

# **Oxidation of Benzyl Alcohols with Tetraalkylammonium Dichlorobromate(1-)**

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## **Abstract**

The reaction of various substituted benzyl alcohols with an equimolar amount of tetraalkylammonium dichlorobromate(1-) (**1**) in water-hexane in the presence of sodium hydroxide gave the corresponding benzaldehydes and with 2 equiv. of **1** in water in the presence of sodium hydroxide (subsequent acid hydrolysis) gave the corresponding benzoic acids, respectively, in good yields. Meanwhile, the reaction of methoxy-substituted benzyl alcohols in the presence of disodium phosphate gave nuclear bromo-substituted benzyl alcohols.

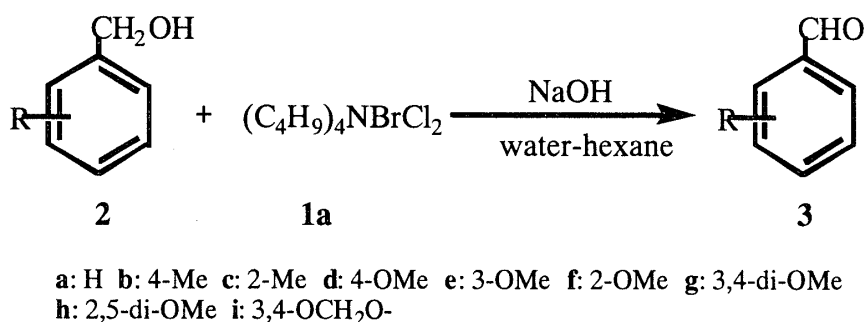
## **1. Introduction**

The oxidation of benzyl alcohols to benzaldehydes or benzoic acids is one of the most important transformation in organic chemistry both at a laboratory and industrial scale, and several methods are available to accomplish this conversion under a variety of reaction conditions.<sup>1,2,3)</sup> Okamoto *et al.* have reported that benzyltrimethylammonium tribromide is an useful oxidation agent for benzyl alcohols.<sup>4)</sup> However, no study has been carried out on the oxidation of alcohols with bromine chloride such as tetraalkylammonium dichlorobromate(1-) (**1**). It has been reported in our laboratory that the bromination of phenols,<sup>5,6)</sup> aromatic ethers,<sup>7)</sup>

aromatic amines,<sup>8,9)</sup> acetanilides,<sup>10)</sup> and aromatic hydrocarbons<sup>11,12)</sup> with **1** gives nuclear bromo-substituted compounds in good yields. We have recently reported that the oxidation of hydroquinones with **1** in aqueous acetic acid or in ether-water-sodium acetate hydrate gave quinones in good yields.<sup>13)</sup> These reagents have a synthetic merit to be handled quantitatively compared with liquid bromine chloride and bromine due to its solid character. As a continuation of our studies for **1** we wish to report the oxidation of benzyl alcohols (**2**) with **1**.

## 2. Results and Discussion

The reaction of an equimolar amount of tetrabutylammonium dichlorobromate(1-) (**1a**) with benzyl alcohols (**2a-i**) was conducted in water-hexane (to avoid the production of carboxylic acid) in the presence of sodium hydroxide to give aldehydes (**3a-i**) in good yields. The reaction scheme can be represented in Scheme 1. The results are summarized in Table 1.



Scheme 1

In the absence of sodium hydroxide, however, the reaction proceeded slowly and gave **3** in low yields. The reaction of benzyl alcohol (**2a**) in aqueous sodium hydroxide solution with **1a** gave a mixture of benzaldehyde (**3a**) and **2a** with significant amounts of benzoic acid (**4a**).

