(Supporting Information)

Diastereoselective Addition of γ -Substituted Allylic Nucleophiles to Ketones: Highly Stereoselective Synthesis of Tertiary Homoallylic Alcohols Using an Allylic Tributylstannane/Stannous Chloride System

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Determination of Relative Stereochemistry.

Determination of Stereochemistry of 3. The esterification of homoallylic alcohol **3a** by Vedejs's method¹ gave ester **3aE**, which was recrystallized to afford a suitable crystalline for X-ray analysis. The relative configuration was unambiguously assigned to $2S^*,3R^*$ -form as shown in Scheme 4.² The transformation of Br on the aryl ring in **3c** to H, Cl, Me, or OMe was performed with retention of the stereochemistry of the carbon chain, and the relative configuration of all products **3a-e** was thus proved to be $2S^*,3R^*$ -form (Scheme 4).

Scheme 4. Determination of Stereochemistry of **3**

Determination of Stereochemistry of 6. The alcohol **6b** was also esterified to give **6bE** as a solid, whose structure was determined by X-ray analysis. The relative configuration was unambiguously assigned as shown in Scheme 5. The alcohol **6c** was treated by the same procedures as in Scheme 4 and all products **6a-e** have the same relative stereochemistry.

Scheme 5. Determination of Stereochemistry of **6**

Determination of Stereochemistry of 7 and 8. The stereochemistry of **7a** and **8a** was determined according to the reported spectroscopic data.³ The product **8b** was transformed to the corresponding *p*-nitrobenzoyl ester **8bE** by Greenwald's method,⁴ whose stereochemistry was determined by X-ray analysis (Scheme 6). The stereochemistry of **7f** was also determined by the X-ray analysis of the ester **7fE** (Scheme 7).

Scheme 6. Determination of Stereochemistry of **8b**

Scheme 7. Determination of Stereochemistry of **7f**

Determination of Stereochemistry of 17. The product **17d** was esterified as shown in Scheme 8 to give **17dE** as a solid, which was crystallized for X-ray analysis and proved to have (3*R**,4*S**)-configuration. Methylation of **17d** led to the compound **17a**, which shows identical spectra with the product obtained in Table 4, entry 1. The product **17e** gave a suitable crystalline for X-ray analysis and its stereochemistry was determined as shown in Scheme 8. Since the compound **17c** was derived from **17e** by methylation of the hydroxyl group, it was also found to have the same stereochemistry.

Scheme 8. Determination of Stereochemistry of **17**

Determination of Stereochemistry of 18. The stereochemistry of the products **18** was determined by the procedure as shown in Scheme 9. A transformation of **18e** to dioxolane **19** by an acetone/ TsOH system gave a suitable crystalline for X-ray analysis. The methoxy compound **18c** was obtained from **18e** and proved to be the same configuration.

Scheme 9. Determination of Stereochemistry of **18c** and **18d**.

Experimental Section

General. IR spectra were recorded as thin films on a Hitachi 260-30 or a HORIBA FT-720 spectrophotometer. ^1H and ^{13}C NMR spectra were obtained with a JEOL JNM-GSX-270 (270 and 67.9 MHz) spectrometers, respectively, with TMS as internal standard. Mass spectra were recorded on a JEOL JMS-DS303 spectrometer. GLC analyses were performed on a Shimadzu GC-8A with FID using a $2\text{ m} \times 3\text{ mm}$ column packed with SE-52. Column chromatography was performed on silica gel (Fuji Silysia BW-200). Bulb-to-bulb distillation (Kugelrohr) was accomplished in a Sibata GTO-250RS at the oven temperature and pressure indicated.

Materials. Acetonitrile was distilled from P_2O_5 . Dichloromethane was distilled from CaH_2 . Tributylcinnamyltin (**1a**),⁵ 3-tributylstannylcyclohexene (**1b**),⁶ 3-tributylstannylcyclopentene (**1c**),⁶ and tributylcrotyltin (**1d**⁷ and **1e**⁶) were prepared by known methods. The ketones **2a-j**, **16a-e**, SnCl_2 , and $\text{BF}_3 \cdot \text{OEt}_2$ were commercially available.

Grignard Reaction with Acetophenone (eq 1). A flask was charged with dried Et_2O (5 mL) and 0.0535 g of magnesium turnings under nitrogen and was cooled to 0°C . Cinnamyl chloride (2 mmol) was slowly added and the mixture was stirred vigorously for 30 min. Acetophenone (1.0 mmol) was added to the Grignard reagent solution at 0°C . After stirring for 1 h, water (5 mL) was added to quench the reaction. The water phase was extracted with Et_2O and the organic layer was dried over MgSO_4 . The solvent was distilled off under reduced pressure. The formation of **3a** (62%) and **3b** (26%) was confirmed by NMR spectra of the crude mixture.

General Procedure (I) for Synthesis of Homoallylic Alcohols Using SnCl_2 . To a mixture of SnCl_2 (1.0 mmol) and ketone **2** or **16** (1.0 mmol) in dry acetonitrile (2 mL) was added allylic stannane **1** (1.0 mmol) under nitrogen. The reaction mixture was stirred under the reaction conditions noted in the text. Diethyl ether (30 mL) and an aqueous NH_4F (15%; 15 mL) were added and the resulting Bu_3SnF was filtered off. The filtrate was washed with water ($30\text{ mL} \times 2$), dried (MgSO_4) and evaporated. Column chromatography of the resultant residue on silica gel followed by distillation or recrystallization gave pure products.

General Procedure (II) for Synthesis of Homoallylic Alcohols Using $\text{BF}_3 \cdot \text{OEt}_2$. To a mixture of $\text{BF}_3 \cdot \text{OEt}_2$ (2.0 mmol) and ketone **2** or **16** (1.0 mmol) in dry dichloromethane (1 mL)

was added allylic tributylstannane **1** (1.0 mmol) under nitrogen. The reaction mixture was stirred under the reaction conditions noted in the text. Diethyl ether (30 mL) and aqueous NH_4F (15%; 15 mL) were added and the resulting Bu_3SnF was filtered off. The filtrate was washed with water (30 mL $\times$ 2), dried (MgSO_4) and evaporated. Column chromatography of the resultant residue on silica gel followed by distillation or recrystallization gave pure products.

Product data. The spectral data of **7a** and **8a** were in an excellent agreement with the reported data.³

(2S*,3R*)-2,3-Diphenyl-4-penten-2-ol (3a). According to the general procedure (I), this compound was prepared from **1a**, SnCl_2 , and **2a** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 105 °C/2 mmHg; IR: (neat) 3556, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.40-7.10 (m, 10H, aroma), 6.12 (ddd, $J = 17.1, 10.3, 8.8$ Hz, 1H, 4-H), 5.05 (ddd, $J = 10.3, 2.0, 1.0$ Hz, 1H, 5- H^{A}), 4.94 (ddd, $J = 17.1, 2.0, 1.0$ Hz, 1H, 5- H^{B}), 3.63 (d, $J = 8.8$ Hz, 1H, 3-H), 2.04 (brs, 1H, OH, D_2O -exchangeable), 1.44 (s, 3H, 1- H_3); ^{13}C NMR: (67.9 MHz, CDCl_3) 146.37 (s, ipso), 140.11 (s, ipso), 137.37 (d, C-4), 129.59 (d), 128.05 (d), 127.74 (d), 126.77 (d), 126.54 (d), 125.48 (d), 117.98 (t, C-5), 76.25 (s, C-2), 61.84 (d, C-3), 28.47 (q, C-1); MS: (CI, 70 eV) m/z 239 ($\text{M}^+ + 1$, 2.7), 221 (97), 195 (2.5), 171 (4), 161 (7), 143 (100), 121 (96), 105 (24), 91 (17); HRMS: (CI, 70 eV) calcd for $\text{C}_{17}\text{H}_{19}\text{O}$ 239.1358, found m/z 239.1426, 239.1452 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{17}\text{H}_{18}\text{O}$: C, 85.67; H, 7.61. Found: C, 85.27; H, 7.64.

(2R*,3R*)-2,3-Diphenyl-4-penten-2-ol (4a). According to the general procedure (II), this compound was prepared from **1a**, $\text{BF}_3 \cdot \text{OEt}_2$, and **2a** in dichloromethane to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 125 °C/4 mmHg; IR: (neat) 3563, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.40-6.90 (m, 10H, aroma), 6.26 (m, 1H, 4-H), 5.18 (dd, $J = 10.3, 1.5$ Hz, 1H, 5- H^{A}), 5.10 (dd, $J = 17.1, 1.5$ Hz, 1H, 5- H^{B}), 3.60 (d, $J = 9.3$ Hz, 1H, 3-H), 2.09 (br, 1H, OH, D_2O -exchangeable), 1.56 (s, 3H, 1- H_3); ^{13}C NMR: (67.9 MHz, CDCl_3) 146.08 (s, ipso), 140.06 (s, ipso), 137.19 (d, C-4), 129.32 (d), 127.78 (d), 127.50 (d), 126.49 (d), 125.62 (d), 117.94 (t, C-5), 76.53 (s, C-2), 61.19 (d, C-3), 27.85 (q, C-1); MS: (CI, 70 eV) m/z 239 ($\text{M}^+ + 1$, 0.04), 221 (49), 161 (2.8), 143 (38), 121 (100), 105 (10), 91 (6); HRMS: (CI, 70 eV) calcd for $\text{C}_{17}\text{H}_{19}\text{O}$ 239.1358, found m/z 239.1420, 239.1429 ($\text{M}^+ + 1$).

(2S*,3R*)-2-(4-Chlorophenyl)-3-phenyl-4-penten-2-ol (3b). According to the general procedure (I), this compound was prepared from **1a**, SnCl_2 , and ketone **2b** in acetonitrile to give the product as a white solid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by recrystallization (hexane): mp: 73-74 °C; IR: (KBr) 3583, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.30-7.05 (m, 9H, aroma), 6.11 (ddd, $J = 17.1, 10.3, 8.8$ Hz, 1H, 4-H), 5.06 (d, $J = 10.3$ Hz, 1H, 5- H^{A}), 4.92 (d, $J = 17.1$ Hz, 1H, 5- H^{B}), 3.56 (d, $J = 8.8$ Hz, 1H, 3-H), 1.99 (brs, 1H, OH, D_2O -exchangeable), 1.42 (s, 3H, 1- H_3); ^{13}C NMR: (67.9 MHz, CDCl_3) 144.87 (s, ipso), 139.75 (s, ipso), 136.97 (d, C-4), 132.34 (s, ipso), 129.47 (d), 128.16 (d), 127.79 (d), 127.07 (d), 126.90 (d), 118.30 (t, C-5), 75.96 (s, C-2), 61.87 (d, C-3), 28.30 (q, C-1); MS: (CI, 70 eV) m/z 275 ($\text{M}^+ + 3$, 1.3), 273 ($\text{M}^+ + 1$, 4), 257 (36), 255 (100), 227 (2), 195 (3), 179 (5), 177 (15), 171 (6), 157 (24), 155 (73), 143 (82), 133 (4), 119 (33), 105 (26), 91 (12); HRMS: (CI, 70 eV) calcd for $\text{C}_{17}\text{H}_{18}\text{ClO}$ 273.0968, found m/z 273.1065, 273.1025 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{ClO}$: C, 74.86; H, 6.28; Cl, 13.00. Found: C, 74.81; H, 6.36; Cl, 12.99.

(2R*,3R*)-2-(4-Chlorophenyl)-3-phenyl-4-penten-2-ol (4b). According to the general procedure (II), this compound was prepared from **1a**, $\text{BF}_3 \cdot \text{OEt}_2$, and **2b** in dichloromethane to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 140 °C/0.1 mmHg; IR: (neat) 3563, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.30-7.05 (m, 9H, aroma), 6.22 (ddd, $J = 17.1, 10.3, 9.8$ Hz, 1H, 4-H), 5.18 (d, $J = 10.3$ Hz, 1H, 5- H^{A}), 5.09 (d, $J = 17.1$ Hz, 1H, 5- H^{B}), 3.54 (d, $J = 9.8$ Hz, 1H, 3-H), 2.19 (br, 1H, OH, D_2O -exchangeable), 1.56 (s, 3H, 1- H_3); ^{13}C NMR: (67.9 MHz, CDCl_3) 144.63 (s, ipso), 139.69 (s, ipso), 136.76 (d, C-4), 132.34 (s, ipso), 129.27 (d), 127.96 (d), 127.59 (d), 127.19 (d), 126.72 (d), 118.38 (t, C-5), 75.79 (s, C-2), 62.12 (d, C-3), 27.89 (q, C-1); MS: (CI, 70 eV) m/z 275 ($\text{M}^+ + 3$, 0.2), 273 ($\text{M}^+ + 1$, 0.7), 257 (38), 255 (100), 177 (19), 157 (15), 155 (42), 143 (71), 117 (14); HRMS: (EI, 70 eV) calcd for $\text{C}_{17}\text{H}_{15}\text{Cl}$ 254.0862, found m/z 254.0864, 254.0879, 254.0872 ($\text{M}^+ - 18$). Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{ClO}$: C, 74.86; H, 6.28. Found: C, 75.15; H, 6.47.

(2S*,3R*)-2-(4-Bromophenyl)-3-phenyl-4-penten-2-ol (3c). According to the general procedure (I), this compound was prepared from **1a**, SnCl_2 , and **2c** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 115 °C/0.1 mmHg; IR: (neat) 3563, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.45-7.00 (m, 9H, aroma), 6.11 (ddd, $J = 17.1, 10.3, 8.8$ Hz, 1H, 4-H), 5.07 (d, $J = 10.3$ Hz, 1H, 5- H^{A}), 4.94 (d, $J = 17.1$ Hz, 1H, 5- H^{B}), 3.57 (d, $J = 8.8$ Hz, 1H, 3-H), 2.02 (brs, 1H, OH, D_2O -exchangeable), 1.42 (s, 3H, 1-H); ^{13}C NMR: (67.9 MHz, CDCl_3) 145.39 (s, ipso), 139.71 (s, ipso), 136.93 (d, C-4), 130.76 (d), 129.47 (d), 128.19 (d), 127.45 (d), 126.92 (d), 120.55 (s, ipso), 118.36 (t, C-5), 75.99 (s, C-2), 61.78 (d, C-3), 28.32 (q, C-1); MS: (CI, 70 eV) m/z 319 ($\text{M}^+ + 3$, 2.2), 317 ($\text{M}^+ + 1$, 2.4), 301 (77), 299 (79), 229 (4), 227 (4), 223 (16), 221 (16), 201 (60), 199 (61), 183 (4), 181 (4), 161 (11), 143 (100), 119 (29), 105 (22), 91 (14); HRMS: (CI, 70 eV) calcd for $\text{C}_{17}\text{H}_{18}\text{BrO}$ 317.0463, found m/z 317.0553, 317.0555, 317.0525 ($\text{M}^+ + 1$).

(2R*,3R*)-2-(4-Bromophenyl)-3-phenyl-4-penten-2-ol (4c). According to the general procedure (II), this compound was prepared from **1a**, $\text{BF}_3 \cdot \text{OEt}_2$, and **2c** in dichloromethane to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 125 °C/1 mmHg; IR: (neat) 3564, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.40-6.90 (m, 9H, aroma), 6.22 (ddd, $J = 17.1, 10.3, 9.3$ Hz, 1H, 4-H), 5.20 (dd, $J = 10.3, 1.5$ Hz, 1H, 5- H^{A}), 5.11 (dd, $J = 17.1, 1.5$ Hz, 1H, 5- H^{B}), 3.56 (d, $J = 9.3$ Hz, 1H, 3-H), 2.08 (s, 1H, OH, D_2O -exchangeable), 1.57 (s, 3H, 1- H_3); ^{13}C NMR: (67.9 MHz, CDCl_3) 145.21 (s, ipso), 139.71 (s, ipso), 136.78 (d, C-4), 130.57 (d), 129.30 (d), 127.99 (d), 127.61 (d), 126.75 (d), 120.58 (s, ipso), 118.41 (t, C-5), 75.87 (s, C-2), 62.09 (d, C-3), 27.91 (q, C-1); MS: (CI, 70 eV) m/z 301 (99), 299 (100), 223 (12), 221 (12), 201 (10), 199 (10), 143 (71); HRMS: (CI, 70 eV) calcd for $\text{C}_{17}\text{H}_{18}\text{BrO}$ 317.0463, found m/z 317.0516, 317.0528, 317.0544 ($\text{M}^+ + 1$).

(2S*,3R*)-2-(4-Methylphenyl)-3-phenyl-4-penten-2-ol (3d). According to the general procedure (I), this compound was prepared from **1a**, SnCl_2 , and **2d** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 125 °C/2 mmHg; IR: (neat) 3563, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.20-7.00 (m, 9H, aroma), 6.11 (ddd, $J = 17.1, 10.3, 8.8$ Hz, 1H,

4-H), 5.04 (ddd, $J = 10.3, 2.0, 1.0$ Hz, 1H, 5-H^A), 4.92 (ddd, $J = 17.1, 2.0, 1.0$ Hz, 1H, 5-H^B), 3.62 (d, $J = 8.8$ Hz, 1H, 3-H), 2.32 (s, 3H, ArMe), 2.02 (s, 1H, OH, D₂O-exchangeable), 1.41 (s, 3H, 1-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 143.45 (s, ipso), 140.21 (s, ipso), 137.52 (d, C-4), 135.97 (s, ipso), 129.62 (d), 128.42 (d), 127.99 (d), 126.68 (d), 125.38 (d), 117.84 (t, C-5), 76.13 (s, C-2), 61.74 (d, C-3), 28.66 (q, ArMe), 20.92 (q, C-1); MS: (CI, 70 eV) m/z 253 ($M^+ + 1$, 1.5), 235 (100), 171 (4), 157 (11), 143 (44), 135 (61), 119 (7), 105 (10), 91 (3), 84 (2); HRMS: (CI, 70 eV) calcd for C₁₈H₂₁O 253.1514, found m/z 253.1575, 253.1614 ($M^+ + 1$). Anal. Calcd for C₁₈H₂₀O: C, 85.67; H, 7.99. Found: C, 85.56; H, 7.99.

