## Anodic Oxidation of Mixed Ethers of Hydroquinones. A Complementary Route to Benzoquinone Monoketals

Mark G. Dolson and John S. Swenton*

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210

Quinone monoketals are of demonstrated utility in organic synthesis.[1] These compounds are prepared from oxidation of *p*-methoxyphenols[1,2] or by regioselective hydrolysis of quinone bisketals.[1,3] The former method is dependent upon the availability of the requisite phenol; the latter procedure affords the quinone monoketal wherein the less hindered quinone carbonyl is present as the free carbonyl group. We report here a technique for preparation of selected quinone monoketals wherein the more hindered carbonyl group of the quinone is unprotected.
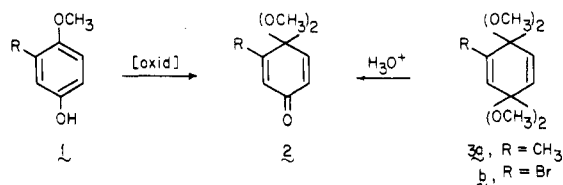
A key aspect of the work was the finding that mixed aromatic ethers (i.e., **4**) undergo anodic oxidation to give predominantly **5**.[4] The lesser amount of **6** formed in the reaction is readily separated by chromatography on neutral alumina. The products **5** and **6** showed spectroscopic (IR, NMR) properties in agreement with the assigned structures.

(1) For leading references, see: Henton, D. R.; Anderson, D. K.; Manning, M. J.; Swenton, J. S. *J. Org. Chem.* **1980**, *45*, 3422–3433.

(2) (a) McKillip, A.; Perry, D. H.; Edwards, M.; Antus, S.; Farkas, L.; Nogradi, M.; Taylor, E. C. *J. Org Chem.* **1976**, *41*, 282–287. (b) Büchi, G.; Chu, P.; Hoppmann, A.; Mak, C.; Pearce, A. *Ibid.* **1978**, *43*, 3983–3985. (c) Crouse, D. J.; Wheeler, D. M. S. *Tetrahedron Lett.* **1979**, 4797–4798. (d) Hart, W.; Scheinmann, F. *Ibid.* **1980**, *21*, 2295–2296.

(3) (a) Manning, M. J.; Henton, D. R.; Swenton J. S. *Tetrahedron Lett.* **1977**, 333–337. (b) Henton, D. R.; Chenard, B. L.; Swenton, J. S. *J. Chem. Soc., Chem. Commun.* **1979**, 326–327.

(4) The anodic oxidation of the 1,4-bis(2-hydroxyethoxy)benzene has been reported: Margaretha, P.; Tissot, P. *Helv. Chim. Acta* **1975**, *58*, 933–936.

(5) An alternate hydrolysis product was detected but not isolated.
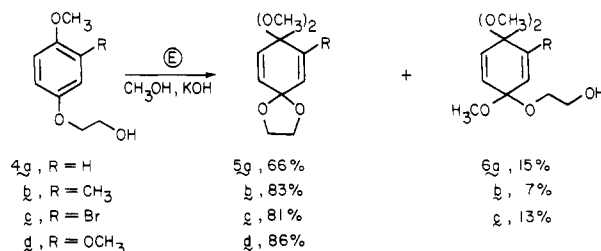
The monohydrolysis of bisketals **5a–c** gave monoketals **7a–c** in the indicated yields after recrystallization or molecular distillation. In the case of **5d**, two products were formed in ca. 7:1 ratio. The major product, **8d**, was obtained by direct recrystallization in 68% yield. The minor product (presumably **7d**) was difficultly separable from **8d** by chromatography and was not rigorously characterized.
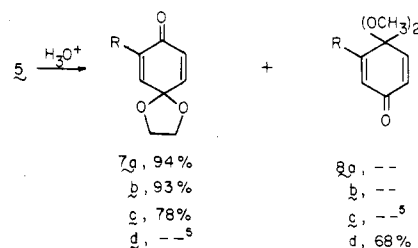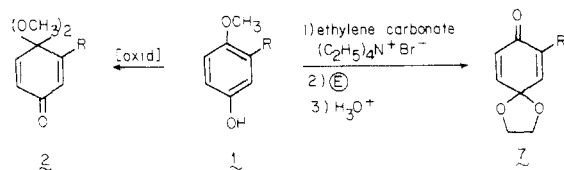