Table 1. Oxidation of Benzyl Alcohols (**2**) to Benzaldehydes (**3**) with **1a** in the Presence of Sodium Hydroxide<sup>a)</sup>

Entry	Substrate	Reaction time/h	Product	Yield (%) <sup>b)</sup>
1	<b>2a</b>	2.5	<b>3a</b>	50 <sup>c)</sup>
2	<b>2b</b>	20.5	<b>3b</b>	42 <sup>c)</sup>
3	<b>2c</b>	18	<b>3c</b>	36 <sup>c)</sup>
4	<b>2d</b>	10 min	<b>3d</b>	80
5	<b>2e</b>	35 min	<b>3e</b>	60
6	<b>2f</b>	3.5	<b>3f</b>	62
7	<b>2g</b>	40 min	<b>3g</b>	79
8	<b>2h</b>	43	<b>3h</b>	52
9	<b>2i</b>	15 min	<b>3i</b>	77

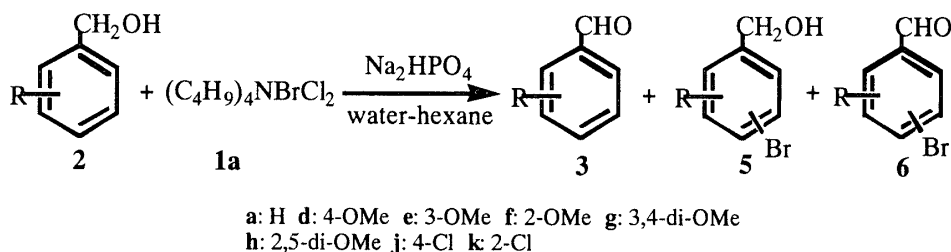
a) Reactions were carried out with substrate (5 mmol), **1a** (5 mmol), and NaOH (15 mmol) in water (10 cm<sup>3</sup>) -hexane (60 cm<sup>3</sup>) at room temperature. b) Isolated yield. c) Benzaldehydes (**3**) were separated from alcohols (**2**) by column chromatography on alumina.

The use of a two-liquid phase system composed of water and hexane appears, therefore, to be suited to the production of aldehydes by oxidation of benzyl alcohols with **1a**. As shown in Table 1, the oxidation of particularly active benzyl alcohols such as methoxybenzyl alcohols (**2d**, **2e** and **2f**), 3,4-dimethoxybenzyl alcohol (**2g**), and piperonyl alcohol (**2i**) with **1a** gave the corresponding aldehydes (**3**) in high yields. However, a small amount of nuclear bromo-substituted aldehydes and alcohols (1-3% by GLC analyses) in the reaction of **2d-h** with **1a** were detected by Mass spectrometry and GLC.

Under the same conditions, 4-nitrobenzyl alcohol or 3-nitrobenzyl alcohol with **1a** was much slowly oxidized to give aldehyde in poor yields.

Meanwhile, the reaction of 4-methoxybenzyl alcohol (**2d**) with **1a** gave a small amount of 4-methoxybenzyl 4-methoxybenzoate (Table 1, Entry 4). A similar ester also was given in the reaction of 3-methoxybenzyl alcohol (**2e**) with **1a** (Table 1, Entry 5). A similar dimeric esterification of primary alcohols was also found for the reaction of primary alcohols and benzyltrimethylammonium tribromide in the presence of disodium phosphate.<sup>14)</sup>

Table 2 shows the reaction of benzyl alcohols (**2**) with **1a** in water-hexane in the presence of disodium phosphate (Scheme 2).

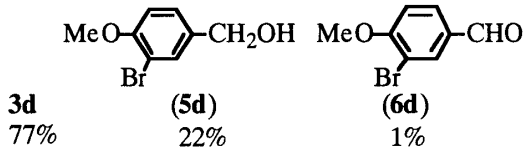
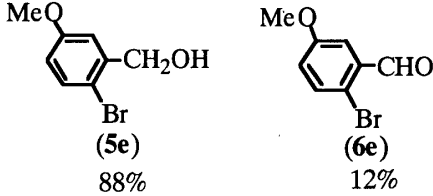
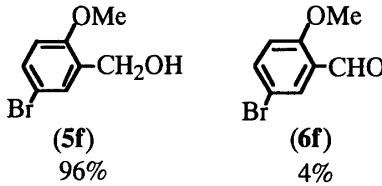
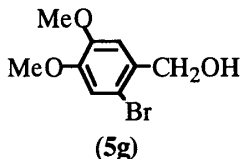
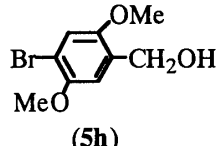


Scheme 2

The reaction of benzyl alcohol (**2a**) with **1a** gave a mixture of **3a** and benzoic acid (**4a**) with a small amount of benzyl benzoate (Table 2, Entry 1). The oxidation of 4-chloro- and 2-chlorobenzyl alcohols (**2j** and **2k**) with **1a** gave the corresponding benzaldehydes (**3j** and **3k**), respectively in good yields (Table 2, Entries 7 and 8).