(2R*,3R*)-2-(4-Methylphenyl)-3-phenyl-4-penten-2-ol (4d). According to the general procedure (II), this compound was prepared from **1a**, BF₃·OEt₂, and **2d** in dichloromethane to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 120 °C/0.1 mmHg; IR: (neat) 3563, 1635 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.30-6.90 (m, 9H, aroma), 6.25 (ddd, $J = 17.1, 10.3, 9.8$ Hz, 1H, 4-H), 5.18 (dd, $J = 10.3, 2.0$ Hz, 1H, 5-H^A), 5.11 (dd, $J = 17.1, 2.0$ Hz, 1H, 5-H^B), 3.61 (d, $J = 9.8$ Hz, 1H, 3-H), 2.30 (s, 3H, ArMe), 2.05 (s, 1H, OH, D₂O-exchangeable), 1.57 (s, 3H, 1-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 143.05 (s, ipso), 140.18 (s, ipso), 137.33 (d, C-4), 136.06 (s, ipso), 129.40 (d), 128.48 (d), 128.23 (d), 127.82 (d), 126.54 (d), 125.61 (d), 117.90 (t, C-5), 75.92 (s, C-2), 62.21 (d, C-3), 27.91 (q, ArMe), 20.94 (q, C-1); MS: (CI, 70 eV) m/z 253 ($M^+ + 1$, 0.6), 235 (100), 157 (20), 143 (53), 135 (30), 117 (7), 105 (5), 91 (3); HRMS: (EI, 70 eV) calcd for C₁₈H₁₈ 234.1409, found m/z 234.1417, 234.1412, 234.1413 ($M^+ - H_2O$).

(2S*,3R*)-2-(4-Methoxyphenyl)-3-phenyl-4-penten-2-ol (3e). According to the general procedure (I), this compound was prepared from **1a**, SnCl₂, and **2e** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 95 °C/0.75 mmHg; IR: (neat) 3541, 1612 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.30-6.80 (m, 9H, aroma), 6.14 (ddd, $J = 17.1, 10.3, 8.8$ Hz, 1H, 4-H), 5.08 (d, $J = 10.3$ Hz, 1H, 5-H^A), 4.97 (d, $J = 17.1$ Hz, 1H, 5-H^B), 3.80 (s, 3H, OMe), 3.60 (d, $J = 8.8$ Hz, 1H, 3-H), 2.00 (s, 1H, OH, D₂O-exchangeable), 1.44 (s, 3H, 1-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 158.21 (s, ipso), 140.21 (s, ipso), 138.51 (s, ipso), 137.52 (d, C-4), 129.58 (d), 128.04 (d), 126.72 (d), 118.00 (t, C-5), 113.02 (d), 75.98 (s, C-2), 62.09 (d, C-3), 55.18 (q, OMe), 28.43 (q, C-1); MS: (CI, 70 eV) m/z 269 ($M^+ + 1$, 11), 251 (100), 173 (6), 161 (10), 151 (62), 143 (14), 135 (3), 121 (2), 105 (4), 84 (5); HRMS: (CI, 70 eV) calcd for C₁₈H₂₁O₂ 269.1463, found m/z 269.1540, 269.1540 ($M^+ + 1$). Anal. Calcd for C₁₈H₂₀O₂: C, 80.56; H, 7.51. Found: C, 80.26; H, 7.57.

(2R*,3R*)-2-(4-Methoxyphenyl)-3-phenyl-4-penten-2-ol (4e). This compound was not purely isolated because this is the minor isomer in the reaction of **1a** with **2e** in the presence of SnCl₂ (Table 1, entry 5). In a crude reaction mixture, the signals for the title compound were overlapped or obscured in the NMR spectra. The only independent signal can be seen in ¹³C NMR: (67.9 MHz, CDCl₃) 62.41 (d, C-3).

(1R*)-1-((1S*)-Cyclohex-2-enyl)-1-phenylethanol (6a). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **2a** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 70 °C/0.11 mmHg; IR: (neat) 3467 cm⁻¹; ¹H

NMR: (270 MHz, CDCl₃) 7.43-7.19 (m, 5H, aroma), 5.92 (tdd, $J = 10.3, 3.4, 2.0$ Hz, 1H, 3'-H), 5.76 (dd, $J = 10.3, 2.0$ Hz, 1H, 2'-H), 2.57-2.50 (m, 1H, 1'-H), 1.95-1.90 (m, 2H, 4'-H₂), 1.81 (s, 1H, OH, D₂O-exchangeable), 1.75-1.60 (m, 1H, 5'-H^A), 1.59 (s, 3H, 2-H₃), 1.55-1.17 (m, 3H, 5'-H^B and 6'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 147.10 (s, ipso), 131.50 (d, C-3'), 127.85 (d), 126.39 (d, C-2'), 126.32 (d), 125.15 (d), 75.95 (s, C-1), 46.50 (d, C-1'), 27.75 (q, C-2), 25.11 (t, C-4'), 24.24 (t, C-6'), 21.89 (t, C-5'); MS: (CI, 70 eV) m/z 203 ($M^+ + 1$, 2), 185 (67), 121 (100); HRMS: (CI, 70 eV) calcd for C₁₄H₁₉O 203.1358, found m/z 203.1463, 203.1426, 203.1467 ($M^+ + 1$). Anal. Calcd for C₁₄H₁₈O: C, 83.12; H, 8.97. Found: C, 82.87; H, 9.14.

(1R*)-1-((1R*)-Cyclohex-2-enyl)-1-phenylethanol (5a). This compound was minor product in the reaction of **1b** with **2a** in the presence of BF₃·OEt₂ (eq. 2) and was not purely isolated. The selected signals in the crude mixture was shown below: ¹H NMR: (270 MHz, CDCl₃) 5.86-5.81 (m, 1H, 3'-H), 5.37 (d, $J = 10.3$ Hz, 1H, 2'-H), 2.69-2.62 (m, 1H, 1'-H), 1.87 (s, 1H, OH, D₂O-exchangeable), 1.49 (s, 3H, 2-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 148.17 (ipso), 76.14 (C-1), 46.25 (C-1'), 23.89 (C-6').

(1R*)-1-(4-Chlorophenyl)-1-((1S*)-cyclohex-2-enyl)ethanol (6b). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **2b** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 77 °C/0.11 mmHg; IR: (neat) 3571 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.38-7.23 (m, 4H, aroma), 5.98-5.90 (m, 1H, 3'-H), 5.76 (d, $J = 10.3$ Hz, 1H, 2'-H), 2.55-2.46 (m, 1H, 1'-H), 1.98-1.91 (m, 2H, 4'-H₂), 1.76 (s, 1H, OH, D₂O-exchangeable), 1.74-1.66 (m, 1H, 5'-H^A), 1.57 (s, 3H, 2-H₃), 1.49-1.14 (m, 3H, 5'-H^B and 6'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 145.58 (s, 1-C in Ar), 132.13 (d, C-3'), 132.13 (s, CCl), 127.97 (d), 126.72 (d), 125.88 (d, C-2'), 75.75 (s, C-1), 46.43 (d, C-1'), 28.04 (q, C-2), 25.09 (t, C-4'), 24.25 (t, C-6'), 21.79 (t, C-5'); MS: (EI) m/z 218 ($M^+ - 18$, 1), 157 (38), 155 (100). Anal. Calcd for C₁₄H₁₇ClO: C, 71.03; H, 7.24; Cl, 14.98. Found: C, 70.98; H, 7.36; Cl, 14.83.

(1R*)-1-(4-Bromo-phenyl)-1-((1S*)-cyclohex-2-enyl)ethanol (6c). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **2c** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 77 °C/0.11 mmHg; IR: (neat) 3464 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.44 (d, $J = 8.8$ Hz, 2H, aroma), 7.29 (d, $J = 8.8$ Hz, 2H, aroma), 5.97-5.89 (m, 1H, 3'-H), 5.76 (d, $J = 10.3$ Hz, 1H, 2'-H), 2.55-2.46 (m, 1H, 1'-H), 1.98-1.89 (m, 2H, 4'-H₂), 1.81 (s, 1H, OH, D₂O-exchangeable), 1.75-1.65 (m, 1H, 5'-H^A), 1.56 (s, 3H, 2-H₃), 1.48-1.14 (m, 3H, 5'-H^B and 6'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 146.10 (s, 1-C in Ar), 132.10 (d, C-3'), 130.91 (d), 127.10 (d), 125.85 (d, C-2'), 120.26 (s, CBr), 75.76 (s, C-1), 46.35 (d, C-1'), 27.95 (q, C-2), 25.06 (t, C-4'), 24.22 (t, C-6'), 21.76 (t, C-5'); MS: (EI) m/z 201 (98), 199 (100). Anal. Calcd for C₁₄H₁₇BrO: C, 59.80; H, 6.09; Br, 28.42. Found: C, 59.52; H, 6.08; Br, 28.31.

(1R*)-1-((1S*)-Cyclohex-2-enyl)-1-*p*-tolylethanol (6d). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **2d** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 75 °C/0.11 mmHg; IR: (neat) 3467 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.31 (d, $J = 8.3$ Hz, 2H, aroma), 7.14 (d, $J = 8.3$ Hz, 2H, aroma), 5.92-5.87 (m, 1H, 3'-H), 5.75 (d, $J = 11.2$ Hz, 1H, 2'-H), 2.52-2.50 (m, 1H, 1'-H), 2.34 (s, 3H, Ar-Me),

1.95-1.93 (m, 2H, 4'-H₂), 1.72 (s, 1H, OH, D₂O-exchangeable), 1.75-1.67 (m, 1H, 5'-H^A), 1.57 (s, 3H, 2-H₃), 1.49-1.18 (m, 3H, 5'-H^B and 6'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 144.20 (s, 1-C in Ar), 135.86 (s, Me-C in Ar), 131.37 (d, C-3'), 128.59 (d), 126.55 (d, C-2'), 125.10 (d), 75.90 (s, C-1), 46.53 (d, C-1'), 27.78 (q, C-2), 25.14 (t, C-4'), 24.30 (t, C-6'), 21.95 (t, C-5'), 20.95 (q, Ar-Me); MS: (CI, 200 eV) *m/z* 217 (M⁺ + 1, 2), 199 (100), 135 (18); HRMS: (CI, 200 eV) calcd for C₁₅H₂₁O 217.1514, found *m/z* 217.1608, 217.1595, 217.1590 (M⁺ + 1). Anal. Calcd for C₁₅H₂₀O: C, 83.28; H, 9.32. Found: C, 83.01; H, 9.40.

(1R*)-1-((1S*)-Cyclohex-2-enyl)-1-(4-methoxyphenyl)ethanol (6e). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **2e** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 110 °C/0.11 mmHg; IR: (neat) 3464 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.33 (d, *J* = 8.8, 2H, aroma), 6.86 (d, *J* = 8.8, 2H, aroma), 5.90-5.85 (m, 1H, 3'-H), 5.76 (d, *J* = 10.3 Hz, 1H, 2'-H), 3.80 (s, 3H, MeO), 2.51-2.47 (m, 1H, 1'-H), 1.94-1.92 (m, 2H, 4'-H₂), 1.79 (s, 1H, OH, D₂O-exchangeable), 1.75-1.64 (m, 1H, 5'-H^A), 1.56 (s, 3H, 2-H₃), 1.50-1.23 (m, 3H, 5'-H^B and 6'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 158.04 (s, MeO-C in Ar), 139.33 (s, 1-C in Ar), 131.18 (d, olefin), 126.61 (d, olefin), 126.37 (d), 113.14 (d), 75.75 (s, C-1), 55.15 (q, MeO), 46.72 (d, C-1'), 27.60 (q, C-2), 25.13 (t, C-4'), 24.30 (t, C-6'), 21.93 (t, C-5'); MS: (EI) *m/z* 232 (M⁺, 2), 214 (12), 151 (100); HRMS: (EI) calcd for C₁₅H₂₀O₂ 232.1463, found *m/z* 232.1464, 232.1456, 232.1465 (M⁺). Anal. Calcd for C₁₅H₂₀O₂: C, 77.55; H, 8.68. Found: C, 77.32; H, 8.78.

(1S*)-1-((1S*)-Cyclohex-2-enyl)-1-(4-methoxyphenyl)-ethanol (5e). This compound was a minor product in the reaction of **1b** with **2e** in the presence of SnCl₂ (Table 2, entry 5) and was not purely isolated. The selected signals in the crude mixture was shown below: ¹H NMR: (270 MHz, CDCl₃) 5.42 (d, *J* = 9.8); ¹³C NMR: (67.9 MHz, CDCl₃) 139.32 (1-C in Ar), 127.00 (olefin), 126.16, 113.27, 75.90 (C-1), 46.39 (C-1').

(1R*)-1-((1S*)-Cyclopent-2-enyl)-1-phenylethanol (6f). According to the general procedure (I), this compound was prepared from **1c**, SnCl₂, and **2a** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 55 °C/0.11 mmHg; IR: (neat) 3463 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.45 (d, *J* = 7.3 Hz, 2H, aroma), 7.33 (t, *J* = 7.6 Hz, 2H, aroma), 7.23 (d, *J* = 6.8 Hz, 1H, aroma), 6.00-5.95 (m, 1H), 5.79-5.74 (m, 1H), 3.27-3.18 (m, 1H, 1'-H), 2.36-2.15 (m, 2H, 4'-H₂), 1.79 (s, 1H, OH, D₂O-exchangeable), 1.55 (s, 3H, 2-H₃), 1.72-1.48 (m, 2H, 5'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 147.79 (s, ipso), 135.34 (d, olefin), 129.84 (d, olefin), 127.88 (d), 126.25 (d), 125.04 (d), 75.75 (s, C-1), 57.27 (d, C-1'), 32.26 (t, C-4'), 28.95 (q, C-2), 24.74 (t, C-5'); MS: (CI) *m/z* 189 (M⁺ + 1, 2), 171 (100), 121 (37); HRMS: (CI) calcd for C₁₃H₁₇O 189.1201, found *m/z* 189.1263, 189.1256, 189.1295 (M⁺ + 1). Anal. Calcd for C₁₃H₁₆O: C, 82.94; H, 8.57. Found: C, 82.59; H, 8.53.

(2R*, 3S*)-2-(4-Chlorophenyl)-3-methyl-4-pentene-2-ol (8b). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **2b** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 100 °C/2 mmHg; IR: (neat) 3467, 1639 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.40-7.20 (m, 4H, aroma), 5.78 (ddd, *J* = 17.1, 10.3, 8.3 Hz, 1H, 4-H), 5.14-5.06 (m, 2H, 5-H₂), 2.49 (m, 1H, 3-H), 1.88 (brs, 1H, OH, D₂O-exchangeable), 1.50 (s,

3H, 1-H₃), 0.85 (d, $J = 6.8$ Hz, 3H, 3-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 145.47 (s, ipso), 139.42 (d, C-4), 132.22 (s, ipso), 127.94 (d), 126.77 (d), 116.68 (t, C-5), 75.52 (s, C-2), 48.81 (d, C-3), 28.49 (q, C-1), 14.66 (q, 3-Me); MS: (CI, 70 eV) m/z 211 ($M^+ + 1$, 2), 193 (100), 155 (13), 99 (3), 84 (12); HRMS: (CI, 70 eV) calcd for C₁₂H₁₅ClO 211.0811, found m/z 211.0882, 211.0906 ($M^+ + 1$). Anal. Calcd for C₁₂H₁₅ClO: C, 68.40; H, 7.18; Cl, 16.83. Found: C, 68.24; H, 7.07; Cl, 16.69.

(2S*, 3S*)-2-(4-Chlorophenyl)-3-methyl-4-pentene-2-ol (7b). According to the general procedure (II), this compound was prepared from **1b**, BF₃·OEt₂, and **2b** in dichloromethane to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 100 °C/2 mmHg; cm^{-1} ; ¹H NMR: (270 MHz, CDCl₃) 7.40-7.20 (m, 4H, aroma), 5.68 (ddd, $J = 17.1, 10.3, 8.3$ Hz, 1H, 4-H), 5.13-5.08 (m, 2H, 5-H₂), 2.55 (m, 1H, 3-H), 1.94 (brs, 1H, OH, D₂O-exchangeable), 1.51 (s, 3H, 1-H₃), 0.96 (d, $J = 6.8$ Hz, 3H, 3-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 145.53 (s, ipso), 139.56 (d, C-4), 132.45 (s, ipso), 127.97 (d), 127.04 (d), 117.03 (t, C-5), 75.44 (s, C-2), 48.87 (d, C-3), 25.95 (q, C-1), 14.09 (q, 3-Me).

(2S*, 3S*)-3-methyl-2-(1'-naphthyl)-4-pentene-2-ol (7f). According to the general procedure (I), this compound was prepared from **1e**, SnCl₂, and **2f** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 78 °C/0.1 mmHg; IR: (neat) 3559, 1635 cm^{-1} ; ¹H NMR: (270 MHz, CDCl₃) 7.35-8.88 (m, 7H, aroma), 5.93 (ddd, $J = 17.6, 10.3, 9.8$ Hz, 1H, 4-H), 5.23 (ddd, $J = 17.6, 2.0, 1.0$ Hz, 1H, 5-H^B), 5.21 (ddd, $J = 10.3, 2.0, 1.0$ Hz, 1H, 5-H^A), 3.40 (m, 1H, 3-H), 2.28 (s, 1H, OH, D₂O-exchangeable), 1.73 (s, 3H, 1-H₃), 0.85 (d, $J = 6.8$ Hz, 3H, 3-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 141.76 (s, ipso), 139.85 (d, C-4), 134.99 (s, ipso), 130.82 (s, ipso), 129.17 (d), 128.63 (d), 127.16 (d), 125.13 (d), 125.07 (d), 124.81 (d), 124.52 (d), 117.09 (t, C-5), 78.13 (s, C-2), 46.01 (d, C-3), 25.13 (q, C-1), 15.04 (q, 3-Me); MS: (EI, 70 eV) m/z 226 (M^+ , 0.3), 172 (18), 171 (100), 155 (13), 128 (12), 127 (19), 43 (54); HRMS: (EI, 70 eV) calcd for C₁₆H₁₈O 226.1358, found m/z 226.1363, 226.1369, 226.1362 (M^+). Anal. Calcd for C₁₆H₁₈O: C, 84.91; H, 8.02. Found: C, 84.81; H, 8.01.

