

# Phenol Esters of 2,3,5-Triiodobenzoic Acid

DAVID C. O'DONNELL, FRANCIS G. FALVEY, and ALFRED C. MOLLA, JR.

Department of Chemistry, Boston College, Chestnut Hill, Mass.

The 2,3,5-triiodobenzoic esters of thirty-seven phenols have been prepared by reacting the various phenols with 2,3,5-triiodobenzoyl chloride.

PHENOL ESTERS of 3,4,5-triiodobenzoic acid were reported (2) and the phenol esters of 2,3,5-triiodobenzoic acid have now been prepared.

## EXPERIMENTAL

The phenols were used as obtained from commercial sources. The acid chloride was prepared by the method of Klemme and Hunter (1) except that cyclohexane was used for crystallization.

The method described in (3) was used except that 2 ml. of pyridine was added to the reaction mixture. Unless otherwise indicated in Table I, 1-butanol was used as the solvent.

## LITERATURE CITED

- (1) Klemm, C.J., Hunter, J.H., *J. Org. Chem.* **5**, 508 (1940).
- (2) O'Donnell, D.C., Isaacs, V.A., Kiely, L.E., Millard, R.J., Welchlin, J.A., *J. CHEM. ENG. DATA* **8**, 608 (1963).
- (3) O'Donnell, D.C., Kelley, J.K., Jr., O'Malley, R.F., Upham, R.H., *J. Am. Chem. Soc.* **70**, 1657 (1948).

RECEIVED for review June 22, 1964. Accepted October 20, 1964. This work is taken from theses submitted by Francis G. Falvey and Alfred C. Molla, Jr., in partial fulfillment for the M.S. degree at Boston College, Chestnut Hill, Mass.

