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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- (3) O'Donnell, D.C., Mariani, H.A., Downing, D.J., ibid. 77, 3154 (1955).
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Table I. 3,4,5-Triiodobenzamides

Amineof amide, °C.°Yield, °C.°Calc.FoundCalcd.Found n -Propylamine $206-206.5^\circ$ 91 22.20 22.11 1.86 2.00 iso-Propylamine $243-243.5^\circ$ 93 22.20 22.40 1.86 1.88 Diethylamine $148.5-149^\circ$ 97 23.80 23.95 2.16 2.35 n -Butylamine $198.3-199^\circ$ 95 23.80 24.02 2.16 2.20 iso-Butylamine $210.5-211^\circ$ 97 23.80 24.07 2.16 2.32 tert-Butylamine $230-230.5^\circ$ 95 23.80 24.07 2.16 2.32 Diallylamine $112.0-112.5^\circ$ 82 26.97 26.62 2.09 2.20 Di- n -propylamine $124.7-125.5^\circ$ 96 26.78 26.94 2.77 2.84 Di-isopropylamine $129.8-119^\circ$ 93 29.48 29.36 3.30 3.32 Di-isobtylamine $198.5-199^\circ$ 93 29.48 29.36 3.30 3.32 Cyclohexylamine $243.5-244^\circ$ 91 26.87 26.80 2.43 2.50 Pyrrolidine $223.5-224^\circ$ 92 23.89 24.17 1.82 1.99 Morpholine $236.3-237^\circ$ 91 23.22 23.29 1.77 1.85 2 -Methylpiperidine $147.5-148^\circ$ 95 26.82 26.82 24.3 2.56 Piperazine $310-310.2^{\circ 4}$ 85 20.59 20.76 <t< th=""><th></th><th></th><th></th><th colspan="4">Analysis, %^b</th></t<>				Analysis, % ^b			
Amineof amide, $^{\circ}$ C. $^{\circ}$ %Calc.FoundCalcd.Foundn-Propylamine $206-206.5^{\circ}$ 91 22.20 22.11 1.86 2.00 iso-Propylamine $243-243.5^{\circ}$ 93 22.20 22.40 1.86 1.88 Diethylamine $148.5-149^{\circ}$ 97 23.80 23.95 2.16 2.35 n-Butylamine $198.3-199^{\circ}$ 95 23.80 24.02 2.16 2.20 iso-Butylamine $210.5-211^{\circ}$ 97 23.80 24.07 2.16 2.32 tert-Butylamine $230-230.5^{\circ}$ 95 23.80 23.99 2.16 2.42 Diallylamine $112.0-112.5^{\circ}$ 82 26.97 26.62 2.09 2.20 Di-n-propylamine $124.7-125.5^{\circ}$ 96 26.78 26.82 2.77 2.84 Di-isopropylamine $209.8-210^{\circ}$ 85 26.78 26.83 2.77 2.81 Di-n-butylamine $118.5-119.2^{\circ}$ 93 29.48 29.36 3.30 3.32 Di-isobtylamine $198.5-199^{\circ}$ 93 29.48 29.57 3.30 3.37 Cyclohexylamine $223.5-224^{\circ}$ 91 26.87 26.80 2.43 2.50 Pyrrolidine $223.5-224^{\circ}$ 92 23.89 24.17 1.82 1.99 Morpholine $236.3-237^{\circ}$ 91 23.22 23.29 1.77 1.85 2-Methylpiperidine $147.5-148^{\circ}$ 95 26.82 26.82 2		M.P.	Yield.	С		Н	
iso-Propylamine $243-243.5^{\circ}$ 93 22.20 22.40 1.86 1.88 Diethylamine $148.5-149^{\circ}$ 97 23.80 23.95 2.16 2.35 n -Butylamine $198.3-199^{\circ}$ 95 23.80 24.02 2.16 2.20 iso-Butylamine $210.5-211^{\circ}$ 97 23.80 24.07 2.16 2.32 tert-Butylamine $230-230.5^{\circ}$ 95 23.80 24.07 2.16 2.32 Diallylamine $112.0-112.5^{\circ}$ 82 26.87 26.62 2.09 2.20 Di-n-propylamine $124.7-125.5^{\circ}$ 96 26.78 26.94 2.77 2.84 Di-isopropylamine $20.8-210^{\circ}$ 85 26.78 26.83 2.77 2.84 Di-isobtylamine $118.5-119.2^{\circ}$ 93 29.48 29.36 3.30 3.32 Di-isobtylamine $198.5-199^{\circ}$ 93 29.48 29.57 3.30 3.37	Amine			Calc.	Found	Calcd.	Found
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 $120.5-211^c$ 95 23.80 24.07 2.16 2.32 Dialylamine $112.0-112.5^d$ 82 26.97 26.62 2.09 2.20 Di-n-propylamine $124.7-125.5^d$ 96 26.78 26.83 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 $198.5-199^c$ 93 29.48 29.57 3.30 3.37 Cyclohexylam	n-Propylamine	$206-206.5^{\circ}$	91	22.20	22.11	1.86	2.00
