REVISED

Supporting Information

Known Compounds (CAS registry number in the parentheses):

1b[1] [131356-52-0], **1d**[2], **1e**[3] [60512-57-4], **1f**[3], **1g**[2] [90585-32-3], **1h**[4] [7436-90-0], **1j**[5] [56017-84-6],], **2k**[6] [15022-93-2], **3k** [58-72-0, commercially available from Aldrich], **4k** [501-65-5, commercially available from Aldrich], **4l**[7] [13633-26-6].

Methyl 4-(2,2-Dibromovinyl)benzoate (1a): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 (dt, J = 1.9, 8.4 Hz, 2H), 7.61 (dt, J = 1.8, 8.5 Hz, 2H), 7.52 (s, 1H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 139.0, 135.5, 129.4, 129.1, 127.8, 91.4, 51.7. Anal. calcd for $C_{10}H_8Br_2O_2$: C, 37.54; H, 2.52. Found: C, 37.74; H, 2.63.

1,1-Dibromo-2-(3-cyanophenyl)ethene (1c): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, J = 0.7 Hz, 1H), 7.66 (d, J = 6.8 Hz, 1H), 7.56 (d, J = 8.1Hz, 1H), 7.43 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.35, 134.48, 132.47, 131.63, 131.52, 129.24, 118.07, 112.06, 92.71. Anal. Calcd for $C_9H_5NBr_2$: C, 37.67; H, 1.76; N, 4.88. Found: C, 37.60; H, 1.64; N, 4.86.

1,1-Dibromo-2-(2-nitrophenyl)ethene (1i): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 (td, J = 2.2, 8.8 Hz, 2H), 7.70 (td, J = 2.1, 8.8 Hz, 2H), 7.56 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.2, 141.4, 134.9, 125.0, 123.7, 94.1. Anal. calcd for $C_8H_5NBr_2O_2$: C, 31.31; H, 1.64; N, 4.56. Found: C, 31.28; H, 1.82; N, 4.50.

Ethyl *trans*-2-(2,2-Dibromovinyl)cyclopropane-1-carboxylate (1k): ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.88 (d, *J* = 8.9 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 2.20 (m, 1H), 1.77 (m, 1H), 1.48 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.05 (m, 1 H).); ^{13}C NMR (75 MHz, $CDCl_3$) □ 172.2, 138.0, 89.1, 60.8, 25.1, 21.3, 14.9, 14.1. Anal. calcd for $C_8H_{10}Br_2O_2$: C, 32.25; H, 3.38. Found: C, 32.49; H, 3.39.

Products

Methyl (*Z*)-4-(2-Bromo-2-phenylvinyl)benzoate (2a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (td, *J* = 1.7, 8.5 Hz, 2H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.62 (m, 2H), 7.35 (m, 3H), 7.20 (s, 1H), 3.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 140.7, 140.5, 129.4, 129.3, 129.06, 129.03, 128.9, 128.3, 127.7, 126.2, 52.0; Anal. calcd for $C_{16}H_{13}BrO_2$: C, 60.59; H, 4.13. Found: C, 60.69; H, 4.28.

Methyl (*Z*)-4-[2-Bromo-2-(2-furanyl)vinyl]benzoate (2b): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (m, 2H), 7.79 (m, 2H), 7.54 (s, 1H), 7.48 (dd, *J* = 2, 0.7 Hz, 1H), 6.74 (d, *J* = 3.4 Hz, 1H), 6.47 (dd, *J* = 3.4, 1.7 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 152.1, 144.0, 140.0, 129.4, 129.20, 129.18, 112.4, 112.1, 112.0, 52.2. Anal. calcd for $C_{14}H_{11}BrO_3$: C, 54.75; H, 3.61. Found: C, 54.69, H; 3.51.

Methyl (*Z*)-4-[2-Bromo-2-(3-furanyl)vinyl]benzoate (2c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.1 Hz, 2H), 7.70 (s, 1H), 7.68 (d, *J* = 8.1 Hz, 2H), 7.41 (s, 1H), 7.12 (s, 1H), 6.62 (s, 1H), 3.87 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 143.9, 143.3, 140.0, 129.2, 129.02, 128.96, 127.7, 125.7, 115.4, 107.8, 51.9. Anal. calcd for $C_{14}H_{11}BrO_3$: C, 54.75; H, 3.61. Found: C, 54.86; H, 3.55.

Methyl (*Z*)-4-(2-Bromo-1,3-butadienyl)benzoate (2d): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 (m, 2H), 7.76 (m, 2H), 7.01 (s, 1H), 6.51 (dd, *J* = 15.9, 10.8 Hz, 1H), 5.79 (d, *J* = 16.2 Hz, 1H), 5.41 (d, *J* = 10.5 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 140.0, 136.8, 131.2, 129.4, 129.3, 128.9, 125.8, 120.3, 52.1. Anal. calcd for $C_{12}H_{11}BrO_2$: C, 53.96; H, 4.15. Found: C, 53.68; H, 3.98.

(*Z*)-1-Bromo-2-(4-cyanophenyl)-1-phenylethene (2e): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (m, 2H), 7.74-7.70 (m, 4H), 7.50-7.41 (m, 3H), 7.28 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.7, 140.2, 131.9, 129.7, 129.3, 128.4, 128.0, 127.7, 127.4, 118.7, 111.2, 105.3. Anal. calcd for $C_{15}H_{10}NBr$: C, 63.40; H, 3.55; N, 4.93. Found: C, 63.28; H, 3.67; N, 4.82.

(*Z*)-1-Bromo-2-(3-cyanophenyl)-1-phenylethene (2f): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.09 (d, *J* = 1.7 Hz, 1H), 7.98 (d, *J* = 6.8 Hz, 1H), 7.75 (m, 2H), 7.70 (dd, *J* = 1.3, 7.6 Hz, 1H), 7.60 (t, *J* = 8 Hz, 1H), 7.50 (m, 3H), 7.27 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.1, 137.5, 133.4, 132.5, 131.2, 129.3, 129.0, 128.4, 127.7, 127.6, 127.4, 126.9, 118.5, 112.4. Anal. calcd for $C_{15}H_{10}NBr$: C, 63.40; H, 3.55; N, 4.93. Found: C, 63.26; H, 3.41; N, 4.96.

(*Z*)-1-Bromo-2-(2-cyanophenyl)-1-phenylethene (2g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.68 (m, 3H), 7.62 (dt, *J* = 1.4, 7.6 Hz, 1H), 7.49 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ

140.0, 139.7, 132.7, 132.2, 129.56, 129.50, 129.4, 128.4, 128.05, 127.95, 125.8, 117.6, 112.7. Anal. calcd for $C_{15}H_{10}BrN$: C, 63.40; H, 3.55; N, 4.93. Found: C, 63.69; H, 3.69; N, 4.76.

(*Z*)-1-Bromo-2-(4-methoxyphenyl)-1-phenylethene (2h): Inseparable mixture with **3h** (ratio **2h**:**3h** = 88:12), ^{1}H NMR for **2h** (400 MHz, $CDCl_3$) δ 7.68 (td, *J* = 2.1, 8.9 Hz, 2H), 7.61 (m, 2H), 7.37-7.25 (m, 3H), 7.13 (s, 1H), 6.91 (td, *J* = 2.5, 8.9 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR for **2h** (100 MHz, $CDCl_3$) δ 159.3, 141.2, 130.7, 129.4, 128.7, 128.4, 128.3, 127.7, 122.0, 113.6, 55.2.

(*Z*)-1-Bromo-2-(3-methoxyphenyl)-1-phenylethene (2i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (td, *J* = 1.7, 6.4 Hz, 2H), 7.42-7.24 (m, 6H), 7.20 (s, 1H), 6.89 (ddd, *J* = 1.0, 2.4, 8.0 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 140.9, 137.4, 129.8, 129.1, 128.7, 128.3, 127.7, 124.2, 121.9, 114.3, 113.9, 55.2. Anal. calcd for $C_{15}H_{13}BrO$: C, 62.30; H, 4.53. Found: C, 62.64; H, 4.78.