These results most reasonably arise from the slower rate of hydrolysis of the ethylene glycol ketal relative to the dimethyl ketal. For the dimethyl ketal a trans-periplanar arrangement between the departing methoxy group and the electron pair on the oxygen of the remaining methoxy group is possible (i.e., **9**), whereas for an ethylene glycol ketal this most favorable situation is not available (i.e., **10**) (Figure 1).[6] While the change in direction of ketal hydrolysis in going from the bisketals such as **3a** and **3b** to **5b** and **5c** was anticipated, the high selectivity of the hydrolysis in the latter two systems would not be expected from literature rate data.[7] The rate difference for hydrolysis of an ethylene glycol ketal vs. a dimethyl ketal is a factor of ~20. In the monohydrolysis of the bis(dimethoxy ketals) **3a** and **3b**, the regioselectivity of the hydrolysis is 85:15 and 95:5, respectively, in favor of the monoketal **2**.[3b] Assuming a rate retardation of 20 upon replacement of a dimethyl ketal by an ethylene glycol ketal, some regioselectivity of hydrolysis for **5b** and **5c** would have been predicted. The highly selective hydrolysis for **5b** and **5c** suggests there must be a small rate alteration in these systems upon replacement of a dimethyl ketal by an ethylene glycol ketal. In the case of **5d**, the stabilization of the carbonium ion formed in the hydrolysis by the methoxy group outweighs other considerations, and the major monoketal formed is **8d**, the same product obtained from the bis(dimethoxy ketal) **3d** (R = OCH₃).

As noted earlier conventional preparation of quinone monoketals often gives access to only one of the regioisomers. These results demonstrate that both benzoquinone monoketal regioisomers can be obtained from common intermediates—the *p*-methoxyphenol or the bisketal—provided good carbonium ion stabilizing groups are not substituted on the bisketal.

(6) (a) For a general discussion of ketal and orthoester hydrolysis, see: Cordes, E. H.; Bull, H. G. *Chem. Rev.* **1974**, *74*, 581–603; Fife, T. *Acc. Chem. Res.* **1972**, *5*, 264–272. (b) Kirby, A. J.; Martin, R. J. *J. Chem. Soc., Chem. Commun.* **1978**, 803–804.

(7) (a) Fife, T. H.; Bord, L. H. *J. Org. Chem.* **1968**, *33*, 4136–4140. (b) Slavin, M. N.; Levi, I. S.; Matveeva, A. A.; Kikot, V. S.; Berlin, A. Y. *J Org. Chem. USSR (Engl. Transl.),* **1969**, *5*, 448–450.

Figure 1. Newman projection of ketal hydrolysis intermediates looking down the C–O bond of the ketal.

Experimental Section[8]

2-(4-Methoxy-3-methylphenoxy)ethanol (4b). A mixture of 10.4 g (75 mmol) of 4-methoxy-3-methylphenol,[9] 8.8 g (100 mmol) of ethylene carbonate, 2.5 g of tetraethylammonium bromide, and 5 mL of dimethylformamide was heated to 140 °C for 7 h. The cooled reaction mixture was poured into 100 mL of ether, washed with water (3 × 50 mL) and brine (50 mL), and dried (CaSO₄). Concentration of the ethereal layer gave a light-brown oil which was distilled [bp 115–118 °C (0.35 mm)] to afford 11.9 g (87%) of **4b** as a white solid: mp 41–42.5 °C; IR (KBr) 3509 (s), 2976 (s), 1605 (w), 1499 (s), 1460 (s), 1370 (w), 1279 (s), 1212 (s), 1164 (s), 1126 (m), 1086–1010 (s, br), 958 (m), 893 (s), 868 (s), 789 (s), 749 (m), 733 cm⁻¹ (m); NMR δ 6.64–6.44 (str m, 3 H), 3.80 (br s, 4 H), 3.67 (s, 3 H), 3.27 (br s, 1 H), 2.08 (s, 3 H); exact mass calcd for C₁₀H₁₄O₃ *m/e* 182.0943, found *m/e* 182.0947.

