[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 McElvain[2] 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 decomposed by an equivalent amount of anhydrous pyridine. The mixture was fractionated 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 $C_7H_4N_2OS$: S, 19.54. Found: S, 19.83.

THIOUREAS

	Thiourea	Formula	M. p., °C.	Analyses, % Calcd. N	Calcd. S	Found N	Found S
I	α-Nicotinyl-β-phenyl-	$C_{13}H_{11}N_3OS$	154–155	16.34	12.46	15.75	12.35
II	α-Nicotinyl-β-(*o*-tolyl)-	$C_{14}H_{13}N_3OS$	160–161	15.49	11.82	15.39	11.61
III	α-Nicotinyl-β-(*m*-tolyl)-	$C_{14}H_{13}N_3OS$	149–150		11.82		12.09
IV	α-Nicotinyl-β-(*p*-tolyl)-	$C_{14}H_{13}N_3OS$	174–175	15.49	11.82	15.33	11.78
V	β-(α-Naphthyl)-α-nicotinyl-	$C_{17}H_{13}N_3OS$	171–172	13.68		13.76	
VI	β-(β-Naphthyl)-α-nicotinyl-	$C_{17}H_{13}N_3OS$	193–194	13.68	10.44	13.34	10.69
VII	α-Methyl-β-nicotinyl-α-phenyl-	$C_{14}H_{13}N_3OS$	156–157	15.49		15.31	
VIII	α-Ethyl-β-nicotinyl-α-phenyl-	$C_{15}H_{15}N_3OS$	123–124		11.24		11.46
IX	β-Nicotinyl-α,α-diphenyl-	$C_{19}H_{15}N_3OS$	162–163		9.62		10.02
X	α-Benzyl-β-nicotinyl-α-phenyl-	$C_{20}H_{17}N_3OS$	150–151	12.10		11.81	
XI	Nicotinyl-	$C_7H_7N_3OS$	209–210		17.70		17.53

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).

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