

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF NORTH DAKOTA AGRICULTURAL COLLEGE]

Nicotinyl Isothiocyanate and Some of its Derivatives

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A number of acyl isothiocyanates are known but only one derived from a heterocyclic acid has been reported.¹ This note describes another, prepared from nicotinic acid.

Experimental

Nicotinic acid nitrate was prepared by the method of McElvain² and was transformed into nicotinyl chloride by a modification of the method of Meyer and Graf.³ The nitrate was allowed to react with thionyl chloride for two hours on the steam-bath. After removal of the excess thionyl chloride, the nicotinyl chloride hydrochloride was

under vacuum and the portion boiling at 70–90° at 10 mm. was collected. Redistillation gave a 64% yield of nicotinyl chloride.

Nicotinyl isothiocyanate was prepared by allowing the acid chloride to react with ammonium thiocyanate suspended in anhydrous toluene. The mixture was stirred and heated at 125° for five minutes, after which it was quickly filtered and fractionated. A small amount of the isothiocyanate was obtained as a straw colored oil which polymerized on standing: b. p. 121° at 12 mm.; index of refraction, 1.640 at 25°. *Anal.* Calcd. for C₇H₄N₂OS: S, 19.54. Found: S, 19.83.

THIOUREAS

	Thiourea	Formula	M. p., °C.	Analyses, %			
				Calcd.		Found	
				N	S	N	S
I	α -Nicotinyl- β -phenyl-	C ₁₃ H ₁₁ N ₃ OS	154–155	16.34	12.46	15.75	12.35
II	α -Nicotinyl- β -(<i>o</i> -tolyl)-	C ₁₄ H ₁₃ N ₃ OS	160–161	15.49	11.82	15.39	11.61
III	α -Nicotinyl- β -(<i>m</i> -tolyl)-	C ₁₄ H ₁₃ N ₃ OS	149–150		11.82		12.09
IV	α -Nicotinyl- β -(<i>p</i> -tolyl)-	C ₁₄ H ₁₃ N ₃ OS	174–175	15.49	11.82	15.33	11.78
V	β -(α -Naphthyl)- α -nicotinyl-	C ₁₇ H ₁₃ N ₃ OS	171–172	13.68		13.76	
VI	β -(β -Naphthyl)- α -nicotinyl-	C ₁₇ H ₁₃ N ₃ OS	193–194	13.68	10.44	13.34	10.69
VII	α -Methyl- β -nicotinyl- α -phenyl-	C ₁₄ H ₁₃ N ₃ OS	156–157	15.49		15.31	
VIII	α -Ethyl- β -nicotinyl- α -phenyl-	C ₁₅ H ₁₅ N ₃ OS	123–124		11.24		11.46
IX	β -Nicotinyl- α , α -diphenyl-	C ₁₉ H ₁₅ N ₃ OS	162–163		9.62		10.02
X	α -Benzyl- β -nicotinyl- α -phenyl-	C ₂₀ H ₁₇ N ₃ OS	150–151	12.10		11.81	
XI	Nicotinyl-	C ₇ H ₇ N ₃ OS	209–210		17.70		17.53

decomposed by an equivalent amount of anhydrous pyridine. The mixture was fractionated

Nicotinyl thioureas were prepared from acetone solutions of the mustard oil by a method already described.¹

(1) Douglass and Dains, *THIS JOURNAL*, **56**, 719 (1934).

(2) "Organic Syntheses," 1924, Vol. IV, p. 49; Coll. Vol. I, p. 378.

(3) Meyer and Graf, *Ber.*, **61B**, 2205–2206 (1928).