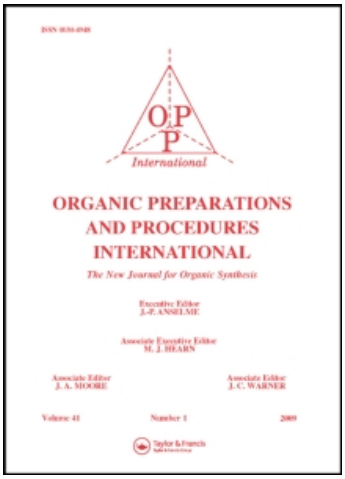

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SOME 5,6- AND 2,5-DIHALONICOTINAMIDES

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yl-3-piperidone (VI), bp. 87-90°/38 mm; HCl salt, mp. 109-111°, lit.[6] mp. 110-111°.

^{1}H NMR ($DMSO_6$): δ 2.78 (s, 2H), 2.21 (s, 3H), 2.65-1.7 (m, 6H). IR (film): ν_{max} 2940, 2775, 1720, 1452, 1140 cm^{-1}.

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SOME 5,6- AND 2,5-DIHALONICOTINAMIDES

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(8/20/79)

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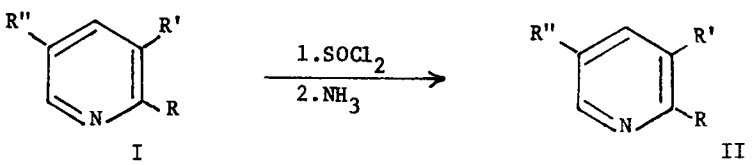
A number of dihalonicotinamides (IIa-f) have been prepared.



I II

I	II
a) R = Cl; R' = Br; R'' = CO_2H	a) R = Cl; R' = Br; R'' = $CONH_2$
b) R = Cl; R' = Cl; R'' = CO_2H	b) R = Cl; R' = Cl; R'' = $CONH_2$
c) R = Cl; R' = CO_2H; R'' = Br	c) R = Cl; R' = $CONH_2$; R'' = Br
d) R = Cl; R' = CO_2H; R'' = Cl	d) R = Cl; R' = $CONH_2$; R'' = Cl
e) R = Cl; R' = CO_2H; R'' = I	e) R = Cl; R' = $CONH_2$; R'' = I
f) R = Cl; R' = CO_2H; R'' = F	f) R = Cl; R' = $CONH_2$; R'' = F

Table 1. Data on Dihalonicotinamides[a]

II.	Mp. (°C)	Yield (%)	Elemental Analyses C	H	N	Found C	H	N
a	237-238°	73	30.57	1.69	11.89	30.53	1.67	11.82
b	222-224°	56	37.70	2.09	14.66	37.52	2.14	14.45
c	187-188°	58	30.57	1.69	11.89	30.67	1.65	11.94
d	176-177°	61	37.70	2.09	14.66	37.59	2.17	14.44
e	184-185°	59	25.50	1.42	9.91	25.27	1.36	9.80
f	137-139°[b]	35	41.26	2.29	16.05	41.05	2.39	15.94

a. Mps. and yields are those of analytical samples obtained by recrystallization from aqueous ethanol; Mps. are uncorrected. Elemental analyses were performed by Galbraith Laboratories, Knoxville, Tennessee. The starting acids (Ia-f) were obtained as previously described [J. Chem. Eng. Data, 15, 590 (1970); 17, 515 (1972); 21, 246 (1976)].
b. Recrystallized from water.

Typical Procedure. 5-Bromo-6-chloronicotinamide (IIa).-5-Bromo-6-chloronicotinic acid (Ia, 2.4g; 0.01 mole) and thionyl chloride (12ml) were mixed and stirred under gentle reflux for 2 hr. The reaction mixture was then cooled to room temperature, and the excess thionyl chloride was removed on a rotary evaporator. The crude acid chloride (heavy oil) was quickly dissolved in dry chloroform (25ml) and the resulting solution was transferred to a small flask with a gas inlet port. The chloroform solution was cooled to 10° with stirring while a slow stream of anhydrous ammonia was passed in for approximately 5 min. The crude amide which precipitated was recrystallized from aqueous ethanol to yield 1.75g (73%) of pure IIa, mp. 237-238°.