2-(3-Bromo-4-methoxyphenoxy)ethanol (4c). The title compound was prepared as above in 86% yield from 3-bromo-4-methoxyphenol:[10] bp 142–149 °C (0.5 mm); mp 37–38 °C; IR (neat) 3460 (m), 2967 (m), 1488 (s), 1449 (m), 1433 (m), 1263 (s), 1208 (s), 1071 (m), 1044 (s), 1011 (m), 929 (m), 796 (m), 740 cm⁻¹ (m); NMR δ 7.05 (str m, 1 H), 6.72 (s, 2 H), 4.04–3.63 (m, 4 H), 3.78 (s, 3 H), 2.20 (br s, 1 H); exact mass calcd for C₉H₁₁O₃⁷⁹Br *m/e* 245.9892, found *m/e* 245.9896.

2-(3,4-Dimethoxyphenoxy)ethanol (4d). The title compound was prepared as above in 63% yield from 3,4-dimethoxyphenol:[11] mp 69–70.5 °C; IR (KBr) 3425 (m), 3333 (m), 2967 (m), 1608 (m), 1595 (m), 1504 (s), 1460 (s), 1437 (s), 1408 (m), 1381 (w), 1355 (w), 1277 (s), 1250 (s), 1220 (s), 1192 (s), 1179 (s), 1157 (s), 1131 (s), 1070 (s), 1057 (s), 1017 (s), 962 (s), 897 (s), 884 (m), 867 (w), 835 (m), 819 (w), 775 (s), 751 (m), 731 cm⁻¹ (m); NMR (CDCl₃) δ 6.56 (AB, Δν = 23 Hz, J = 8 Hz, with the upfield component further split into a doublet, J = 3 Hz, 2 H), 6.51 (d, J = 3 Hz, 1 H), 3.97 (br s, 4 H), 3.82 (s, 6 H), 2.88 (br s, 1 H); exact mass calcd for C₁₀H₁₄O₄ *m/e* 198.0892, found *m/e* 198.0897.

Electrolysis of 4a. A solution of 2.5 g (0.015 mol) of **4a**[12] in 75 mL of solvent was electrolyzed under the standard conditions[13]

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(8) All melting points were taken with a Thomas-Hoover capillary melting-point apparatus and are uncorrected. Measurements of standard samples indicated that the observed melting points were probably 1–2 °C lower than the corrected value. Infrared spectra were recorded on a Perkin-Elmer Infracord and calibrated by using the polystyrene band at 1601.4 cm⁻¹. ¹H NMR spectra were taken at 60 MHz with a Varian EM-360 or EM-360L spectrometer in carbon tetrachloride, unless indicated otherwise. ¹³C NMR spectra (tetramethylsilane reference) were recorded on a Bruker WP-80 instrument at 20.1 MHz by Dr. C. Cottrell. Mass spectra and exact mass measurements were obtained by Mr. C. R. Weisenberger on a Consolidated Electronics MS-9 double-focusing mass spectrometer (ionization potential 70 eV). Analytical samples were determined by Scandinavian Microanalytical Laboratory, Herlev, Denmark, and were within 0.3% of calculated values. Aluminum oxide and silica gel were from E. Merck Co. Ether (E) and hexane (H), bp 35–50 °C, were distilled prior to use. All GLC analyses were done on a 1 ft × ¹/₈ in. column of 5% SE-30 on 60/80-mesh Chromosorb G at 120 °C. "Workup" consisted of extraction with ether, drying over Drierite, and concentration in vacuo. Where an extraction was already performed, only the last two operations were valid.

(9) Baker, W.; Brown, N. C. *J. Chem. Soc.* **1948**, 2303–2307.

(10) Irvine, F. M.; Smith, J. C. *J. Chem. Soc.* **1927**, 74–77.

(11) Baker, W.; Evans, C. *J. Chem. Soc.* **1938**, 372–375.

