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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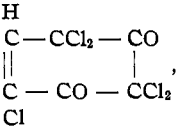
THE SUBLIMATION AND DECOMPOSITION OF ACETYLUREA.

By Rasik Lal Datta and Satyaranjan Das Gupta.

Received September 30, 1913.

The action of heat on acetylurea has been studied only qualitatively by Zinin[2] 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,

[1] A pentachloro substitution derivative of this constitution,  , has been described (Beilstein, *Org. Chem., Spl. Bd. I*, 539).

[2] *Ann.*, **92**, 405 (1854).

it was always noticed, especially when there was a delay in raising the temperature, that a minute quantity of white needles made their appearance at the upper and cooler parts of the tube, above the level of the sulfuric acid in the bath. This led us to suspect that the substance itself or one of its decomposition products might have sublimed. With a view toward collecting enough of the substance for an examination, acetylurea was placed in glass tubes and dipped in sulfuric acid bath whose temperature was kept between 180 and 190°, *i. e.*, much below the temperature of its melting point (218–219°). The sublimation is facilitated by the employment of tubes which are a little constricted at the points where the crystals would collect. Very gradually, a sublimate, consisting of fine white needles, made its appearance and this increased up to a definit quantity, after which no further deposition took place even on keeping the temperature of the bath at the melting point. As the quantity turned out at each operation is small, the operation was carried out in series of tubes, sometimes heated by the same bath. During the process, it was noticed that above the sublimate, a liquid collects in droplets, which, on cooling the tube, solidifies to well foliated crystals sticking to the sides of the tube; at the same time an odor similar to acetamide is produced. The sublimate from several tubes was collected and dried in the desiccator. The dried substance was analyzed and gave a melting point of 216–217°. Evidently the substance is acetylurea. The minute

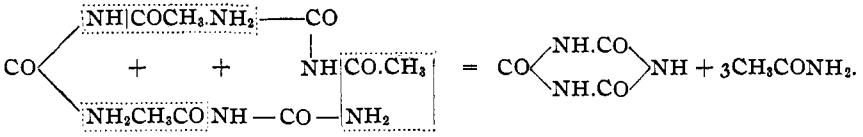
Found: N = 27.29; Calc.: N = 27.45.

drops of liquid were found, on examination, to be pure acetamide. Hence, the substance undergoes sublimation only partly, with a tendency to decompose into acetamide and cyanuric acid.

Now diacetylurea[1] sublimes completely without being changed, and it is quite expected that mono-acetylurea would behave similarly, had there been no chance for the substance to decompose into acetamide and cyanuric acid. The sole reason for the sublimation stopping at a certain point is that the slight decomposition which takes place during the process covers the substance with a coating of the decomposition products. It may be noted that, when no more of the product sublimes, the mass becomes pasty and melts at a much lower temperature, as would be expected from the presence of the decomposition products. It is for this reason that a low melting point is obtained, especially when the bath is heated slowly to the melting temperature, and this might account for the low melting points obtained by different observers (212°). On the other hand, if the substance be heated rapidly, it is found that it decomposes quantitatively into acetamide and cyanuric acid according to the following scheme:

[1] Schmidt, *J. prakt. Chem.*, [2] **5**, 63 (1872).

[2] Behrend, *Ann.*, **229**, 30 (1885).



For showing the quantitative production of acetamide, the compound is subjected to dry distillation with a free flame, when the substance melts and decomposes rapidly, and almost pure acetamide distils over between 221° to 223° in quantitative yield. On conducting the distillation to completion, the product left within the flask chars and from this cyanuric acid can be obtained. To show the quantitative production of cyanuric acid the distillation is stopped when the least sign of charring is noticeable. Consequently some of the acetamide remains behind in the flask, along with all of the cyanuric acid. The residue in the flask is treated with a small quantity of water, whereupon the acetamide dissolves, leaving nearly pure cyanuric acid in quantitative yield. The perfectly pure product is obtained after recrystallization from water.

Found: N = 32.64; Calc.: N = 32.56.

Hence, acetylurea decomposes quantitatively into acetamide and cyanuric acid; a part also sublimes if the temperature be kept between 180° and 190°.

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THE LEAF OIL OF DOUGLAS FIR.

BY A. W. SCHORGER.

Received September 26, 1913.

The Douglas fir[1] occurs in the Rocky Mountain region of the United States, extending northward into British Columbia. The six samples of the leaf oil examined were distilled by the Forest Service near Northfolk, Cal., the yield of oil being 0.11–0.20%, with an average of 0.163%.

Brandel and Sweet[2] obtained a yield of 0.8–1.0% of oil from small trees and underbrush collected in the State of Washington. These authors showed the presence in the oil of free borneol, bornyl acetate, and camphene. Pinene and limonene were thought to be present but not identified.

The California oil was found to consist mainly of β-pinene, and contained in addition furfurol, α-pinene, dipentene, free borneol, bornyl acetate, and an unidentified green oil, apparently a sesquiterpene.

Experimental.

The six samples gave the following range of properties: density, $d_{15°}$, 0.8727–0.8759; refractive index, $n_{D15°}$, 1.4758–1.4780; rotation,[3] $\alpha_{D20°}$,

[1] Pseudotsuga taxifolia Britt.

[2] *Pharm. Rev.*, **26**, 326–8 (1908).

[3] The rotation readings as given are the readings in a 100 mm. tube at 20° C.