LXX.—*Further Observations on the Action of Hexabromacetone on Urea.*

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IN a recent communication (*ante*, p. 693) I described a series of experiments showing that of the two isomeric cyanuric acids which Herzig (*Ber.*, **12**, 170) claims to have obtained by the action of hexabromacetone on urea, the one—the α -acid—is identical with ordinary cyanuric acid, whilst in the case of the other—the β -acid—further experiments were needed before a decisive opinion could be formed. These have now been made. I have prepared several specimens of the β -acid, carefully attending to the directions given by Herzig; they were all similar in appearance, and consisted of crystals unlike cyanuric acid, and exhibited generally the properties which Herzig observed. On repeated recrystallisations from water, however, the crystals were found

gradually to assume the character of the ordinary acid and the other distinctions one by one to disappear. The properties given by Herzig as distinguishing the β from the α and from the ordinary acid are :—

- I. When heated it does not yield cyanic acid.
- II. When treated with phosphorus pentachloride cyanuric chloride is not formed.
- III. It does not give Hofmann's test for cyanuric acid.
- IV. The crystals do not contain water.
- V. It is more soluble in alcohol than either the (α) or the ordinary acid.
- VI. A barium salt of constant composition could not be obtained.

I. *Action of Heat.*

None of the specimens which I have examined appeared to yield cyanic acid when heated in small tubes until they had been three or four times recrystallised from water; when this was done, however, the odour of cyanic acid was easily obtained, and could be recognised beyond any doubt.

II. *Action of Phosphorus Pentachloride.*

By carefully treating the product obtained by heating the β -acid with phosphorus pentachloride in a closed tube, in accordance with the directions of Beilstein (*Annalen*, **116**, 357), I have isolated cyanuric chloride as announced in my previous paper. The contents of the tube are in the first place distilled at 150° , which removes the phosphorus oxychloride formed, the cyanuric chloride remaining with other products in the retort. The mixture in the retort is extracted with boiling dry ether, which, on evaporation, leaves crude cyanuric chloride. This must now be washed quickly with water, dried in a vacuum, and again extracted with ether. The ethereal solution thus obtained, when evaporated, gives the cyanuric chloride in crystals, which are readily identified by their melting point and characteristic odour.

III. *Hofmann's Test for Cyanuric Acid.*

This test is based on the characteristic property of cyanuric acid of giving a trisodium salt less soluble in warm than in cold concentrated solution of sodium hydroxide; I have applied it successfully in the case of all the specimens of the β -acid which I have prepared, even after the first recrystallisation.

IV. *Water of Crystallisation.*

Contrary to Herzig's experience, my specimens of the β -acid when purified by recrystallisation from water and dried by pressing between paper and subsequent exposure to the air for an hour, contain water which is given off below 120° , the proportion increasing with purification. The product of a fourth crystallisation gave the following percentage:—

Theory.	Experiment.
$(C_3N_3)(OH)_3 \cdot 2H_2O$.	
21·8	15·8

The same specimen once more recrystallised retained a larger proportion of water. An experiment was made at the same time under precisely the same conditions with the ordinary acid, thus:—

Theory.	Experiment, ordinary acid.	Experiment, β -acid.
$(C_3N_3)(OH)_3 \cdot 2H_2O$.		
21·8	21·9	19·7

It is clear then that the β -acid when purified does contain water of crystallisation, and that this is increased by purification, until, as seen in my last experiment, it amounts approximately to the two molecules contained in the crystals of the ordinary acid.

V. *Solubility in Alcohol.*

Herzig gives no determination of the solubility of the β -acid in alcohol. It is indeed, as he points out, more soluble in alcohol than the ordinary or the α -acid, but by successive recrystallisations its solubility is gradually reduced; thus:—

	1st crystallisation.	5th crystallisation.	6th crystallisation.
In 100 grams alcohol } at $22-24^\circ$ }	0·425	0·240	0·156

This greater solubility, therefore, is due to the influence of some impurity which recrystallisation removes, and is not characteristic of the β -acid.

VI. *Barium Salt.*

Herzig did not succeed in preparing a definite barium salt from the β -acid. By carefully following the directions of Wöhler (*Annalen*, **62**, 252), however, I have obtained, as in the case of the α -acid, the monobarium compound in beautiful needles. It has the formula $(C_3N_3)(OH)_2(OBa) + H_2O$, which requires—

	Theory.	Experiment.
Barium	31·93	32·71

It thus appears that by simple recrystallisation from water " β -cyanuric" acid may be so purified that each of the properties which were supposed to distinguish it from the ordinary acid in turn disappears. By this means undoubtedly, small quantities of some impurity is taken away, which, although not sufficient to materially affect the analytical numbers as given by Herzig is, nevertheless, enough to modify in a remarkable manner the properties of the acid. β -cyanuric acid therefore, like the α -compound, is identical with ordinary cyanuric acid.

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