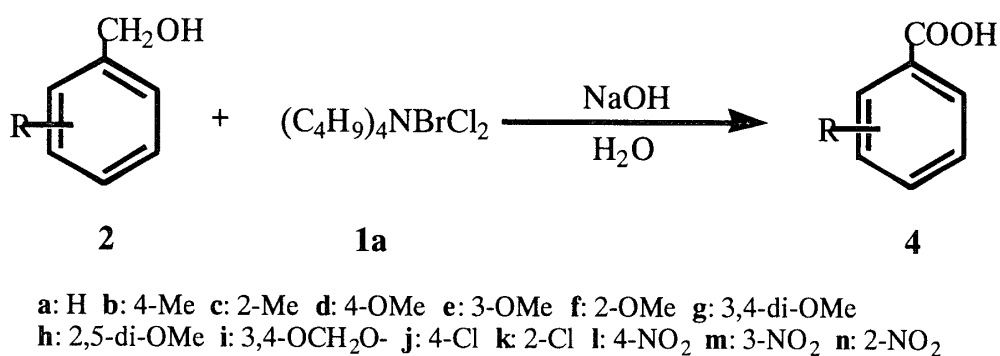
Meanwhile, the reaction of 4-methoxybenzyl alcohol (**2d**) with **1a** gave 4-methoxybenzaldehyde (**3d**) with a considerable amount of nuclear bromo-substituted methoxybenzyl alcohol (**5d**) and benzaldehyde (**6d**) (Table 2, Entry 2). The reactions of 3- and 2-methoxybenzyl alcohols (**2e** and **2f**) with **1a** gave nuclear bromo-substituted methoxybenzyl alcohol (**5e** and **5f**) with a small amount of nuclear bromo-substituted methoxybenzaldehydes, respectively. In the reaction of 3,4- and 2,4-dimethoxybenzyl alcohols (**2g** and **2h**) with **1a** in the presence of disodium phosphate, the exclusive nuclear bromo-substituted benzyl alcohols (**5g** and **5h**) were given in high yields. Thus, in less basic reaction conditions, active benzyl alcohols (**2**) such as **2d-2h** tend to react with **1a** *via* electrophilic aromatic substitution to give bromo compounds.

Table 2. Oxidation of Benzyl Alcohols (**2**) with **1a** in the Presence of Disodium Phosphate<sup>a)</sup>

Entry	Substrate	Reaction conditions		Product <sup>b)</sup>	Yield (%) <sup>c)</sup>
		Temp/°C	Time/h		
1	<b>2a</b>	60	13.5	<b>3a</b> <b>4a</b> 47% 53%	-
2	<b>2d</b>	60	6	 <b>3d</b> <b>(5d)</b> <b>(6d)</b> 77% 22% 1%	-
3	<b>2e</b>	60	0.5	 <b>(5e)</b> <b>(6e)</b> 88% 12%	56 <sup>d)</sup>
4	<b>2f</b>	60	5	 <b>(5f)</b> <b>(6f)</b> 96% 4%	60 <sup>e)</sup>
5	<b>2g</b>	60	3	 <b>(5g)</b>	92
6	<b>2h</b>	60	5.5	 <b>(5h)</b>	86
7	<b>2j</b>	80	13	<b>3j</b>	60
8	<b>2k</b>	80	11.5	<b>3k</b>	56 <sup>f)</sup>

a) Reactions were carried out with substrate (5 mmol), **1a** (5.5 mmol), and Na<sub>2</sub>HPO<sub>4</sub> · 12H<sub>2</sub>O (11 mmol) in water (10cm<sup>3</sup>)-hexane (60cm<sup>3</sup>). b) Determined by glc before isolation. Percentages are normalized to 100% c) Isolated yield. d) Yield of **5e**. e) Yield of **5f**. f) Isolated by column chromatography on alumina.

The reaction of 2 equiv. of tetrabutylammonium dichlorobromate(1-) (**1a**) with benzyl alcohols (**2**) was conducted in the presence of sodium hydroxide to give carboxylic acids (**4**) in high yields (Scheme 3).



