

6-Iodo-2-chloro-7-methylpurine

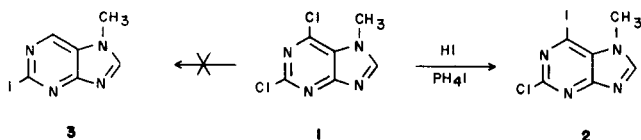
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The structure of the compound obtained upon treatment of 2,6-dichloro-7-methylpurine with hydroiodic acid and phosphonium iodide is revised.

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In 1898 Emil Fisher reported the reaction of 2,6-dichloro-7-methylpurine (**1**) with hydroiodic acid and phosphonium iodide [1]. Fisher isolated a single product with a melting point of 223° (corrected 229°) and assigned the structure of this compound as 2-iodo-7-methylpurine (**3**). This structure assignment was based on elemental analysis (Found: C, 27.80; H, 2.04; N, 21.33; I, 49.27), the reduction of this substance to 7-methylpurine with zinc dust and water, and the conversion of the product to an oxo compound (mp 323°) and to an amino compound (mp 274°, corrected 283°) with aqueous alkali and alcoholic ammonia respectively. A mercapto derivative (mp 295° dec) was also reported but not characterized.



We have repeated this reaction and have isolated the same compound that was obtained by Fisher (our melting point 214.5-216°). The ¹H nmr spectrum of this compound showed a single downfield resonance ($\delta = 8.62$) and mass spectroscopy confirmed that the compound contains one chlorine atom ($m/e = 294$ and 296). Because of the greater reactivity of the 6-position of the purine ring system, [2] we assign the structure of this compound as 2-chloro-6-iodo-7-methylpurine (**2**).

Support for our structural assignment is provided by

the amino derivative prepared by Fisher. The 6-amino-2-chloro-7-methylpurine has been prepared by at least two independent routes [3,4]. In fact, it was prepared earlier in 1898 by Fisher [3] who reports a melting point of 275°, (corrected 284°) and this is almost certainly the amino compound he reported in reference [1]. The 6-oxo- and 6-thio-2-chloro-7-methylpurines have also been prepared by an independent route but their melting points could not be determined and so comparison with the compounds prepared in reference [1] is not possible.

EXPERIMENTAL

2-Chloro-6-iodo-7-methylpurine (**2**).

2,6-Dichloro-7-methylpurine (1.00 g, Vega) was added to hydroiodic acid (10 g, 55%, Fisher) which was cooled to 0°. Phosphonium iodide (0.2 g, Pfaltz and Bauer) was added and the solution was stirred for 5 hours. Additional phosphonium iodide (0.07 g) was added and solution was allowed to stand for 2 additional hours. The solution was poured into 15 ml of water at 0° and the product collected by filtration and washed with cold water. Recrystallization from hot water gave a white solid, mp 215°, 0.63 g (52%); nmr (DMSO-*d*₆): δ 4.22 (s, CH₃), 8.89 (s, CH); ms: (relative intensity) m/e 100 (100), 199 (33), 270 (231), 294 (6.5); ir (Nujol): 3090 (CH), 1575 (CC), 1520, 1083 cm^{-1} (CCI).

Anal. Calcd. for C₆H₄ClIN₄: C, 24.48; H, 1.37; N, 19.03. Found: C, 24.46; H, 1.41; N, 18.99.

REFERENCES AND NOTES

- [1] E. Fisher, *Ber.*, **31**, 2550 (1898).
- [2] E. B. Elion and G. H. Hitchings, *J. Am. Chem. Soc.*, **78**, 3508 (1956).
- [3] E. Fisher, *Ber.*, **31**, 104 (1898).
- [4] R. R. Adams and F. C. Whitmore, *J. Am. Chem. Soc.*, **67**, 1271 (1945).