3,3,3-Tris-(4-Chloro-phenyl)-propionic acid (2S*,3R*)-diphenyl-4-pentenyl ester (3aE). The esterification was performed by known method.¹ A flask was charged with dried dichloromethane (6 mL) and **3a** (1.5 mmol) under nitrogen. To the solution were sequentially added MgBr₂ (3.0 mmol), Et₃N (4.5 mmol), and tris(*p*-chlorophenyl)propionic acid anhydride (3.0 mmol).⁸ The mixture was stirred for 20 h, water (10 mL) was added. The mixture was extracted with ethyl acetate and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. Column chromatography (hexane/AcOEt, 9/1) of the resultant residue on silica gel to give the esterified product (50% yield). Recrystallization from hexane gave the suitable crystalline for X-ray analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: mp: 145-147 °C; IR: (neat) 1728 cm^{-1} ; ¹H NMR: (270 MHz, CDCl₃) 7.22-7.07 (m, 18H), 6.82 (dd, $J = 7.8, 2.0$ Hz, 2H, aroma), 6.67 (dd, $J = 7.8, 2.0$ Hz, 2H, aroma), 6.00 (ddd, $J = 17.1, 10.3, 8.8$ Hz, 1H, 4-H), 5.05 (dd, $J = 10.3, 1.5$ Hz, 1H, 5-H^A), 4.91 (d, $J = 8.8$ Hz, 1H, 5-H^B), 3.72 (d, $J = 15.6$ Hz, 1H, 2'-H^A), 3.66 (d, $J = 15.6$ Hz, 1H, 2'-H^B), 3.53 (d, $J = 8.8$ Hz, 1H, 3-H), 1.58 (s, 3H, 1-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 168.31 (s, C=O), 144.14 (s), 141.54 (s), 139.20 (s), 136.36 (d, olefin), 132.54 (s), 130.46 (d),

129.70 (d), 128.20 (d), 127.65 (d), 127.38 (d), 126.89 (d), 126.72 (d), 125.56 (d), 118.06 (t, olefin), 86.30 (s, C-2), 61.75 (d, C-3), 54.55 (s, C-3'), 46.59 (t, C-2), 22.09 (q, C-1); MS: (EI) m/z 389 (3), 387 (3), 347 (99), 345 (100). Anal. Calcd for $C_{38}H_{31}Cl_3O_2$: C, 72.91; H, 4.99; Cl, 16.99. Found: C, 72.63; H, 5.00; Cl, 16.99.

Procedure for Transformation from 3c to 3a. A flask was charged with dried Et_2O (10 mL) and **3c** (1.0 mmol) under nitrogen and was cooled to $-78\text{ }^{\circ}C$. A 1.56 M solution of $n\text{-BuLi}$ in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. To the resulting yellow suspension was added 0.5 M of HCl aq (10 mL). The water phase was extracted with Et_2O and the organic layer was dried over $MgSO_4$. The solvent was distilled off under reduced pressure. The formation of **3a** (97%) was confirmed by NMR spectra of the crude mixture.

Procedure for Transformation from 3c to 3b. A flask was charged with dried Et_2O (10 mL) and **3c** (1.0 mmol) under nitrogen and was cooled to $-78\text{ }^{\circ}C$. A 1.56 M solution of $n\text{-BuLi}$ in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. To the resulting yellow suspension was added hexachloroethane (5.0 mmol) at $0\text{ }^{\circ}C$, and the mixture was stirred for an additional 16 h at room temperature. 0.5 M of HCl aq (10 mL) was added to the reaction mixture. The water phase was extracted with Et_2O and the organic layer was dried over $MgSO_4$. The solvent was distilled off under reduced pressure. The formation of **3b** (69%) and **3a** (17%) was confirmed by NMR spectra of the crude mixture.

Procedure for Transformation from 3c to 3d. A flask was charged with dried Et_2O (10 mL) and **3c** (1.0 mmol) under nitrogen and was cooled to $-78\text{ }^{\circ}C$. A 1.56 M solution of $n\text{-BuLi}$ in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. To the resulting yellow suspension was added MeI (10.0 mmol) at room temperature, and the mixture was stirred for an additional 2 h at room temperature. 0.5 M of HCl aq (10 mL) was added to the reaction mixture. The water phase was extracted with Et_2O and the organic layer was dried over $MgSO_4$. The solvent was distilled off under reduced pressure. The formation of **3d** (71%) and **3a** (20%) was confirmed by NMR spectra of the crude mixture.

Procedure for Transformation from 3c to 3e. A flask was charged with dried Et_2O (10 mL) and **3c** (1.0 mmol) under nitrogen and was cooled to $-78\text{ }^{\circ}C$. A 1.56 M solution of $n\text{-BuLi}$ in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. The reaction mixture was cooled to $-78\text{ }^{\circ}C$, and then oxygen was slowly bubbled through the solution. The mixture was warmed to room temperature with bubbling and stirring for 20 h. The quenching by HCl aq (0.5 M, 10 mL), extraction with Et_2O , drying over $MgSO_4$, concentration under reduced pressure gave a crude mixture including phenolic compound (**3y**; X = OH, 50%) and **3a** (35%) by NMR. The crude mixture was roughly purified by column chromatography to yield **3a** (eluted by hexane/ Et_2O = 3/1) and **3y** (X = OH) (eluted by Et_2O).

(1S*,2R*)-4-(1-Hydroxy-1-methyl-2-phenyl-3-butenyl)phenol (3y). 1H NMR: (270 MHz, $CDCl_3$) 7.35-7.00 (m, 8H, aroma), 6.70 (d, J = 8.3 Hz, 2H, 2'3'-H), 6.13 (ddd, J = 17.1, 10.3, 8.8 Hz, 1H, 3-H), 6.04 (brs, 1H, ArOH, D_2O -exchangeable), 5.09 (d, J = 10.3 Hz, 1H, 4- H^A), 4.99 (d, J = 17.1 Hz, 1H, 4- H^B), 3.59 (d, J = 8.8 Hz, 1H, 2-H), 2.26 (s, 1H, OH, D_2O -

exchangeable), 1.44 (s, 3H, 1-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 154.35 (s, ipso), 139.95 (s, ipso), 138.09 (s, ipso), 137.33 (d, C-3), 129.53 (d), 127.99 (d), 126.90 (d), 126.72 (d), 118.20 (t, C-4), 114.60 (d), 76.31 (s, C-1), 62.13 (d, C-2), 28.47 (q, 1-Me); MS: (CI, 70 eV) *m/z* 255 (M⁺ + 1, 3), 237 (97), 177 (9), 161 (13), 159 (13), 143 (43), 137 (100), 117 (11); HRMS: (CI, 70 eV) calcd for C₁₇H₁₉O₂ 255.1307, found *m/z* 255.1385, 255.1382, 255.1391 (M⁺ + 1). 0.7 mmol of the phenolic compound **3y** was dissolved in acetonitrile (15 mL) and the solution was cooled to -40 °C under nitrogen. To the mixture were sequentially added MeI (7.0 mmol) and NaH (0.7 mmol), and the solution was warmed to room temperature. After stirring for 16 h, 0.5 M of HCl aq (10 mL) was added to the reaction mixture. The water phase was extracted with Et₂O and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. The formation of **3e** (57%) was confirmed by NMR spectra of the crude mixture accompanied with *p*-methoxyacetophenone (13%) which came from a retro-aldol reaction.

4-Nitrobenzoic acid (1R*)-1-(4-Chlorophenyl)-1-((1S*)-cyclohex-2-enyl)ethyl ether (6bE). The esterification was performed by known method.¹ A flask was charged with dried dichloromethane (10 mL) and **6b** (2.0 mmol) under nitrogen and was cooled to 0 °C. To the solution were sequentially added MgBr₂ (4.0 mmol), Et₃N (6.0 mmol), and *p*-nitrobenzoic acid anhydride (4.0 mmol).⁸ The mixture was warmed to rt. After stirring for 24 h, water (10 mL) was added. The water phase was extracted with ethyl acetate and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. Column chromatography (hexane/AcOEt, 3/1) of the resultant residue on silica gel to give the esterified product (27% yield). Recrystallization from hexane/EtOH (99/1) gave the suitable crystalline for X-ray analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: mp: 137-138 °C; IR: (neat) 1720 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 8.30 (d, *J* = 8.9, 2H, *p*-NO₂-aroma), 8.18 (d, *J* = 8.9 Hz, 2H, *p*-NO₂-aroma), 7.31-7.26 (m, 4H, *p*-Cl-aroma), 5.83-5.77 (m, 1H, 3'-H), 5.32 (d, *J* = 10.4 Hz, 1H, 2'-H), 2.90-2.84 (m, 1H, 1'-H), 1.98 (s, 3H, 2-H₃), 2.13-1.81 (m, 4H), 1.66-1.45 (m, 2H); ¹³C NMR: (67.9 MHz, CDCl₃) 162.97 (s, C=O), 150.42 (s), 141.58 (s), 136.62 (s), 133.25 (s), 130.77 (d, olefin), 130.59 (d), 128.36 (d), 126.94 (d), 125.19 (d, olefin), 123.56 (d), 87.12 (s, C-1), 47.72 (d, C-1'), 25.11 (t), 24.62 (t), 21.90 (t), 19.91 (q, C-2); MS: (CI) *m/z* 386 (M⁺ + 1, 11), 304 (5), 275 (4), 221 (38), 219 (100); HRMS: (CI) calcd for C₂₁H₂₁ClNO₄ 386.1081, found *m/z* 386.1141, 386.1174, 386.1150 (M⁺ + 1). Anal. Calcd for C₂₁H₂₀ClNO₄: C, 65.37; H, 5.22; N, 3.63; Cl, 9.19. Found: C, 65.08; H, 5.06; N, 3.66; Cl, 8.88.

Procedure for Transformation from 6c to 6a. A flask was charged with dried Et₂O (8 mL) and **6c** (1.0 mmol) under nitrogen and was cooled to -78 °C. A 1.56 M solution of *n*-BuLi in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. To the resulting yellow suspension was added 0.5 M of HCl aq (10 mL). The water phase was extracted with Et₂O and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. The formation of **6a** (94%) was confirmed by NMR spectra of the crude mixture.

Procedure for Transformation from 6c to 6b. A flask was charged with dried Et₂O (8 mL) and **6c** (1.0 mmol) under nitrogen and was cooled to -78 °C. A 1.56 M solution of *n*-BuLi in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. To the resulting yellow suspension was added hexachloroethane (5.0 mmol) at 0 °C, and the mixture was stirred for an additional 16 h at room

temperature. 0.5 M of HCl aq (10 mL) was added to the reaction mixture. The water phase was extracted with Et₂O and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. The formation of **6b** (78%) was confirmed by NMR spectra of the crude mixture.

Procedure for Transformation from 6c to 6d. A flask was charged with dried Et₂O (8 mL) and **6c** (1.0 mmol) under nitrogen and was cooled to -78 °C. A 1.56 M solution of *n*-BuLi in hexane (1.5 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 9 h. To the resulting yellow suspension was added MeI (10.0 mmol) at room temperature, and the mixture was stirred for an additional 2 h at room temperature. 0.5 M of HCl aq (10 mL) was added to the reaction mixture. The water phase was extracted with Et₂O and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. The formation of **6d** (75%) was confirmed by NMR spectra of the crude mixture.

Procedure for Transformation from 6c to 6e. A flask was charged with dried Et₂O (25 mL) and **6c** (3.0 mmol) under nitrogen and was cooled to -78 °C. A 1.6 M solution of *n*-BuLi in hexane (4 mL) was added at the same temperature. The mixture was warmed to room temperature and stirring was continued for 6 h. Oxygen was slowly bubbled into the solution. The bubbling was continued for 20 h. The quenching by HCl aq (0.5 M, 10 mL), extraction with Et₂O, drying over MgSO₄, concentration under reduced pressure gave a crude mixture including phenolic compound (**6y**; X = OH, 14%) confirmed by NMR. The crude mixture was roughly purified by column chromatography to yield **6y** (eluted by Et₂O).

3,4-Diphenyl-hex-5-en-3-ol (6y). Only a part of signals for the compound can be observed: ¹H NMR: (270 MHz, CDCl₃) 7.25 (d, *J* = 8.3 Hz), 6.77 (d, *J* = 8.3 Hz), 6.19-6.12 (m). 0.43 mmol of the phenolic compound **6y** (X = OH) was dissolved in acetonitrile (10 mL) and the solution was cooled to -40 °C under nitrogen. To the mixture were sequentially added MeI (4.3 mmol) and NaH (0.43 mmol), and the solution was warmed to room temperature. After stirring for 22 h, 0.5 M of HCl aq (10 mL) was added to the reaction mixture. The water phase was extracted with ethyl acetate and the organic layer was dried over MgSO₄. The solvent was distilled off under reduced pressure. The formation of **6e** (80%) was confirmed by NMR spectra of the crude mixture.

4-Chloro-1-[(1R*, 2S*)-1,2-dimethyl-1-[(4-nitrophenyl)carboniloxy]-3-butenyl]benzene (8bE). The esterification was performed by known method.⁴ The mixture of **8b** (1.3 mmol), *p*-nitrobenzoic acid (4.1 mmol), 4-(dimethylamino)pyridine (4.1 mmol), Sc(OTf)₃ (0.8 mmol) in dry CH₂Cl₂ (15 mL) was cooled to -8 °C for 30 min. Diisopropylcarbodiimide (4.2 mmol) was added and the reaction mixture stirred at -8 °C for 30 min and then warmed to room temperature and stirred for 4 h. After filtration of the mixture, the filtrate was washed by HCl (0.1 N), and NaHCO₃ (0.1 N), and water successively. The organic layer was dried over MgSO₄ and evaporated to give the white solid (52% yield). Recrystallization from hexane/ethanol (10/1) gave the suitable crystalline for X-ray analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: mp: 107-108 °C; IR: (KBr) 1720, 1639 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 8.32-8.18 (m, 4H, aroma), 7.31-7.20 (m, 4H, aroma), 5.61 (m, 1H, 3-H), 5.05 (d, *J* = 9.3 Hz, 1H, 4-H^A), 5.01 (d, *J* = 15.6 Hz, 1H, 4-H^B), 2.80 (m, 1H, 2-H), 1.98 (s, 3H, 1-Me), 1.18 (d, *J* = 6.8 Hz, 3H, 2-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 162.92 (s, C=O),

150.50 (s, ipso), 141.13 (s, ipso), 137.66 (d, C-3), 136.60 (s, ipso), 133.15 (s, ipso), 130.57 (d), 128.25 (d), 126.81 (d), 123.59 (d), 117.05 (t, C-4), 76.79 (s, C-1), 49.51 (d, C-2), 20.62 (q, 1-Me), 14.77 (q, 2-Me); MS: (CI, 70 eV) m/z 360 ($M^+ + 1$, 43), 304 (9), 221 (6), 193 (100), 168 (32), 150 (23); HRMS: (CI, 70 eV) calcd for $C_{19}H_{18}ClNO_4$ 360.0924, found m/z 360.1017, 360.0984 ($M^+ + 1$). Anal. Calcd for $C_{19}H_{18}ClNO_4$; C, 63.42; H, 5.04; Cl, 9.85; N, 3.89. Found: C, 63.21; H, 5.01; Cl, 9.57; N, 3.83.

(3*S, 4*S**)-3-Methyl-4-(1'-naphthyl)-4-(*p*-nitrobenzoyloxy)-4-pentene-2**

-ol (7fE). The esterification was performed by known method.¹ *p*-Nitrobenzoic anhydride (2.0 mmol) was added to a stirred suspended mixture of **7f** (1.0 mmol), $MgBr_2$ (2.0 mmol) and Et_3N (3.0 mmol) in dry CH_2Cl_2 (10 mL) at room temperature. The mixture stirred for 20 h and then quenched by the addition of water. The product was extracted with ethyl acetate (50 mL $\times$ 2). The organic layer was washed with water and then dried ($MgSO_4$). The solvent was evaporated and the residue was purified by flash chromatography (hexane/ Et_2O , 3/1) on silica gel and recrystallization (hexane/ $EtOH$, 1/1) to give the product (0.26g, 68%). The crystal obtained was suitable for X-ray crystallographic analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: mp: 147-148 °C; IR: (KBr) 1716, 1600 cm^{-1} ; 1H NMR: (270 MHz, $CDCl_3$) 7.25-8.50 (m, 11H, aroma), 6.33 (ddd, $J = 17.6, 10.3, 10.3$ Hz, 1H, 2-H), 5.40 (d, $J = 17.6$ Hz, 1H, 1- H^B), 5.35 (d, $J = 10.3$ Hz, 1H, 5- H^A), 3.67 (m, 1H, 3-H), 2.12 (s, 3H, 5- H_3), 0.72 (d, $J = 6.8$ Hz, 3H, 3-Me); ^{13}C NMR: (67.9 MHz, $CDCl_3$) 162.35 (s, C=O), 150.31 (s, ipso), 139.42 (d, C-2), 137.84 (s, ipso), 136.55 (s, ipso), 134.73 (s, ipso), 130.56 (d), 129.85 (d), 129.82 (d), 129.24 (d), 125.91 (d), 125.62 (d), 125.16 (d), 124.78 (d), 124.73 (d), 123.51 (d), 116.47 (t, C-1), 89.50 (s, C-4), 45.72 (d, C-3), 19.30 (q, C-5), 14.84 (q, 3-Me); MS: (EI, 70 eV) m/z 375 (M^+ , 7), 321 (11), 320 (45), 153 (31), 150 (100), 104 (10); HRMS: (EI, 70 eV) calcd for $C_{23}H_{21}NO_4$ 375.1471, found m/z 375.1472, 375.1495, 375.1449 (M^+). Anal. Calcd for $C_{23}H_{21}NO_4$; C, 73.58; H, 5.64; N, 3.73. Found: C, 73.42; H, 5.45; N, 3.76.

(*E*)-3,6-Diphenyl-5-hexene-3-ol (9). According to the general procedure (I), this compound was prepared from **1a**, $SnCl_2$, and **2g** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 120 °C/2 mmHg; IR: (neat) 3564 cm^{-1} ; 1H NMR: (270 MHz, $CDCl_3$) 7.50-7.10 (m, 10H, aroma), 6.47 (d, $J = 16.1$ Hz, 1H, 6-H), 5.97 (ddd, $J = 16.1, 8.8, 6.4$ Hz, 1H, 5-H), 2.85 (ddd, $J = 13.7, 6.4, 1.5$ Hz, 1H, 4- H^A), 2.61 (ddd, $J = 13.7, 8.8, 1.0$ Hz, 1H, 4- H^B), 2.03 (s, 1H, OH, D_2O -exchangeable), 1.90 (q, $J = 7.3$ Hz, 1H, 2- H^A), 1.88 (q, $J = 7.3$ Hz, 1H, 2- H^B), 0.78 (t, $J = 7.3$ Hz, 3H, 1- H_3); ^{13}C NMR: (67.9 MHz, $CDCl_3$) 145.79 (s, ipso), 137.07 (s, ipso), 134.44 (d, C-6), 128.48, 128.11, 127.33, 126.49, 126.17, 125.38, 124.95, 76.53 (s, C-3), 46.24 (t, C-4), 35.13 (t, C-3), 7.84 (q, C-1); MS: (CI, 70 eV) m/z 253 ($M^+ + 1$, 3), 235 (48), 223 (3), 175 (9), 157 (15), 147 (4), 135 (100), 119 (20), 105 (6), 91 (12); HRMS: (CI, 70 eV) calcd for $C_{18}H_{21}O$ 253.1514, found m/z 253.1609, 253.1602 ($M^+ + 1$). Anal. Calcd for $C_{18}H_{20}O$; C, 85.67; H, 7.99. Found: C, 85.27; H, 8.02.