Scheme 3

The results are summarized in Table 3. In the case of the reaction of **2d-h**, a small amount of nuclear bromo-substituted benzoic acids were detected by Mass spectrometry.

Table 3. Oxidation of Benzyl Alcohols (**2**) to Benzoic Acids (**4**) with **1a**<sup>a)</sup>

Entry	Substrate	Reaction time/h	Product	Yield (%) <sup>b)</sup>
1	<b>2a</b>	20	<b>4a</b>	97
2	<b>2b</b>	18	<b>4b</b>	92
3	<b>2c</b>	18	<b>4c</b>	91
4	<b>2d</b>	23	<b>4d</b>	73
5	<b>2e</b>	18	<b>4e</b>	72
6	<b>2f</b>	6	<b>4f</b>	74
7	<b>2g</b>	18	<b>4g</b>	98
8	<b>2h</b>	50	<b>4h</b>	67
9	<b>2i</b>	23	<b>4i</b>	81
10	<b>2j</b>	20	<b>4j</b>	97
11	<b>2k</b>	20	<b>4k</b>	97
12	<b>2l</b>	48 <sup>c)</sup>	<b>4l</b>	98
13	<b>2m</b>	24	<b>4m</b>	98
14	<b>2n</b>	24	<b>4n</b>	95

a) Reactions were carried out with substrate (5 mmol), **1a** (5 mmol), and NaOH (20 mmol) in water (30 cm<sup>3</sup>) at room temperature. b) Isolated yield. c) at 70 °C

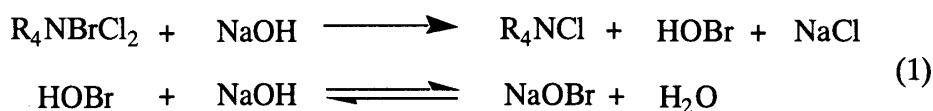
Similarly, the reaction of 2 equiv. of benzyltrimethylammoniumdichlorobromate(1-) (**1b**) with benzyl alcohols (**2**) gave benzoic acids in high yields (Table 4).

Table 4. Oxidation of Benzyl Alcohols (**2**) to Benzoic Acids (**4**) with **1b**<sup>a)</sup>

Entry	Substrate	Reaction time/h	Product	Yield (%) <sup>b)</sup>
1	<b>2a</b>	3.5	<b>4a</b>	82
2	<b>2b</b>	22	<b>4b</b>	91
3	<b>2c</b>	5	<b>4c</b>	94
4	<b>2d</b>	23	<b>4d</b>	60
5	<b>2h</b>	35	<b>4h</b>	93
6	<b>2i</b>	22	<b>4i</b>	95
7	<b>2j</b>	6	<b>4j</b>	95
8	<b>2k</b>	9.5	<b>4k</b>	96
9	<b>2l</b>	22 <sup>c)</sup>	<b>4l</b>	89
10	<b>2m</b>	22.5	<b>4m</b>	86
11	<b>2n</b>	19	<b>4n</b>	81

a) Reactions were carried out with substrate (2.5 mmol), **1b** (5 mmol), and NaOH (20 mmol) in water (30 cm<sup>3</sup>) at room temperature. b) Isolated yield. c) at 70 °C

It is reasonable to assume that R<sub>4</sub>NBrCl<sub>2</sub> (**1**) can be dissociated by sodium hydroxide or water as shown in the following equation (1).



The sodium hypobromite (or hypobromous acid) thus formed may act as the attacking species and may convert alcohols into aldehydes or carboxylic acids.

The structures of the products were easily determined by NMR and Mass spectrometry. In conclusion, we suggested that **1** is an efficient oxidizing reagent for benzyl alcohols because of its easeful handling, simplicity, and good product yields.

### 3. Experimental

**General.** NMR spectra were recorded on a JEOL JNM-EX90 spectrometer, using TMS as the internal standard. High-resolution mass spectra were recorded on a

JEOL JMS-AX505HA mass spectrometer operating at 70 or 30 eV. The GLC analyses were performed on a Yanako Gas Chromatograph with a Silicon DC 550 (25%)-Chromosorb WAW (1m) column with helium as a carrier gas. Melting points were recorded on a Yanako Micro Melting Point Apparatus and are uncorrected. Tetrabutylammonium dichlorobromate(1-) (**1a**) and benzyltrimethylammonium dichlorobromate(1-) (**1b**) were prepared by the previous method.<sup>8,15</sup> All products were characterized by comparing their <sup>1</sup>H and <sup>13</sup>C NMR spectra and mp or bp with those of authentic samples or reported data.