n-Butylamine 198.3–199° 95 23.80 24.02 2.16 2.20 iso-Butylamine 210.5–211° 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 26.84 2.77 2.84 Di-isopropylamine 209.8–210° 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° 93 29.48 29.36 3.30 3.37 Cyclohexylamine 198.5–199° 93 29.48 29.57 3.30 3.37 Cyclohexylamine 243.5–244° 91 26.87 26.80 2.43 2.50 Pyrrolidine 223.5–224° 92 23.89 24.17 </td <td>iso-Propylamine</td> <td>$243-243.5^{\circ}$</td> <td>93</td> <td>22.20</td> <td>22.40</td> <td>1.86</td> <td>1.88</td>	iso-Propylamine	$243-243.5^{\circ}$	93	22.20	22.40	1.86	1.88
iso-Butylamine $210.5-211^{\circ}$ 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 $209.8-210^{\circ}$ 85 26.78 26.83 2.77 2.84 Di-isopropylamine $118.5-119.2^d$ 93 29.48 29.36 3.30 3.32 Di-isobtylamine $198.5-199^{\circ}$ 93 29.48 29.57 3.30 3.32 Cyclohexylamine $243.5-244^{\circ}$ 91 26.87 26.80 2.43 2.50 Pyrrolidine $223.5-224^{\circ}$ 91 23.22 23.29 1.77 1.82 1.99 Morpholine $236.3-237^{\circ}$ 91 23.22 23.29 1.77 1.85 <td>Diethylamine</td> <td>148.5-149°</td> <td>97</td> <td>23.80</td> <td>23.95</td> <td>2.16</td> <td>2.35</td>	Diethylamine	148.5-149°	97	23.80	23.95	2.16	2.35
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 $310-310.2^{e/}$ 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 σ -Toluidine $247.8-248.5^c$ 98 28.55 28.78 1.71 1.88 m -Anisidine $224.2-224.8^d$ 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- σ -toluidine $130.5-131^d$ 87 29.87 29.80 2.01	n-Butylamine	$198.3 - 199^{\circ}$	95	23.80	24.02	2.16	2.20
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	iso-Butylamine	210.5-211°	97	23.80	24.07	2.16	2.32
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	tert-Butylamine	$230-230.5^{d}$	95	23.80	23.99	2.16	2.42
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		$112.0-112.5^d$	82	26.97	26.62	2.09	2.20
Di-isopropylamine $209.8-210^{\circ}$ 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 $198.5-199^{\circ}$ 93 29.48 29.57 3.30 3.37 Cyclohexylamine $243.5-244^{\circ}$ 91 26.87 26.80 2.43 2.50 Pyrrolidine $223.5-224^{\circ}$ 92 23.89 24.17 1.82 1.99 Morpholine $236.3-237^{\circ}$ 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 $310-310.2^{e/}$ 85 20.59 20.76 1.15 1.33 Benzylamine $211.8-212.2^{\circ}$ 97 28.55 28.79 1.71 1.95 α -Phenylethylamine $208-208.5^{\circ}$ 90 29.87 30.10 2.00 2.07	Di-n-propylamine	$124.7 - 125.5^d$	96	26.78	26.94	2.77	2.84
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		$209.8 - 210^{\circ}$	85	26.78	26.83	2.77	2.81
$ \begin{array}{c} \text{Cyclohexylamine} & 243.5-244^c & 91 & 26.87 & 26.80 & 2.43 & 2.50 \\ \text{Pyrrolidine} & 223.5-224^c & 92 & 23.89 & 24.17 & 1.82 & 1.99 \\ \text{Morpholine} & 236.3-237^c & 91 & 23.22 & 23.29 & 1.77 & 1.85 \\ \text{2-Methylpiperidine} & 147.5-148^d & 95 & 26.82 & 26.82 & 2.43 & 2.56 \\ \text{Piperazine} & 310-310.2^{\epsilon/} & 85 & 20.59 & 20.76 & 1.15 & 1.33 \\ \text{Benzylamine} & 211.8-212.2^c & 97 & 28.55 & 28.79 & 1.71 & 1.95 \\ \alpha\text{-Phenylethylamine} & 208-208.5^c & 90 & 29.87 & 30.10 & 2.00 & 2.07 \\ \text{Aniline} & 251.2-252^c & 98 & 27.16 & 26.92 & 1.40 & 1.55 \\ o\text{-Toluidine} & 266.8-267.5^c & 97 & 28.55 & 28.66 & 1.71 & 1.83 \\ m\text{-Toluidine} & 247.8-248.5^c & 98 & 28.55 & 28.78 & 1.71 & 1.88 \\ m\text{-Anisidine} & 224.2-224.8^d & 95 & 27.79 & 27.60 & 1.67 & 1.69 \\ \text{Ethyl p-aminobenzoate} & 222.2-222.8^c & 90 & 29.70 & 29.98 & 1.87 & 2.00 \\ N\text{-Methyl-o-toluidine} & 130.5-131^d & 87 & 29.87 & 29.80 & 2.01 & 2.22 \\ \end{array} $		$118.5 - 119.2^d$	93	29.48	29.36	3.30	3.32