3,4-Diphenyl-hex-5-en-3-ol (10). This compound was not purely isolated because this is the minor isomer in the reaction of **1a** with **2g** using $SnCl_2$ (eq. 3). The diastereomeric ratio (62:38) was determined by 1H NMR of the reaction mixture. In a crude mixture, the signals for the title compound were overlapped or obscured in the NMR spectra. The only independent signals are shown below: 1H NMR: (270 MHz, $CDCl_3$) 5.11-4.81 (m, 6-H), 3.67 (d, $J = 8.3$ Hz, minor

product of 4-H), 3.61 (d, $J = 9.7$ Hz, major product of 4-H); ^{13}C NMR: (67.9 MHz, CDCl_3) major product: 117.47 (6-C), 61.86 (4-C) and minor product: 117.28 (6-C), 61.31 (4-C).

(1R*)-1-((1S*)-Cyclohex-2-enyl)-1-phenylpropan-1-ol (11). According to the general procedure (I), this compound was prepared from **1b**, SnCl_2 , and **2g** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 78 °C/0.11 mmHg; IR: (neat) 3571 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.38-7.17 (m, 5H, aroma), 5.98-5.88 (m, 2H, 2'-H and 3'-H), 2.60-2.53 (m, 1H, 1'-H), 2.10-1.83 (m, 2H, 2-H₂), 1.99-1.91 (m, 2H, 4'-H₂), 1.62 (s, 1H, OH, D_2O -exchangeable), 1.74-1.60 (m, 1H, 5'-H^A), 1.47-1.13 (m, 3H, 5'-H^B and 6'-H₂), 0.69 (t, $J = 7.3$ Hz, 3H, 3-H₃); ^{13}C NMR: (67.9 MHz, CDCl_3) 144.37 (s, ipso), 132.13 (d, C-3'), 127.71 (d), 126.22 (d, C-2'), 125.99 (d), 125.73 (d), 78.67 (s, C-1), 45.77 (d, C-1'), 32.60 (t, C-2), 25.19 (t, C-4'), 24.38 (t, C-6'), 21.89 (t, C-5'), 7.78 (q, C-3); MS: (CI, 200 eV) m/z 217 ($\text{M}^+ + 1$, 1), 199 (100), 135 (14); HRMS: (CI, 200 eV) calcd for $\text{C}_{15}\text{H}_{21}\text{O}$ 217.1514, found m/z 217.1588, 217.1584, 217.1581 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{15}\text{H}_{20}\text{O}$: C, 83.28; H, 9.32. Found: C, 83.04; H, 9.35.

(1S*)-1-((1R*)-Cyclohex-2-enyl)-1-phenylpentan-1-ol (12). According to the general procedure (I), this compound was prepared from **1b**, SnCl_2 , and **2h** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 85 °C/0.11 mmHg; IR: (neat) 3579 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.38-7.16 (m, 5H, aroma), 5.97-5.88 (m, 2H, 2'-H and 3'-H), 2.58-2.51 (m, 1H, 1'-H), 2.02-1.81 (m, 2H, 4'-H₂ and 2-H₂), 1.72-1.61 (m, 1H, 5'-H^A), 1.67 (s, 1H, OH, D_2O -exchangeable), 1.46-1.12 (m, 6H, 3-H₂ and 4H₂ and 5'-H^B and 6'-H^A), 0.93-0.85 (m, 1H, 6'-H^B), 0.81 (t, $J = 7.1$ Hz, 3H, 5-H₃); ^{13}C NMR: (67.9 MHz, CDCl_3) 144.84 (s, ipso), 132.05 (d, olefin), 127.71 (d), 126.29 (d, olefin), 125.94 (d), 125.61 (d), 78.38 (s, C-1), 46.04 (d, C-1'), 39.96 (t, C-2), 25.60 (t, C-6'), 25.20 (t, C-4'), 24.30 (t), 23.12 (t), 21.89 (t, C-5'), 13.97 (q, C-5); MS: (CI) m/z 245 ($\text{M}^+ + 1$, 3), 227 (100), 121 (37); HRMS: (CI) calcd for $\text{C}_{17}\text{H}_{25}\text{O}$ 245.1827, found m/z 245.1898, 245.1888, 245.1924 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{17}\text{H}_{24}\text{O}$: C, 83.55; H, 9.90. Found: C, 83.15; H, 9.84.

(1R*)-1-((1S*)-Cyclopent-2-enyl)-1-phenylpropan-1-ol (13). According to the general procedure (I), this compound was prepared from **1c**, SnCl_2 , and **2g** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 58 °C/0.11 mmHg; IR: (neat) 3563 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.44-7.11 (m, 5H, aroma), 6.02-5.97 (m, 1H), 5.86-5.81 (m, 1H), 3.32-3.24 (m, 1H, 1'-H), 2.36-2.10 (m, 2H, 4'-H₂), 1.99-1.78 (m, 2H, 2-H₂), 1.58 (s, 1H, OH, D_2O -exchangeable), 1.68-1.45 (m, 2H, 5'-H₂), 0.68 (t, $J = 7.6$ Hz, 3H, 3-H₃); ^{13}C NMR: (67.9 MHz, CDCl_3) 144.90 (s, ipso), 135.80 (d, olefin), 129.49 (d, olefin), 127.79 (d), 125.96 (d), 125.74 (d), 78.51 (s, C-1), 56.53 (d, C-1'), 34.11 (t, C-2), 32.06 (t, C-4'), 24.80 (t, C-5'), 7.87 (q, C-3); MS: (CI) m/z 203 ($\text{M}^+ + 1$, 3), 185 (100), 135 (28); HRMS: (CI) calcd for $\text{C}_{14}\text{H}_{19}\text{O}$ 203.1358, found m/z 203.1419, 203.1429, 203.1454 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12; H, 8.97. Found: C, 82.88; H, 8.92.

4-Methyl-3-phenyl-hept-1-en-4-ol (14). According to the general procedure (I), this compound was prepared from **1a**, SnCl_2 , and **2i** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ AcOEt , 7/3). The diastereomeric mixture was obtained and showed NMR signals as shown below.

¹H NMR: (270 MHz, CDCl₃) 7.38-7.15 (m, 5H, Ph), 6.42-6.22 (m, 1H, 2-H), 5.19-5.08 (m, 2H, 1-H), 3.33-3.26 (m, 1H, 3-H), 1.52-1.33 (m, 5H), 1.15 (s, 3H, minor product of 4-Me), 1.05 (s, 3H, major product of 4-Me), 0.98-0.83 (m, 3H, 7-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) major product 141.16 (ipso), 137.72, 129.13, 128.18, 126.47, 117.41, 73.93 (C-4), 60.33 (C-3), 42.32, 24.76, 16.80, 14.57; minor product 141.00 (ipso), 137.84, 129.19, 128.18, 126.47, 117.34, 73.93 (C-4), 60.29 (C-3), 42.47, 24.46, 16.72, 14.52; MS: (CI) *m/z* 205 (M⁺ + 1, 1), 187 (100); HRMS: (CI) calcd for C₁₄H₂₁O 205.1545, found *m/z* 205.1574, 205.1584 (M⁺ + 1).

2,3-Dimethyl-4-phenyl-hex-5-en-3-ol (15). According to the general procedure (I), this compound was prepared from **1a**, SnCl₂, and **2j** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/AcOEt, 8/2): ¹H NMR: (270 MHz, CDCl₃) 7.39-7.19 (m, 5H, aroma), 6.47-6.24 (m, 1H, 2-H), 5.17-5.06 (m, 2H, 1-H₂), 3.42 (d, *J* = 9.8 Hz, 1H, 3-H), 2.01-1.91 (m, 1H, 5-H), 0.98 (d, *J* = 6.3 Hz, 3H), 0.92 (d, *J* = 6.8 Hz, 3H), 0.87 (s, 3H, 4-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 141.86 (ipso), 137.86, 129.30, 128.25, 126.45, 116.87, 76.07 (C-4), 57.63 (C-3), 34.00, 20.14, 17.53, 16.86; MS: (CI) *m/z* 205 (M⁺ + 1, 4), 187 (99), 117 (100); HRMS: (CI) calcd for C₁₄H₂₁O 205.1545, found *m/z* 205.1609 (M⁺ + 1).

(2S*, 3R*)-1-Methoxy-2-methyl-3-phenyl-4-penten-2-ol (17a). According to the general procedure (I), this compound was prepared from **1a**, SnCl₂, and **16a** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 60 °C/0.1 mmHg; IR: (neat) 3467, 1635 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.18-7.34 (m, 5H, aroma), 6.30 (ddd, *J* = 17.6, 10.3, 9.8 Hz, 1H, 4-H), 5.15 (d, *J* = 10.3 Hz, 1H, 5-H^A), 5.14 (d, *J* = 17.6 Hz, 1H, 5-H^B), 3.47 (d, *J* = 9.8 Hz, 1H, 3-H), 3.32 (s, 3H, OMe), 3.22 (d, *J* = 8.8 Hz, 1H, 1-H^A), 3.09 (d, *J* = 8.8 Hz, 1H, 1-H^B), 2.15 (s, 1H, OH, D₂O-exchangeable), 1.18 (s, 3H, 2-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 140.61 (s, ipso), 137.43 (d, C-4), 129.20 (d), 128.16 (d), 126.57 (d), 117.48 (t, C-5), 78.31 (t, C-1), 73.44 (s, C-2), 59.00 (q, OMe), 57.63 (d, C-3), 22.67 (q, 2-Me); MS: (CI, 70 eV) *m/z* 207 (M⁺ + 1, 8), 189 (100), 157 (33), 118 (17), 117 (18); HRMS: (CI, 70 eV) calcd for C₁₃H₁₉O₂ 207.1307, found *m/z* 207.1383, 207.1383, 207.1391 (M⁺ + 1). Anal. Calcd for C₁₃H₁₈O₂: C, 75.69; H, 8.80. Found: C, 75.45; H, 8.54.

(2S*, 3R*)-2-methyl-1-phenoxy-3-phenyl-4-penten-2-ol (17b). According to the general procedure (I), this compound was prepared from **1a**, SnCl₂, and **16b** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 115 °C/0.1 mmHg; IR: (neat) 3467, 1635 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 6.82-7.34 (m, 10H, aroma), 6.37 (ddd, *J* = 16.6, 10.3, 9.8 Hz, 1H, 4-H), 5.19 (d, *J* = 10.3 Hz, 1H, 5-H^A), 5.18 (d, *J* = 16.6 Hz, 1H, 5-H^B), 3.76 (d, *J* = 8.8 Hz, 1H, 1-H^A), 3.65 (d, *J* = 8.8 Hz, 1H, 1-H^B), 3.61 (d, *J* = 9.8 Hz, 1H, 3-H), 2.21 (s, 1H, OH, D₂O-exchangeable), 1.31 (s, 3H, 2-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 158.44 (s, ipso), 140.24 (s, ipso), 137.10 (d, C-4), 129.41 (d), 129.14 (d), 128.43 (d), 128.33 (d), 126.75 (d), 120.99 (d), 117.94 (t, C-5), 114.53 (d), 73.30 (s, C-2), 73.17 (t, C-1), 57.66 (d, C-3), 22.97 (q, 2-Me); MS: (EI, 70 eV) *m/z* 268 (M⁺, 0.7), 151 (62), 150 (33), 133 (30), 119 (14), 118 (100), 115 (16), 107 (11), 94 (11), 91 (10), 77 (12), 57 (12), 43 (17); HRMS: (EI, 70 eV) calcd for C₁₈H₂₀O₂ 268.1463, found *m/z* 268.1472, 268.1459, 268.1463 (M⁺). Anal. Calcd for C₁₈H₂₀O₂: C, 80.56; H, 7.51. Found: C, 80.41; H, 7.47.

(2R*, 3R*)-1-Methoxy-2,3-diphenyl-4-penten-2-ol (17c). According to the general

procedure (I), this compound was prepared from **1a**, SnCl₂, and **16c** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 100 °C/0.1 mmHg; IR: (neat) 3552, 1635 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.09-7.35 (m, 10H, aroma), 6.10 (ddd, *J* = 17.1, 10.3, 8.8 Hz, 1H, 4-H), 5.00 (dd, *J* = 10.3, 1.5 Hz, 1H, 5-H^A), 4.89 (ddd, *J* = 17.1, 1.5, 1.5 Hz, 1H, 5-H^B), 3.71 (d, *J* = 8.8 Hz, 1H, 3-H), 3.62 (d, *J* = 9.3 Hz, 1H, 1-H^A), 3.47 (d, *J* = 9.3 Hz, 1H, 1-H^B), 3.21 (s, 3H, OMe), 2.90 (s, 1H, OH, D₂O-exchangeable); ¹³C NMR: (67.9 MHz, CDCl₃) 142.95 (s, ipso), 139.86 (s, ipso), 137.14 (d, C-4), 129.59 (d), 127.85 (d), 127.64 (d), 126.78 (d), 126.61 (d), 125.97 (d), 117.32 (t, C-5), 77.93 (s, C-2), 77.60 (t, C-1), 59.17 (q, OMe), 58.40 (d, C-3); MS: (CI, 70 eV) *m/z* 251 (9), 219 (21), 167 (10), 151 (100), 117 (57), 105 (10), 91 (14); HRMS: (EI, 70 eV) calcd for C₁₈H₁₈O 250.1358, found *m/z* 250.1355, 250.1345, 250.1358 (M⁺ - OH₂). Anal. Calcd for C₁₈H₂₀O₂: C, 80.56; H, 7.51. Found: C, 80.83; H, 7.62.

(2S*, 3R*)-2-Methyl-3-phenyl-4-penten-1,2-diol (17d). According to the general procedure (I), this compound was prepared from **1a**, SnCl₂, and **16d** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 1/1). Further purification was performed by distillation under reduced pressure: bp: 85 °C/0.1 mmHg; IR: (neat) 3401, 1635 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.15-7.30 (m, 5H, aroma), 6.24 (ddd, *J* = 17.6, 9.8, 9.8 Hz, 1H, 4-H), 5.15 (m, 2H, 5-H₂), 3.46 (dd, *J* = 11.2, 5.9 Hz, 1H, 1-H^A), 3.45 (d, *J* = 9.8 Hz, 1H, 3-H), 3.32 (dd, *J* = 11.2, 5.9 Hz, 1H, 1-H^B), 2.69 (t, *J* = 5.9 Hz, 1H, 1-OH, D₂O-exchangeable), 2.35 (s, 1H, 2-OH, D₂O-exchangeable), 1.12 (s, 3H, 2-Me); ¹³C NMR: (67.9 MHz, CDCl₃) 140.08 (s, ipso), 136.96 (d, C-4), 129.17 (d), 128.25 (d), 126.72 (d), 117.78 (t, C-5), 74.14 (s, C-2), 68.20 (t, C-1), 57.30 (d, C-3), 21.55 (q, 2-Me); MS: (CI, 70 eV) *m/z* 175 (56), 157 (79), 147 (33), 118 (33), 117 (100), 75 (15); HRMS: (EI, 70 eV) calcd for C₁₂H₁₄O 174.1045, found *m/z* 174.1046, 174.1018, 174.1040 (M⁺ - H₂O). Anal. Calcd for C₁₂H₁₆O₂: C, 74.97; H, 8.39. Found: C, 75.32; H, 8.37.

(2S*,3R*)-2,3-Diphenyl-4-pentene-1,2-diol (17e). According to the general procedure (I), this compound was prepared from **1a**, SnCl₂, and **16e** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 6/4). Further purification was performed by distillation under reduced pressure to give a white solid. The crystal obtained was suitable for X-ray crystallographic analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: bp: 138 °C/0.1 mmHg; mp: 115 °C; IR: (neat) 3502, 3398 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.36-7.09 (m, 10H, aroma), 6.06 (ddd, *J* = 17.1, 10.3, 8.8 Hz, 1H, 4-H), 5.01 (dd, *J* = 10.3, 1.5 Hz, 1H, 5-H^A), 4.90 (dd, *J* = 17.1, 1.5 Hz, 1H, 5-H^B), 3.81 (dd, *J* = 11.7, 4.8 Hz, 1H, 1-H^A), 3.75 (d, *J* = 8.8 Hz, 1H, 3-H), 3.69 (dd, *J* = 11.7, 7.8 Hz, 1H, 1-H^B), 2.84 (s, 1H, OH, D₂O-exchangeable), 1.62 (dd, *J* = 7.8, 4.8 Hz, 1H, OH, D₂O-exchangeable); ¹³C NMR: (67.9 MHz, CDCl₃) 142.28 (s, ipso), 139.42 (s, ipso), 136.91 (d, C-4), 129.52 (d), 128.23 (d), 128.04 (d), 127.13 (d), 127.00 (d), 126.13 (d), 117.63 (t, C-5), 78.50 (s, C-2), 68.49 (t, C-1), 57.98 (d, C-3); MS: (CI) *m/z* 237 (95), 219 (100). Anal. Calcd for C₁₇H₁₈O₂: C, 80.28; H, 7.13. Found: C, 80.28; H, 7.08.

(3R*,4S*)-4-Methyl-4,5-di(*p*-nitrobenzoyloxy)-3-phenyl-1-pentene (17dE). The esterification was performed by known method.¹ *p*-Nitrobenzoic anhydride (4.0 mmol) was added to a stirred suspended mixture of **17d** (1.0 mmol), MgBr₂ (4.0 mmol) and Et₃N (6.0 mmol) in dry CH₂Cl₂ (10 mL) at room temperature. The mixture was refluxed (40 °C) and stirred for 2 h

and then quenched by the addition of water. The product was extracted with ethyl acetate (50 mL $\times$ 2). The organic layer was washed with water and then dried (MgSO_4). The solvent was evaporated and the residue was purified by flash chromatography (hexane/ Et_2O , 3/1) on silica gel and recrystallization (hexane-ethanol-chloroform, 5 : 5 : 1) to give the product (0.35g, 88%). The crystal obtained was suitable for X-ray crystallographic analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: mp: 111-112 °C; IR: (KBr) 1724, 1635 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 8.00-8.30 (m, 8H, *p*- NO_2 -aroma), 7.20-7.40 (m, 5H, aroma), 6.40 (m, 1H, 2-H), 5.27 (d, J = 15.1 Hz, 1H, 1- H^{B}), 5.26 (d, J = 11.7 Hz, 1H, 1- H^{A}), 4.96 (d, J = 11.8 Hz, 1H, 5- H^{A}), 4.83 (d, J = 11.8 Hz, 1H, 5- H^{B}), 4.32 (d, J = 9.8 Hz, 1H, 3-H); ^{13}C NMR: (67.9 MHz, CDCl_3) 163.94 (s, C=O), 163.61 (s, C=O), 150.56 (s, *p*- NO_2 -ipso), 150.40 (s, *p*- NO_2 -ipso), 138.72 (s, ipso), 136.33 (s, ipso), 135.16 (d, C-2), 134.90 (s, ipso), 130.59 (d), 130.48 (d), 129.30 (d), 128.49 (d), 127.38 (d), 123.60 (d), 123.51 (d), 119.45 (t, C-1), 85.24 (s, C-4), 66.98 (t, C-5), 55.87 (d, C-3), 19.58 (q, 4-Me); MS: (CI, 70 eV) m/z 491 ($\text{M}^+ + 1$, 5), 325 (20), 324 (100), 224 (14), 168 (19), 157 (25), 138 (18); HRMS: (CI, 70 eV) calcd for $\text{C}_{26}\text{H}_{23}\text{N}_2\text{O}_8$ 491.1376, found m/z 491.1429, 491.1426, 491.1463 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{26}\text{H}_{22}\text{N}_2\text{O}_8$: C, 63.67; H, 4.52; N, 5.71. Found: C, 63.53; H, 4.47; N, 5.65.