(*Z*)-1-Bromo-2-(2-methoxyphenyl)-1-phenylethene (2j). Inseparable mixture with **3j** (ratio **2j**:**3j** = 66:34), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (from **3j**, dd, *J* = 1.7, 7.8 Hz, 2H), 7.68(from **2j**, td, *J* = 1.7, 6.8 Hz, 2H), 7.45 (m), 7.24 (m), 7.14 (dt, *J* = 7.0 Hz), 7.02 (dt, *J* = 1.1, 7.6 Hz), 6.90 (dd, *J* = 1.0, 8.1 Hz), 6.82 (dd, *J* = 1.0, 8.1 Hz), 6.73 (dd, 1.7, 7.8 Hz), 6.10 (dt, *J* = 1.0, 7.8 Hz), 3.84 (from **2j**, s, 3H), 3.82 (from **3j**, s, 3H).

(*Z*)-2-Bromo-1-phenyl-1,3-butadiene (2l): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 1H), 6.95 (s, 1H), 6.47 (dd, *J* = 10.2, 16.1 Hz, 1H), 5.72 (d,

J = 16.1 Hz, 1H), 5.31 (d, *J* = 10.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.1, 135.5, 132.3, 125, 128.2, 128.1, 123.8, 119.0. Anal. calcd for $C_{10}H_9Br$: C, 57.45; H, 4.34. Found: C, 57.12; H, 4.20.

(*Z*)-1-Bromo-2-(2-nitrophenyl)-1-phenylethene (2m): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 (td, *J* = 1.7, 6.8 Hz, 2H), 7.86 (td, *J* = 1.8, 6.9 Hz, 2H), 7.66 (m, 2H), 7.40 (m, 3H), 7.26 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.9, 142.8, 140.2, 129.9, 129.5, 128.5, 128.2, 127.82, 127.77, 123.5. Anal. calcd for $C_{14}H_{10}BrNO_2$: C, 55.29; H, 3.31; N, 4.61. Found: C, 55.59; H, 3.36; N, 4.56.

***4S*-4-[(*Z*)-2-Bromo-2-phenylvinyl]-2,2-dimethyl-1,3-dioxolane (2n):** ^{1}H NMR (400 MHz, dmso-d_6) δ 7.60 (m, 2H), 7.40 (m, 3H), 6.52 (d, *J* = 7.4 Hz, 1H), 4.93 (q, *J* = 6.9 Hz, 1H), 4.24 (dd, *J* = 6.5, 8.5 Hz, 1H), 3.74 (dd, *J* = 6.6, 8.3 Hz, 1H), 1.39 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (100 MHz, dmso-d_6) δ 138.3, 130.6, 129.5, 128.8, 127.6, 126.8, 109.3, 76.3, 68.2, 26.7, 25.9. Anal. calcd for $C_{13}H_{15}BrO_2$: C, 55.14; H, 5.34. Found: C, 54.85; H, 5.26.

Ethyl *trans*-2-[(*Z*)-2-Bromo-2-phenylvinyl]cyclopropane-1-carboxylate (2o): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (m, 2H), 7.29 (m, 3H), 5.65 (d, *J* = 8.9 Hz, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 2.54 (m, 1H), 1.81 (m, 1H), 1.56 (m, 1H), 1;28 (t, *J* = 7.0 Hz, 3H), 1.09 (t, 1 0.H); ^{13}C NMR (100 MHz, $CDCl_3$) d 172.7, 139.3, 131.3, 128.5, 128.2, 127.3, 125.7, 60.8, 25.5, 22.2, 15.8, 14.2; . Anal. calcd for $C_{14}H_{15}BrO_2$: C, 56.97; H, 5.12. Found: C, 57.32; H, 5.18.

Ethyl *trans*-2-[(*Z*)-2-Bromo-1,3-butadienyl]cyclopropane-1-carboxylate (2p): ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.26 (dd, *J* = 10.5, 16.2 Hz, 1H), 5.54 (d, *J* = 16.1 Hz, 1H), 5.43 (d, *J* = 9.3 Hz, 1H), 5.18 (d, *J*

= 10.6 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.50 (m, 1H), 1.76 (m, 1H), 1.53 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.05 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 135.1, 134.3, 125.9, 117.8, 60.7, 24.5, 22.3, 15.9, 14.2. Anal. calcd for $C_{10}H_{13}BrO2$: C, 49.00; H, 5.35. Found: C, 48.83; H, 5.41

Methyl 4-(2,2-Diphenylvinyl)benzoate (3a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.2 Hz, 2H),7.25 (m, 8H), 7.16 (m, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.95 (s, 1H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 144.9, 142.8, 142.0, 139.7, 130.1, 129.3, 129.1, 128.6, 128.2, 127.88, 127.85, 127.67, 127.61, 126.9, 51.8. Anal. calcd for $C_{20}H_{18}O_2$: C, 84.05; H, 5.77. Found: C, 83.82; H, 5.82.

(4*S*)-2,2-Dimethyl-4-(2,2-diphenylvinyl)-1,3-dioxolane (3n): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (m, 3H), 7.24 (m, 5H), 7.17 (m, 2H), 6.05 (d, *J* = 8.9 Hz, 1H), 4.56 (m, 1H), 4.03 (dd, *J* = 5.9, 8.0 Hz, 1H), 3.71 (t, *J* = 7.8 Hz, 1H), 1.47 (s, 3H), 1.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 141.5, 138.9, 129.7, 128.14, 128.09, 127.72, 127.62, 127.57, 126.1, 109.2, 74.0, 69.7, 26.8, 25.9. Anal. calcd for $C_{19}H_{20}O_2$: C, 81.40; H, 7.19. Found: C, 81.24, H, 7.23.

Methyl 4-(2-Phenylethynyl)benzoate (4a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (td, *J* = 1.7, 8.5 Hz, 2H), 7.55 (td, *J* =1.7, 8.5 Hz, 2H), 7.52 (m, 2H), 7.32 (m, 3H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 131.6, 131.3, 129.35, 129.33, 128.6, 128.3, 127.9, 122.6, 92.3, 88.5, 52.0. Anal. calcd for $C_{16}H_{12}O_2$: C, 81.34; H, 5.12. Found: C, 81.06; H, 5.24.

Methyl 4-[2-(2-Furanyl)ethynyl]benzoate (4b): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (m, 2H), 7.59 (m, 2H), 7.46 (dd, *J* = 1.7, 0.7 Hz, 1H), 6.71 (dd, *J* = 3.4, 0.6 Hz, 1H), 6.45 (dd, *J* = 3.7, 2 Hz, 1H), 3.92 (s,

3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 144.1, 136.7, 131.2, 129.8, 129.5, 127.0, 116.1, 111.2, 92.6, 82.3, 52.2. Anal. calcd for $C_{14}H_{10}O_3$: C, 74.33; H, 4.46. Found: C, 74.16; H, 4.25.

Methyl 4-[2-(3-Furanyl)ethynyl]benzoate (4c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (td, *J* = 1.8, 8.5 Hz, 2H), 7.72 (dd, *J* = 1.3, 2.1 Hz, 1H), 7.54 (td, *J* = 1.8, 8.5 Hz, 2H), 7.42 (t, *J* = 1.7 Hz, 1H), 6.53 (dd, *J* = 1.0, 1.7 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 146.0, 143.0, 131.3, 129.5, 129.4, 127.9, 112.4, 107.3, 90.4, 83.6, 52.2. Anal. calcd for $C_{14}H_{10}O_3$: C, 74.33; H, 4.46. Found: C, 74.33; H, 4.22.