Table I. Phenol Esters of 2,3,5-Triiodobenzoic Acid

Phenol Used	M.P., °C.	Yield, %	Formula	Iodine, %	
				Calcd.	Found
2-Aceto-1-naphthol	181.5-182.0	27	C <sub>19</sub> H <sub>13</sub> O <sub>3</sub> I <sub>3</sub>	57.0	57.02
Acetyl- <i>p</i> -methylaminophenol	172.4-173.8 <sup>a</sup>	19	C <sub>16</sub> H <sub>12</sub> O <sub>3</sub> NI <sub>3</sub>	58.85	59.30
<i>p</i> -Benzylphenol	147.4-148.2	50	C <sub>20</sub> H <sub>13</sub> O <sub>2</sub> I <sub>3</sub>	57.16	57.44
<i>p</i> -Bromphenol	162.8-163.8 <sup>b</sup>	36	C <sub>13</sub> H <sub>6</sub> O <sub>2</sub> BrI <sub>3</sub>	58.14	57.73
Catechol	174.6-175.2 <sup>a</sup>	42	C <sub>20</sub> H <sub>8</sub> O <sub>4</sub> I <sub>6</sub>	70.92	71.01
2-Chloro-5-hydroxytoluene	156.6-157.4	58	C <sub>14</sub> H <sub>9</sub> O <sub>2</sub> ClI <sub>3</sub>	60.98	61.43
<i>o</i> -Chlorophenol	139.3-140.6 <sup>b</sup>	64	C <sub>13</sub> H <sub>6</sub> O <sub>2</sub> ClI <sub>3</sub>	62.39	62.10
<i>m</i> -Chlorophenol	130.6-132.3 <sup>b</sup>	21	C <sub>13</sub> H <sub>6</sub> O <sub>2</sub> ClI <sub>3</sub>	62.39	62.17
<i>p</i> -Chlorothymol	124.8-125.4 <sup>c</sup>	52	C <sub>17</sub> H <sub>14</sub> O <sub>2</sub> ClI <sub>3</sub>	57.14	57.52
2,4-Dichloro-1-naphthol	234.0-235.2 <sup>d</sup>	37	C <sub>17</sub> H <sub>7</sub> O <sub>2</sub> Cl <sub>2</sub> I <sub>3</sub>	54.81	54.48
2,6-Dichloro-4-nitrophenol	157.0-158.6	30	C <sub>13</sub> H <sub>6</sub> O <sub>4</sub> NCl <sub>2</sub> I <sub>3</sub>	55.19	54.96
2,4-Dichlorophenol	170.8-172.2 <sup>b</sup>	32	C <sub>13</sub> H <sub>5</sub> O <sub>2</sub> Cl <sub>2</sub> I <sub>3</sub>	59.05	59.12
2,4-Dihydroxybenzaldehyde	175.5-176.2	27	C <sub>21</sub> H <sub>8</sub> O <sub>5</sub> I <sub>6</sub>	69.10	68.58
2,2'-Dihydroxybinaphthyl-1,1'	259.4-260.4 <sup>d</sup>	42	C <sub>34</sub> H <sub>16</sub> O <sub>4</sub> I <sub>6</sub>	60.92	61.25
2,7-Dihydroxynaphthalene	218.0-220.8 <sup>e</sup>	31	C <sub>24</sub> H <sub>10</sub> O <sub>4</sub> I <sub>6</sub>	67.76	67.87
2,5-Dihydroxytoluene	268.0-269.8 <sup>f</sup>	25	C <sub>21</sub> H <sub>10</sub> O <sub>4</sub> I <sub>6</sub>	70.00	69.90
3,5-Dinitro- <i>o</i> -cresol	171.0-171.8	18	C <sub>14</sub> H <sub>6</sub> O <sub>6</sub> N <sub>2</sub> I <sub>3</sub>	56.00	56.49
2,4-Dinitrophenol	154.6-155.8 <sup>b</sup>	31	C <sub>13</sub> H <sub>5</sub> O <sub>6</sub> N <sub>2</sub> I <sub>3</sub>	57.18	57.40
Eugenol	123.5-125.0 <sup>b</sup>	45	C <sub>17</sub> H <sub>13</sub> O <sub>3</sub> I <sub>3</sub>	58.94	58.81
Hydroquinone monobenzylether	137.7-138.6	45	C <sub>20</sub> H <sub>13</sub> O <sub>3</sub> I <sub>3</sub>	55.85	56.01
Hydroquinone monomethylether	152.0-154.0 <sup>b</sup>	36	C <sub>14</sub> H <sub>9</sub> O <sub>3</sub> I <sub>3</sub>	62.84	63.01
4-Hydroxy-1,2-dimethylbenzene	137.0-137.6	49	C <sub>15</sub> C <sub>11</sub> O <sub>2</sub> I <sub>3</sub>	63.05	63.34
4-Hydroxy-1,3-dimethylbenzene	125.0-126.4	47	C <sub>15</sub> H <sub>11</sub> O <sub>2</sub> I <sub>3</sub>	63.05	62.90
2-Hydroxy-1,4-dimethylbenzene	112.6-113.4 <sup>c</sup>	55	C <sub>15</sub> H <sub>11</sub> O <sub>2</sub> I <sub>3</sub>	63.05	62.80
<i>o</i> -Hydroxydiphenyl	143.0-145.0	56	C <sub>19</sub> H <sub>11</sub> O <sub>2</sub> I <sub>3</sub>	58.39	58.50
<i>p</i> -Hydroxydiphenyl	169.8-170.6	64	C <sub>19</sub> H <sub>11</sub> O <sub>2</sub> I <sub>3</sub>	58.39	58.87
<i>o</i> -Iodophenol	183.6-185.6 <sup>b</sup>	70	C <sub>13</sub> H <sub>6</sub> O <sub>2</sub> I <sub>4</sub>	72.32	72.13
1-Naphthol	145.2-147.2	42	C <sub>17</sub> H <sub>8</sub> O <sub>2</sub> I <sub>3</sub>	60.82	60.82
2-Naphthol	157.2-158.0 <sup>b</sup>	33	C <sub>17</sub> H <sub>8</sub> O <sub>2</sub> I <sub>3</sub>	60.82	60.80
<i>o</i> -Nitrophenol	192.8-193.6	67	C <sub>13</sub> H <sub>6</sub> O <sub>4</sub> NI <sub>3</sub>	61.34	61.01
<i>m</i> -Nitrophenol	176.2-177.2 <sup>b</sup>	34	C <sub>13</sub> H <sub>6</sub> O <sub>4</sub> NI <sub>3</sub>	61.34	61.21
<i>p</i> -Nitrophenol	179.4-180.4	42	C <sub>13</sub> H <sub>6</sub> O <sub>4</sub> NI <sub>3</sub>	61.34	61.48
Phenol	132.4-133.3 <sup>c</sup>	71	C <sub>13</sub> H <sub>7</sub> O <sub>2</sub> I <sub>3</sub>	66.11	66.22
Pyrogallol-1,3-dimethyl ether	147.2-148.0	57	C <sub>15</sub> H <sub>10</sub> O <sub>3</sub> I <sub>3</sub>	59.87	59.74
Resorcinol monoethyl ether	106.0-106.8	29	C <sub>15</sub> H <sub>11</sub> O <sub>3</sub> I <sub>3</sub>	61.41	61.30
Thymol	98.6-100.6	29	C <sub>17</sub> H <sub>13</sub> O <sub>2</sub> I <sub>3</sub>	60.24	60.40
2,4,6-Trichlorophenol	148.4-149.8 <sup>b</sup>	46	C <sub>13</sub> H <sub>4</sub> O <sub>2</sub> Cl <sub>3</sub> I <sub>2</sub>	56.05	56.05

<sup>a</sup> 50% Dioxane and 50% water. <sup>b</sup> Ethyl acetate. <sup>c</sup> Ethanol. <sup>d</sup> Benzene. <sup>e</sup> Toluene. <sup>f</sup> 75% Dioxane and 25% water.