Procedure for Transformation from 17d to 17a.

To a solution of **17d** (0.5 mmol) and MeI (5 mmol) in THF (6 mL) at room temperature was added sodium hydride (0.5 mmol). The mixture was stirred for 24 h and then quenched by H_2O (20 mL). The resultant mixture was extracted with diethyl ether (100 mL), and the organic layer was washed with water and then dried over MgSO_4 . The solvent evaporated under reduced pressure to give **17a** (59%) confirmed by ^1H NMR.

Procedure for Transformation from 17e to 17c.

To a solution of **17e** (1 mmol) and MeI (10 mmol) in THF (10 mL) at room temperature was added sodium hydride (1 mmol). The mixture was stirred for 32 h and then quenched by H_2O (20 mL). The resultant mixture was extracted with ethyl acetate (50 mL $\times$ 2), and the organic layer was washed with water and then dried over MgSO_4 . The solvent evaporated under reduced pressure to give **17c** (11%) confirmed by ^1H NMR.

Alkylations in Table 4. The reaction in entry 1 in Table 4 was run according to the general procedure (I) using **1a** (1.0 mmol), SnCl_2 (1.0 mmol), and **16a** (1.0 mmol). The reaction in entry 2 in Table 4 was run according to the general procedure (II) using **1a** (1.0 mmol), $\text{BF}_3 \cdot \text{OEt}_2$ (1.0 mmol), and **16a** (1.0 mmol). The reaction in entry 3 in Table 4 was as follows: To a suspension of Zinc powder (2.5 mmol) and **16a** (1.0 mmol) in THF (0.2 mL) and saturated NH_4Cl aq (2 mL) at room temperature was added cinnamyl chloride (2.5 mmol). The mixture was stirred for 3 h at rt. H_2O (20 mL) was added and the resultant mixture was extracted with diethyl ether (50 mL $\times$ 2), and the organic layer was washed with water and then dried over MgSO_4 . The solvent evaporated under reduced pressure to give the product (58%, 78: 22) in crude mixture confirmed by ^1H NMR. The reaction in entry 4 in Table 4 was as follows: A flask was charged with dried Et_2O (10 mL) and 0.15 g of magnesium turnings under nitrogen and was cooled to 0 °C. Cinnamyl chloride (4.5 mmol) was slowly added and the mixture was stirred vigorously for 30 min and cooled to -78 °C. The ketone **16a** (3.0 mmol) was added to the Grignard reagent solution at -78 °C. After stirring for 1 h, H_2O was added to quench the reaction. The mixture was extracted with Et_2O and the organic layer was dried over MgSO_4 . The solvent

was distilled off under reduced pressure to give the product (75%, 54:46) in crude mixture confirmed by ^1H NMR.

Competitive Alkylations in Table 4. The reaction in entry 1 in Table 4 was run according to the general procedure (I) using **1a** (1.0 mmol), SnCl_2 (1.0 mmol), **16a** (1.0 mmol), and **2i** (1.0 mmol). The reaction in entry 2 in Table 4 was run according to the general procedure (II) using **1a** (1.0 mmol), $\text{BF}_3 \cdot \text{OEt}_2$ (1.0 mmol), **16a** (1.0 mmol), and **2i** (1.0 mmol). The reaction in entry 3 in Table 4 was as follows: To a suspension of zinc powder (2.5 mmol), **16a** (1.0 mmol), and **2i** (1.0 mmol) in THF (0.2 mL) and saturated NH_4Cl aq (2 mL) at room temperature was added cinnamyl chloride (1.0 mmol). The mixture was stirred for 5 h at rt. H_2O (20 mL) was added and the resultant mixture was extracted with diethyl ether (50 mL $\times$ 2), and the organic layer was washed with water and then dried over MgSO_4 . The solvent evaporated under reduced pressure to give the products in crude mixture, which were analyzed by ^1H NMR. The reaction in entry 4 in Table 4 was as follows: A flask was charged with dried Et_2O (10 mL) and 0.15 g of magnesium turnings under nitrogen and was cooled to 0 $^\circ\text{C}$. Cinnamyl chloride (3.0 mmol) was slowly added and the mixture was stirred vigorously for 30 min and cooled to -78 $^\circ\text{C}$. The mixture of **16a** (3.0 mmol) and **2i** (3.0 mmol) in 3 mL of Et_2O was added to the Grignard reagent solution at -78 $^\circ\text{C}$. After stirring for 1 h, H_2O was added to quench the reaction. The mixture was extracted with Et_2O and the organic layer was dried over MgSO_4 . The solvent was distilled off under reduced pressure to give the products in crude mixture, which were analyzed by ^1H NMR.

(2R*)-2-((1R*)-Cyclohex-2-enyl)-1-methoxypropan-2-ol (18a). According to the general procedure (I), this compound was prepared from **1b**, SnCl_2 , and **16a** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 62 $^\circ\text{C}$ /0.11 mmHg; IR: (neat) 3464 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 5.85-5.76 (m, 2H, 2'-H and 3'-H), 3.38 (s, 3H, MeO), 3.34 (d, $J = 9.3$ Hz, 1H, 1-H^A), 3.21 (d, $J = 9.3$ Hz, 1H, 1-H^B), 2.37 (s, 1H, OH, D_2O -exchangeable), 2.41-2.37 (m, 1H, 1'-H), 1.98-1.94 (m, 2H, 4'-H₂), 1.84-1.68 (m, 2H, 5'-H^A and 6'-H^A), 1.59-1.43 (m, 1H, 5'-H^B), 1.28-1.13 (m, 1H, 6'-H^B), 1.03 (s, 3H, 3-H₃); ^{13}C NMR: (67.9 MHz, CDCl_3) 128.74 (d), 127.44 (d), 78.42 (t, C-1), 73.90 (s, C-2), 59.20 (q, MeO), 42.74 (d, C-1'), 25.00 (t, C-4'), 24.59 (t, C-6'), 24.22 (t, C-5'), 19.82 (q, C-3); MS: (EI, 70 eV) m/z 125 (18), 89 (100). Anal. Calcd for $\text{C}_{10}\text{H}_{18}\text{O}_2$: C, 70.55; H, 10.66. Found: C, 70.22; H, 10.43.

(2R*)-2-((1R*)-Cyclohex-2-enyl)-1-phenoxypropan-2-ol (18b). According to the general procedure (I), this compound was prepared from **1b**, SnCl_2 , and **16b** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/ Et_2O , 3/1). Further purification was performed by distillation under reduced pressure: bp: 95 $^\circ\text{C}$ /0.11 mmHg; IR: (neat) 3464 cm^{-1} ; ^1H NMR: (270 MHz, CDCl_3) 7.31-7.22 (m, 2H, aroma), 6.986.89 (m, 3H, aroma), 5.90-5.79 (m, 2H, 2'-H and 3'-H), 3.91 (d, $J = 9.3$ Hz, 1H, 1-H^A), 3.79 (d, $J = 9.3$ Hz, 1H, 1-H^B), 2.61-2.55 (m, 1H, 1'-H), 2.39 (s, 1H, OH, D_2O -exchangeable), 1.99-1.94 (m, 2H, 4'-H₂), 1.90-1.12 (m, 4H, 5'-H₂ and 6'-H₂), 1.19 (s, 3H, 3-H₃); ^{13}C NMR: (67.9 MHz, CDCl_3) 158.62 (s, ipso), 129.40 (d), 129.24 (d, olefin), 127.00 (d, olefin), 120.99 (d), 114.54 (d), 73.81 (s, C-2), 73.28 (t, C-1), 42.62 (d, C-1'), 24.96 (t, C-4'), 24.54 (t, C-6'), 22.16 (t, C-5'), 19.98 (q, C-3); MS: (EI) m/z 232 (M^+ , 1), 151 (100), 133 (52); HRMS: (EI) calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$ 232.1463, found m/z 232.1471, 232.1464, 232.1434 (M^+). Anal. Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$: C, 77.55; H, 8.68. Found: C, 77.36; H, 8.61.

(2R*)-2-((1S*)-Cyclohex-2-enyl)-1-phenoxypropan-2-ol (18b'). This compound was not purely isolated because this is the minor isomer in the reaction of **1b** with **16b** in the presence of SnCl₂ (Table 9, entries 2 or 3). In a crude reaction mixture, the signals for the title compound were overlapped or obscured in the NMR spectra. The only independent signals are shown below.

¹H NMR: (270 MHz, CDCl₃) 5.71-5.68 (m, 2'-H and 3'-H), 3.92 (d, *J* = 9.3 Hz, 1-H^A), 3.83 (d, *J* = 9.3 Hz, 1-H^B), 2.36 (s, OH, D₂O-exchangeable), 1.27 (s, 3H, 3-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 158.68 (s, ipso), 129.59, 126.77 (olefin), 120.96, 73.72 (C-2), 72.78 (C-1), 42.67 (C-1'), 25.05, 23.52, 22.04 (C-5').

(1R*)-1-((1S*)-Cyclohex-2-enyl)-2-methoxy-1-phenylethanol (18c). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **16c** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 96 °C/0.11 mmHg; IR: (neat) 3564 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.44-7.41 (m, 2H, aroma), 7.34-7.31 (m, 2H, aroma), 7.29-7.19 (m, 1H, aroma), 5.85-5.76 (m, 2H, 2'-H and 3'-H), 3.77 (d, *J* = 9.3 Hz, 1H, 2-H^A), 3.71 (d, *J* = 9.3 Hz, 1H, 2-H^B), 3.34 (s, 3H, MeO), 2.79 (s, 1H, OH, D₂O-exchangeable), 2.62-2.59 (m, 1H, 1'-H), 1.91-1.87 (m, 2H, 4'-H₂), 1.72-1.63 (m, 1H, 5'-H^A), 1.49-1.20 (m, 3H, 5'-H^B and 6'-H₂); ¹³C NMR: (67.9 MHz, CDCl₃) 143.47 (s, ipso), 130.05 (d, olefin), 127.67 (d), 126.55 (d), 126.55 (d, olefin), 125.73 (d), 77.63 (t, C2), 77.32 (s, C-1), 59.29 (q, MeO), 43.66 (d, C-1'), 24.96 (t, C-4'), 24.09 (t, C-6'), 21.95 (t, C-5'); MS: (CI) *m/z* 233 (*M*⁺ + 1, 1), 215 (100), 151 (25); HRMS: (CI) calcd for C₁₅H₂₁O₂ 233.1463, found *m/z* 233.1515, 233.1550, 233.1527 (*M*⁺ + 1). Anal. Calcd for C₁₅H₂₀O₂: C, 77.55; H, 8.68. Found: C, 77.26; H, 8.71.

(2S*)-2-((1S*)-Cyclohex-2-enyl)-propan-1, 2-diol (18d). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **16d** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 65 °C/0.11 mmHg; IR: (neat) 3394 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 5.87-5.75 (m, 2H), 3.57 (d, *J* = 11.2 Hz, 1H, 1-H^A), 3.42 (d, *J* = 11.2 Hz, 1H, 1-H^B), 3.07 (s, 1H, OH, D₂O-exchangeable), 2.73 (s, 1H, OH, D₂O-exchangeable), 2.41-2.35 (m, 1H, 1'-H), 1.98-1.94 (m, 2H, 4'-H₂), 1.83-1.73 (m, 2H, 5'-H^A and 6'-H^A), 1.59-1.42 (m, 1H, 5'-H^B), 1.28-1.11 (m, 1H, 6'-H^B), 1.05 (s, 3H, 3-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 129.32 (d), 126.97 (d), 75.04 (s, C-2), 68.34 (t, C-1), 42.33 (d, C-1'), 24.99 (t, C-4'), 24.50 (t, C-6'), 22.15 (t, C-5'), 19.49 (q, C-3); MS: (EI) *m/z* 125 (21), 75 (100). Anal. Calcd for C₉H₁₆O₂: C, 69.19; H, 10.32. Found: C, 68.80; H, 10.20.

(1R*)-1-((1S*)-Cyclohex-2-enyl)-1-phenylethane-1,2-diol (18e). According to the general procedure (I), this compound was prepared from **1b**, SnCl₂, and **16e** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 1/1). Further purification was performed by distillation under reduced pressure to give a white solid: bp: 132 °C/0.1 mmHg; mp: 112 °C; IR: (neat) 3340 cm⁻¹; ¹H NMR: (270 MHz, CDCl₃) 7.39-7.20 (m, 5H, aroma), 5.86-5.73 (m, 2H, 2'-H and 3'-H), 3.87 (d, *J* = 11.2 Hz, 1H, 2-H^A), 3.82 (d, *J* = 11.2 Hz, 1H, 2-H^B), 2.84 (s, 1H, OH, D₂O-exchangeable), 2.55-2.52 (m, 1H, 1'-H), 2.39 (s, 1H, OH, D₂O-exchangeable), 1.90-1.88 (m, 2H, 4'-H₂), 1.68-1.62 (m, 1H, 5'-H^A), 1.47-1.32 (m, 2H, 5'-H^B and 6'-H^A), 1.27-1.13 (m, 1H, 6'-H^B); ¹³C NMR: (67.9 MHz, CDCl₃) 142.51 (s, ipso), 130.79 (d, C-3'), 127.96 (d), 126.75 (d), 126.08 (d, C-2'), 125.97 (d), 78.59 (s, C-1), 67.86 (s, C-2),

42.99 (d, C-1'), 24.93 (t, C-4'), 24.03 (t, C-6'), 21.79 (t, C-5'); MS: (EI) m/z 187 (21), 137 (100), 119 (64). Anal. Calcd for $C_{14}H_{18}O_2$: C, 77.03; H, 8.31. Found: C, 76.95; H, 8.53.

(2R*)-2-((1R*)-Cyclopent-2-enyl)-1-methoxypropan-2-ol (18f). According to the general procedure (I), this compound was prepared from **1c**, $SnCl_2$, and **16a** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 32 °C/0.11 mmHg; IR: (neat) 3464 cm^{-1} ; ¹H NMR: (270 MHz, CDCl₃) 5.88-5.84 (m, 1H), 5.78-5.74 (m, 1H), 3.39 (s, 3H, OMe), 3.31 (d, J = 9.3 Hz, 1H, 1-H^A), 3.23 (d, J = 9.3 Hz, 1H, 1-H^B), 2.96-2.88 (m, 1H, 1'-H), 2.32-2.28 (m, 2H, 4'-H₂), 2.26 (s, 1H, OH, D₂O-exchangeable), 1.98-1.85 (m, 1H, 5'-H^A), 1.70-1.57 (m, 1H, 5'-H^B), 1.08 (s, 3H, 3-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 132.83 (d), 130.77 (d), 79.29 (t, C-1), 73.85 (s, C-2), 59.29 (q, OMe), 53.32 (d, C-1'), 32.26 (t, C-4'), 24.71 (t, C-5'), 21.12 (q, C-3); MS: (EI) m/z 138 (1), 111 (18), 89 (100). Anal. Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 68.79; H, 10.10.

(2R*)-2-((1R*)-Cyclopent-2-enyl)-1-phenoxypropan-2-ol (18g). According to the general procedure (I), this compound was prepared from **1c**, $SnCl_2$, and **16b** in acetonitrile to give the product as a colorless liquid after chromatography (hexane/Et₂O, 3/1). Further purification was performed by distillation under reduced pressure: bp: 106 °C/0.11 mmHg; IR: (neat) 3456 cm^{-1} ; ¹H NMR: (270 MHz, CDCl₃) 7.31-7.23 (m, 2H, aroma), 6.98-6.90 (m, 3H, aroma), 5.92-5.87 (m, 1H), 5.81-5.78 (m, 1H), 3.86 (d, J = 9.3 Hz, 1H, 1-H^A), 3.80 (d, J = 9.3 Hz, 1H, 1-H^B), 3.13-3.06 (m, 1H, 1'-H), 2.38-2.30 (m, 2H, 4'-H₂), 2.27 (s, 1H, OH, D₂O-exchangeable), 2.02-1.89 (m, 1H, 5'-H^A), 1.77-1.64 (m, 1H, 5'-H^B), 1.22 (s, 3H, 3-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 158.75 (s, ipso), 133.38 (d, olefin), 130.39 (d, olefin), 129.43 (d), 120.99 (d), 114.59 (d), 74.14 (t, C-1), 73.70 (s, C-2), 53.26 (d, C-1'), 32.32 (t, C-4'), 24.68 (t, C-5'), 21.24 (q, C-3); MS: (EI) m/z 218 (M⁺, 4), 151 (100), 133 (49); HRMS: (EI) calcd for $C_{14}H_{18}O_2$ 218.1307, found m/z 218.1314, 218.1315, 218.1306 (M⁺). Anal. Calcd for $C_{14}H_{18}O_2$: C, 77.03; H, 8.31. Found: C, 76.79; H, 8.34.

(2R*)-2-((1S*)-Cyclopent-2-enyl)-1-phenoxypropan-2-ol (18g'). This compound was not purely isolated because this is the minor isomer in the reaction of **1c** with **16b** in the presence of $SnCl_2$ (Table 9, entry 8). In a crude reaction mixture, the signals for the title compound were overlapped or obscured in the NMR spectra. The only independent signals are shown below. ¹H NMR: (270 MHz, CDCl₃) 5.70-5.63 (m), 2.23 (s, OH, D₂O-exchangeable), 1.25 (s, 3H, 3-H₃); ¹³C NMR: (67.9 MHz, CDCl₃) 133.46 (olefin), 130.22 (olefin), 73.70 (C-2), 53.06 (C-1'), 32.37 (C-4'), 24.04 (C-5'), 21.96 (C-3).

