

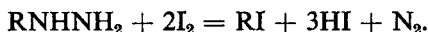
A NOTE ON THE ESTIMATION OF ISONICOTINIC ACID HYDRAZIDE

BY TEODOR CANBÄCK

From Apotekens kontrollaboratorium, Stockholm, Sweden

Received April 4, 1952

THE current interest in isonicotinic acid hydrazide has prompted the need for a rapid assay method. It is well known that hydrazines in slightly alkaline solutions are oxidised by iodine according to the scheme:—



Mono-acylhydrazides are said to behave in a different way¹:—



However, isonicotinic acid hydrazide consumes 4 equivalents of iodine when oxidised as described below. Thus the mechanism of the reaction seems to be analogous to that of hydrazines.

Isonicotinic acid hydrazide may be titrated in the following manner:— Dissolve 50 mg. of the substance in 50 ml. of water, add 1 g. of sodium bicarbonate and 25 ml. of 0.1N iodine. Allow to stand for 15 minutes. Cautiously add 10 ml. of 5N hydrochloric acid and titrate the excess of iodine with 0.1N sodium thiosulphate using starch solution as indicator. 1 ml. of 0.1N iodine corresponds to 0.003429 g. of $\text{C}_6\text{H}_7\text{ON}_3$.

Two samples of the hydrazide from different manufacturers, m.pt. 168° to 169° C. and 170° to 171° C. (Kofler), assayed 97.8, 97.8, 98.0 and 98.4, 98.6, 98.7, 98.2 per cent.

Commercial tablets containing 50 mg. of the hydrazide were analysed in a similar way, the only difference being that the suspension of the tablet powder was centrifuged before the addition of the sodium bicarbonate. The following results were obtained:—47.7, 48.1, 48.3 and 48.2 mg. per tablet.

REFERENCE

1. *Chemistry of Carbon Compounds*, edited by E. H. Rodd, Elsevier, London, 1951, 1A, 601.

Titration in non-aqueous media. References continued.

5. Lavine and Toennies, *Amer. J. med. Sci.*, 1933, **185**, 302.
6. Blumrich and Bandel, *Angew. Chem.*, 1941, **54**, 374.
7. Tomicek, *Collection. Czechoslov. Chem. Commun.*, 1948, **13**, 116.
8. Markunas and Riddick, *Anal. Chem.*, 1951, **23**, 337.
9. Pifer and Wollish, *J. Amer. pharm. Ass., Sci. Ed.*, 1951, **40**, 609.
10. Seaman and Allen, *Anal. Chem.*, 1951, **23**, 592.
11. Moore, McCutchan and Young, *ibid.*, 1951, **23**, 1639.
12. Pifer and Woolish, *ibid.*, 1952, **24**, 300.
13. Fritz, *ibid.*, 1952, **24**, 306.
14. Fritz and Keen, *ibid.*, 1952, **24**, 308.
15. Markunas and Riddick, *ibid.*, 1952, **24**, 312.
16. Terry, Eilar and Moe, *ibid.*, 1952, **24**, 313.
17. Higuchi and Concha, *J. Amer. pharm. Ass., Sci. Ed.*, 1951, **40**, 173.
18. Morton, details of this instrument are to be published later.