(12) Yoshino, T.; Inaba, S.; Ishido, Y. *Bull. Chem. Soc. Jpn.* **1973**, *46*, 553–557.

(13) All electrolyses were performed in 1% methanolic potassium hydroxide, using a platinum gauze anode, a platinum sheet cathode, and a platinum wire reference electrode. A potentiostat constructed from a KEPCO BOP72-5M bipolar operational amplifier[14] was used. In the single-cell electrolyses, the applied potential was increased in order to maintain a current of ~1 A until the UV absorption maximum at 288 nm indicated most of the starting material had reacted. The potential was held constant at this point until the UV absorption decreased to <5% of its initial value. Double-cell electrolyses were conducted similarly with a current of ~0.5 A being maintained.
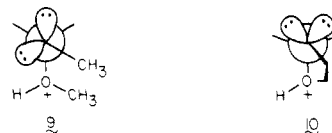

[2980 C, 97% current efficiency]. After the solvent was removed in vacuo, 20 mL of water was added, and the reaction mixture was extracted with chloroform (3 × 15 mL). Workup afforded a colorless oil which was chromatographed on neutral alumina (activity III, 2.5 × 25 cm column, slurry packed with 5% ether/hexane) as follows: 80 mL of 5% E/H, nil; 320 mL of 5% E/H, 80 mL of 10% E/H, 1.96 g (66%) of **5a** as a white crystalline solid. **5a**: mp 62–63 °C; IR (KBr) 3003 (m), 2950 (m), 1460 (w), 1406 (s), 1307 (m), 1170 (m), 1151–1066 (s, br), 1042–1001 (s, br), 973–923 (s, br), 903 (m), 766 (m), 758 (w), 707 cm⁻¹ (w); NMR δ 5.80 (s, 4 H), 3.92 (s, 4 H), 3.18 (s, 6 H); exact mass calcd for C₁₀H₁₄O₄ *m/e* 198.0892, found *m/e* 198.0897.

Elution was continued as follows: 120 mL of 10% E/H, nil; 240 mL of 10% E/H, 0.52 g (15%) of **6a** as a colorless oil. **6a**: IR (neat) 3559 (m), 3003 (m), 1502 (w), 1456 (m), 1399 (m), 1304 (w), 1217 (m), 1181 (m), 1120–1005 (s, br), 971–934 (s, br), 800–772 (m, br), 766–743 cm⁻¹ (m, br); NMR δ 5.95 (s, 4 H), 3.51 (br s, 4 H), 3.19 (s, 6 H).[15]

Electrolysis of 4b. A solution of 3 g (0.016 mol) of **4b** in 100 mL of solvent was electrolyzed under the standard conditions[13] [3218 C, 98% current efficiency]. The methanol was removed in vacuo, 40 mL of water was added, and the residue was extracted with methylene chloride (4 × 25 mL). Workup afforded a colorless oil which was chromatographed on neutral alumina (activity III, 1.8 × 15 cm column, slurry packed with 5% ether/hexane) as follows: 60 mL of 5% E/H, nil; 120 mL of 5% E/H, 2.88 g (83%) of **5b** as a colorless oil. **5b**: IR (neat) 2967 (m), 1435 (m), 1381 (m), 1359 (m), 1342 (w), 1282 (m), 1195 (m), 1145 (s), 1098 (s), 1079–1044 (s, br), 1019 (s), 1008 (s), 969–945 (s, br), 939 (s), 756 cm⁻¹ (w); NMR δ 5.95 (AB, Δν = 14.4 Hz, J = 12 Hz, with the downfield component being further split into a doublet, J = 2 Hz, 2 H), 5.65 (m partially obscured by AB, 1 H), 3.93 (s, 4 H), 3.08 (s, 6 H), 1.66 (d, J = 1.5 Hz, 3 H); exact mass calcd for C₁₁H₁₆O₄ *m/e* 212.1048, found *m/e* 212.1044.