(4R*)-4-((1S*)-Cyclohex-2-enyl)-2,2-dimethyl-4-phenyl-1,3-dioxolane (19). *p*-Toluenesulfonic Acid (0.4 mmol) was added to a stirred solution of **18e** (2.0 mmol) and acetone (20 mmol) in dry benzene (5 mL) at room temperature. The mixture was stirred for 6 h and then quenched by H₂O. The resultant mixture was extracted with ethyl acetate (50 mL × 2), and the organic layer was washed with water and then dried over MgSO₄. The solvent evaporated under reduced pressure and the residue was purified by column chromatography (hexane/ethyl acetate, 6/1) on silica gel and recrystallization (hexane) to give the product (0.42g, 82%). The crystal obtained was suitable for X-ray crystallographic analysis. Details of the crystal structure data and analysis are provided in the Supporting Information: mp: 68 °C; IR: (KBr) 1211, 1057 cm^{-1} ; ¹H NMR: (270 MHz, CDCl₃) 7.35-7.20 (m, 5H, aroma), 5.85-5.74 (m, 2H, 2'-H and 3'-H), 4.36 (d,

$J = 8.8$ Hz, 1H, 5-H^A), 4.17 (d, $J = 8.8$ Hz, 1H, 5-H^B), 2.53-2.48 (m, 1H, 1'-H), 1.91-1.88 (m, 2H, 4'-H₂), 1.70-1.62 (m, 1H, 5'-H^A), 1.51 (s, 3H, 2-Me^A), 1.46-1.23 (m, 3H, 5'-H^B and 6'-H₂), 1.21 (s, 3H, 2-Me^B); ¹³C NMR: (67.9 MHz, CDCl₃) 144.77 (s, ipso), 129.43 (d, vinyl), 127.71 (d), 127.10 (d, vinyl), 126.64 (d), 125.68 (d), 109.91 (s, C-2), 86.72 (s, C-4), 72.75 (t, C-5), 45.72 (d, C-1'), 26.64 (q, 2-Me^A), 25.72 (q, 2-Me^B), 25.08 (t, C-4'), 24.97 (t, C-6'), 21.99 (t, C-5'); MS: (CI) m/z 259 ($M^+ + 1$, 11), 201 (100), 177 (46), 119 (11); HRMS: (CI) calcd for C₁₇H₂₃O₂ 259.1620, found m/z 259.1695 ($M^+ + 1$). Anal. Calcd for C₁₇H₂₂O₂: C, 79.03; H, 8.58. Found: C, 79.00; H, 8.46.

Procedure for Transformation from 18e to 18c. To a solution of **18e** (0.5 mmol) and MeI (5mmol) in THF (5 mL) at room temperature was added sodium hydride (1 mmol). The mixture was stirred for 15 h and then quenched by H₂O (20 mL). The resultant mixture was extracted with ethyl acetate (50 mL × 2), and the organic layer was washed with water and then dried over MgSO₄. The solvent evaporated under reduced pressure to give **18c** (72%) confirmed by ¹H NMR.

References and Notes.

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X-ray Crystallographic Data

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X-ray Structure Report for **3aE**

Experimental

Data Collection

A colorless prism crystal of $C_{38}H_{31}O_2Cl_3$ having approximate dimensions of 0.10 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-K α radiation.

Indexing was performed from 2 oscillations which were exposed for 5.0 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned} a &= 14.200(1) \text{ \AA} \\ b &= 9.8919(7) \text{ \AA} & \beta &= 92.757(2)^\circ \\ c &= 23.384(2) \text{ \AA} \\ V &= 3281.0(4) \text{ \AA}^3 \end{aligned}$$

For $Z = 4$ and F.W. = 626.02, the calculated density is 1.27 g/cm³. The systematic absences of:

$$\begin{aligned} h0l: & l \neq 2n \\ 0k0: & k \neq 2n \end{aligned}$$

uniquely determine the space group to be:

$$P2_1/c (\#14)$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ to a maximum 2θ value of 55.0° . A total of 55 images, corresponding to 220.0° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 3.30 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 29225 reflections which were collected, 7500 were unique ($R_{\text{int}} = 0.083$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 3.1 cm⁻¹. A symmetry-related absorption correction using the program ABSCOR¹ was applied which resulted in transmission factors ranging from 0.71 to 0.97. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods² and expanded using Fourier techniques³. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement⁴ was based on 2220 observed reflections ($I > 2.00\sigma(I)$, $2\theta < 54.96$) and 388 variable parameters and converged (largest parameter shift was 2.69 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.134$$

$$R_w = [(\sum w (|F_o| - |F_c|)^2 / \sum w F_o^2)]^{1/2} = 0.213$$

$$R_w = [(\sum w (F_o^2 - F_c^2)^2 / \sum w (F_o^2)^2)]^{1/2} = 0.000 \quad \text{for all data}$$

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.134 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁵ was 4.38. The weighting scheme was based on counting statistics and included a factor ($p = 0.050$) to downweight the intense reflections. Plots of $\sum w (|F_o| - |F_c|)^2$ versus $|F_o|$, 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 0.68 and -0.39 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁶. Anomalous dispersion effects were included in Fcalc⁷; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁸. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁹. All calculations were performed using the teXsan¹⁰ crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(3) 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.

(4) Least-Squares:

Function minimized: $\sum w(|F_o| - |F_c|)^2$ where
 $w = 1/[\sigma^2(F_o)] = [\sigma_c^2(F_o) + p^2 F_o^2/4]^{-1}$
 $\sigma_c(F_o) = \text{e.s.d. based on counting statistics}$
 $p = \text{p-factor}$

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

$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$
 where: N_o = number of observations
 N_v = number of variables

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- (10) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₃₈ H ₃₁ O ₂ Cl ₃
Formula Weight	626.02
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.10 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	16009 (3.4 - 54.8 $^{\circ}$)
Indexing Images	2 oscillations at 5.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 14.200(1) Å b = 9.8919(7) Å c = 23.384(2) Å β = 92.757(2) $^{\circ}$ V = 3281.0(4) Å ³
Space Group	P2 ₁ /c (#14)
Z value	4
D _{calc}	1.267 g/cm ³
F ₀₀₀	1304.00
μ (MoK α)	3.11 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Temperature	23.0 $^{\circ}$ C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm

Data Images	55 exposures at 3.3 minutes per degree
Oscillation Range ($\phi=0.0^\circ, \chi=45.0^\circ$)	ω 130.0 - 190.0 $^\circ$ with 4.0 $^\circ$ step
Oscillation Range ($\phi=180.0^\circ, \chi=45.0^\circ$)	ω 0.0 - 160.0 $^\circ$ with 4.0 $^\circ$ step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	55.0 $^\circ$
No. of Reflections Measured	Total: 29225 Unique: 7500 ($R_{\text{int}} = 0.083$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.7074 - 0.9694)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o - F_c)^2$
Least Squares Weights	$1/\sigma^2(F_o) = 4F_o^2/\sigma^2(F_o^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I > 2.00\sigma(I)$, $2\theta < 54.96^\circ$)	2220
No. Variables	388
Reflection/Parameter Ratio	5.72
Residuals: R; R_w	0.134 ; 0.213
Residuals: R_1	0.134
No. of Reflections to calc R_1	2220
Goodness of Fit Indicator	4.38
Max Shift/Error in Final Cycle	2.690
Maximum peak in Final Diff. Map	0.68 e $^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	-0.39 e $^-/\text{\AA}^3$

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

atom	x	y	z	B_{eq}
Cl(1)	0.3429(6)	0.0327(8)	0.8857(3)	7.0(2)
Cl(2)	0.3379(6)	0.5120(10)	0.5414(3)	8.6(3)
Cl(3)	-0.0127(6)	-0.3799(8)	0.5457(3)	7.2(2)
O(1)	-0.093(1)	0.046(2)	0.7383(7)	4.6(4)
O(2)	-0.1644(9)	0.138(1)	0.6597(5)	2.9(3)
C(1)	-0.217(2)	0.191(4)	0.524(1)	7.5(10)
C(2)	-0.254(2)	0.084(3)	0.5511(10)	4.8(7)
C(3)	-0.304(1)	0.113(2)	0.6057(8)	3.4(5)
C(4)	-0.252(1)	0.054(2)	0.6608(9)	3.1(6)
C(5)	-0.308(2)	0.086(3)	0.7132(9)	3.6(6)
C(6)	-0.363(2)	-0.015(3)	0.7348(10)	4.2(6)
C(7)	-0.422(2)	0.015(3)	0.780(1)	4.6(7)
C(8)	-0.422(2)	0.142(3)	0.804(1)	5.5(8)
C(9)	-0.370(2)	0.248(4)	0.782(1)	6.1(8)
C(10)	-0.307(2)	0.210(3)	0.737(1)	4.7(7)
C(11)	-0.405(2)	0.058(3)	0.597(1)	5.1(8)
C(12)	-0.428(2)	-0.067(4)	0.574(1)	6.9(9)
C(13)	-0.521(3)	-0.110(4)	0.568(1)	8(1)
C(14)	-0.582(2)	-0.019(6)	0.590(2)	9(1)
C(15)	-0.561(3)	0.110(6)	0.613(2)	11(1)
C(16)	-0.472(2)	0.153(4)	0.617(1)	6.6(9)
C(17)	-0.225(2)	-0.094(2)	0.6544(9)	3.8(6)
C(18)	-0.092(2)	0.120(3)	0.698(1)	4.0(6)
C(19)	-0.007(1)	0.214(2)	0.6880(8)	2.5(5)
C(20)	0.088(2)	0.138(2)	0.6739(8)	3.1(5)
C(21)	0.156(2)	0.110(2)	0.7284(8)	3.2(5)
C(22)	0.117(2)	0.086(2)	0.7824(8)	3.6(6)
C(23)	0.182(2)	0.061(3)	0.8297(8)	5.0(7)
C(24)	0.278(2)	0.061(2)	0.8224(10)	3.8(6)
C(25)	0.315(2)	0.082(2)	0.7727(10)	4.4(6)
C(26)	0.255(2)	0.107(2)	0.7250(9)	3.9(6)
C(27)	0.148(1)	0.234(2)	0.6350(8)	2.2(5)
C(28)	0.167(2)	0.361(3)	0.6548(9)	4.0(6)
C(29)	0.228(2)	0.448(2)	0.625(1)	4.2(6)
C(30)	0.265(2)	0.405(3)	0.575(1)	5.8(8)
C(31)	0.241(2)	0.283(3)	0.554(1)	5.2(7)
C(32)	0.187(2)	0.195(3)	0.5830(10)	4.6(7)
C(33)	0.067(1)	0.006(2)	0.6422(8)	2.6(5)
C(34)	0.099(2)	-0.120(2)	0.6635(10)	4.0(6)
C(35)	0.073(2)	-0.240(3)	0.634(1)	5.1(7)
C(36)	0.017(2)	-0.233(3)	0.584(1)	4.6(7)
C(37)	-0.019(1)	-0.110(3)	0.5634(8)	3.3(5)

C(38)	0.009(1)	0.009(2)	0.5916(8)	3.0(5)
H(1)	-0.2235	0.2800	0.5389	8.8
H(2)	-0.1847	0.1792	0.4892	8.8
H(4)	-0.2482	-0.0060	0.5363	5.8
H(5)	-0.3082	0.2093	0.6101	4.0
H(6)	-0.3600	-0.1050	0.7194	5.1
H(7)	-0.4632	-0.0522	0.7941	5.6
H(8)	-0.4594	0.1581	0.8363	6.6
H(9)	-0.3739	0.3388	0.7961	7.3
H(10)	-0.2637	0.2758	0.7231	5.6
H(11)	-0.3792	-0.1258	0.5627	8.1
H(12)	-0.5414	-0.1930	0.5506	9.5
H(13)	-0.6457	-0.0457	0.5909	10.8
H(14)	-0.6100	0.1683	0.6256	12.8
H(15)	-0.4541	0.2420	0.6307	7.7
H(16)	-0.2807	-0.1485	0.6554	4.7
H(17)	-0.1966	-0.1075	0.6191	4.7
H(18)	-0.1827	-0.1197	0.6851	4.7
H(19)	-0.0241	0.2725	0.6567	2.9
H(20)	0.0048	0.2681	0.7215	2.9
H(21)	0.0500	0.0864	0.7867	4.4
H(22)	0.1593	0.0447	0.8670	5.6
H(23)	0.3822	0.0796	0.7699	5.2
H(24)	0.2818	0.1224	0.6886	4.8
H(25)	0.1399	0.3916	0.6889	4.9
H(26)	0.2435	0.5360	0.6403	4.9
H(27)	0.2622	0.2580	0.5169	6.1
H(28)	0.1746	0.1061	0.5684	5.6
H(29)	0.1385	-0.1237	0.6976	4.8
H(30)	0.0932	-0.3262	0.6491	6.1
H(31)	-0.0606	-0.1072	0.5303	3.9
H(32)	-0.0111	0.0940	0.5760	3.6

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	0.100(6)	0.094(6)	0.069(4)	-0.001(5)	-0.040(4)	0.011(4)
Cl(2)	0.127(7)	0.115(7)	0.087(5)	-0.060(6)	0.032(5)	0.026(5)
Cl(3)	0.115(6)	0.068(5)	0.094(5)	-0.023(5)	0.026(5)	-0.045(5)
O(1)	0.05(1)	0.07(1)	0.06(1)	-0.018(10)	0.005(8)	

	0.011(10)					
O(2)	0.023(8)	0.050(10)	0.034(8)	0.006(8)	-0.004(7)	0.004(7)
C(1)	0.07(2)	0.14(3)	0.07(2)	0.00(2)	0.00(2)	0.01(2)
C(2)	0.05(2)	0.08(2)	0.05(2)	0.01(2)	0.00(1)	0.01(2)
C(3)	0.04(1)	0.06(2)	0.03(1)	0.00(1)	0.001(10)	0.00(1)
C(4)	0.02(1)	0.04(2)	0.06(1)	-0.01(1)	-0.01(1)	0.01(1)
C(5)	0.03(1)	0.07(2)	0.04(1)	0.01(1)	0.00(1)	0.00(1)
C(6)	0.04(1)	0.06(2)	0.07(2)	0.00(1)	0.01(1)	0.02(1)
C(7)	0.04(2)	0.07(2)	0.06(2)	0.00(2)	0.01(1)	0.00(2)
C(8)	0.05(2)	0.08(2)	0.08(2)	-0.02(2)	0.02(1)	0.01(2)
C(9)	0.05(2)	0.11(3)	0.07(2)	0.01(2)	0.01(1)	-0.03(2)
C(10)	0.04(2)	0.06(2)	0.08(2)	0.00(1)	-0.02(1)	0.00(2)
C(11)	0.04(2)	0.10(2)	0.05(2)	-0.02(2)	-0.01(1)	0.02(2)
C(12)	0.07(2)	0.11(3)	0.08(2)	-0.02(2)	-0.03(2)	0.02(2)
C(13)	0.08(3)	0.14(3)	0.08(2)	-0.01(3)	-0.02(2)	0.03(2)
C(14)	0.05(2)	0.23(5)	0.09(3)	-0.05(3)	-0.01(2)	0.05(3)
C(15)	0.10(4)	0.23(6)	0.09(3)	-0.02(4)	-0.01(2)	0.03(3)
C(16)	0.04(2)	0.15(3)	0.06(2)	0.03(2)	0.01(1)	0.01(2)
C(17)	0.05(2)	0.04(2)	0.06(2)	0.00(1)	0.01(1)	0.01(1)
C(18)	0.06(2)	0.04(2)	0.05(2)	-0.01(2)	0.03(1)	-0.01(1)
C(19)	0.02(1)	0.04(1)	0.04(1)	0.01(1)	0.012(9)	0.00(1)
C(20)	0.05(2)	0.04(1)	0.03(1)	0.00(1)	0.01(1)	0.00(1)
C(21)	0.07(2)	0.03(1)	0.02(1)	0.00(1)	0.00(1)	0.00(1)
C(22)	0.07(2)	0.04(1)	0.04(1)	0.00(1)	0.00(1)	0.00(1)
C(23)	0.13(2)	0.05(2)	0.01(1)	-0.01(2)	-0.01(1)	0.01(1)
C(24)	0.03(1)	0.04(1)	0.07(2)	0.00(1)	-0.02(1)	0.01(1)
C(25)	0.08(2)	0.05(2)	0.03(1)	0.00(1)	0.01(1)	0.02(1)
C(26)	0.03(1)	0.06(2)	0.06(2)	0.01(1)	0.00(1)	0.00(1)
C(27)	0.01(1)	0.04(1)	0.03(1)	0.00(1)	0.003(9)	0.01(1)
C(28)	0.05(2)	0.04(2)	0.06(1)	0.00(1)	-0.01(1)	-0.01(1)
C(29)	0.03(1)	0.05(2)	0.08(2)	-0.01(1)	0.01(1)	0.02(1)
C(30)	0.10(2)	0.07(2)	0.05(2)	-0.02(2)	0.01(2)	0.01(2)
C(31)	0.06(2)	0.08(2)	0.06(2)	-0.03(2)	0.02(1)	0.02(2)
C(32)	0.07(2)	0.06(2)	0.05(1)	0.00(2)	0.01(1)	-0.02(1)
C(33)	0.01(1)	0.05(1)	0.04(1)	0.00(1)	0.012(9)	0.00(1)
C(34)	0.07(2)	0.03(1)	0.05(2)	0.01(1)	0.00(1)	-0.01(1)
C(35)	0.11(2)	0.05(2)	0.04(1)	0.00(2)	0.01(1)	0.00(1)
C(36)	0.08(2)	0.04(2)	0.06(2)	-0.01(2)	0.03(1)	-0.02(1)
C(37)	0.03(1)	0.05(2)	0.05(1)	-0.01(1)	0.02(1)	-0.01(1)
C(38)	0.05(1)	0.04(1)	0.03(1)	0.00(1)	-0.011(10)	0.01(1)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Cl(1)	C(24)	1.73(2)	Cl(2)	C(30)	1.70(3)
Cl(3)	C(36)	1.75(3)	O(1)	C(18)	1.19(3)
O(2)	C(4)	1.50(2)	O(2)	C(18)	1.35(3)
C(1)	C(2)	1.35(4)	C(2)	C(3)	1.52(3)
C(3)	C(4)	1.57(3)	C(3)	C(11)	1.54(3)
C(4)	C(5)	1.52(3)	C(4)	C(17)	1.52(3)
C(5)	C(6)	1.37(3)	C(5)	C(10)	1.35(3)
C(6)	C(7)	1.42(3)	C(7)	C(8)	1.37(4)
C(8)	C(9)	1.39(4)	C(9)	C(10)	1.47(3)
C(11)	C(12)	1.38(4)	C(11)	C(16)	1.43(4)
C(12)	C(13)	1.38(4)	C(13)	C(14)	1.36(5)
C(14)	C(15)	1.41(6)	C(15)	C(16)	1.33(5)
C(18)	C(19)	1.55(3)	C(19)	C(20)	1.60(3)
C(20)	C(21)	1.58(3)	C(20)	C(27)	1.59(3)
C(20)	C(33)	1.52(3)	C(21)	C(22)	1.43(3)
C(21)	C(26)	1.41(3)	C(22)	C(23)	1.43(3)
C(23)	C(24)	1.38(3)	C(24)	C(25)	1.31(3)
C(25)	C(26)	1.39(3)	C(27)	C(28)	1.37(3)
C(27)	C(32)	1.41(3)	C(28)	C(29)	1.42(3)
C(29)	C(30)	1.38(3)	C(30)	C(31)	1.35(4)
C(31)	C(32)	1.37(3)	C(33)	C(34)	1.41(3)
C(33)	C(38)	1.40(3)	C(34)	C(35)	1.41(3)
C(35)	C(36)	1.39(3)	C(36)	C(37)	1.39(3)
C(37)	C(38)	1.39(3)			