Methyl 4-(But-3-en-1-ynyl)benzoate (4d): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 6.04 (dd, *J* = 17.3, 10.9 Hz, 1H), 5.78 (dd, *J* = 17.6, 2 Hz, 1H), 5.60 (dd, *J* = 11.2, 2 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5, 131.4, 129.6, 129. 5, 127. 9, 116.9, 90.9, 89.9, 52.2. Anal. calcd for $C_{10}H_{10}O_2$: C, 77.40; H, 5.41. Found: C, 74.10; H, 5.28.

1-(4-Cyanophenyl)-2-phenylethyne (4e): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51-7.42 (m, 6H), 7.28-7.24 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 131.92, 131.89, 131.67, 129.01, 128.39, 128.08, 122.11, 118.37, 111.35, 93.68, 87.65. Anal. calcd for $C_{15}H_9N$: C, 88.64; H, 4.46; N, 6.89. Found: C, 88.40; H, 4.58; N, 6.83.

1-(3-Cyanophenyl)-2-phenylethyne (4f): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (t, *J* = 1.7 Hz, 1H), 7.82 (dt, *J* = 6.7, 0.3 Hz, 1H), 7.69-7.62 (m, 3H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.48 (m, 3H); ^{13}C NMR (75 MHz,

$CDCl_3$) δ 135.4, 134.7, 131.6, 131.2, 129.1, 128.9, 128.36, 128.29, 128.27, 124.8, 122.2, 117.9, 112.7, 91.7, 86.8. Anal. calcd for $C_{15}H_9N$: C, 88.64; H, 4.46; N, 6.89. Found: C, 88.31; H, 4.77; N, 6.90.

1-(2-Cyanophenyl)-2-phenylethyne (4g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.57 (m, 4H), 7.52 (dt, *J* = 1.3, 7.8 Hz, 1H), 7.39-7.31 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.5, 132.3, 131.9, 131.8, 129.1, 128.3, 128.1, 127.0, 121.9, 117.4, 115.1, 95.8, 85.5. Anal. calcd for $C_{15}H_9N$: C, 88.65; H, 4.46; N, 6.89. Found: C, 88.39; H, 4.58; N, 6.97.

1-(4-Methoxyphenyl)-2-phenylethyne (4h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (m, 2H), 7.44 (td, *J* = 2.5, 8.9 Hz, 2H), 7.28 (m, 3H), 6.82 (td, *J* = 2.5, 8.9 Hz, 2H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 133.0, 131.4, 128.2, 127.8, 123.5, 115.3, 113.9, 89.4, 88.0, 55.1. Anal. calcd for $C_{15}H_{12}O$: C, 86.51; H, 5.81. Found: C, 86.23; H, 5.78.

1-(3-Methoxyphenyl)-2-phenylethyne (4i): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (m, 2H), 7.52 (m, 2H), 7.34 (m, 3H), 7.23 (d, *J* = 8.2 Hz, 1H), 7.13 (td, *J* = 1.2, 7.5 Hz, 1H), 7.06 (dd, *J* = 1.3, 2.6 Hz, 1H), 6.89 (ddd, *J* = 1.1, 2.4, 8.2 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.4, 131.6, 129.4, 128.32, 128.27, 124.26, 124.18, 123.2, 116.3, 114.9, 89.3, 89.2, 55.3. Anal. calcd for $C_{15}H_{12}O$: C, 86.51; H, 5.81. Found: C, 86.33; H, 5.80.

1-(2-Methoxyphenyl)-2-phenylethyne (4j): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55 (m, 2H), 7.49 (dd, *J* = 1.7, 7.5 Hz, 1H), 7.38-7.27 (m, 4H), 6.94 (m, 2H), 3.92 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.9,

133.5, 131.6, 129.7, 128.2, 128.1, 123.6, 120.5, 112.5, 110.7, 93.4, 85.7, 55.8. Anal. calcd for $C_{15}H_{12}O$: C, 86.51; H, 5.81. Found: C, 86.45; H, 5.65.

1-(2-Nitrophenyl)-2-phenylethyne (4m): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 (td, *J* = 2.1, 9.1 Hz, 2H), 7.67 (td, *J* = 2.1, 9.2 Hz, 2H), 7.56 (m, 2H), 7.40 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.0, 132.3, 131.8, 130.3, 129.3, 128.5, 123.6, 122.1, 94.7, 87.5. Anal. calcd for $C_{14}H_9NO_2$: C, 75.33; H, 4.06; N, 6.27. Found: C, 75.56; H, 4.37; N, 6.17.

(4*S*)-2,2-Dimethyl-4-(2-phenylethynyl)-1,3-dioxolane (4n): ^{1}H NMR (400 MHz, dmso-d_6) δ 7.40 (m, 2H), 7.40 (m, 3H), 5.02 (t, *J* = 6.0 Hz, 1H), 4.21 (dd, *J* = 6.3, 8.0 Hz, 1H), 3.93 (dd, *J* = 6.0, 8.0 Hz, 1H), 1.45 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (100 MHz, dmso-d_6) δ 131.6, 129.2, 128.9, 121.9, 109.8, 88.0, 85.1, 69.7, 65.6, 26.5, 26.0. Anal. calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 77.27; H, 6.98.

Ethyl *trans*-2-(2-phenylethynyl)cyclopropane-1-carboxylate (4o): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (m, 2H), 7.27 (m, 3H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.07 (m, 1H), 2.02 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.28 (m, 1H), ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 131.6, 128.2, 127.9, 123.1, 89.5, 77.7, 60.9, 23.1, 17.0, 14.2, 10.9. Anal. calcd for $C_{14}H_{14}O_2$: C, 78.48; H, 6.59. Found: C, 78.69; H, 6.49.

Ethyl *trans*-2-(but-3-en-1-ynyl)cyclopropane-1-carboxylate (4p): ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.73 (ddd, *J* = 1.7, 11.0, 17.8 Hz, 1H), 5.56 (dd, *J* = 2.4, 17.8 Hz, 1H), 5.41 (dd, *J* = 2.5, 11.0 Hz, 1H), 4.15 (q, *J* = 6.8 Hz, 2H), 1.95 (m, 1H), 1.42 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.20 (m, 1H); ^{13}C NMR

(100 MHz, $CDCl_3$) δ 172.1, 126.6, 116.9, 90.2, 76.5, 60.9, 23.0, 16.9, 14.2, 10.8. Anal. calcd for $C_{10}H_{12}O_2$: C, 73.15; H, 7.37. Found: C, 73.01; H, 7.57.

Methyl 4-[(*E*)-2-Methyl-2-phenylvinyl]benzoate (5a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (td, J = 1.7, 8.5 Hz, 2H), 7.49 (m, 2H), 7.39 (d, J = 8.2 Hz, 2H), 7.35 (m, 2H), 7.28 (m, 1H), 6.81 (s, 1H), 3.88 (s, 3H), 2.26 (d, J = 1.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 143.4, 142.9, 139.5, 129.4, 128.9, 128.3, 127.9, 127.5, 126.7, 125.9, 51.9, 17.6. Anal. calcd for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39. Found: C, 80.81; H, 6.44.

Methyl 4-[(*Z*)-2-(2-furanyl)-2-phenylvinyl]benzoate (5b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.0 Hz, 2H), 7.40 (m, 2H), 7.33 (m, 4H), 7.16 (d, J = 8.0 Hz, 2H), 6.80 (s, 1H), 6.40 (m, 1H), 6.21 (dd, J = 0.6, 3.5 Hz, 1H) 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 152.1, 142.14, 142.05, 140.9, 133.6, 129.2, 128.9, 128.6, 128.30, 128.22, 128.12, 112.1. 111.3, 51.9. Anal. calcd for $C_{20}H_{16}O_3$: C, 78.93; H, 5.30. Found: C, 78.82; H, 5.19.