Elution was continued as follows: 20 mL of 5% E/H, nil; 60 mL of 10% E/H, nil; 220 mL of 10% E/H, 0.285 g (7%) of **6b** as a colorless oil. **6b**: IR (neat) 3597 (w), 3003 (m), 1443 (w), 1387 (w), 1284 (w), 1211 (w), 1196 (w), 1149 (m), 1099 (s), 1068–1043 (s, br), 1019 (m), 957 (s), 785 (w), 755 cm⁻¹ (w); NMR δ 6.12 (partially obscured AB, Δν = 17 Hz, J = 11 Hz, with the downfield component being further split into a doublet, J = 2 Hz, 2 H), 5.82 (m partially obscured by AB, 1 H), 3.54 (br s, 4 H), 3.38 (br s, 1 H), 3.22 (s, 3 H), 3.12 (s, 6 H), 1.70 (d, J = 1.5 Hz, 3 H).[15]

Electrolysis of 4c. Into the anode compartment of a divided cell was placed a solution of 2.7 g (0.011 mol) of **4c** in 60 mL of 1% methanolic potassium hydroxide. An equal volume of the same solvent was placed in the cathode compartment, and this solution was electrolyzed[13] until the UV absorption maximum at 292 nm had decreased to ~5% of its initial value (2908 C, 73% current efficiency). The methanol was removed from the anolyte, 30 mL of water was added, and the residue was extracted with methylene chloride (3 × 20 mL). Workup afforded a yellowish oil which was chromatographed on alumina (activity III, 1.8 × 15 cm column, slurry packed with 10% ether/hexane) as follows: 20 mL of 10% E/H, nil; 160 mL of 10% E/H, 2.45 g (81%) of **5c** as white crystals. **5c**: mp 59.5–61 °C; IR (KBr) 2990 (m), 2950 (m), 2925 (m), 2890 (m), 2875 (m), 2830 (m), 1672 (m), 1638 (m), 1461 (m), 1396 (s), 1317 (m), 1286 (m), 1218 (m), 1147–1077 (s, br), 1067 (s), 1018 (s), 983 (s), 959 (s), 793 (m), 753 (m), 663 cm⁻¹ (m); NMR δ 6.37 (d, J = 2 Hz, 1 H), 5.96 (AB, Δν = 16 Hz, J = 10 Hz, with downfield component further split into a doublet,

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(14) Henton, D. R.; McCreery, R. L.; Swenton, J. S. *J. Org. Chem.* **1980**, *45*, 369–378.

(15) Compounds **6a–c** did not give parent ions in the mass spectrometer nor did they give satisfactory combustion analyses.

J = 2 Hz, 2 H), 3.98 (s, 3 H), 3.13 (s, 6 H); exact mass calcd for $C_{10}H_{13}O_4{}^{79}Br$ *m/e* 275.9998, found *m/e* 276.0003.

Elution was continued as follows: 240 mL of 10% E/H, nil; 160 mL of 10% E/H, 0.46 g (13%) of **6c** as a colorless oil. **6c**: IR (neat) 3534 (m), 2976 (s), 1603 (m), 1449 (s), 1374 (s), 1299 (m), 1287 (m), 1261 (m), 1199 (m), 1127–996 (s, br), 969 (s), 925 (s), 762 cm^{-1} (w); NMR δ 6.51 (d, *J* = 2 Hz, 1 H), 6.13 (AB, $\Delta\nu$ = 17 Hz, *J* = 11 Hz, with the downfield component further split into a doublet, *J* = 2 Hz, 2 H), 3.57 (br s, 4 H), 3.27 (s, 3 H), 3.18 (s, 6 H), 2.48 (br s, 1 H).[15]

