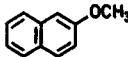
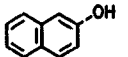
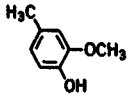
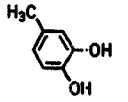
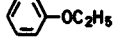
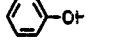
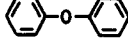
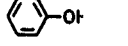
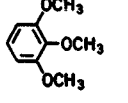
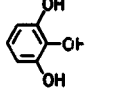
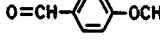
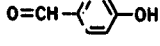
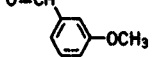
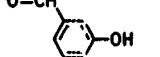
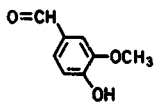
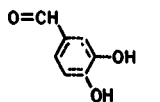
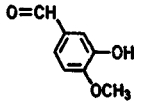
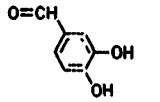
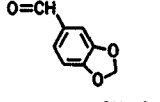
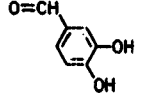
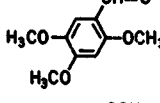
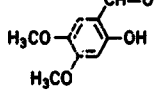
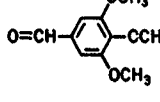
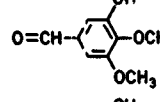
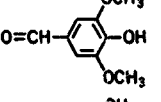
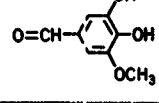
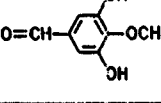




**Table 2.** Cleavage of Ethers 1a–l by Aluminium Triiodide in the Presence of Catalytic Amounts of Tetra-*n*-butylammonium Iodide

Ether	Product: <sup>a</sup>	Solvent	Molar Ratios of AlI <sub>3</sub> /1		Reaction Time <sup>b</sup>	Yield [%] <sup>c</sup>
<b>a</b> 		benzene	1	360	0.3 h	94
<b>b</b> 		cyclohexane	2	100	0.7 h	98
<b>c</b> 		cyclohexane	1	360	1.0 h	90
<b>d</b> 		benzene	1	360	1.0 h	trace
<b>e</b> 		benzene	3	180	0.5 h	95
<b>f</b> 		benzene	1	360	0.5 h	78
<b>g</b> 		cyclohexane	1	360	0.5 h	80
<b>h</b> 		benzene	1	360	0.5 h	84
<b>i</b> 		benzene	4.8	360	1.5 h	88 <sup>d</sup>
<b>j</b> 		cyclohexane	5.5	120	2.5 h	14 <sup>d</sup>
<b>k</b> 		cyclohexane	4.5	120	1.5 h	84 <sup>d</sup>
<b>l</b> 	<b>A:</b>  + 	cyclohexane	1.5	100	14.0 h	88 <sup>d</sup>
	<b>B:</b>  + 	cyclohexane	3.3	100	1.5 h	90 <sup>d</sup>
		cyclohexane	5.5	100	1.5 h	91 <sup>d</sup>
		cyclohexane	10.0	50	10.0 h	85 <sup>d</sup>
		cyclohexane	1.5	100	12.0 h	7(A) <sup>d,e</sup> ; 41(B) <sup>d,e</sup>
		cyclohexane	3.3	100	6.0 h	16(A) <sup>d,e</sup> ; 84(B) <sup>d,e</sup>
		cyclohexane	5.5	100	15.0 h	10(A) <sup>d,e</sup> ; 81(B) <sup>d,e</sup>

<sup>a</sup> The products were identified by b.p., m.p., or <sup>1</sup>H-N.M.R. (JEOL PMX 60 spectrometer) data.

<sup>b</sup> The reactions were monitored by T.L.C. (SiO<sub>2</sub>).

<sup>c</sup> Yield of pure, isolated product.

<sup>d</sup> Products isolated by column chromatography (silica, Merck 60).

<sup>e</sup> The different isomers of A and B could not be completely separated on a silica column.

We also found that the reactions are preferably conducted in inert solvents such as benzene and cyclohexane, which are more suitable for large scale handling.

In contrast to earlier studies<sup>1</sup>, this ether cleaving reagent was applied to aromatic systems containing formyl and hydroxy functions, in addition to the alkoxy group in the ring. It was found that the AlI<sub>3</sub>/ether 1 ratio may be critical, both for the reaction time and the product distribution (Table 2).

#### Aluminium Triiodide<sup>1,2</sup>:

Aluminium powder (2.5 g, 93 mmol) and iodine (19.0 g, 150 mmol) are mixed and refluxed in benzene (120 ml, or cyclohexane) until the red colour of iodine has disappeared (1.5–2 h).

#### Cleavage of Anisole (1; R = CH<sub>3</sub>, Ar = C<sub>6</sub>H<sub>5</sub>); Typical Procedure:

To a cooled suspension of aluminium triiodide (50 mmol) in cyclohexane (120 ml), a solution of anisole (5.4 g, 50 mmol) and tetra-*n*-butylammonium iodide (0.05 g, 0.14 mmol) in cyclohexane (25 ml) is added dropwise. The mixture is refluxed for 20 min, then cooled in an ice/water bath and hydrolysed with water (150 ml). The resulting two layers are separated, and the water phase is extracted with diethyl ether (2 × 25 ml). The extracts are collected and extracted with 2 molar sodium hydroxide solution (30 ml). The water phase is separated, acidified with concentrated hydrochloric acid (to pH = 1), and extracted with diethyl ether (3 × 25 ml). The organic phases are dried with sodium sulphate and evaporated to give pure, crystalline phenol (2; Ar = C<sub>6</sub>H<sub>5</sub>); yield: ~ 100%; m.p. 41–42°C (Ref.<sup>1,3</sup>, m.p. 39–42°C).

**Cleavage of 1,2,3-Trimethoxybenzene (1e); Typical Procedure:**

To a cooled suspension of aluminium triiodide (150 mmol) in benzene (120 ml), a solution of 1,2,3-trimethoxybenzene (**1e**; pyrogallol trimethyl ether; 8.4 g, 50 mmol) and tetra-*n*-butylammonium iodide (0.1 g, 0.28 mmol) in benzene (25 ml) is added dropwise. After refluxing for 0.5 h, the mixture is hydrolysed with water (150 ml). The benzene phase is separated and washed with water (2 × 20 ml). The water phases are collected and extracted with ethyl acetate (6 × 25 ml). The ethyl acetate extracts are dried with sodium sulphate, and the solvent is evaporated to give pure, crystalline pyrogallol (**2e**); yield: 95% m.p. 131–133 °C (Ref.<sup>3</sup>, m.p. 132–133 °C).

*The Chemical Dynamics Development Corp. is thanked for financial support and Dr. Robert E. Carter for linguistic criticism.*

Received: October, 22, 1984

<sup>1</sup> Bhatt, M. V., Kulkarni, S. U. *Synthesis* **1983**, 253.

<sup>2</sup> Cabiddu, S., Gelli, G., Maccioni, A., Secci, M. *Ann. Chim. (Rome)* **1972**, 62, 505.

<sup>3</sup> *The Merck Index*, 9th Edn., Merck & Co. Inc., Rahway, N.J., U.S.A. 1976.