Acid hydrolysis of II followed by acetylation and chromatographic separation4 yields N-methyl- $\alpha$ -L-glucosamine pentaacetate,  $[\alpha]^{29}D$   $-99 \pm 2^{\circ}$ (c 1, chloroform), m. p. 158°, unchanged on admixture with an authentic synthetic specimen. These data demonstrate that in streptomycin, the carbonyl group of N-methyl-L-glucosamine is glycosidically joined to the central portion, which is in turn similarly linked to streptidine.

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## 2,2,5,5-TETRAMETHYL-3-HEXENE

Sir:

The properties of sym-di-t-butylethylene, as recently reported by Hennion and Banigan, are not in agreement with the properties measured in these laboratories on 2,2,5,5-tetramethyl-3hexene, nor with those reported by Moersch.2 However, the properties of the paraffin hydrocarbon (2,2,5,5-tetramethylhexane) prepared from the olefin by each investigator are in close agreement. The properties of the compounds in question are listed in Table I.

## TABLE I Bn°C Fn°C

d 20

as 20 m

	D. p., C.	r. p., c.	<b>G</b>	WD
2,2,5,5-Me4-3-				
hexenea (trans)	$125.013^{b}$	- 4.75	0.71673°	$1.41148^d$
2,2,5,5-Me <sub>4</sub> -3-				
hexene <sup>e</sup> (trans)	$123.2^{f}$		.7176	1,4117
2,2,5,5-Me <sub>4</sub> -3-				
hexene* (cis)	$144.2 - 144.4^h$	<-65	.7439	1.4271
2,2,5,5-Me4-hexanea	137 . 457 <sup>i</sup>		$.71875^{j}$	$1.40550^{k}$
2,2,5,5-Me <sub>4</sub> -hexane <sup>g</sup>	$136.2 - 136.4^{l}$	<b>-13</b>	.7189	1.4060
2,2,5,5-Me <sub>4</sub> -hexane <sup>m</sup>	$135.8 - 136.1^n$	-11.6		1.4057
<sup>a</sup> This work. <sup>b</sup> At 760 mm., $dT/dP = 0.048^{\circ}/mm$ .				
range (20-80% d	istilled) =	0.010°. •	$d^{25} =$	0.71223.
1 10000 t Manuals and D 1 At 707 2 mm				

Tange (20-80%) is shirted -0.016 . At 727.3 mm. If n = 0.016 Hennion and Banigan, ref. 2. At 727.3 mm. Hennion and Banigan, ref. 1. At 748 mm. At 760 mm.,  $dT/dP = 0.050^{\circ}/\text{mm}$ . range (20-80% distilled)  $= 0.004^{\circ}$ .  $d^{25} = 0.71480$ .  $d^{25} = 0.40315$ . At 739 mm. Buechele, Thesis, Pennsylvania State Columbia. lege, 1937. <sup>n</sup> At 731 mm.

An examination of the experimental evidence reveals that there exists no reasonable doubt concerning the identity of the di-t-butylethylene (2,2,-5,5-tetramethyl-3-hexene) reported by each investigator. We suggest that the two sets of physical properties may correspond to the two geometric isomers, and that our low-boiling, high-melting olefin is the trans isomer, and that of Hennion and Banigan (low-melting, high-boiling) is the cis isomer. The methods of preparation strengthen this conjecture. Additional confirmation lies in the fact that the molecular model for cis-2,2,5,5tetramethyl-3-hexene shows very large steric hindrance among four of the methyl groups.

Our synthesis was carried out as follows: 2,2,5,5-Tetramethyl-3-hexanol was prepared by

- (1) Hennion and Banigan, This Journal, 68, 1202 (1946).
- (2) Moersch, Ph. D. Thesis. Pennsylvania State College, 1942.

the method described by Moersch.<sup>2</sup> Methyl tbutylacetate (6815 g., 52.4 moles) was added to tbutylmagnesium chloride (about 300 moles). The product was recovered by orthodox methods and distilled in a 30-plate column. There was obtained 6507 g. (41.2 moles, 79%) of carbinol collected at  $166-170^{\circ}$ . The carbinol was dehydrated by J. T. Baker Hydralo at 300–305° to a mixture of olefins (4999 g., 86.6%). The mixture was separated into concentrates boiling at 121–127° (2,2,5,5-tetramethyl-3-hexene, 34.5 mole per cent. of product), 139-145° (2,3,5,5-tetramethyl-1-hexene, 35.2%), and  $152-158^{\circ}$  (2,3,5,5tetramethyl-2-hexene, 30.2%). The first concentrate was redistilled in a 30-plate column, and the best portion was fractionated in a 6-foot Podbielniak column. Physical constants were measured on a selected best fraction from the latter distillation. Ozonolysis of a sample of 2,2,5,5tetramethyl-3-hexene gave only trimethylacetaldehyde (m. p. and mixed m. p. of 2,4-dinitrophenylhydrazone, 207–208°).

High-pressure hydrogenation of this olefin gave 2,2,5,5-tetramethylhexane. The other two olefins yielded 2,2,4,5-tetramethylhexane. Purification by distillation in a 6-foot Podbielniak column gave a high-purity sample for measurement<sup>3</sup> of physical constants.

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(3) Brooks, Howard and Crafton, J. Research Natl. Bur. Standards, 23, 637 (1939).

HYDROCARBON RESEARCH LABORATORY AUTOMOTIVE SECTION FRANK L. HOWARD NATIONAL BUREAU OF STANDARDS THOMAS W. MEARS Washington, D. C. A. Fookson

PHILIP POMERANTZ RECEIVED AUGUST 9, 1946

## 2,3-DIAMINOQUINOXALINE

Sir:

Our attention has been called to the publication by Gowenlock, Newbold and Spring,  $\bar{J}$ . Chem. Soc., 622 (1945), describing the preparation of 2,3diaminoquinoxaline by essentially the same procedure as that described in our recent publication, This Journal, **68**, 1035 (1946). We are extremely sorry that we neglected to include this reference in our publication.

MERCK AND CO. RAHWAY, N. J.

J. R. STEVENS K. PFISTER F. J. Wolf

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## PREPARATION OF 2-THIENYLMETHYLCARBINOL AND a-VINYLTHIOPHENE

 $\alpha$ -Vinylthiophene was obtained in quantitative yields by dehydrating 2-thienylmethylcarbinol