Methyl 4-[(*E*)-2-(2-furanyl)-2-phenylvinyl]benzoate (5c): 1H NMR (400 MHz, CDCl3) d 7.75 (d, J = 8.1 Hz, 2H), 7.45 (dd, J = 0.6, 1.1 Hz, 1H), 7.36 (m,3H), 7.27 (m, 2H), 7.19 (s, 1H), 7.00 (d, J = 8.1 Hz, 2H), 6.35 (m, 1H), 5.95 (d, J = 3.4 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 155.5, 142.8, 141.3, 137.2, 133.7, 129.7, 129.16, 129.13, 128.8, 128.1, 127.9, 123.2, 111.7, 110.6, 51.9. Anal. calcd for $C_{20}H_{16}O_3$: C, 78.93; H, 5.30. Found: C, 78.80; H, 5.20.

(4*S*)-2,2-Dimethyl-4-[(*E*)-2-phenyl-1,3-butadienyl]-1,3-dioxolane (5d): ^{1}H NMR (400 MHz, CD_3OD) δ 7.35-7.24 (m, 5H), 6.85 (ddd, *J* = 0.8, 11.0, 17.4 Hz, 1H), 5.55 (d, *J* = 8.5 Hz, 1H), 5.36 (td, *J* = 1.8, 11.0 Hz, 1H), 5.10 (m, 2H), 4.17 (dd, *J* = 6.4, 8.1 Hz, 1H), 3.63 (t, *J* = 7.8 Hz, 1H), 1.41 (s, 3H), 1.40 (s, 3H); ^{13}C NMR (100 MHz, CD_3OD) δ 143.7, 140.0, 132.5, 127.9, 127.7, 127.1, 119.2, 109.0, 73.7, 72.3, 69.1, 25.6, 24.7. Anal. calcd for $C_{15}H_{18}O_2$: C, 78.23; H, 7.88. Found: C, 78.25; H, 7.98.

(4*S*)-2,2-Dimethyl-4-[(*Z*)-2-phenyl-1,3-butadienyl]-1,3-dioxolane (5e): ^{1}H NMR (400 MHz, CD_3OD) δ 7.36 (m, 3H), 7.11 (m, 2H), 6.62 (dd, *J* = 10.6, 17.3 Hz, 1H), 5.71 (d, 8.9 Hz, 1H), 5.14 (dd, *J* = 0.8, 10.6 Hz, 1H), 4.78 (dd, *J* = 0.8, 17.3 Hz, 1H), 4.33 (m, 1H), 3.89 (dd, *J* = 5.9, 8.0 Hz, 1H), 3.59 (t, 8.0 Hz, 1H), 1.38 (s, 3H), 1.21 (s, 3H); ^{13}C NMR (100 MHz, CD_3OD) δ 146.5, 140.9, 137.7, 130.7, 130.4, 129.3, 128.7, 118.1, 110.3, 74.9, 70.3, 27.0, 26.1. Anal. calcd for $C_{15}H_{18}O_2$: C, 78.23; H, 7.88. Found: C, 78.50; H, 8.02.

Ethyl *trans*-2-[(*Z*)-2-(2-furanyl)-2-phenylvinyl]cyclopropane-1-carboxylate (5f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (dd, *J* = 0.8, 1.7 Hz, 1H), 7.28 (m, 5H), 6.40 (dd, *J* = 1.7, 3.4 Hz, 1H), 6.25 (d, *J* = 3.4 Hz, 1H), 5.10 (d, *J* = 10.2 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.84 (m, 1H), 1.77 (m, 1H), 1.56 (m, 1H), 1.27 (t, *J* = 7.0 Hz, 3H), 1.06 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 153.0, 142.1, 140.8, 132.6, 131.2, 128.10, 128.06, 127.5, 111.2, 110.8, 60.6, 23.4, 23.2, 16.9, 14.2. Anal. calcd for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43. Found: C, 76.66; H, 6.34

Ethyl *trans*-2-[(*E*)-2-(2-furanyl)-2-phenylvinyl]cyclopropane-1-carboxylate (5g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 6H), 6.30 (dd, *J* = 2.1, 3.4 Hz, 1H), 5.83 (d, *J* = 3.4 Hz, 1H), 5.68 (d, *J* = 10.2 Hz, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 2.00 (m, 1H), 1.78 (m, 1H), 1.51 (m, 1H), 1.21 (t, *J* = 7.2 Hz, 3H),

1.06 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 154.9, 141.7, 136.9, 132.6, 129.8, 128.3, 127.7, 126.7, 111.3, 107.8, 60.5, 23.2, 22.9, 16.7, 14.2. Anal. calcd for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43. Found: C, 76.41; H, 6.56.

(1) (a) Lu, Y.-H.; Hsu, C.-S.; Wu, S.-T. Mol. *Cryst. Liq. Cryst. Sci. Technol., Sect. A* **1993**, *225*; 1. (b) Upasani, R. B.; Yang, Kevin C.; Acosta-Burruel, M.; Konkoy, C. S.; McLellan, J. A. *J. Med. Chem.* **1997**; *40*, 73.

(2) Topolski, M. *J. Org. Chem.* **1995**, *60*, 5588.

(3) Morris, J.; Wishka, D. G. *Synthesis* **1994**, 43.

(4) Ramirez, F.; Desai, N. B.; McKelvie, N. *J. Am. Chem. Soc.* **1962**, *84*, 1745.

(5) Gooding, O. W.; Beard, C. C.; Jackson, D. Y.; Wren, D. L.; Cooper, G. F. *J. Org. Chem.* **1991**, *56*, 1083.

(6) Kropp, P. J.; Crawford, S. D.; *J. Org. Chem.* **1994**, *59*, 3102.

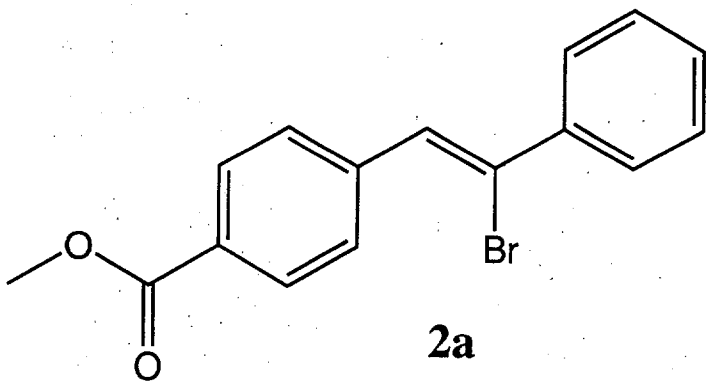
(7) Krijnen, E. S.; Zuilhof, H.; Lodder, G.; *J. Org. Chem.* **1994**, *59*, 8139.

Sample: XRAY1073

X-ray Structure Report

for

Wang Shen



2a

52271-69

Mon Nov 10 1997

11-12-97

Experimental

Data Collection

A clear rod crystal of $C_{16}H_{13}BrO_2$ having approximate dimensions of 0.60 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on an unknown diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 999 carefully centered reflections in the range $3.44 < 2\theta < 46.55°$ corresponded to a primitive monoclinic cell with dimensions:

$a = 12.3593(5)\ \AA$
$b = 20.4171(8)\ \AA \qquad \beta = 106.816(1)°$
$c = 11.6027(2)\ \AA$
$V = 2802.6(1)\ \AA^3$

For Z = 8 and F.W. = 317.18, the calculated density is 1.50 g/cm^3. The systematic absences of:

h0l: $l \neq 2n$
0k0: $k \neq 2n$

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of 20 $\pm$ 1°C using the ω scan technique to a maximum 2θ value of 46.6°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.00° with a take-off angle of 6.0°. Scans of (3.44 + 3.44 tan θ)° were made at a speed of 0.0°/min (in omega).

