

Table 2

Substance	No. rats per group	Wt. of substance giving same response as standard		M.W.	No. moles eqivalent to 1 mole DOCA	Mean
		Subst. (mgm.)	Standard (DOCA) (mgm.)			
(I)	5	3	0.33	176	17	16
	5	3	0.375		10	
	5	3	0.33		17	
	8-10	1	0.087		21	
(II) <i>cis</i> configuration?	5	3	0.6	182	8.2	10.7
	6	2.5	0.5		3.9	
	6	2	0.5		7.2	
	4	5	0.418		21.7	
(III) R = H		1.25	0.25	252	6.6	
(III) R = OCH <sub>3</sub>		1.25	0.5	282	2.9	
(IV)		1.25	0.5	282	2.9	

The error is of the order  $100 \pm 80$  ( $P = 0.95$ ).

Simpson (salt balance) of the Courtauld Institute of Biochemistry.

Full details of the chemistry and pharmacology will be published elsewhere.

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<sup>1</sup> Steiger and Reichstein, *Helv. Chim. Acta*, **20**, 1164 (1937).

<sup>2</sup> Selye and Schenker, *Proc. Soc. Exp. Biol., N.Y.*, **39**, 518 (1938).

### Structure of Adenylthiomethylpentose

It has been shown in a previous communication<sup>1</sup> that the structure of adenylthiomethylpentose is 9'-(5-thiomethyl-ribofuranosyl)-adenine, based on periodate titration of adenylthiomethylpentose, thio-sugar and thiopentitol, and from the result of the Boeseken test upon adenylthiomethylpentose. We have now obtained further evidence for this structural formula using synthetic methods.

Adenylthiomethylpentose gave a tritosyl derivative (m.p. 158–160°. Found: N, 9.27; calc. for C<sub>32</sub>H<sub>33</sub>O<sub>9</sub>N<sub>5</sub>S<sub>4</sub>: N, 9.21 per cent) on treatment with *p*-tosyl chloride in dry pyridine. Since none of its tosyl residue was replaced by iodine on treatment with sodium iodide in acetone during 2 hr. at 100°, it follows that there is no tosyl group at position 5 of the sugar portion.

Condensation of adenylthiomethylpentose with acetone in the presence of zinc chloride gave mono-*isopropylidene* adenylthiomethylpentose (m.p. 143°. Found: N, 19.9; calc. for C<sub>14</sub>H<sub>19</sub>O<sub>3</sub>N<sub>5</sub>S: N, 20.7 per cent. Picrate, m.p. 175°. Found: N, 19.76, S, 5.8; calc. for C<sub>14</sub>H<sub>19</sub>O<sub>3</sub>N<sub>5</sub>S.C<sub>6</sub>H<sub>3</sub>N<sub>3</sub>O<sub>7</sub>: N, 19.78, S, 5.66 per cent), from which the *isopropylidene* group was hydrolysed by dilute acid to give adenylthiomethylpentose again, though the yield was very poor.

On the other hand, monoacetone adenosine, obtained by condensing with acetone in the presence of zinc

chloride by the method of Levene and Tipson<sup>2</sup>, was converted to 5-tosylmonoacetone adenosine (found: N, 15.52; calc. for C<sub>20</sub>H<sub>23</sub>O<sub>6</sub>N<sub>5</sub>S: N, 15.18 per cent) by treating with *p*-tosyl chloride in dry pyridine at room temperature for 24 hr. Dry tosyl monoacetone adenosine and dry sodium methyl mercaptan was dissolved in acetone and the solution heated in a sealed tube at 100° for 2 hr. The reaction product (5-thiomethylmonoacetoneadenosine) after conversion into the picrate, which had a melting point of 172–174° (decomp.) (found: N, 19.3, S, 5.3; calc. for C<sub>14</sub>H<sub>19</sub>O<sub>3</sub>N<sub>5</sub>S.C<sub>6</sub>H<sub>3</sub>N<sub>3</sub>O<sub>7</sub>: N, 19.7, S, 5.6 per cent), was compared with the monoacetone derivative of the natural compound, and no melting-point depression occurred when admixed with the picrate of monoacetone adenylthiomethylpentose.

Also, from 5-tosyl-2,3-*isopropylidene* inosine<sup>3</sup> (m.p. 185°) 5-thiomethyl-2,3-*isopropylidene* inosine was obtained by heating with sodium methyl mercaptan in acetone. The crude reaction product was converted into picrate (m.p. 102° (decomp.); found: N, 16.4; calc. for C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>N<sub>4</sub>S.C<sub>6</sub>H<sub>3</sub>N<sub>3</sub>O<sub>7</sub>: N, 17.2 per cent).

Treating adenylthiomethylpentose with nitrous acid gave hypoxanthinylthiomethylpentose (m.p. 221°) as described by Kuhn and Henkel<sup>4</sup>, which yielded a monoacetone derivative (m.p. 85° (decomp.)) on condensing with acetone in the presence of zinc chloride. The picrate of the latter had a melting point of 105° (decomp.) (found: N, 16.75; calc. for C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>N<sub>4</sub>S.C<sub>6</sub>H<sub>3</sub>N<sub>3</sub>O<sub>7</sub>: N, 17.2 per cent), and showed no melting-point depression when admixed with the picrate of the monoacetone-derivative of 5-thiomethyl-inosine.

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<sup>1</sup> Satoh and Makino, *Nature*, **165**, 769 (1950).

<sup>2</sup> Levene and Tipson, *J. Biol. Chem.*, **121**, 131 (1937).

<sup>3</sup> Levene and Tipson, *J. Biol. Chem.*, **111**, 313 (1935).

<sup>4</sup> Kuhn and Henkel, *Chem. Abstr.*, **38**, 6241 (1942).

### An Isomeric Benzylideneguanosine

ALTHOUGH various investigators<sup>1</sup> have prepared benzylideneguanosine, Michelson and Todd are the only ones who report a specific rotation as well as a melting point for this compound. Recently it became desirable to prepare benzylideneguanosine in this laboratory. Using the same general method described previously<sup>1</sup>, we have obtained a benzylideneguanosine with a melting point in agreement with those already reported, but with a specific rotation which is quite different. On the other hand, benzylideneguanosine has also been prepared and has been found to be identical in physical properties with the substance described by Michelson and Todd<sup>1</sup>.

Anhydrous guanosine from yeast nucleic acid (m.p. 235° C. decomp. (all melting points are uncorrected);  $[\alpha]_D^{26} - 60.2^\circ$  in 0.1 *N* sodium hydroxide\*, *c* 3.05;  $[\alpha]_D^{21} - 34.1^\circ$  in dimethylformamide, *c* 0.122) was used as the starting material in the preparation of the benzylidene derivative.

\* This specific rotation is for guanosine containing two molecules of water of crystallization; all other specific rotations given in this communication are calculated on an anhydrous basis.