# Notes

## ANALYSES

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In. %	C, %	н. %	C1, %
Calcd. Found	Caled. Found	Caled. Found	Calcd. Found
41.31 $41.40$ $41.48$	25.91 $25.72$ $25.80$	3.99  3.93  4.02	
37.54  37.67  37.62	31.38 31.32 31.20	$4.94 \ 4.76 \ 4.84$	
25.12 24.96 25.03	10.51  10.61  10.56	0.22  0.25  0.29	46.60 $46.63$ $46.38$
$30.71 \ \ 30.71 \ \ 30.48$	44.94 $44.82$ $44.75$	2.94 $2.91$ $3.01$	
28.56 $28.62$ $28.42$	47.77 $47.62$ $47.71$	3.76  3.72  3.67	
28.56 $28.63$ $28.52$	47.77 47.70 47.83	3.76  3.65  3.60	
28.56 $28.41$ $28.39$	47.77 47.70 47.86	3.76  3.71  3.80	
	Calcd. Found   41.31 41.40 41.48   37.54 37.67 37.62   25.12 24.96 25.03   30.71 30.71 30.48   28.56 28.62 28.42   28.56 28.63 28.52	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

the salt from an acid indium sulfate solution by means of a solution of sodium benzoate and washing the precipitate with alcohol and ether; yield quantitative.

The ortho, meta and para toluates were each prepared by adding indium hydroxide to a slight excess over the theoretical quantity of the melted acid and the mixture kept in the melted state until all the hydroxide was dissolved. After being cooled, the excess acid was removed by boiling the melt in water, filtering off the salt and washing it with alcohol and ether; yield quantitative.

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## d-Ribose from the Croton Bean

By Joseph R. Spies with Nathan L. Drake

Cherbuliez and Bernhard<sup>1</sup> have recently isolated from the croton bean, *Croton tiglium* (L), a new glycoside, which they identified as 2-hydroxy-6-amino-purine-*d*-riboside, and which they proposed to call a "crotonoside." They did not, however, succeed in crystallizing the sugar residue obtained by hydrolysis.

The accumulation of a quantity of this material has afforded us an opportunity to examine it. We have succeeded in crystallizing the sugar residue and have conclusively established its identity as d-ribose, thus confirming the findings of these workers.

#### Experimental

Isolation of the Crotonoside.—The glycoside was extracted from the ground unshelled beans with methanol and isolated as described by Cherbuliez and Bernhard.<sup>1</sup> From 125 kg, of beans was obtained 345.0 g, of crude product which contained about 25% of pure crotonoside (0.07%). When recrystallized to constant melting point and dried in the Abderhalden at 110°, it decomposed at 245–247°. (All melting points were taken with standardized Anschütz thermometers.) Anal. Caled. for  $C_{10}H_{13}O_5N_5;$  C, 42.37; H, 4.59; N, 24.73. Found: C, 42.01; H, 4.47; N, 24.56.²

**Crystallization of** *d*-**Ribose**.—The crotonoside was hydrolyzed and the ribose isolated as described by Cherbuliez and Bernhard.<sup>1</sup> The sirup obtained was dried in a vacuum over phosphorus pentoxide, and when nucleated with an authentic specimen of *d*-ribose it slowly crystallized.<sup>3</sup> Recrystallization three times from absolute ethanol and once from dry isopropanol gave a product that melted constantly at  $83-87^{\circ}$  with previous softening at  $80^{\circ}$ . The melt, however, was cloudy;  $[\alpha]^{20-25}D - 17.5^{\circ}$ (c = 5.00 g./100 ml.); (Levene and Jacobs,  $85^{\circ}$ ,  $[\alpha]^{29}D$  $-19.2^{\circ})^4$  (Phelps, Isbell and Pigman,  $87^{\circ}$ ,  $[\alpha]^{1}D - 23.7^{\circ}.)^5$ 

**Tetraacetate.**—The tetraacetate (tetraacetylribose) was prepared as described by Levene and Tipson,<sup>6</sup> who give 110° as the melting point and  $[\alpha]^{24} D - 52.0^{\circ}$  and  $[\alpha]^{25} D$  $-54.3^{\circ}$  in chloroform. The present writers' product was recrystallized to constant melting point from ethanol; m. p. 108–109°; in chloroform  $[\alpha]^{24} D - 54.1^{\circ}$  (c, 6.52 g./100 ml.).

Anal. Calcd. for C<sub>13</sub>H<sub>18</sub>O<sub>9</sub>: C, 49.06; H, 5.66. Found: C, 49.33; H, 5.88.

**Phenylosazone.**—The phenylosazone was recrystallized to constant melting point from 40% ethanol; m. p. 165–165.5° (Levene and LaForge, 166°).<sup>7</sup>

*p*-Bromophenylhydrazone.—The *p*-bromophenylhydrazone was prepared as described by Cherbuliez and Bernhard.<sup>1</sup> It was recrystallized to constant melting point from an absolute ethanol-ether solution; m. p. 164° (Ekenstein and Blanksma,<sup>8</sup> 164°) (Levene and Jacobs,<sup>9</sup> 170°).

(2) The authors are indebted to Mr. H. M. Duvall of the University of Maryland for the micro Kjeldahl determination.

- (3) The writers are indebted to Mr. F. P. Phelps, of the Bureau of Standards of the U. S. Dept. of Commerce, for this material.
  - (4) Levene and Jacobs. Ber., 42, 2469, 2474 (1909).
  - (5) Phelps, Isbell and Pigman. THIS JOURNAL. 56, 747 (1934).
  - (6) Levene and Tipson, J. Biol. Chem., 92, 109 (1931).
  - (7) Levene and LaForge, *ibid.*, **20**, 429 (1915).
  - (8) Ekenstein and Blanksma, Chem. Weekblad. 10, 664 (1913).
  - (9) Levene and Jacobs, Ber., 42, 2703 (1909).
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and Plant Quarantine

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### The Decomposition of Acetylphthalimide

By Charles D. Hurd, Malcolm F. Dull and J. W. Williams

It was reported earlier<sup>1</sup> that acetylphthalimide decomposed at 240-325° into phthalimide, acetic (1) Hurd and Dull, THIS JOURNAL, **54**, 2437 (1932).

<sup>(1)</sup> Cherbuliez and Bernhard, Helv. Chim. Acta, 15, 464 (1932).