Data Reduction

Of the 5363 reflections which were collected, 3651 were unique (R_{int} = 0.147); equivalent reflections were merged. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 29.4 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-

squares refinement[3] was based on 2285 observed reflections ($I > 3.00\sigma(I)$) and 343 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.086$$

$$R_w = \sqrt{(\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2)}] = 0.087$$

The standard deviation of an observation of unit weight[4] was 3.13. The weighting scheme was based on counting statistics. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.59 and -0.68 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] .The values for the mass attenuation coefficients are those of Creagh and Hubbel[6]. All calculations were performed using the teXsan[7] crystallographic software package of Molecular Structure Corporation.

References

(1) ORIENT: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., Garcia-Granda, S., Gould, R.O., Smits, J.M.M. and Smykalla, C. (1992). The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(7) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{16}H_{13}BrO_2$
Formula Weight	317.18
Crystal Color, Habit	clear, rod
Crystal Dimensions	0.60 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	999 (3.4 - 46.5°)
Omega Scan Peak Width at Half-height	0.00°
Lattice Parameters	a = 12.3593(5)Å b = 20.4171(8) Å c = 11.6027(2) Å β = 106.816(1)°
	V = 2802.6(1) Å^3
Space Group	$P2_1/c$ (#14)
Z value	8
D_{calc}	1.503 g/cm^3
F_{000}	1280.00
μ(MoKα)	29.36 cm^{-1}

B. Intensity Measurements

Diffractometer	unknown

Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Take-off Angle	2.8°
Detector Aperture	2.0 - 2.5 mm horizontal 2.0 mm vertical
Crystal to Detector Distance	21 mm
Temperature	20.0°C
Scan Type	ω
Scan Rate	0.0°/min (in ω) (up to 0 scans)
Scan Width	(3.44 + 3.44 tan θ)°
$2\theta_{max}$	46.6°
No. of Reflections Measured	Total: 5363 Unique: 3651 (R_{int} = 0.147)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0000
No. Observations (I>3.00σ(I))	2285
No. Variables	343
Reflection/Parameter Ratio	6.66
Residuals: R; Rw	0.086 ; 0.087
Goodness of Fit Indicator	3.13
Max Shift/Error in Final Cycle	0.01
Maximum peak in Final Diff. Map	0.59 $e^-/Å^3$

Minimum peak in Final Diff. Map	-0.68 $e^-/\AA^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Br(1)	0.0279(1)	0.14384(8)	-0.2019(1)	7.36(4)
Br(2)	0.5052(1)	0.12927(8)	0.5928(1)	7.29(4)
O(3)	0.9740(6)	-0.1154(4)	0.5010(7)	6.1(2)
O(5)	1.0039(7)	-0.0740(5)	0.6848(8)	6.7(2)
O(6)	0.5113(6)	-0.0907(4)	0.1171(7)	5.5(2)
O(7)	0.4410(6)	-0.1033(4)	-0.0815(7)	6.1(2)
C(1)	-0.288(1)	0.2271(6)	-0.002(2)	6.4(4)
C(2)	0.297(1)	0.1726(7)	0.375(1)	5.6(3)
C(3)	0.4916(9)	0.1263(6)	0.4250(10)	4.4(3)
C(4)	0.8509(8)	-0.0316(6)	0.5289(9)	4.4(3)
C(6)	-0.1049(9)	0.1614(5)	-0.0371(10)	4.1(3)
C(7)	0.209(1)	0.2055(8)	0.302(1)	6.7(4)
C(8)	0.7884(9)	-0.0370(6)	0.4095(10)	4.9(3)
C(9)	-0.194(1)	0.1788(7)	-0.133(1)	6.2(4)
C(10)	0.3981(9)	0.1618(5)	0.3467(10)	4.2(3)
C(11)	0.3387(9)	-0.0369(5)	0.0185(10)	4.1(3)
C(12)	0.6621(9)	0.0483(6)	0.4412(9)	4.3(3)
C(13)	0.4335(10)	-0.0799(6)	0.010(1)	4.9(3)
C(15)	-0.287(1)	0.2119(7)	-0.115(1)	6.7(4)
C(16)	0.3384(9)	-0.0034(5)	0.1199(10)	4.8(3)
C(17)	0.244(1)	-0.0308(6)	-0.084(1)	5.3(3)
C(18)	0.9511(10)	-0.0762(6)	0.580(1)	4.8(3)
C(19)	0.2468(10)	0.0371(6)	0.1220(10)	4.6(3)
C(20)	0.5589(9)	0.0892(7)	0.380(1)	5.5(3)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(21)	0.1564(9)	0.0095(6)	-0.0832(10)	4.5(3)
C(22)	0.1550(8)	0.0443(5)	0.0188(9)	3.7(3)
C(23)	0.0596(9)	0.0842(6)	0.0255(10)	4.3(3)
C(24)	0.4030(10)	0.1876(6)	0.239(1)	5.3(3)
C(25)	-0.1101(10)	0.1763(6)	0.076(1)	5.3(3)
C(26)	-0.202(1)	0.2106(6)	0.093(1)	5.9(4)
C(27)	0.725(1)	0.0521(7)	0.557(1)	6.3(4)
C(28)	0.6956(9)	0.0038(6)	0.364(1)	5.3(3)
C(29)	0.822(1)	0.0116(6)	0.6046(9)	5.6(3)
C(30)	0.605(1)	-0.1323(7)	0.117(1)	6.9(4)
C(31)	-0.0033(9)	0.1242(6)	-0.052(1)	4.8(3)
C(32)	0.314(1)	0.2196(7)	0.162(1)	6.6(4)
C(33)	1.071(1)	-0.1592(8)	0.546(1)	7.4(4)
C(34)	0.214(1)	0.2282(7)	0.194(2)	7.1(4)
H(1)	-0.3528	0.2479	0.0106	7.3699
H(2)	0.2878	0.1556	0.4488	6.7987
H(3)	0.1388	0.2122	0.3198	7.5458
H(4)	0.8057	-0.0693	0.3586	6.7002
H(5)	-0.1957	0.1677	-0.2165	7.8862
H(6)	-0.3505	0.2234	-0.1793	8.4019
H(7)	0.3975	-0.0083	0.1904	6.3103
H(8)	0.2443	-0.0549	-0.1538	5.9917
H(9)	0.2475	0.0595	0.1929	5.5690
H(10)	0.5385	0.0874	0.2964	6.1884

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(11)	0.0968	0.0145	-0.1503	5.7442
H(12)	0.0406	0.0816	0.0986	5.3594
H(13)	0.4726	0.1835	0.2168	6.2344
H(14)	-0.0465	0.1644	0.1474	5.8084
H(15)	-0.1985	0.2229	0.1737	7.6845
H(16)	0.7060	0.0825	0.6104	8.2508
H(17)	0.6516	0.0017	0.2817	6.1486
H(18)	0.8671	0.0162	0.6871	7.4780
H(19)	0.5775	-0.1734	0.0825	8.6346
H(20)	0.6473	-0.1122	0.0683	8.6346
H(21)	0.6523	-0.1375	0.1956	8.6346
H(22)	0.3217	0.2366	0.0880	8.0845
H(23)	1.0618	-0.1841	0.6152	9.2653
H(24)	1.0828	-0.1865	0.4900	9.2653
H(25)	1.1390	-0.1321	0.5812	9.2653
H(26)	0.1513	0.2511	0.1376	7.4742

