Two other experiments of the same kind gave similar results.

Hexabromodihydrobenzene is rather more soluble in glacial acetic acid than in most solvents, but even in this molecular weight determinations could not be made. It is difficultly soluble in absolute alcohol, hot and cold, in benzene, nitrobenzene and aniline. It dissolves readily in ethylene bromide, in which the molecular weight was determined.

0.3090 g. in 42.56 g. $C_2H_4Br_2$ gave lowering 0.166°; 0.5236 g. gave 0.282°.

0.1661 g. gave 0.0073 g. H_2O, 0.0802 g. CO_2; 0.1685 g. gave 0.0077 g. H_2O, 0.0813 g. CO_2. 0.1562 g. gave 0.3215 g. AgBr; 0.1542 g. gave 0.3170 g. AgBr.

	Theory.	Found.	
	$C_6H_2Br_6$.	Theurer.	Thomas.
C	13.00	13.26, 13.09, 13.14	13.17, 13.16
H	0.36	0.79, 0.50, 0.50	0.49, 0.51
Br	86.63	86.8, 86.72, 86.41	87.50, 87.60
M. wt.	554	....	514, 516

Summary.

I. It has been shown that the hitherto accepted formula for xanthogallol is inconsistent with its molecular weight and with the fact that it contains no hydrogen.

II. A new cyclic formula has been suggested based essentially upon several series of reactions leading to oxalic acid and brominated acetones. This formula also permits a simple interpretation of the reaction by which xanthogallol is formed from pyrogallol.

III. The genetic relationships studied in these reactions serve to establish the constitution of nearly twenty other substances closely allied to xanthogallol.

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[Contribution from Chemistry Section, Iowa State College.]

SYNTHESIS OF A NAPHTHOTETRAZINE FROM DIETHYL SUCCINYLOSUCCINATE AND DICYANODIAMIDE.

By Arthur W. Dox.

Received February 27, 1917.

On account of the ease with which dicyanodiamide can be prepared in quantity and at very small cost from the crude calcium cyanamide of commerce, this substance is beginning to find numerous applications in organic syntheses. Among other properties, the amidine structure of dicyanodiamide has been taken advantage of for the preparation of certain nitrogen heterocycles. For example, by condensation with such substances as α-ketone acid esters, various pyrimidine derivatives are obtained. Thus, dicyanodiamide condenses with malonic ester derivatives[1] and with acetoacetic ester[2] to form substituted pyrimidines. It is not

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[1] German Patent 165,223 (1905).

[2] Söll and Stutzer, *Ber.*, 42, 4534 (1910).

improbable that dicyanodiamide is capable of entering into the same condensation reactions and yielding cyanamino derivatives of the various heterocycles now prepared from guanidine.

The readiness with which dicyanodiamide yields pyrimidine derivatives suggested to the writer the possibility of preparing a 1,3,6,8-naphthotetrazine, or symmetrical benzodipyrimidine, by condensation with succinylosuccinic ester. Other amidines have been condensed with succinylsuccinic ester, forming substituted naphthotetrazines. Thus, benzamidine[1] yielded 2,7-diphenyl-4,9-diketotetrahydro-1,3,6,8-naphthotetrazine, guanidine[2] the corresponding 2,7-diamino, and acetamidine[3] the corresponding 2,7-dimethyl derivatives. Other derivatives of this heterocycle have been prepared by Bogert and Nelson[4] from *p*-diaminoterephthalic acid and its derivatives. They all appear to be characterized by insolubility, infusibility and general inertness.

Experimental.

Dicyanodiamide was prepared by the method of Söll and Stutzer[5] from commercial calcium cyanamide. The product was obtained in large white crystals, melting at 209° (corr.). Succinylosuccinic ester, prepared in the usual way from diethyl succinate and sodium, was suspended in ten times its weight of water and an equal volume of 5% sodium hydroxide solution added. After the ester had dissolved to a bright yellow solution, dicyanodiamide, equivalent in amount to two molecules for every molecule of succinylosuccinic ester, was added in the solid form, and the mixture gradually warmed on an electric stove. As the temperature rose, the dicyanodiamide went into solution and at about 50° a pale yellow granular precipitate began to form. Heating was continued until the mixture just began to boil. After cooling, the precipitate was filtered with suction, washed with water, dilute hydrochloric acid, alcohol and finally ether, and dried in the oven at 100°. The yield was 37% of the theory. In a second and third preparation, equal weights of dicyanodiamide and succinylosuccinic ester were used, and the yields on the basis of the latter substance were 72% and 61%, respectively.

