methyl and 9-methyladenine.[7] We believe these findings to be consistent with structure I.[8]

Confirmation of this structure has been obtained by condensing D-psicosyl chloride tetraacetate with chloromercuri-6-acetamidopurine and deacylating the resulting product.[9] Countercurrent distribution of the reaction mixture afforded synthetic 6-amino-9-D-psicofuranosylpurine, identical with the natural material.

The successful extension of this synthetic method to the preparation of other keto nucleosides will be reported in detail at a later date.

D-Psicose has been reported once before[10] to be a naturally occurring sugar, although this claim was subject to question.[11]

The present finding constitutes the first demonstration of a biologically produced ketose nucleoside and provides good evidence that D-psicose can be elaborated by microörganisms.[12]

We wish to thank Dr. W. G. Jackson for his interest in this problem and Mr. W. A. Struck and associates for the microanalyses.

(7) J. M. Gulland and E. R. Holiday, *J. Chem. Soc.*, 765 (1936); J. M. Gulland and L. F. Story, *ibid.*, 259 (1938).

(8) The n.m.r. spectrum, as interpreted by Dr. George Slomp of these laboratories, is also in accord with this proposal.

(9) This general procedure for nucleoside syntheses from aldo sugar halides was developed by J. Davoll and B. A. Lowy, THIS JOURNAL, **73**, 1650 (1951). The present report is the first recorded instance of its use in the synthesis of a keto sugar nucleoside.

(10) F. W. Zerban and L. Sattler, *Ind. Eng. Chem.*, **34**, 1180 (1942); THIS JOURNAL, **64**, 1740 (1942).

(11) L. Hough, J. K. N. Jones and E. L. Richards, *J. Chem. Soc.*, 2005 (1953).

(12) After submission of this paper, we received the paper [Hsü. Yüntsen, *J. Antibiotics (Japan)*, **11A**, 244 (1958)] in which structure I was assigned to angustmycin C.

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ORGANOBORON COMPOUNDS. XI. TRIALKYLBORANES HAVING TWO *t*-BUTYL GROUPS ATTACHED TO BORON[1,2]

Sir:

It is now clear[3] that no authentic organoboron compound having more than one *t*-butyl group attached to the same boron atom has been described previously.

We now report the first trialkylboranes having two *t*-butyl groups attached to boron, *viz.*, di-*t*-butyl-*n*-butylborane (I) and di-*t*-butyl-*n*-amylborane (II). Each of these substances was fractionally distilled at least twice *in vacuo*, below 50°, without evidence of decomposition, rearrangement or disproportionation. Oxidation of I with alkaline hydrogen peroxide gave a 2:1 mixture of *t*-butyl and *n*-butyl alcohols in high yield; II similarly treated produced *t*-butyl and *n*-amyl alcohols in the proper ratio. The infrared spectra of I and II are similar and different from the spectra of related trialkylboranes previously described.[3]

Property		Compd. I	Compd. II
B.p., °C. (mm.)		47.7 (1.7)	42.5–42.7 (0.5)
$n^{25}D$		1.4373	1.4397
d^{25}		0.7608	0.7668
B	Calcd., %	5.94	5.52
	Found, %	6.08	5.56
MR_D	Calcd.	62.67	67.30
	Obsd.	62.76	67.38

When II was heated under nitrogen at 205° for fifteen minutes rearrangement and disproportionation occurred and a 2:1 mixture of triisobutylborane and tri-*n*-amylborane was produced in quantitative yield. It may be noted that the same mixture was produced when *t*-butyl-isobutyl-*n*-amylborane[1] was heated in the same manner.

I was prepared in 41% yield by the alkylation of boron fluoride with *t*-butylmagnesium chloride in anhydrous ether containing a large excess of 1-butene. II was made in the same way (32–39% yields) except that 1-pentene was employed in place of 1-butene. It is noteworthy that attempts to prepare di-*t*-butyl-isobutylborane by this procedure failed. When the reaction of boron fluoride with *t*-butylmagnesium chloride was carried out in the presence of isobutylene, the product proved to be *t*-butyl-diisobutylborane.[3] Furthermore, I did not react with isobutylmagnesium bromide by alkyl exchange.[1] It now appears likely that di-*t*-butyl-isobutylborane, if formed under any conditions, is unstable due to steric hindrance and rearranges rapidly at low temperature to *t*-butyl-diisobutylborane and at high temperature to triisobutylborane.

The mechanisms of the reactions mentioned above are now under investigation in This Laboratory and will be discussed at a later date.

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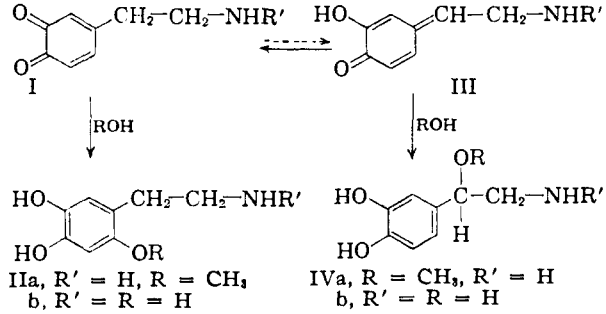
RECEIVED FEBRUARY 6, 1959

2,4,5-TRIHYDROXYPHENETHYLAMINE, A NEW METABOLITE OF 3,4-DIHYDROXYPHENETHYLAMINE

Sir:

The 1,4-addition of nucleophilic agents to *o*-quinones of acylated dopamine derivatives I leads to 6-methoxydopamine (IIa, R = CH_3) and 2,4,5-trihydroxyphenethylamine (IIb, R = H). Concomitant 1,6-addition to the tautomeric quinonemethine III yields <0.1% norepinephrine (IVb).[1]



IIa, R' = H, R = CH_3 — IVa, R = CH_3, R' = H
b, R' = R = H — b, R' = R = H

(1) Previous paper, G. F. Hennion, P. A. McCusker and J. V. Marra, THIS JOURNAL, **80**, 3481 (1958).

(2) Contribution from the Radiation Project operated by the University of Notre Dame and supported in part under Atomic Energy Commission Contract AT-(11-1)-38.

(3) G. F. Hennion, P. A. McCusker, *et al.*, THIS JOURNAL, **79**, 5190, 5192, 5194 (1957); **80**, 617 (1958).

(1) S. Senoh and B. Witkop, A. C. S. Meeting, Chicago, Sept., 1958, Abstracts p. 64-P.