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	0.111(1)	0.097(1)	0.089(1)	0.0137(9)	0.0569(9)	0.0301(8)
Br(2)	0.0834(10)	0.135(1)	0.0643(8)	0.0125(9)	0.0310(7)	-0.0001(8)
O(3)	0.077(6)	0.086(7)	0.067(5)	0.017(5)	0.019(4)	-0.010(5)
O(5)	0.080(6)	0.096(7)	0.068(6)	0.023(5)	0.006(5)	0.004(5)
O(6)	0.063(5)	0.077(6)	0.064(5)	0.006(5)	0.011(4)	-0.006(4)
O(7)	0.069(5)	0.098(7)	0.071(5)	0.006(5)	0.030(4)	-0.010(5)
C(1)	0.072(10)	0.061(9)	0.12(1)	0.008(7)	0.050(10)	0.005(9)
C(2)	0.065(8)	0.075(9)	0.066(8)	-0.012(7)	0.011(7)	-0.012(7)
C(3)	0.051(7)	0.058(7)	0.061(7)	-0.017(6)	0.019(6)	-0.013(6)
C(4)	0.044(6)	0.064(8)	0.051(7)	-0.011(6)	0.003(6)	0.000(6)
C(6)	0.044(6)	0.049(7)	0.060(7)	-0.011(5)	0.008(6)	0.004(6)
C(7)	0.064(9)	0.11(1)	0.08(1)	-0.005(8)	0.022(8)	-0.009(9)
C(8)	0.048(7)	0.082(9)	0.052(7)	0.002(7)	0.006(6)	-0.012(6)
C(9)	0.064(8)	0.09(1)	0.090(10)	-0.010(8)	0.029(8)	-0.004(8)
C(10)	0.050(7)	0.062(8)	0.059(7)	-0.007(6)	0.031(6)	-0.009(6)
C(11)	0.052(7)	0.052(7)	0.056(7)	-0.010(6)	0.022(6)	0.001(6)
C(12)	0.051(7)	0.073(9)	0.037(6)	-0.003(6)	0.008(6)	-0.007(5)
C(13)	0.068(8)	0.060(8)	0.067(8)	-0.008(7)	0.032(8)	-0.009(7)
C(15)	0.051(8)	0.08(1)	0.11(1)	0.002(7)	0.004(8)	0.000(9)
C(16)	0.068(8)	0.049(8)	0.064(8)	-0.004(6)	0.018(6)	-0.003(6)
C(17)	0.085(9)	0.054(8)	0.071(8)	-0.017(7)	0.040(8)	-0.005(6)
C(18)	0.057(7)	0.069(9)	0.041(7)	-0.018(7)	-0.008(6)	0.008(6)
C(19)	0.068(8)	0.058(8)	0.054(7)	-0.005(6)	0.024(7)	-0.015(6)
C(20)	0.057(7)	0.09(1)	0.055(7)	0.009(7)	0.010(6)	-0.015(7)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(21)	0.045(7)	0.066(8)	0.052(7)	-0.001(6)	0.001(5)	-0.007(6)
C(22)	0.047(6)	0.044(7)	0.049(7)	-0.015(5)	0.011(6)	-0.001(5)
C(23)	0.053(7)	0.046(8)	0.065(7)	0.001(6)	0.017(6)	0.005(6)
C(24)	0.055(7)	0.070(9)	0.078(9)	0.003(7)	0.022(7)	0.003(7)
C(25)	0.079(9)	0.058(8)	0.066(8)	-0.004(7)	0.026(7)	0.003(6)
C(26)	0.071(9)	0.072(9)	0.085(9)	0.019(8)	0.030(8)	-0.010(7)
C(27)	0.077(9)	0.09(1)	0.065(9)	0.025(8)	0.011(7)	-0.009(7)
C(28)	0.049(7)	0.09(1)	0.053(7)	-0.008(7)	0.006(6)	0.000(6)
C(29)	0.075(8)	0.094(10)	0.037(6)	0.003(8)	0.005(6)	-0.024(6)
C(30)	0.073(8)	0.08(1)	0.11(1)	0.022(8)	0.028(8)	-0.003(8)
C(31)	0.066(7)	0.056(8)	0.068(7)	-0.014(7)	0.032(7)	-0.003(7)
C(32)	0.09(1)	0.070(10)	0.10(1)	0.005(8)	0.036(9)	-0.003(8)
C(33)	0.080(9)	0.11(1)	0.10(1)	0.035(9)	0.030(8)	0.005(8)
C(34)	0.063(9)	0.07(1)	0.12(1)	0.021(8)	-0.003(9)	0.004(8)

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
Br(1)	C(31)	1.92(1)	Br(2)	C(3)	1.91(1)
O(3)	C(18)	1.31(1)	O(3)	C(33)	1.47(2)
O(5)	C(18)	1.20(1)	O(6)	C(13)	1.35(1)
O(6)	C(30)	1.44(1)	O(7)	C(13)	1.19(1)
C(1)	C(15)	1.36(2)	C(1)	C(26)	1.33(2)
C(2)	C(7)	1.35(2)	C(2)	C(10)	1.40(1)
C(3)	C(10)	1.44(2)	C(3)	C(20)	1.34(1)
C(4)	C(8)	1.38(1)	C(4)	C(18)	1.51(2)
C(4)	C(29)	1.37(2)	C(6)	C(9)	1.37(2)
C(6)	C(25)	1.37(2)	C(6)	C(31)	1.52(2)
C(7)	C(34)	1.34(2)	C(8)	C(28)	1.39(2)
C(9)	C(15)	1.39(2)	C(10)	C(24)	1.37(2)
C(11)	C(13)	1.49(1)	C(11)	C(16)	1.36(1)
C(11)	C(17)	1.41(2)	C(12)	C(20)	1.52(2)
C(12)	C(27)	1.35(2)	C(12)	C(28)	1.42(2)
C(16)	C(19)	1.41(2)	C(17)	C(21)	1.37(2)
C(19)	C(22)	1.40(1)	C(21)	C(22)	1.39(1)
C(22)	C(23)	1.45(1)	C(23)	C(31)	1.30(1)
C(24)	C(32)	1.36(2)	C(25)	C(26)	1.39(2)
C(27)	C(29)	1.42(2)	C(32)	C(34)	1.40(2)

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.95	C(2)	H(2)	0.96
C(7)	H(3)	0.97	C(8)	H(4)	0.95
C(9)	H(5)	0.99	C(15)	H(6)	0.94
C(16)	H(7)	0.93	C(17)	H(8)	0.94
C(19)	H(9)	0.94	C(20)	H(10)	0.93
C(21)	H(11)	0.91	C(23)	H(12)	0.95
C(24)	H(13)	0.97	C(25)	H(14)	0.99
C(26)	H(15)	0.96	C(27)	H(16)	0.96
C(28)	H(17)	0.96	C(29)	H(18)	0.96
C(30)	H(19)	0.95	C(30)	H(20)	0.96
C(30)	H(21)	0.94	C(32)	H(22)	0.95
C(33)	H(23)	0.99	C(33)	H(24)	0.90
C(33)	H(25)	0.99	C(34)	H(26)	0.98

