Acid hydrolysis of II followed by acetylation and chromatographic separation[4] yields N-methyl-α-L-glucosamine pentaacetate, $[\alpha]^{29}\text{D}$ −99 ± 2° (*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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RECEIVED AUGUST 16, 1946

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

Sir:

The properties of *sym*-di-*t*-butylethylene, as recently reported by Hennion and Banigan,[1] are not in agreement with the properties measured in these laboratories on 2,2,5,5-tetramethyl-3-hexene, 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

	B. p., °C.	F. p., °C.	d^{20}	$n^{20}\text{D}$
2,2,5,5-Me_4-3-hexene[a] (*trans*)	125.013[b]	− 4.75	0.71673[c]	1.41148[d]
2,2,5,5-Me_4-3-hexene[e] (*trans*)	123.2[f]	.....	.7176	1.4117
2,2,5,5-Me_4-3-hexene[g] (*cis*)	144.2–144.4[h]	<−65	.7439	1.4271
2,2,5,5-Me_4-hexane[a]	137.457[i]	−12.64	.71875[j]	1.40550[k]
2,2,5,5-Me_4-hexane[g]	136.2–136.4[l]	−13	.7189	1.4060
2,2,5,5-Me_4-hexane[m]	135.8–136.1[n]	−11.6		1.4057

[a] This work. [b] At 760 mm., d*T*/d*P* = 0.048°/mm. range (20–80% distilled) = 0.010°. [c] d^{25} = 0.71223. [d] $n^{25}\text{D}$ = 1.40890. [e] Moersch, ref. 2. [f] At 727.3 mm. [g] Hennion and Banigan, ref. 1. [h] At 748 mm. [i] At 760 mm., d*T*/d*P* = 0.050°/mm. range (20–80% distilled) = 0.004°. [j] d^{25} = 0.71480. [k] $n^{25}\text{D}$ = 1.40315. [l] At 739 mm. [m] Buechele, Thesis, Pennsylvania State College, 1937. [n] 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*-2,2,5,5-tetramethyl-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 the method described by Moersch.[2] Methyl *t*-butylacetate (6815 g., 52.4 moles) was added to *t*-butylmagnesium 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°. 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° (2,3,5,5-tetramethyl-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 2,2,5,5-tetramethyl-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[3] of physical constants.

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[1] Hennion and Banigan, THIS JOURNAL, **68**, 1202 (1946).

[2] Moersch, Ph. D. Thesis, Pennsylvania State College, 1942.

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

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RECEIVED AUGUST 9, 1946

2,3-DIAMINOQUINOXALINE

Sir:

Our attention has been called to the publication by Gowenlock, Newbold and Spring, *J. Chem. Soc.*, 622 (1945), describing the preparation of 2,3-diaminoquinoxaline 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.

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RECEIVED SEPTEMBER 18, 1946

PREPARATION OF 2-THIENYLMETHYLCARBINOL AND α-VINYLTHIOPHENE

Sir:

α-Vinylthiophene was obtained in quantitative yields by dehydrating 2-thienylmethylcarbinol