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 $310-310.2^{e/l}$ 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	Di-isobtylamine	$198.5 - 199^{\circ}$	93	29.48	29.57	3.30	3.37
Morpholine 236.3–237° 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 $310-310.2^{e/}$ 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 ο-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 $224.2-224.8^d$ 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		243.5-244°	91	26.87	26.80	2.43	2.50
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Pyrrolidine	$223.5 - 224^{\circ}$	92	23.89	24.17	1.82	1.99
Piperazine $310-310.2^{e/}$ 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 28.66 1.71 1.88 m -Anisidine $224.2-224.8^d$ 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	Morpholine	$236.3 - 237^{\circ}$	91	23.22	23.29	1.77	1.85
Piperazine $310-310.2^{e/}$ 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 $224.2-224.8^d$ 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	2-Methylpiperidine	$147.5 - 148^d$	95	26.82	26.82	2.43	2.56
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		$310-310.2^{e,f}$	85	20.59	20.76	1.15	1.33
Aniline $251.2-252^{\circ}$ 98 27.16 26.92 1.40 1.55 o-Toluidine $266.8-267.5^{\circ}$ 97 28.55 28.66 1.71 1.83 m-Toluidine $247.8-248.5^{\circ}$ 98 28.55 28.78 1.71 1.88 m-Anisidine $224.2-224.8^{s}$ 95 27.79 27.60 1.67 1.69 Ethyl p -aminobenzoate $222.2-222.8^{\circ}$ 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	Benzylamine	$211.8 - 212.2^{\circ}$	97	28.55	28.79	1.71	1.95
o-Toluidine $266.8-267.5^{\circ}$ 97 28.55 28.66 1.71 1.83 m-Toluidine $247.8-248.5^{\circ}$ 98 28.55 28.78 1.71 1.88 m-Anisidine $224.2-224.8^{s}$ 95 27.79 27.60 1.67 1.69 Ethyl p-aminobenzoate $222.2-222.8^{\circ}$ 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	α -Phenylethylamine	$208-208.5^{\circ}$	90	29.87	30.10		2.07
m -Toluidine $247.8-248.5^{\circ}$ 98 28.55 28.78 1.71 1.88 m -Anisidine $224.2-224.8^{\circ}$ 95 27.79 27.60 1.67 1.69 Ethyl p -aminobenzoate $222.2-222.8^{\circ}$ 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	Aniline	$251.2 - 252^{\circ}$	98	27.16	26.92	1.40	1.55
m -Anisidine $224.2-224.8^s$ 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	o-Toluidine	$266.8 - 267.5^{\circ}$	97	28.55	28.66	1.71	1.83
Ethyl p-aminobenzoate 222.2-222.8° 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	m-Toluidine	$247.8 - 248.5^{\circ}$	98	28.55	28.78		
N-Methyl-o-toluidine 130.5–131 ^d 87 29.87 29.80 2.01 2.22	m-Anisidine	$224.2 - 224.8^s$	95	27.79	27.60	1.67	1.69
1. Divily a delatation to the second	Ethyl p-aminobenzoate	222.2-222.8°	90	29.70	29.98		
N Mothyl m toluiding $128.128.5^d$ 85 29.87 30.07 2.01 2.11	N-Methyl- o -toluidine		87	29.87	29.80		
17-14-coldidite 120-120.0 00 25.01 00.01 2.01 2.11	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. 'Recrystallized from 95% ethanol. ^d Recrystallized from ethanol-water mixture. ^e Re-

crystallized from ethanol-pyridine mixture. $^{\prime}$ Data given for diamide. $^{\ell}$ Recrystallized from n-propyl alcohol.