Electrolysis of 4d. A solution of 1 g (0.010 mol) of **4d** in 80 mL of solvent was electrolyzed under the standard conditions[13] [2061 C, 95% current efficiency]. The methanol was removed in vacuo, 25 mL of water was added, and the residue was extracted with methylene chloride (3 × 20 mL). Workup afforded a colorless oil which was chromatographed on neutral alumina (activity III, 1.8 × 20 cm column, slurry packed with 10% ether/hexane) as follows: 260 mL of 10% E/H, nil; 120 mL of 10% E/H, 120 mL of 14% E/H, 1.98 g (86%) of **5d** as white crystals. **5d**: mp 49.5–51 °C; IR (KBr) 2941 (m), 1597 (m), 1443 (s), 1376 (s), 1342 (s), 1227 (s), 1198 (s), 1170 (s), 1130–1028 (br, s), 1002 (s), 971 (m), 943 (s), 872 (m), 810 (m), 709 cm^{-1} (m); NMR δ 5.81 (AB, $\Delta\nu$ = 19 Hz, *J* = 11 Hz, with downfield component further split into a doublet, *J* = 2 Hz, 2 H), 4.83 (d, *J* = 2 Hz, 1 H), 3.94 (s, 4 H), 3.61 (s, 3 H), 3.16 (s, 6 H); exact mass calcd for $C_{11}H_{16}O_5$ *m/e* 228.0997, found *m/e* 228.0992.

Hydrolysis of 5a. To a solution of 1.0 g (5.1 mmol) of **5a** in 25 mL of tetrahydrofuran was added 12.5 mol of 2% acetic acid. The hydrolysis was followed by VPC and quenched with 10 mL of saturated bicarbonate solution after 1.5 h. Workup afforded 0.73 g (94%) of **7a** as an off-white crystalline solid which was recrystallized from methylene chloride/hexane; mp 50–51 °C (lit.[16] mp 51–52 °C).

Hydrolysis of 5b. To a solution of 1.78 g (8.4 mmol) of **5b** in 50 mL of tetrahydrofuran was added 25 mL of 2% acetic acid. The hydrolysis was followed by VPC and quenched with 30 mL of saturated bicarbonate solution after 1.5 h. The organic layer was separated and the aqueous portion extracted with 2 × 30 mL of ether. Workup of the combined organic solutions afforded 1.36 g of **7b** as a light-yellow oil. Short-path distillation (70 °C bath, 0.40 mm) afforded 1.30 g (93%) of **7b** as a colorless oil: IR (neat) 3040 (w), 2950 (w), 1678 (s), 1647 (s), 1443 (w), 1368 (m), 1355 (m), 1285 (m), 1235 (w), 1156 (s), 1111 (s), 1071 (s), 1011 (s), 963 (s), 940 (m), 869 (w), 810 cm^{-1} (m); NMR δ 6.38 (AB, $\Delta\nu$ = 31 Hz, *J* = 10 Hz, with the downfield component further split into a doublet, *J* = 3 Hz, 2 H), 6.35 (m, 1 H), 4.05 (s, 4 H), 1.83 (d, *J* = 2 Hz, 3 H); exact mass calcd for $C_9H_{10}O_3$ *m/e* 166.0630, found *m/e* 166.0635.

Hydrolysis of 5c. To a solution of 1.69 g (6.1 mmol) of **5c** in 25 mL of tetrahydrofuran was added 12.5 mL of 2% acetic acid. The hydrolysis was followed by VPC and quenched with 20 mL of saturated bicarbonate solution after 40 h. The organic layer was separated, and the aqueous layer was extracted with 20 mL of ether. Workup of the combined organic solutions afforded 1.38 g of an oily solid shown to be a 10:1 mixture of **7c** and **8c** (not rigorously characterized) by NMR. Recrystallization from carbon tetrachloride afforded 1.10 g (78%) of **7c** as white needles: mp 81–82 °C; IR (KBr) 3115 (w), 3030 (w), 2941 (w), 1675 (s), 1639 (m), 1608 (m), 1366 (m), 1325 (s), 1264 (m), 1209 (m), 1135 (s), 1110 (s), 1088 (m), 1003 (s), 965 (s), 947 (s), 819 (s), 796 (m), 775 cm^{-1} (w); NMR δ 7.03 (d, *J* = 3 Hz, 1 H), 6.51 (AB, $\Delta\nu$ = 25 Hz, *J* = 10 Hz, with the downfield component further split into a doublet, *J* = 3 Hz, 2 H), 4.12 (s, 4 H); exact mass calcd for $C_8H_7O_3{}^{79}Br$ *m/e* 229.9578, found *m/e* 229.9583.

