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The Identification of Drugs

VI. Nitration of Acetarsol *

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In order to obtain a suitable derivative of acetarsol (3-acetamido-4-hydroxyphenylarsonic acid) as an identity test in the Scandinavian Pharmacopoeia nitration was tried. Burschkies and Rothermundt¹ state that acetarsol treated with a mixture of concentrated nitric acid and concentrated sulphuric acid at a temperature below 0°C yields 5-nitroacetarsol (m.p. 218–220°C).

In the present work 2 M nitric acid was added to acetarsol and the solution was boiled for 15 sec and then cooled. A yellow derivative immediately crystallised and it did not contain arsenic.

We have now proved that the derivative is identical with 2-acetamido-4,6-dinitrophenol which means that besides the expected introduction of a nitro group *ortho* to the hydroxyl group another nitro group has replaced the arsonic group to give a dinitro derivative. The identity was confirmed by quantitative titration with titanous chloride and mixed melting point

determination with an authentic sample prepared by nitration of 2-acetamidophenol. Furthermore, the free phenol group of the derivative was acetylated and the resulting product was found to be identical with O,N-diacetylpicramic acid. This compound was prepared by acetylation of picramic acid.

The proposed derivative is specific for acetarsol as none of the other arsenicals used as drugs will give this derivative.

Experimental. 2-Acetamido-4,6-dinitrophenol from acetarsol. 0.1 g acetarsol is added 5 ml of 2 M nitric acid and the solution is boiled about 15 sec. and then immediately cooled with tap-water. The yellow precipitate is recrystallised from 10 ml of water. M.p. (corr.) 206–207°C (lit.² 205–206°C). Titanometric equiv. weight, found: 20.2, 20.1. Calc. for $C_8H_7O_6N_3$: 20.1. If the mixture is boiled for 5 min no precipitate is formed, probably due to an oxidation.

Acetylation of picramic acid and 2-acetamido-4,6-dinitrophenol. The substance is refluxed for 30 min with acetic anhydride and a few drops of pyridine. After cooling water is added and the precipitate is recrystallised from glacial acetic acid. Nearly colourless crystals, m.p. (corr.) 191°C (lit.² 190–191°C).

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Correction to "Least Square Method for Computer Calculations of Stability Constants" *

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- P. 187, right column, first line from above,
for $L = 35$ read $L = 46$.

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