**Oxidation of Piperonyl Alcohol (2i) to Piperonal (3i) with Tetrabutylammonium Dichlorobromate(1-) (1a) in the Presence of Sodium Hydroxide. General Procedure:** Tetrabutylammonium dichlorobromate(1-) (**1a**) (1.966 g, 5.00 mmol) was added in portions with stirring at room temperature to a mixture of **2i** (0.760 g, 5.00 mmol) and sodium hydroxide (0.600 g, 15 mmol) in water (10 cm<sup>3</sup>) and hexane (60 cm<sup>3</sup>). The mixture was stirred for 15 min until the initial yellow color of **1a** faded. To the reaction mixture were added water (50 cm<sup>3</sup>) and a small amount of sodium sulfite. After the reaction mixture was neutralized with dilute hydrochloric acid, hexane layer was separated and then water layer was extracted with ether. The organic layer was dried over magnesium sulfate and then concentrated in vacuo. GLC analyses of the residue showed one peak of piperonal (**3i**); yield 0.578 g (77%); Mp 36-37 °C (lit,<sup>16</sup> mp 37 °C); <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ = 6.04 (2H, s), 6.85-7.44 (3H, m), and 9.77 (1H, s); MS (30 eV) *m/z* (rel intensity) 150 (M<sup>+</sup>, 100), 149 (47), and 121 (7).

**Oxidation of 4-Methoxybenzyl Alcohol (2d) with Tetrabutylammonium Dichlorobromate(1-) (1a) in the Presence of Disodium Phosphate. General Procedure:** Tetrabutylammonium dichlorobromate(1-) (**1a**) (2.167 g, 5.50 mmol) was added to a mixture of **2d** (0.690 g, 5.00 mmol) and Na<sub>2</sub>HPO<sub>4</sub> · 12H<sub>2</sub>O (3.939 g, 11 mmol) in water (10 cm<sup>3</sup>) and hexane (60 cm<sup>3</sup>). The mixture was stirred for 6 h at



60 °C until the initial yellow color of **1a** faded. The solution was then cooled to room temperature and treated with a small amount of sodium sulfite in water (50 cm<sup>3</sup>). Hexane layer was separated and then water layer was extracted with ethyl acetate. The organic layer was dried over magnesium sulfate and then concentrated in vacuo. GLC analyses of the residue showed four peaks (4-methoxybenzaldehyde, 4-methoxybenzyl alcohol, 3-bromo-4-methoxybenzaldehyde, and 3-bromo-4-methoxybenzyl alcohol). Repeated recrystallization of the residue from pentane afforded 50 mg of almost pure 3-bromo-4-methoxybenzyl alcohol (**5d**); Mp 53-56 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ = 1.71 (1H, br. s), 3.89 (3H, s), 4.60 (2H, s), and 6.82-7.55 (3H, m); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ = 56.3, 64.3, 111.7, 111.9, 127.3, 132.3, 134.6, and 155.4; MS (30 eV) *m/z* (rel intensity) 218 (M<sup>+</sup> + 2, 39), 216 (M<sup>+</sup>, 43), 137 (92) and 77 (100).

**2-Bromo-5-Methoxybenzyl Alcohol (5e).** Prepared from 3-methoxybenzyl alcohol (**2e**) and **1a** in the presence of Na<sub>2</sub>HPO<sub>4</sub> · 12H<sub>2</sub>O in a 56% yield: Mp 34-36 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ = 2.95 (1H, br. s), 3.68 (3H, s), 4.57 (2H, s), 6.59 (1H, dd, J = 8.8 and 3.2 Hz), 6.97 (1H, d, 3.2 Hz), and 7.30 (1H, d, J = 8.8 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ = 55.4, 64.4, 112.2, 113.9, 114.4, 132.9, 140.7, and 159.1; MS (30 eV) *m/z* (rel intensity) 218 (M<sup>+</sup> + 2, 93), 216 (M<sup>+</sup>, 100), 137 (48) and 109 (66).