Monohydrolysis of 5d. To a solution of 1.5 g (6.6 mmol) of **5d** in 25 mL of tetrahydrofuran was added 12.5 mL of 2% aqueous acetic acid. The hydrolysis was followed by VPC and quenched with 20 mL of saturated sodium bicarbonate. Workup gave 1.15 g of an oily solid which was ca. 7:1 mixture of **8d** and an uncharacterized compound (presumably **7d**). Recrystallization of this material from ether/hexane gave 0.83 g (68%) of **8d** as white crystals, mp 62–64 °C (lit.[3b] mp 63.5–64.5 °C).

A Boron-Stabilized Carbanion from the Reaction of Trimesitylborane with Strong Bases in Tetrahydrofuran and Dimethyl Sulfoxide

Brian G. Ramsey*[1a] and Lorne M. Isabelle[1b]

Departments of Chemistry, University of Oregon, Eugene, Oregon 94703, The Oregon Graduate Center, Beaverton, Oregon 97005, and San Francisco State University, San Francisco, California 94132

At the 1980 IUPAC Conference on Boron Chemistry, we presented[2] the preliminary report of the first spectroscopic observation of a carbanion, obtained from trimesitylborane, **1**, which was clearly stabilized by boron–carbon 2p-π bonding and existed as either an intimate or solvent-separated ion pair. Boron-stabilized "carbanions" have been implicated as intermediates in a number of reactions[3], and the syntheses of *B*-(lithiomethyl)-9-borabicyclonane and (3-lithio-2-octenyl)diisiamylborane have been reported[4] as examples of the formation of boron-stabilized "carbanions". More recently, Wilson has reported[5] the formation of (1-lithioalkyl)dimesitylboranes in tetrahydrofuran (THF) as examples of boron-stabilized carbanions. The degree of ionic character in the carbon–lithium bonds and the extent to which the α boryl substituent stabilized these "carbanions" was not discussed, however, in these papers. We present here a more detailed account of our own results which addresses these questions.

Trimesitylborane, **1**, reacts with *n*-butyllithium or methyllithium in THF and with dimsylsodium or dimsyllithium in dimethyl sulfoxide to form the anion **2**, (4-methylene-2,6,-dimethylphenyl)bis(2,4,6-trimethylphenyl)boron anion.

1   **2**
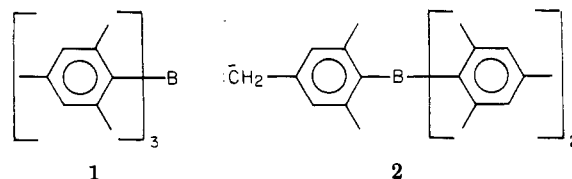
Solutions of the lithium or sodium salt of **2** in THF or Me$_2$SO are rose to purple red, depending on concentration, and exhibit a broad absorption band in the visible spectrum, λ_{max} 505 nm (log ϵ 4.4). The addition of lithium-

(1) (a) San Francisco State University;   (b) The Oregon Graduate Center.

(2) Brian G. Ramsey and Lorne Isabelle, Program Abstracts of International Union of Pure and Applied Chemistry, IMEBORON IV Conf., 1979, p 44.

(3) (a) D. S. Matteson, *Synthesis*, 147 (1975); (b) D. S. Matteson and J. R. Thomas, *J. Organomet. Chem.*, **24**, 263 (1970); (c) G. Cainelli, G. Dal Bello, and G. Zubiana, *Tetrahedron Lett.*, 3429 (1975); (d) G. Zweifel and H. Avzomanian, *ibid.*, 2535 (1966).

(4) M. W. Rathke and R. Kow, *J. Am. Chem. Soc.*, **94**, 6854 (1972); **95**, 2715 (1973).

(5) John Wilson, *J. Organomet. Chem.*, **186**, 297 (1980).

(16) Heller, J. E.; Dreiding, A. S.; O'Connor, B. R.; Simmons, H. E.; Buchanan, G. L.; Raphael, R. A.; Taylor, R. *Helv. Chim. Acta* **1973**, *56*, 272–280.