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(18)	O(3)	C(33)	116.4(10)	C(13)	O(6)	C(30)	117.1(9)
C(15)	C(1)	C(26)	120(1)	C(7)	C(2)	C(10)	122(1)
Br(2)	C(3)	C(10)	116.5(7)	Br(2)	C(3)	C(20)	122.2(9)
C(10)	C(3)	C(20)	121.1(10)	C(8)	C(4)	C(18)	120(1)
C(8)	C(4)	C(29)	121(1)	C(18)	C(4)	C(29)	118(1)
C(9)	C(6)	C(25)	118(1)	C(9)	C(6)	C(31)	122(1)
C(25)	C(6)	C(31)	119(1)	C(2)	C(7)	C(34)	121(1)
C(4)	C(8)	C(28)	119(1)	C(6)	C(9)	C(15)	120(1)
C(2)	C(10)	C(3)	122(1)	C(2)	C(10)	C(24)	115(1)
C(3)	C(10)	C(24)	121.9(9)	C(13)	C(11)	C(16)	123(1)
C(13)	C(11)	C(17)	117(1)	C(16)	C(11)	C(17)	118(1)
C(20)	C(12)	C(27)	126(1)	C(20)	C(12)	C(28)	114.8(9)
C(27)	C(12)	C(28)	118(1)	O(6)	C(13)	O(7)	122(1)
O(6)	C(13)	C(11)	113(1)	O(7)	C(13)	C(11)	124(1)
C(1)	C(15)	C(9)	119(1)	C(11)	C(16)	C(19)	120(1)
C(11)	C(17)	C(21)	120(1)	O(3)	C(18)	O(5)	124(1)
O(3)	C(18)	C(4)	114.1(10)	O(5)	C(18)	C(4)	121(1)
C(16)	C(19)	C(22)	120.3(10)	C(3)	C(20)	C(12)	131(1)
C(17)	C(21)	C(22)	121(1)	C(19)	C(22)	C(21)	118.0(10)
C(19)	C(22)	C(23)	118.9(9)	C(21)	C(22)	C(23)	122.9(10)
C(22)	C(23)	C(31)	130(1)	C(10)	C(24)	C(32)	123(1)
C(6)	C(25)	C(26)	120(1)	C(1)	C(26)	C(25)	120(1)
C(12)	C(27)	C(29)	122(1)	C(8)	C(28)	C(12)	120(1)
C(4)	C(29)	C(27)	118.1(10)	Br(1)	C(31)	C(6)	112.8(8)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
Br(1)	C(31)	C(23)	121.6(8)	C(6)	C(31)	C(23)	125.5(10)
C(24)	C(32)	C(34)	118(1)	C(7)	C(34)	C(32)	118(1)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(15)	C(1)	H(1)	119.8	C(26)	C(1)	H(1)	119.6
C(7)	C(2)	H(2)	118.0	C(10)	C(2)	H(2)	119.8
C(2)	C(7)	H(3)	122.6	C(34)	C(7)	H(3)	116.1
C(4)	C(8)	H(4)	121.1	C(28)	C(8)	H(4)	119.4
C(6)	C(9)	H(5)	121.0	C(15)	C(9)	H(5)	118.4
C(1)	C(15)	H(6)	117.4	C(9)	C(15)	H(6)	122.7
C(11)	C(16)	H(7)	120.5	C(19)	C(16)	H(7)	118.6
C(11)	C(17)	H(8)	118.3	C(21)	C(17)	H(8)	121.1
C(16)	C(19)	H(9)	119.9	C(22)	C(19)	H(9)	119.9
C(3)	C(20)	H(10)	114.6	C(12)	C(20)	H(10)	113.6
C(17)	C(21)	H(11)	120.6	C(22)	C(21)	H(11)	117.9
C(22)	C(23)	H(12)	115.6	C(31)	C(23)	H(12)	114.0
C(10)	C(24)	H(13)	119.2	C(32)	C(24)	H(13)	117.8
C(6)	C(25)	H(14)	119.8	C(26)	C(25)	H(14)	119.5
C(1)	C(26)	H(15)	122.6	C(25)	C(26)	H(15)	117.2
C(12)	C(27)	H(16)	120.2	C(29)	C(27)	H(16)	117.5
C(8)	C(28)	H(17)	121.2	C(12)	C(28)	H(17)	118.3
C(4)	C(29)	H(18)	121.0	C(27)	C(29)	H(18)	120.8
O(6)	C(30)	H(19)	109.4	O(6)	C(30)	H(20)	108.8
O(6)	C(30)	H(21)	110.0	H(19)	C(30)	H(20)	108.7
H(19)	C(30)	H(21)	110.8	H(20)	C(30)	H(21)	109.2
C(24)	C(32)	H(22)	120.1	C(34)	C(32)	H(22)	121.0
O(3)	C(33)	H(23)	108.9	O(3)	C(33)	H(24)	114.0
O(3)	C(33)	H(25)	108.3	H(23)	C(33)	H(24)	110.6

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H(23)	C(33)	H(25)	103.6	H(24)	C(33)	H(25)	110.8
C(7)	C(34)	H(26)	123.0	C(32)	C(34)	H(26)	118.1

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Br(1)	C(31)	C(6)	C(9)	30(1)	Br(1)	C(31)	C(6)	C(25)	-151.6(8)
Br(1)	C(31)	C(23)	C(22)	-4(1)	Br(2)	C(3)	C(10)	C(2)	-28(1)
Br(2)	C(3)	C(10)	C(24)	151.3(9)	Br(2)	C(3)	C(20)	C(12)	-6(1)
O(3)	C(18)	C(4)	C(8)	3(1)	O(3)	C(18)	C(4)	C(29)	-179(1)
O(5)	C(18)	O(3)	C(33)	0(1)	O(5)	C(18)	C(4)	C(8)	-177(1)
O(5)	C(18)	C(4)	C(29)	0(1)	O(6)	C(13)	C(11)	C(16)	9(1)
O(6)	C(13)	C(11)	C(17)	-170.3(10)	O(7)	C(13)	O(6)	C(30)	0(1)
O(7)	C(13)	C(11)	C(16)	-170(1)	O(7)	C(13)	C(11)	C(17)	9(1)
C(1)	C(15)	C(9)	C(6)	0(2)	C(1)	C(26)	C(25)	C(6)	2(1)
C(2)	C(7)	C(34)	C(32)	-2(2)	C(2)	C(10)	C(3)	C(20)	146(1)
C(2)	C(10)	C(24)	C(32)	-2(1)	C(3)	C(10)	C(2)	C(7)	-179(1)
C(3)	C(10)	C(24)	C(32)	177(1)	C(3)	C(20)	C(12)	C(27)	-16(2)
C(3)	C(20)	C(12)	C(28)	165(1)	C(4)	C(8)	C(28)	C(12)	-2(1)
C(4)	C(18)	O(3)	C(33)	179.3(10)	C(4)	C(29)	C(27)	C(12)	0(2)
C(6)	C(31)	C(23)	C(22)	177(1)	C(7)	C(2)	C(10)	C(24)	0(1)
C(7)	C(34)	C(32)	C(24)	1(2)	C(8)	C(4)	C(29)	C(27)	0(1)
C(8)	C(28)	C(12)	C(20)	-179(1)	C(8)	C(28)	C(12)	C(27)	2(1)
C(9)	C(6)	C(25)	C(26)	-2(1)	C(9)	C(6)	C(31)	C(23)	-150(1)
C(9)	C(15)	C(1)	C(26)	0(2)	C(10)	C(2)	C(7)	C(34)	2(2)
C(10)	C(3)	C(20)	C(12)	178(1)	C(10)	C(24)	C(32)	C(34)	1(2)
C(11)	C(13)	O(6)	C(30)	179.4(9)	C(11)	C(16)	C(19)	C(22)	0(1)
C(11)	C(17)	C(21)	C(22)	-1(1)	C(13)	C(11)	C(16)	C(19)	179.3(10)
C(13)	C(11)	C(17)	C(21)	-178.0(10)	C(15)	C(1)	C(26)	C(25)	-1(2)
C(15)	C(9)	C(6)	C(25)	1(1)	C(15)	C(9)	C(6)	C(31)	178(1)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(16)	C(11)	C(17)	C(21)	2(1)	C(16)	C(19)	C(22)	C(21)	1(1)
C(16)	C(19)	C(22)	C(23)	177.3(10)	C(17)	C(11)	C(16)	C(19)	0(1)
C(17)	C(21)	C(22)	C(19)	0(1)	C(17)	C(21)	C(22)	C(23)	-175(1)
C(18)	C(4)	C(8)	C(28)	179(1)	C(18)	C(4)	C(29)	C(27)	-178(1)
C(19)	C(22)	C(23)	C(31)	141(1)	C(20)	C(3)	C(10)	C(24)	-33(1)
C(20)	C(12)	C(27)	C(29)	-179(1)	C(21)	C(22)	C(23)	C(31)	-42(1)
C(23)	C(31)	C(6)	C(25)	26(1)	C(26)	C(25)	C(6)	C(31)	179(1)
C(28)	C(8)	C(4)	C(29)	1(1)	C(28)	C(12)	C(27)	C(29)	-1(1)