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.96	C(1)	H(2)	0.95
C(2)	H(4)	0.96	C(3)	H(5)	0.96
C(6)	H(6)	0.96	C(7)	H(7)	0.96
C(8)	H(8)	0.96	C(9)	H(9)	0.96
C(10)	H(10)	0.96	C(12)	H(11)	0.96
C(13)	H(12)	0.96	C(14)	H(13)	0.95
C(15)	H(14)	0.97	C(16)	H(15)	0.97

C(17)	H(16)	0.96	C(17)	H(17)	0.95
C(17)	H(18)	0.95	C(19)	H(19)	0.95
C(19)	H(20)	0.96	C(22)	H(21)	0.96
C(23)	H(22)	0.96	C(25)	H(23)	0.96
C(26)	H(24)	0.96	C(28)	H(25)	0.95
C(29)	H(26)	0.96	C(31)	H(27)	0.96
C(32)	H(28)	0.96	C(34)	H(29)	0.95
C(35)	H(30)	0.96	C(37)	H(31)	0.96
C(38)	H(32)	0.96			

Table 5. Bond Angles($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(4)	O(2)	C(18)	121(1)	C(1)	C(2)	C(3)	117(2)
C(2)	C(3)	C(4)	113(1)	C(2)	C(3)	C(11)	106(1)
C(4)	C(3)	C(11)	111(1)	O(2)	C(4)	C(3)	97(1)
O(2)	C(4)	C(5)	111(1)	O(2)	C(4)	C(17)	108(1)
C(3)	C(4)	C(5)	109(1)	C(3)	C(4)	C(17)	113(1)
C(5)	C(4)	C(17)	114(1)	C(4)	C(5)	C(6)	118(2)
C(4)	C(5)	C(10)	121(2)	C(6)	C(5)	C(10)	120(2)
C(5)	C(6)	C(7)	119(2)	C(6)	C(7)	C(8)	120(2)
C(7)	C(8)	C(9)	121(2)	C(8)	C(9)	C(10)	115(2)
C(5)	C(10)	C(9)	122(2)	C(3)	C(11)	C(12)	125(2)
C(3)	C(11)	C(16)	110(2)	C(12)	C(11)	C(16)	124(2)
C(11)	C(12)	C(13)	121(3)	C(12)	C(13)	C(14)	112(3)
C(13)	C(14)	C(15)	127(3)	C(14)	C(15)	C(16)	119(4)
C(11)	C(16)	C(15)	114(3)	O(1)	C(18)	O(2)	124(2)
O(1)	C(18)	C(19)	122(2)	O(2)	C(18)	C(19)	112(2)
C(18)	C(19)	C(20)	114(1)	C(19)	C(20)	C(21)	113(1)
C(19)	C(20)	C(27)	108(1)	C(19)	C(20)	C(33)	110(1)
C(21)	C(20)	C(27)	104(1)	C(21)	C(20)	C(33)	110(1)
C(27)	C(20)	C(33)	109(1)	C(20)	C(21)	C(22)	119(1)
C(20)	C(21)	C(26)	121(1)	C(22)	C(21)	C(26)	118(1)
C(21)	C(22)	C(23)	116(2)	C(22)	C(23)	C(24)	120(2)
Cl(1)	C(24)	C(23)	112(1)	Cl(1)	C(24)	C(25)	124(2)
C(23)	C(24)	C(25)	123(2)	C(24)	C(25)	C(26)	118(2)
C(21)	C(26)	C(25)	121(2)	C(20)	C(27)	C(28)	117(1)
C(20)	C(27)	C(32)	124(2)	C(28)	C(27)	C(32)	117(2)
C(27)	C(28)	C(29)	120(2)	C(28)	C(29)	C(30)	119(2)
Cl(2)	C(30)	C(29)	117(2)	Cl(2)	C(30)	C(31)	122(2)
C(29)	C(30)	C(31)	119(2)	C(30)	C(31)	C(32)	121(2)
C(27)	C(32)	C(31)	120(2)	C(20)	C(33)	C(34)	121(1)

C(20)	C(33)	C(38)	119(1)	C(34)	C(33)	C(38)	118(2)
C(33)	C(34)	C(35)	120(2)	C(34)	C(35)	C(36)	119(2)
Cl(3)	C(36)	C(35)	120(2)	Cl(3)	C(36)	C(37)	118(2)
C(35)	C(36)	C(37)	121(2)	C(36)	C(37)	C(38)	118(1)
C(33)	C(38)	C(37)	121(2)				

Table 6. Bond Angles($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(1)	H(1)	119.8	C(2)	C(1)	H(2)	121.4
H(1)	C(1)	H(2)	118.8	C(1)	C(2)	H(4)	120.6
C(3)	C(2)	H(4)	122.2	C(2)	C(3)	H(5)	108.2
C(4)	C(3)	H(5)	108.2	C(11)	C(3)	H(5)	107.8
C(5)	C(6)	H(6)	120.2	C(7)	C(6)	H(6)	120.5
C(6)	C(7)	H(7)	120.3	C(8)	C(7)	H(7)	119.0
C(7)	C(8)	H(8)	119.2	C(9)	C(8)	H(8)	118.9
C(8)	C(9)	H(9)	122.9	C(10)	C(9)	H(9)	121.9
C(5)	C(10)	H(10)	118.2	C(9)	C(10)	H(10)	119.5
C(11)	C(12)	H(11)	119.4	C(13)	C(12)	H(11)	119.0
C(12)	C(13)	H(12)	125.3	C(14)	C(13)	H(12)	122.5
C(13)	C(14)	H(13)	116.8	C(15)	C(14)	H(13)	115.5
C(14)	C(15)	H(14)	121.0	C(16)	C(15)	H(14)	119.2
C(11)	C(16)	H(15)	122.8	C(15)	C(16)	H(15)	122.8
C(4)	C(17)	H(16)	109.0	C(4)	C(17)	H(17)	110.1
C(4)	C(17)	H(18)	110.0	H(16)	C(17)	H(17)	108.8
H(16)	C(17)	H(18)	108.7	H(17)	C(17)	H(18)	110.2
C(18)	C(19)	H(19)	108.4	C(18)	C(19)	H(20)	108.3
C(20)	C(19)	H(19)	108.1	C(20)	C(19)	H(20)	108.2
H(19)	C(19)	H(20)	108.7	C(21)	C(22)	H(21)	121.6
C(23)	C(22)	H(21)	121.8	C(22)	C(23)	H(22)	119.6
C(24)	C(23)	H(22)	119.7	C(24)	C(25)	H(23)	119.8
C(26)	C(25)	H(23)	121.3	C(21)	C(26)	H(24)	118.8
C(25)	C(26)	H(24)	119.3	C(27)	C(28)	H(25)	119.2
C(29)	C(28)	H(25)	119.9	C(28)	C(29)	H(26)	120.5
C(30)	C(29)	H(26)	120.2	C(30)	C(31)	H(27)	119.0
C(32)	C(31)	H(27)	119.7	C(27)	C(32)	H(28)	119.0
C(31)	C(32)	H(28)	120.1	C(33)	C(34)	H(29)	119.9
C(35)	C(34)	H(29)	120.0	C(34)	C(35)	H(30)	120.8
C(36)	C(35)	H(30)	119.8	C(36)	C(37)	H(31)	120.5
C(38)	C(37)	H(31)	120.8	C(33)	C(38)	H(32)	119.1
C(37)	C(38)	H(32)	119.2				

Table 7. Torsion Angles($^{\circ}$)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Cl(1)	C(24)	C(23)	C(22)	178(1)	Cl(1)	C(24)	C(25)	C(26)	-178(1)
Cl(2)	C(30)	C(29)	C(28)	179(1)	Cl(2)	C(30)	C(31)	C(32)	176(2)
Cl(3)	C(36)	C(35)	C(34)	177(1)	Cl(3)	C(36)	C(37)	C(38)	-176(1)
O(1)	C(18)	O(2)	C(4)	-5(3)	O(1)	C(18)	C(19)	C(20)	66(2)
O(2)	C(4)	C(3)	C(2)	64(2)	O(2)	C(4)	C(3)	C(11)	-174(2)
O(2)	C(4)	C(5)	C(6)	-151(1)	O(2)	C(4)	C(5)	C(10)	31(2)
O(2)	C(18)	C(19)	C(20)	-117(1)	C(1)	C(2)	C(3)	C(4)	-112(2)
C(1)	C(2)	C(3)	C(11)	123(2)	C(2)	C(3)	C(4)	C(5)	-178(2)
C(2)	C(3)	C(4)	C(17)	-49(2)	C(2)	C(3)	C(11)	C(12)	45(3)
C(2)	C(3)	C(11)	C(16)	-135(2)	C(3)	C(4)	O(2)	C(18)	-176(1)
C(3)	C(4)	C(5)	C(6)	101(2)	C(3)	C(4)	C(5)	C(10)	-76(2)
C(3)	C(11)	C(12)	C(13)	179(2)	C(3)	C(11)	C(16)	C(15)	-177(2)
C(4)	O(2)	C(18)	C(19)	177(1)	C(4)	C(3)	C(11)	C(12)	-79(2)
C(4)	C(3)	C(11)	C(16)	99(2)	C(4)	C(5)	C(6)	C(7)	-173(1)
C(4)	C(5)	C(10)	C(9)	171(2)	C(5)	C(4)	O(2)	C(18)	68(2)
C(5)	C(4)	C(3)	C(11)	-57(2)	C(5)	C(6)	C(7)	C(8)	-3(3)
C(5)	C(10)	C(9)	C(8)	6(3)	C(6)	C(5)	C(4)	C(17)	-27(2)
C(6)	C(5)	C(10)	C(9)	-5(3)	C(6)	C(7)	C(8)	C(9)	5(4)
C(7)	C(6)	C(5)	C(10)	3(3)	C(7)	C(8)	C(9)	C(10)	-6(4)
C(10)	C(5)	C(4)	C(17)	154(2)	C(11)	C(3)	C(4)	C(17)	72(2)
C(11)	C(12)	C(13)	C(14)	-3(4)	C(11)	C(16)	C(15)	C(14)	0(5)
C(12)	C(11)	C(16)	C(15)	1(4)	C(12)	C(13)	C(14)	C(15)	5(5)
C(13)	C(12)	C(11)	C(16)	0(4)	C(13)	C(14)	C(15)	C(16)	-3(6)
C(17)	C(4)	O(2)	C(18)	-59(2)	C(18)	C(19)	C(20)	C(21)	-93(2)
C(18)	C(19)	C(20)	C(27)	151(1)	C(18)	C(19)	C(20)	C(33)	31(2)
C(19)	C(20)	C(21)	C(22)	32(2)	C(19)	C(20)	C(21)	C(26)	-148(2)
C(19)	C(20)	C(27)	C(28)	54(2)	C(19)	C(20)	C(27)	C(32)	-129(2)
C(19)	C(20)	C(33)	C(34)	-122(2)	C(19)	C(20)	C(33)	C(38)	55(2)
C(20)	C(21)	C(22)	C(23)	179(2)	C(20)	C(21)	C(26)	C(25)	-179(2)
C(20)	C(27)	C(28)	C(29)	173(1)	C(20)	C(27)	C(32)	C(31)	-176(2)
C(20)	C(33)	C(34)	C(35)	176(2)	C(20)	C(33)	C(38)	C(37)	-174(1)
C(21)	C(20)	C(27)	C(28)	-67(2)	C(21)	C(20)	C(27)	C(32)	108(2)
C(21)	C(20)	C(33)	C(34)	4(2)	C(21)	C(20)	C(33)	C(38)	-178(1)
C(21)	C(22)	C(23)	C(24)	0(3)	C(21)	C(26)	C(25)	C(24)	0(3)
C(22)	C(21)	C(20)	C(27)	149(1)	C(22)	C(21)	C(20)	C(33)	-92(2)
C(22)	C(21)	C(26)	C(25)	0(3)	C(22)	C(23)	C(24)	C(25)	-1(3)
C(23)	C(22)	C(21)	C(26)	0(3)	C(23)	C(24)	C(25)	C(26)	1(3)
C(26)	C(21)	C(20)	C(27)	-31(2)	C(26)	C(21)	C(20)	C(33)	86(2)
C(27)	C(20)	C(33)	C(34)	118(2)	C(27)	C(20)	C(33)	C(38)	-64(2)

C(27) C(28) C(29) C(30)	1(3)	C(27) C(32) C(31) C(30)	5(4)
C(28) C(27) C(20) C(33)	174(1)	C(28) C(27) C(32) C(31)	0(3)
C(28) C(29) C(30) C(31)	2(3)	C(29) C(28) C(27) C(32)	-2(3)
C(29) C(30) C(31) C(32)	-6(4)	C(32) C(27) C(20) C(33)	-9(2)
C(33) C(34) C(35) C(36)	1(3)	C(33) C(38) C(37) C(36)	-5(3)
C(34) C(33) C(38) C(37)	2(3)	C(34) C(35) C(36) C(37)	-4(3)
C(35) C(34) C(33) C(38)	0(3)	C(35) C(36) C(37) C(38)	5(3)

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
Cl(3)	Cl(3) ¹⁾	3.23(1)	O(1)	C(28) ²⁾	3.31(3)
C(7)	C(29) ²⁾	3.52(3)	C(15)	C(31) ³⁾	3.51(5)
C(17)	C(23) ²⁾	3.49(3)	C(17)	C(24) ²⁾	3.55(3)

Symmetry operations

(1) -X,-Y-1,-Z+1
(3) X-1,Y,Z

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

X-ray Structure Report for **6bE**

Experimental

Data Collection

A colorless prismatic crystal of $C_{21}H_{20}O_4NCl$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 20 carefully centered reflections in the range $25.19 < 2\theta < 28.16^\circ$ corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned} a &= 7.601(6) \text{ \AA} \\ b &= 9.651(4) \text{ \AA} & \beta &= 95.76(3)^\circ \\ c &= 26.999(4) \text{ \AA} \\ V &= 1970(1) \text{ \AA}^3 \end{aligned}$$

For $Z = 4$ and F.W. = 385.85, the calculated density is 1.30 g/cm³. The systematic absences of:

$$\begin{aligned} h0l: & h+1 \pm 2n \\ 0k0: & k \pm 2n \end{aligned}$$

uniquely determine the space group to be:

$$P2_1/n \text{ (#14)}$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 55.0° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.32° with a take-off angle of 6.0° . Scans of $(0.58 + 0.30 \tan \theta)^\circ$ were made at a speed of $16.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum

of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 6.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 4438 reflections which were collected, 3846 were unique ($R_{\text{int}} = 0.087$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 2.2 cm^{-1} . An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.61 to 0.89.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ was based on 3846 all reflections ($2\theta < 54.98$) and 324 variable parameters and converged (largest parameter shift was 0.16 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (F_o^2 - F_c^2) / \Sigma F_o^2 = 0.174$$

$$R_w = [(\Sigma w (F_o^2 - F_c^2)^2 / \Sigma w (F_o^2)^2)]^{1/2} = 0.175$$

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.056 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁴ was 1.00. The weighting scheme was based on counting statistics and included a factor ($p = 0.050$) to downweight the intense reflections.

Plots of $\sum w (F_o^2 - F_c^2)^2$ versus F_o^2 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 0.44 and -0.36 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the *teXsan*⁹ crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(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: $\sum w (F_o^2 - F_c^2)^2$ where

$$w = 1/[\sigma^2(F_o^2)] = [\sigma_c^2(F_o^2) + (p \cdot (\text{Max}(F_o^2, 0) + 2F_c^2)/3)^2]^{-1}$$

 $\sigma_c(F_o^2)$ = e.s.d. based on counting statistics
 p = p-factor

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

$$[\sum w (|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$

where: N_o = number of observations
 N_v = number of variables

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(6) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) 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).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₁ H ₂₀ O ₄ NCl
Formula Weight	385.85
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	20 (25.2 - 28.2°)
Omega Scan Peak Width at Half-height	0.32°
Lattice Parameters	a = 7.601(6) Å b = 9.651(4) Å c = 26.999(4) Å β = 95.76(3)° V = 1970(1) Å ³
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.300 g/cm ³
F ₀₀₀	808.00
μ (MoK α)	2.19 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.69,
12.30, 44.72)	
Temperature	23.0 °C
Collimator Size	6.0 mm

Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm
Scan Type	ω -2 θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	(0.58 + 0.30 tan θ)°
2 $\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 4438 Unique: 3846 ($R_{\text{int}} = 0.087$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.6070 - 0.8862)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/\sigma^2(F_o^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Reflections (All, $2\sigma < 54.98^\circ$)	3846
No. Variables	324
Reflection/Parameter Ratio	11.87
Residuals: R; R_w	0.174 ; 0.175
Residuals: R_1	0.056
No. of Reflections to calc R_1	1149
Goodness of Fit Indicator	1.00
Max Shift/Error in Final Cycle	0.157
Maximum peak in Final Diff. Map	0.44 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.36 e ⁻ /Å ³

