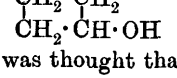
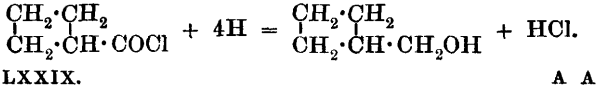
XXXIV.—*Tetramethylenecarbinol.*

By W. H. Perkin, jun.

During the course of an investigation on *hydroxytetramethylene*,



(Trans., 1894, **65**, 960), it was thought that it would be interesting to compare the properties of this substance with those of an alcohol derived from tetramethylene but containing the hydroxyl group outside the ring, and for this reason experiments were made on the reduction of the chloride of tetramethylenecarboxylic acid, in the hope that it might prove possible to prepare tetramethylenecarbinol in quantity by this means :



The result of these experiments showed that, although the reduction does proceed in this way, the yield of tetramethylenecarbinol produced is so small that a sufficiently detailed examination of its behaviour with reagents would be very difficult. It was prepared as follows.

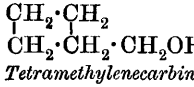
Pure ether was shaken with water until thoroughly wet and then placed on a layer of 25 per cent. caustic soda solution contained in a bottle fitted with a wide reflux condenser and standing in running water. Sodium (10 grams) cut into small pieces, was introduced all at once, and then through the top of the condenser a solution of 5 grams of tetramethylenecarboxylic chloride was run in moderately rapidly.

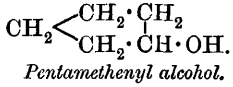
The ethereal solution from five such experiments was washed with dilute hydrochloric acid, the ether distilled off, and the residual oil, which smelt of tetramethylenealdehyde, and which had no constant boiling point, was boiled for one hour with methyl alcoholic potash in order to decompose the tetramethylenecarboxylic ester of tetramethylenecarbinol, which was evidently one of the substances formed in the reaction.

The alkaline solution was mixed with water, extracted with ether,* and the ethereal solution washed with dilute hydrochloric acid and dried over anhydrous potassium carbonate. On distilling off the ether, a small quantity of a colourless oil was obtained, which after twice fractionating distilled constantly at 143—144° under 760 mm. pressure. On analysis:

0·1912 gave 0·4872 CO_2 and 0·2026 H_2O. C = 69·5; H = 11·8.
0·1721 ,, 0·4392 CO_2 ,, 0·1818 H_2O. C = 69·6; H = 11·7.
$C_5H_{10}O$ requires C = 69·7; H = 11·6 per cent.

Tetramethylenecarbinol is a colourless oil which smells like amyl alcohol, and is somewhat soluble in water. It is isomeric with pentamethenyl alcohol, boiling at 139°, which Wislicenus and Hentschel (*Annalen*, 1893, 275, 322) obtained by the reduction of ketopentamethylene.


Tetramethylenecarbinol.


Pentamethenyl alcohol.

The determinations of the density, magnetic rotation and refractive power of tetramethylenecarbinol were carried out by W. H. Perkin, sen., with the following results:

Density: 4°/4° = 0·9162; 15°/15° = 0·9088; 25°/25° = 0·9029.

* By acidifying the alkaline solution and extracting with ether, almost the whole of the unreduced tetramethylenecarboxylic acid may be recovered.

Magnetic rotation.

t.	Sp. rotation.	Mol. rotation.
16·7°	1·0098	5·314.

The probable rotation of a substance of this composition may be calculated from the values of the elements thus:

C_5	= 0·515 × 5	= 2·575
H_{10}	= 0·254 × 10	= 2·540
O (as in alcohols)		= 0·194
Cal. rotation of tetramethylenecarbinol		= 5·309

The number thus obtained agrees well with that actually found.

Refractive power.

d 8·7°/8·7° = 0·9128.

	μ 8·7°.	$\frac{\mu-1}{d}$.	$\frac{\mu-1}{d}p$.
K	1·44096	0·48308	41·544
H_α	1·44368	0·48606	41·801
Na	1·44608	0·48869	42·027
H_β	1·45187	0·49504	42·573
H_γ	1·45645	0·50005	43·005

The calculated refractive power for H_α is obtained thus:

C_5	=	25·00
H_{10}	=	13·00
O (as in alcohols)	=	2·80
$C_5H_{10}O$	=	40·80

The value found is greater than that calculated by no less than 1·0, whereas ring compounds usually give a refractive power which is only 0·29 higher than that calculated in the above way.

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