The mother liquor was bright red, the color evidently being due to an oxidation process, since the red appeared first at the surface of the solution in contact with the air. This color turned yellow on acidifying and then back to the original red on the addition of alkali.

Like the other naphthotetrazine derivatives described, this condensation product is characterized by its insolubility in the neutral solvents

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[1] Pinner, *Ber.*, **22**, 2609 (1889).

[2] Bogert and Dox, THIS JOURNAL, **27**, 1127 (1905).

[3] Bogert and Dox, *Ibid.*, **27**, 1136 (1905).

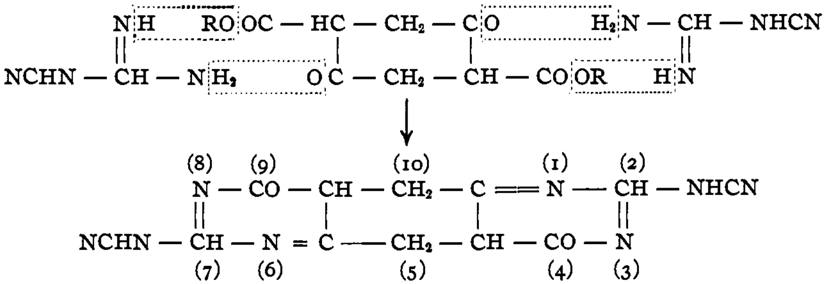
[4] Bogert and Nelson, *Ibid.*, **29**, 729 (1907).

[5] Söll and Stutzer, *loc. cit.*

and by its infusibility. At about 320° it darkens in color without melting. Analysis of the product gave the following results:

Calc. for $C_{12}H_8N_8O_2$: N, 37.8; C, 48.6; H, 3.0. Found: N, 37.2; C, 48.5; H, 2.7.

The condensation evidently takes place as indicated below, and consists in the elimination of two molecules of water and two of alcohol, between one of succinylosuccinic ester and two of dicyanodiamide.



The product is therefore 2,7-dicyanoamino-4,9-diketotetrahydro-1,3,6,8-naphthotetrazine.

AMES, IOWA.

[CONTRIBUTION FROM THE CARBOHYDRATE LABORATORY, BUREAU OF CHEMISTRY, UNITED STATES DEPARTMENT OF AGRICULTURE.]

INDIRECT MEASUREMENTS OF THE ROTATORY POWERS OF SOME ALPHA AND BETA FORMS OF THE SUGARS BY MEANS OF SOLUBILITY EXPERIMENTS.

BY C. S. HUDSON AND E. YANOVSKY.

Received February 5, 1917.

At the present time the crystalline alpha and beta modifications of lactose and of glucose are known in a condition closely approaching purity and their rotatory powers have in consequence been directly measured. The view that the mutarotation of lactose and glucose is due to the slow establishment of an equilibrium in solution between the respective alpha and beta forms of these sugars leads to the presumption that the many other sugars which show mutarotation exist in alpha and beta forms likewise. Such modifications have, indeed, been crystallized in the cases of galactose, rhamnose, gentiobiose and possibly melibiose, but it appears doubtful whether the isomers have been fully separated in these cases. For the remaining crystalline sugars which show mutarotation and, therefore, probably exist in two modifications, namely, xylose, arabinose, lyxose, ribose, mannose, fructose, α-glucoheptose, maltose, cellose, and a few rarer sugars, only one crystalline form has ever been prepared.

It has been suggested by one of us[1] that the difference between the

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[1] Hudson, THIS JOURNAL, 31, 66 (1909).