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
Br(1)	O(3)	3.512(8)	65503	Br(2)	O(6)	3.520(8)	65603
O(5)	C(21)	3.28(1)	65601	O(5)	C(25)	3.41(1)	65603
O(5)	C(17)	3.49(2)	65601	O(7)	C(1)	3.46(1)	3
O(7)	C(24)	3.48(1)	65503	O(7)	C(20)	3.48(1)	65503
C(1)	C(13)	3.49(2)	3	C(17)	C(25)	3.42(2)	3
C(17)	C(28)	3.58(2)	65503	C(21)	C(23)	3.50(1)	3

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ±4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

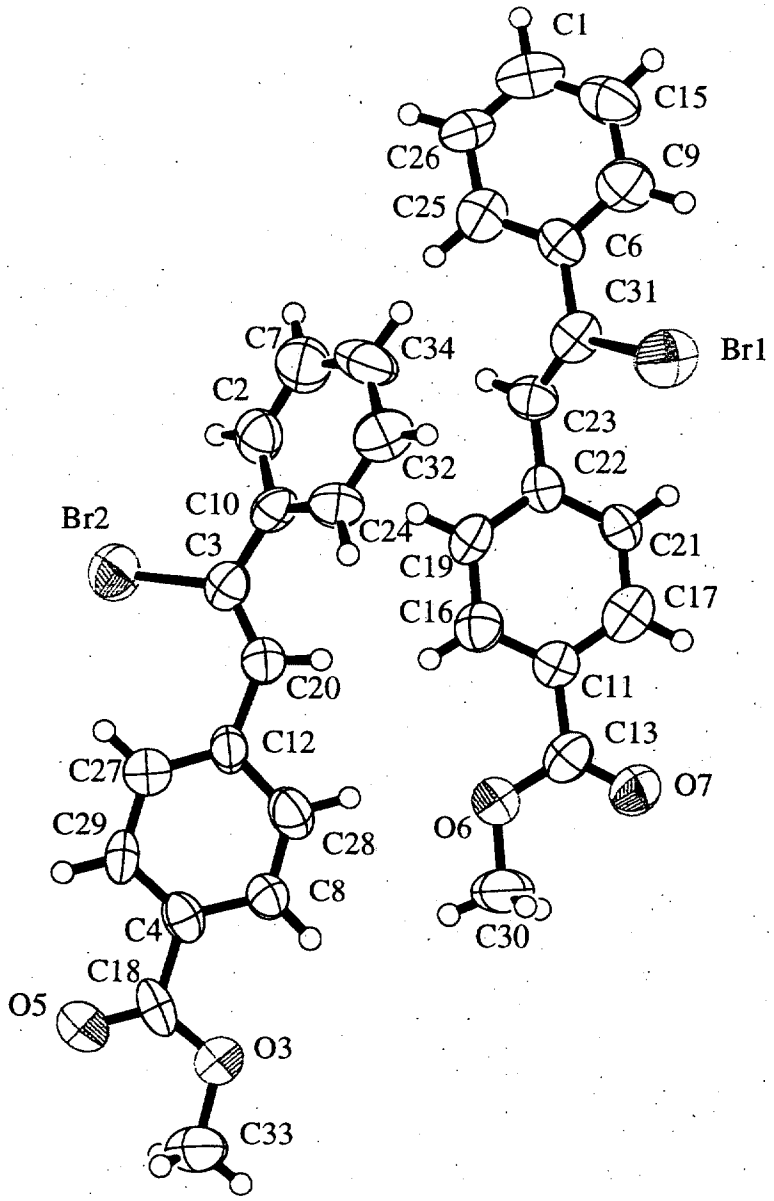
The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	X,	Y,	Z	(2)	-X,	1/2+Y,	1/2-Z
(3)	-X,	-Y,	-Z	(4)	X,	1/2-Y,	1/2+Z

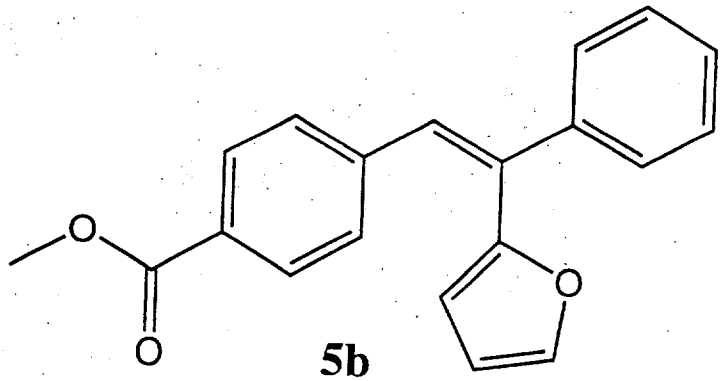

C1
C15
C26
C9
C25
C6
C31
Br1
C7
C34
C2
C23
C32
C22
C10
Br2
C24
C21
C3
C19
C17
C16
C20
C11
C12
C13
C27
O7
C29
O6
C28
C8
C4
C30
C18
O5
O3
C33

Sample: XRAY1074

X-ray Structure Report

for

Wang Shen



5b

52271-77

Mon Nov 10 1997

Experimental

Data Collection

A yellow rod crystal of $C_{20}H_{16}O_3$ having approximate dimensions of 0.50 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on an unknown diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 999 carefully centered reflections in the range $3.96 < 2\theta < 46.51°$ corresponded to a primitive monoclinic cell with dimensions:

$a = 8.8886(1)\ \AA$

$b = 10.2937(2)\ \AA \qquad \beta = 91.842(1)°$

$c = 17.4205(4)\ \AA$

$V = 1593.09(4)\ \AA^3$

For Z = 4 and F.W. = 304.34, the calculated density is 1.27 g/cm^3. The systematic absences of:

h0l: $h+l \neq 2n$

0k0: $k \neq 2n$

uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of 20 $\pm$ 1°C using the ω scan technique to a maximum 2θ value of 46.5°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.00° with a take-off angle of 6.0°. Scans of $(0.00 + 0.00 \tan \theta)°$ were made at a speed of 0.0°/min (in omega).

Data Reduction

Of the 6123 reflections which were collected, 2462 were unique ($R_{int} = 0.040$); equivalent reflections were merged. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.8 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-

matrix least-squares refinement[3] was based on 1737 observed reflections ($I > 3.00\sigma(I)$) and 208 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.055$$

$$R_w = \sqrt{(\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2)}] = 0.067$$

The standard deviation of an observation of unit weight[4] was 4.39. The weighting scheme was based on counting statistics. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.20 and -0.27 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] .The values for the mass attenuation coefficients are those of Creagh and Hubbel[6]. All calculations were performed using the teXsan[7] crystallographic software package of Molecular Structure Corporation.

References

(1) SHELXS86: Sheldrick, G.M. (1985). In: "Crystallographic Computing 3" (Eds G.M. Sheldrick, C. Kruger and R. Goddard) Oxford University Press, pp. 175-189.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(7) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{20}H_{16}O_3$
Formula Weight	304.34
Crystal Color, Habit	yellow, rod
Crystal Dimensions	0.50 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	999 (4.0 - 46.5°)
Omega Scan Peak Width at Half-height	0.00°
Lattice Parameters	a = 8.8886(1)Å b = 10.2937(2) Å c = 17.4205(4) Å β = 91.842(1)° V = 1593.09(4) Å^3
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.269 g/cm^3
F_{000}	640.00
μ(MoKα)	0.85 cm^{-1}

B. Intensity Measurements

Diffractometer	unknown