**5-Bromo-2-Methoxybenzyl Alcohol (5f).** Prepared from 2-methoxybenzyl alcohol (**2e**) and **1a** in the presence of Na<sub>2</sub>HPO<sub>4</sub> · 12H<sub>2</sub>O in a 60% yield: Mp 70-73 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ = 2.95 (1H, s), 3.77 (3H, s), 4.57 (2H, s), 6.67 (1H, d, J = 8.3 Hz), and 7.23-7.39 (2H, m); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ = 55.5, 60.5, 111.8, 112.8, 130.8, 131.0, 131.4, and 156.1; MS (30 eV) *m/z* (rel intensity) 218 (M<sup>+</sup> + 2, 99), 216 (M<sup>+</sup>, 100), 137 (76) and 109 (29).

**6-Bromo-3,4-Dimethoxybenzyl Alcohol (5g).** Prepared from 3,4-dimethoxybenzyl alcohol (**2g**) and **1a** in the presence of Na<sub>2</sub>HPO<sub>4</sub> · 12H<sub>2</sub>O in a 92% yield: Mp 96-98 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ = 3.79 (s), 4.58 (s), 6.92 (s), and 6.96(s); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ = 55.9, 56.1, 64.1, 111.5, 112.0, 115.3, 132.0, 148.4, and 148.6; MS

(30 eV)  $m/z$  (rel intensity) 248 ( $M^+ + 2$ , 96), 246 ( $M^+$ , 100), and 139 (82).

**4-Bromo-2,5-Dimethoxybenzyl Alcohol (5h).** Prepared from 2,5-dimethoxybenzyl alcohol (**2h**) and **1a** in the presence of  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$  in a 86% yield: Mp 104-107 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  = 2.62 (1H, br. s), 3.78 (3H, s), 3.83 (3H, s), 4.60 (2H, s), 6.93 (1H, s), and 7.03 (1H, s);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  = 56.0, 56.9, 60.9, 110.2, 112.7, 115.7, 129.4, 150.1, and 151.3; MS (30 eV)  $m/z$  (rel intensity) 248 ( $M^+ + 2$ , 99) and 246 ( $M^+$ , 100).

**Oxidation of Piperonyl Alcohol (2i) to Piperonylic Acid (4i) with Tetrabutylammonium Dichlorobromate(1-) (1a) in the Presence of Sodium Hydroxide. General Procedure:** Tetrabutylammonium dichlorobromate(1-) (**1a**) (1.966, 5.00 mmol) was added to a mixture of **2i** (0.380 g, 2.50 mmol) and NaOH (0.800 g, 20 mmol) in water (30  $\text{cm}^3$ ). The mixture was stirred for 23 h at room temperature until the initial yellow color of **1a** faded. To the reaction mixture was added a small amount of sodium sulfite in water (50  $\text{cm}^3$ ) and the mixture was then acidified sufficiently with dilute hydrochloric acid. After the solution was extracted with ether, ethereal extract was dried over magnesium sulfate and then concentrated in vacuo to give **4i** as a crystal; yield 0.336 g (81%); Mp 229-231 °C (sublimes) (lit,<sup>17</sup>) mp 229 °C).

**Oxidation of 4-Chlorobenzyl Alcohol (2j) to 4-Chlorobenzoic Acid (4j) with Benzyltrimethylammonium Dichlorobromate(1-) (1b) in the Presence of Sodium Hydroxide. General Procedure:** Benzyltrimethylammonium dichlorobromate(1-) (**1b**) (1.510g, 5.00 mmol) was added to a mixture of **2j** (0.356 g, 2.50 mmol) and NaOH (0.800 g, 20 mmol) in water (30  $\text{cm}^3$ ). The mixture was stirred for 6 h at room temperature until the initial yellow color of **1a** faded. To the reaction mixture was added a small amount of sodium sulfite in water (50  $\text{cm}^3$ ) and the mixture was then acidified sufficiently with dilute hydrochloric acid. After the solution was extracted with ether, ethereal extract was dried over magnesium sulfate

and then concentrated in vacuo to give **4j** as a colorless crystal; yield 0.372 g (95%); Mp 244 °C (sublimes) (lit,<sup>18</sup>) mp 243 °C).

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