3,4,5-Triiodobenzamides as Derivatives of Amines

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3,4,5-Triiodobenzoyl chloride has been utilized in the identification of cellosolves, carbitols (1), alcohols (2) and mercaptans (3). This acid chloride also appears to be ideally suited for the characterization of amines. It may be stored for relatively long periods of time; therefore, preparation immediately before use is not required. In addition, the reaction is rapid, the yields are excellent and the resulting amides are readily crystallized. Table I contains the pertinent data for the amines that were acylated.

EXPERIMENTAL

3,4,5-Triiodobenzoyl chloride was prepared by the method of Klemme and Hunter (4).

Preparation of 3,4,5-Triiodobenzamides. To a solution of 0.5 gram of I in 2 ml. of dioxane was added 1.5 millimoles of solid amine or 1.0 ml. of liquid amine. The resulting mixture was refluxed for 10 min. In those cases when a solid remained after heating, the reaction mixture was filtered with suction and the solid washed with warm 10% hydrochloric acid. When the amide was soluble in dioxane the solution was poured into 30 ml. of cold 10% hydrochloric acid. In most cases this resulted in the formation of a white granular solid or an oil which solidified within a few minutes. In a few instances it was necessary to allow the oil to remain overnight suspended in a 10% hydrochloric acid solution to effect solidification. All solids were washed with water before recrystallization.

LITERATURE CITED

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Table I. 3,4,5-Triiodobenzamides

Amine	M.P. of amide, ° C.[a]	Yield, %	Analysis, %[b] C Calc.	C Found	H Calcd.	H Found
n-Propylamine	206–206.5[c]	91	22.20	22.11	1.86	2.00
iso-Propylamine	243–243.5[c]	93	22.20	22.40	1.86	1.88
Diethylamine	148.5–149[c]	97	23.80	23.95	2.16	2.35
n-Butylamine	198.3–199[c]	95	23.80	24.02	2.16	2.20
iso-Butylamine	210.5–211[c]	97	23.80	24.07	2.16	2.32
tert-Butylamine	230–230.5[d]	95	23.80	23.99	2.16	2.42
Diallylamine	112.0–112.5[d]	82	26.97	26.62	2.09	2.20
Di-*n*-propylamine	124.7–125.5[d]	96	26.78	26.94	2.77	2.84
Di-isopropylamine	209.8–210[c]	85	26.78	26.83	2.77	2.81
Di-*n*-butylamine	118.5–119.2[d]	93	29.48	29.36	3.30	3.32
Di-isobtylamine	198.5–199[c]	93	29.48	29.57	3.30	3.37
Cyclohexylamine	243.5–244[c]	91	26.87	26.80	2.43	2.50
Pyrrolidine	223.5–224[c]	92	23.89	24.17	1.82	1.99
Morpholine	236.3–237[c]	91	23.22	23.29	1.77	1.85
2-Methylpiperidine	147.5–148[d]	95	26.82	26.82	2.43	2.56
Piperazine	310–310.2[e,f]	85	20.59	20.76	1.15	1.33
Benzylamine	211.8–212.2[c]	97	28.55	28.79	1.71	1.95
α-Phenylethylamine	208–208.5[c]	90	29.87	30.10	2.00	2.07
Aniline	251.2–252[c]	98	27.16	26.92	1.40	1.55
o-Toluidine	266.8–267.5[c]	97	28.55	28.66	1.71	1.83
m-Toluidine	247.8–248.5[c]	98	28.55	28.78	1.71	1.88
m-Anisidine	224.2–224.8[g]	95	27.79	27.60	1.67	1.69
Ethyl *p*-aminobenzoate	222.2–222.8[c]	90	29.70	29.98	1.87	2.00
N-Methyl-*o*-toluidine	130.5–131[d]	87	29.87	29.80	2.01	2.22
N-Methyl-*m*-toluidine	128–128.5[d]	85	29.87	30.07	2.01	2.11

[a] All melting points are corrected. [b] Analyses run by Schwarzkopf Microanalytical Laboratory, Woodside, N. Y. [c] Recrystallized from 95% ethanol. [d] Recrystallized from ethanol-water mixture. [e] Recrystallized from ethanol-pyridine mixture. [f] Data given for diamide. [g] Recrystallized from *n*-propyl alcohol.