

NOTE**Recovery of Ammonical Salts from
Mother Liquid of 2-Methyl-5-nitroimidazole**M.K. DONGARE[†], C.V. RODE[†] and J.G. CHANDORKAR**Innovassynth Tech. (I) Ltd., Khopoli, India
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An attempt have been made to recover the ammonical salt (ammonium sulphate and ammonium nitrate) from the mother liquid of 2-methyl-5-nitroimidazole successfully.

Key Words: Recovery, 2-Methyl-5-nitroimidazole, Ammonium sulphate, Ammonium nitrate.

2-Methyl-5-nitroimidazole, a drug intermediate is widely used to manufacture the antiprotozoals like tinidazole¹, metronidazole², secnidazole³, etc. In conventional method, 2-methyl-5-nitroimidazole is manufactured by nitration of 2-methylimidazole and nitric acid in presence of fuming sulphuric acid. The product was precipitated at pH 3 with ammonia and the liquid was sent to effluent treatment plant. This mother liquid contain total dissolved solids (43 %), which are contained ammonium sulphate (42.0-42.5 %) and ammonium nitrate (1 %).

The purpose of this work is to recover the salt from the mother liquid. The mother liquid was treated with 25 % liquid ammonia and was evaporated to dryness. The salt formed was treated with activated charcoal to make almost white to white in appearance.

In modified process, the mother liquid is isolated from the product and then treated with liquid ammonia and finally evaporated the solution. The crystallized product formed are almost white coloured crystalline salt as ammonium sulphate.

During the analysis (Table-1), it is observed that ammonical nitrogen was combination of both ammonium sulphate and ammonium nitrate. As per specification of Fertilizer Control Office (FCO), India for fertilizer grade, ammonium sulphate should not exceed 0.2 % of organic content. Thus, to reduce the organic contents, liquid ammonia was added⁴ (pH 5-6). Therefore, Deverd's alloys was used to determine the total nitrogen content

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TABLE-1
PERCENTAGE OF AMMONIUM SULPHATE AND AMMONIUM
NITRATE WITHOUT TREATING THE MOTHER LIQUID

Quantity taken (mL)	Solid content (as total dissolved solids) (g)	Ammonium sulphate (%)	Ammonium nitrate (%)
500	213	99.75	0.25
500	214	99.74	0.26
500	215	99.70	0.30

in the salt. To reduce the nitrogen content resulting because of ammonium nitrate was reduced by pH of the mother liquid and then mother liquid was evaporated to slurry for crystallization (Table-2). The crystallized mass then filtered and dried at about 60-70 °C.

TABLE-2
REDUCTION PERCENTAGE OF AMMONIUM NITRATE TO
AMMONIUM SULPHATE IN SOLID CONTENT

Quantity taken (mL) (pH 5-6)	Solid content (g)	Ammonium sulphate (%)	Ammonium nitrate (%)
500	207	99.85	0.15
500	210	99.90	0.10
500	209	99.87	0.13

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