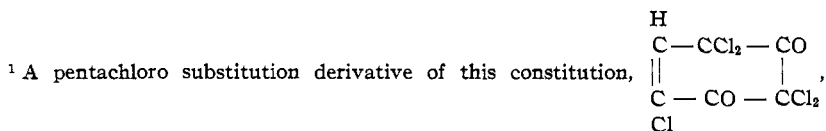
THE SUBLIMATION AND DECOMPOSITION OF ACETYLUREA.

BY RASIK LAL DATTA AND SATYARANJAN DAS GUPTA.

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The action of heat on acetylurea has been studied only qualitatively by Zinin² who, in describing the preparation of this compound from urea and acetyl chloride, mentions incidentally that it breaks up upon heating into cyanuric acid and acetamide. It is the purpose of the present communication to show that acetylurea decomposes quantitatively, upon heating, into cyanuric acid and acetamide, and that under carefully regulated conditions it can be partly sublimed unchanged.

In the course of the determination of the melting point of acetylurea,



has been described (Beilstein, *Org. Chem., Spl. Bd. I*, 539).

² *Ann.*, **92**, 405 (1854).