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## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

### SOME 5,6- AND 2,5-DIHALONICOTINAMIDES

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**To cite this Article** Setliff, Frank L. and Hill, John F.(1980) 'SOME 5,6- AND 2,5-DIHALONICOTINAMIDES', *Organic Preparations and Procedures International*, 12: 3, 259 – 260

**To link to this Article:** DOI: 10.1080/00304948009458567

**URL:** <http://dx.doi.org/10.1080/00304948009458567>

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

<sup>1</sup>H NMR (DMSO<sub>6</sub>): δ 2.78 (s, 2H), 2.21 (s, 3H), 2.65-1.7 (m, 6H). IR (film):  
 $\nu_{\max}$  2940, 2775, 1720, 1452, 1140 cm<sup>-1</sup>.

**Acknowledgements.**— This work was supported in part by grants from the American Cancer Society (CH-57); The Robert A. Welch Foundation (B-702); and North Texas State University Faculty Research.

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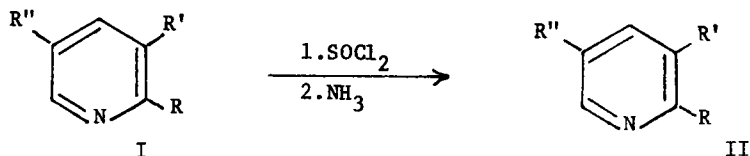
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## SOME 5,6- AND 2,5-DIHALONICOTINAMIDES

Submitted by Frank L. Setliff\* and John F. Hill  
(8/20/79)

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



- | I   | II   |
|---|--|
| a) R = Cl; R' = Br; R'' = CO <sub>2</sub> H | a) R = Cl; R' = Br; R'' = CONH <sub>2</sub>  |
| b) R = Cl; R' = Cl; R'' = CO <sub>2</sub> H | b) R = Cl; R' = Cl; R'' = CONH <sub>2</sub>  |
| c) R = Cl; R' = CO <sub>2</sub> H; R'' = Br | c) R = Cl; R' = CONH <sub>2</sub> ; R'' = Br |
| d) R = Cl; R' = CO <sub>2</sub> H; R'' = Cl | d) R = Cl; R' = CONH <sub>2</sub> ; R'' = Cl |
| e) R = Cl; R' = CO <sub>2</sub> H; R'' = I  | e) R = Cl; R' = CONH <sub>2</sub> ; R'' = I  |
| f) R = Cl; R' = CO <sub>2</sub> H; R'' = F  | f) R = Cl; R' = CONH <sub>2</sub> ; R'' = F  |

Table 1. Data on Dihalonicotinamides<sup>a</sup>

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