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

atom	x	y	z	B_{eq}
Cl(1)	0.7642(2)	-0.0399(2)	0.78037(6)	9.40(6)
O(1)	0.5493(5)	0.2708(4)	0.9697(1)	6.6(1)
O(2)	-0.2534(5)	0.0844(4)	1.0774(1)	6.9(1)
O(5)	-0.0445(5)	-0.0377(5)	1.1131(2)	7.8(1)
O(6)	0.3145(4)	0.3620(4)	0.9235(1)	4.28(8)
N(3)	-0.0991(6)	0.0487(5)	1.0832(2)	4.9(1)
C(2)	0.5143(6)	0.3039(5)	0.8618(2)	4.0(1)
C(3)	0.4212(6)	0.4224(5)	0.8861(2)	4.1(1)
C(4)	0.2766(7)	0.4850(6)	0.8477(2)	4.1(1)
C(5)	0.6841(7)	0.3184(7)	0.8467(2)	5.4(2)
C(6)	0.2029(7)	0.0754(6)	1.0599(2)	4.7(1)
C(7)	0.0818(7)	0.2657(6)	0.9871(2)	4.8(2)
C(8)	-0.0368(7)	0.2084(6)	1.0166(2)	4.4(1)
C(9)	0.0265(6)	0.1146(5)	1.0519(2)	3.8(1)
C(10)	0.3169(7)	0.1357(6)	1.0294(2)	4.7(1)
C(11)	0.5431(9)	0.5337(7)	0.9103(2)	6.0(2)
C(12)	0.1498(8)	0.5845(6)	0.8700(2)	5.2(2)
C(13)	0.3930(7)	0.2881(6)	0.9610(2)	4.6(1)
C(14)	0.5042(8)	0.0745(6)	0.8267(2)	5.4(2)
C(15)	0.7595(8)	0.2121(8)	0.8215(2)	6.0(2)
C(16)	0.1351(9)	0.7432(7)	0.7961(2)	5.9(2)
C(17)	0.4274(7)	0.1816(6)	0.8507(2)	4.4(1)
C(18)	0.3595(8)	0.5522(6)	0.8049(2)	5.4(2)
C(19)	0.0283(8)	0.6537(7)	0.8291(2)	5.9(2)
C(20)	0.6697(8)	0.0929(7)	0.8121(2)	5.7(2)
C(21)	0.2972(8)	0.6664(7)	0.7840(2)	6.0(2)
C(22)	0.2609(6)	0.2299(5)	0.9933(2)	3.8(1)
H(1)	0.736(7)	0.405(5)	0.851(2)	6.7(5)
H(2)	0.252(7)	0.019(5)	1.089(2)	7.8(6)
H(3)	0.044(5)	0.318(4)	0.961(1)	3.1(7)
H(4)	-0.163(6)	0.229(5)	1.011(2)	5.7(7)
H(5)	0.438(6)	0.113(4)	1.036(1)	4.6(7)
H(6)	0.441(6)	-0.011(5)	0.820(2)	6.2(6)
H(7)	0.853(7)	0.222(5)	0.811(2)	6.6(6)
H(8)	0.322(7)	0.167(6)	0.862(2)	7.6(6)
H(9)	0.215(5)	0.405(4)	0.834(1)	3.1(7)
H(10)	0.054(7)	0.756(6)	0.762(2)	8.7(5)
H(11)	0.163(6)	0.826(5)	0.814(2)	4.6(6)
H(12)	0.632(7)	0.489(5)	0.936(2)	7.0(5)
H(13)	0.583(5)	0.589(4)	0.881(2)	4.3(7)
H(14)	0.490(8)	0.578(6)	0.932(2)	8.0(5)
H(15)	0.452(7)	0.497(5)	0.791(2)	7.3(6)

H(16)	0.359(6)	0.703(5)	0.758(2)	5.3(7)
H(17)	0.089(6)	0.532(5)	0.895(2)	6.5(6)
H(18)	0.233(5)	0.661(4)	0.889(2)	4.6(7)
H(19)	-0.034(7)	0.582(5)	0.807(2)	7.6(5)
H(20)	-0.043(6)	0.711(4)	0.847(1)	4.2(7)

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	0.136(2)	0.128(2)	0.099(1)	0.071(1)	0.036(1)	-0.005(1)
O(1)	0.038(2)	0.139(4)	0.073(3)	0.002(2)	0.005(2)	0.026(2)
O(2)	0.047(2)	0.122(4)	0.095(3)	0.006(2)	0.017(2)	0.020(3)
O(5)	0.070(3)	0.124(4)	0.104(3)	-0.004(3)	0.011(2)	0.059(3)
O(6)	0.043(2)	0.072(2)	0.048(2)	0.006(2)	0.008(2)	0.004(2)
N(3)	0.058(3)	0.077(4)	0.051(3)	-0.012(3)	0.005(3)	0.004(3)
C(2)	0.046(3)	0.058(4)	0.048(3)	0.003(3)	0.010(3)	0.006(3)
C(3)	0.046(3)	0.059(4)	0.052(3)	-0.008(3)	0.008(3)	-0.001(3)
C(4)	0.051(3)	0.055(4)	0.048(3)	0.000(3)	0.006(3)	-0.005(3)
C(5)	0.047(4)	0.080(5)	0.078(4)	0.002(3)	0.016(3)	0.015(4)
C(6)	0.055(4)	0.066(4)	0.056(4)	0.002(3)	0.006(3)	0.016(3)
C(7)	0.053(4)	0.082(4)	0.047(3)	0.014(3)	0.000(3)	0.010(3)
C(8)	0.045(3)	0.074(4)	0.049(3)	0.003(3)	0.008(3)	0.009(3)
C(9)	0.047(3)	0.057(3)	0.039(3)	-0.003(3)	0.007(3)	-0.002(3)
C(10)	0.038(3)	0.083(4)	0.057(3)	0.008(3)	-0.002(3)	0.005(3)
C(11)	0.074(4)	0.084(5)	0.068(5)	-0.019(4)	0.004(3)	-0.005(4)
C(12)	0.065(4)	0.072(4)	0.064(4)	0.014(4)	0.016(3)	0.003(3)
C(13)	0.053(4)	0.085(4)	0.037(3)	-0.001(3)	0.006(3)	0.003(3)
C(14)	0.072(5)	0.062(4)	0.071(4)	0.012(4)	0.010(3)	-0.011(3)
C(15)	0.051(4)	0.108(6)	0.073(4)	0.025(5)	0.030(3)	0.031(4)
C(16)	0.082(5)	0.068(4)	0.074(5)	0.014(4)	0.006(4)	0.001(4)
C(17)	0.054(3)	0.062(4)	0.055(3)	-0.004(3)	0.014(3)	-0.002(3)
C(18)	0.071(4)	0.069(4)	0.068(4)	0.017(4)	0.023(3)	0.008(3)
C(19)	0.073(4)	0.079(5)	0.074(4)	0.022(4)	0.018(4)	0.005(4)
C(20)	0.071(4)	0.086(5)	0.061(4)	0.030(4)	0.020(3)	0.009(4)
C(21)	0.073(4)	0.086(5)	0.074(4)	0.010(4)	0.026(3)	0.016(4)
C(22)	0.045(3)	0.059(3)	0.041(3)	0.005(3)	0.002(2)	0.004(3)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Cl(1)	C(20)	1.737(6)	O(1)	C(13)	1.199(6)
O(2)	N(3)	1.217(5)	O(5)	N(3)	1.204(5)
O(6)	C(3)	1.478(5)	O(6)	C(13)	1.330(6)
N(3)	C(9)	1.482(6)	C(2)	C(3)	1.528(6)
C(2)	C(5)	1.399(6)	C(2)	C(17)	1.371(6)
C(3)	C(4)	1.554(6)	C(3)	C(11)	1.522(7)
C(4)	C(12)	1.526(7)	C(4)	C(18)	1.516(7)
C(4)	H(9)	0.96(4)	C(5)	C(15)	1.387(8)
C(5)	H(1)	0.93(5)	C(6)	C(9)	1.389(6)
C(6)	C(10)	1.382(7)	C(6)	H(2)	0.99(5)
C(7)	C(8)	1.377(6)	C(7)	C(22)	1.398(7)
C(7)	H(3)	0.89(4)	C(8)	C(9)	1.365(6)
C(8)	H(4)	0.98(4)	C(10)	C(22)	1.369(6)
C(10)	H(5)	0.95(4)	C(11)	H(12)	1.01(5)
C(11)	H(13)	1.01(4)	C(11)	H(14)	0.86(5)
C(12)	C(19)	1.521(8)	C(12)	H(17)	0.99(5)
C(12)	H(18)	1.06(4)	C(13)	C(22)	1.504(6)
C(14)	C(17)	1.380(7)	C(14)	C(20)	1.367(7)
C(14)	H(6)	0.96(5)	C(15)	C(20)	1.349(8)
C(15)	H(7)	0.80(5)	C(16)	C(19)	1.531(8)
C(16)	C(21)	1.502(8)	C(16)	H(10)	1.06(5)
C(16)	H(11)	0.94(4)	C(17)	H(8)	0.90(5)
C(18)	C(21)	1.306(7)	C(18)	H(15)	0.99(5)
C(19)	H(19)	1.00(5)	C(19)	H(20)	0.95(4)
C(21)	H(16)	0.95(4)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	O(6)	C(13)	119.8(4)	O(2)	N(3)	O(5)	123.0(5)
O(2)	N(3)	C(9)	118.2(5)	O(5)	N(3)	C(9)	118.8(5)
C(3)	C(2)	C(5)	122.1(5)	C(3)	C(2)	C(17)	120.4(4)
C(5)	C(2)	C(17)	117.4(5)	O(6)	C(3)	C(2)	107.9(4)

O(6)	C(3)	C(4)	102.1(3)	O(6)	C(3)	C(11)	109.7(4)
C(2)	C(3)	C(4)	109.4(4)	C(2)	C(3)	C(11)	115.1(4)
C(4)	C(3)	C(11)	111.8(5)	C(3)	C(4)	C(12)	114.5(4)
C(3)	C(4)	C(18)	110.7(4)	C(3)	C(4)	H(9)	103(2)
C(12)	C(4)	C(18)	111.0(5)	C(12)	C(4)	H(9)	110(2)
C(18)	C(4)	H(9)	105(2)	C(2)	C(5)	C(15)	120.5(6)
C(2)	C(5)	H(1)	116(3)	C(15)	C(5)	H(1)	122(3)
C(9)	C(6)	C(10)	116.5(5)	C(9)	C(6)	H(2)	123(2)
C(10)	C(6)	H(2)	119(2)	C(8)	C(7)	C(22)	121.2(5)
C(8)	C(7)	H(3)	120(2)	C(22)	C(7)	H(3)	118(2)
C(7)	C(8)	C(9)	117.6(5)	C(7)	C(8)	H(4)	121(2)
C(9)	C(8)	H(4)	120(2)	N(3)	C(9)	C(6)	117.5(5)
N(3)	C(9)	C(8)	118.7(5)	C(6)	C(9)	C(8)	123.8(5)
C(6)	C(10)	C(22)	122.3(5)	C(6)	C(10)	H(5)	115(2)
C(22)	C(10)	H(5)	121(2)	C(3)	C(11)	H(12)	109(3)
C(3)	C(11)	H(13)	104(2)	C(3)	C(11)	H(14)	109(4)
H(12)	C(11)	H(13)	121(3)	H(12)	C(11)	H(14)	94(4)
H(13)	C(11)	H(14)	117(4)	C(4)	C(12)	C(19)	110.6(5)
C(4)	C(12)	H(17)	107(2)	C(4)	C(12)	H(18)	104(2)
C(19)	C(12)	H(17)	115(2)	C(19)	C(12)	H(18)	109(2)
H(17)	C(12)	H(18)	108(3)	O(1)	C(13)	O(6)	125.6(5)
O(1)	C(13)	C(22)	122.8(5)	O(6)	C(13)	C(22)	111.6(5)
C(17)	C(14)	C(20)	118.9(6)	C(17)	C(14)	H(6)	120(2)
C(20)	C(14)	H(6)	120(2)	C(5)	C(15)	C(20)	119.8(5)
C(5)	C(15)	H(7)	121(4)	C(20)	C(15)	H(7)	118(4)
C(19)	C(16)	C(21)	110.0(5)	C(19)	C(16)	H(10)	105(2)
C(19)	C(16)	H(11)	106(2)	C(21)	C(16)	H(10)	107(2)
C(21)	C(16)	H(11)	112(2)	H(10)	C(16)	H(11)	114(4)
C(2)	C(17)	C(14)	122.0(5)	C(2)	C(17)	H(8)	119(3)
C(14)	C(17)	H(8)	118(3)	C(4)	C(18)	C(21)	122.0(5)
C(4)	C(18)	H(15)	114(3)	C(21)	C(18)	H(15)	122(3)
C(12)	C(19)	C(16)	110.6(5)	C(12)	C(19)	H(19)	110(3)
C(12)	C(19)	H(20)	102(2)	C(16)	C(19)	H(19)	107(3)
C(16)	C(19)	H(20)	109(2)	H(19)	C(19)	H(20)	116(4)
Cl(1)	C(20)	C(14)	119.0(6)	Cl(1)	C(20)	C(15)	119.7(5)
C(14)	C(20)	C(15)	121.3(6)	C(16)	C(21)	C(18)	126.4(6)
C(16)	C(21)	H(16)	116(2)	C(18)	C(21)	H(16)	116(2)
C(7)	C(22)	C(10)	118.6(5)	C(7)	C(22)	C(13)	122.5(5)
C(10)	C(22)	C(13)	118.9(5)				

Table 5. Torsion Angles($^{\circ}$)

atom atom atom atom angle atom atom atom atom angle

Cl(1)	C(20)	C(14)	C(17)	-178.3(4)	Cl(1)	C(20)	C(15)	C(5)	179.9(4)
O(1)	C(13)	O(6)	C(3)	4.7(8)	O(1)	C(13)	C(22)	C(7)	171.1(6)
O(1)	C(13)	C(22)	C(10)	-10.7(8)	O(2)	N(3)	C(9)	C(6)	178.3(5)
O(2)	N(3)	C(9)	C(8)	-2.4(7)	O(5)	N(3)	C(9)	C(6)	-2.0(7)
O(5)	N(3)	C(9)	C(8)	177.4(5)	O(6)	C(3)	C(2)	C(5)	-145.0(4)
O(6)	C(3)	C(2)	C(17)	39.8(6)	O(6)	C(3)	C(4)	C(12)	54.3(6)
O(6)	C(3)	C(4)	C(18)	-179.3(4)	O(6)	C(13)	C(22)	C(7)	-7.3(7)
O(6)	C(13)	C(22)	C(10)	171.0(4)	N(3)	C(9)	C(6)	C(10)	178.5(5)
N(3)	C(9)	C(8)	C(7)	-178.5(4)	C(2)	C(3)	O(6)	C(13)	59.4(5)
C(2)	C(3)	C(4)	C(12)	168.4(4)	C(2)	C(3)	C(4)	C(18)	-65.2(5)
C(2)	C(5)	C(15)	C(20)	-1.1(9)	C(2)	C(17)	C(14)	C(20)	-2.3(8)
C(3)	O(6)	C(13)	C(22)	-177.0(4)	C(3)	C(2)	C(5)	C(15)	-175.2(5)
C(3)	C(2)	C(17)	C(14)	177.0(5)	C(3)	C(4)	C(12)	C(19)	173.0(5)
C(3)	C(4)	C(18)	C(21)	-142.6(6)	C(4)	C(3)	O(6)	C(13)	174.6(4)
C(4)	C(3)	C(2)	C(5)	104.7(5)	C(4)	C(3)	C(2)	C(17)	-70.5(6)
C(4)	C(12)	C(19)	C(16)	-63.5(7)	C(4)	C(18)	C(21)	C(16)	-
3.5(10)									
C(5)	C(2)	C(3)	C(11)	-22.2(7)	C(5)	C(2)	C(17)	C(14)	1.6(8)
C(5)	C(15)	C(20)	C(14)	0.4(9)	C(6)	C(9)	C(8)	C(7)	0.8(8)
C(6)	C(10)	C(22)	C(7)	-0.3(8)	C(6)	C(10)	C(22)	C(13)	-178.7(5)
C(8)	C(7)	C(22)	C(10)	0.3(8)	C(8)	C(7)	C(22)	C(13)	178.6(5)
C(8)	C(9)	C(6)	C(10)	-0.8(8)	C(9)	C(6)	C(10)	C(22)	0.5(8)
C(9)	C(8)	C(7)	C(22)	-0.5(8)	C(11)	C(3)	O(6)	C(13)	-66.7(6)
C(11)	C(3)	C(2)	C(17)	162.7(5)	C(11)	C(3)	C(4)	C(12)	-62.9(6)
C(11)	C(3)	C(4)	C(18)	63.5(6)	C(12)	C(4)	C(18)	C(21)	-14.3(8)
C(12)	C(19)	C(16)	C(21)	44.1(7)	C(15)	C(5)	C(2)	C(17)	0.1(8)
C(15)	C(20)	C(14)	C(17)	1.2(9)	C(18)	C(4)	C(12)	C(19)	46.8(7)
C(18)	C(21)	C(16)	C(19)	-11.8(9)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
Cl(1)	H(10) ¹⁾	3.03(5)	Cl(1)	H(19) ²⁾	3.04(5)
Cl(1)	H(15) ³⁾	3.06(5)	Cl(1)	H(20) ¹⁾	3.26(4)
Cl(1)	H(11) ¹⁾	3.34(5)	Cl(1)	H(7) ³⁾	3.42(5)
Cl(1)	O(5) ⁴⁾	3.485(5)	Cl(1)	C(16) ¹⁾	3.502(7)
Cl(1)	H(2) ⁴⁾	3.55(5)	Cl(1)	H(1) ³⁾	3.59(5)
O(1)	H(4) ⁵⁾	2.39(5)	O(1)	H(14) ⁶⁾	3.06(5)
O(1)	C(8) ⁵⁾	3.327(7)	O(2)	H(5) ⁷⁾	2.51(4)
O(2)	H(18) ⁸⁾	2.62(4)	O(2)	H(8) ⁹⁾	3.00(5)

O(2)	H(11) ⁸⁾	3.08(4)
O(2)	C(10) ⁷⁾	3.427(7)
O(2)	C(17) ⁹⁾	3.553(6)
O(2)	C(10) ⁹⁾	3.575(7)
O(5)	H(8) ⁹⁾	2.59(5)
O(5)	H(11) ⁸⁾	3.04(4)
O(5)	H(3) ⁹⁾	3.37(4)
O(5)	H(20) ⁸⁾	3.37(4)
O(5)	C(7) ⁹⁾	3.477(7)
N(3)	H(20) ⁸⁾	3.11(4)
N(3)	H(8) ⁹⁾	3.14(5)
N(3)	C(7) ⁹⁾	3.589(7)
C(5)	H(10) ²⁾	3.35(6)
C(6)	H(4) ⁹⁾	3.51(4)
C(6)	C(8) ⁹⁾	3.584(8)
C(8)	H(17) ⁸⁾	3.51(5)
C(9)	H(18) ⁸⁾	3.43(4)
C(10)	H(14) ⁶⁾	3.25(6)
C(13)	H(14) ⁶⁾	3.20(6)
C(13)	H(4) ⁵⁾	3.56(5)
C(14)	H(10) ²⁾	2.97(5)
C(15)	H(10) ²⁾	3.14(6)
C(16)	H(6) ¹¹⁾	3.34(5)
C(17)	H(10) ²⁾	3.15(5)
C(17)	H(16) ²⁾	3.48(4)
C(19)	H(1) ⁷⁾	3.36(5)
C(20)	H(10) ²⁾	2.95(6)
C(21)	H(6) ¹¹⁾	3.41(5)
C(22)	H(12) ⁶⁾	3.37(5)
H(1)	H(17) ⁵⁾	3.08(7)
H(2)	H(6) ⁴⁾	3.22(7)
H(3)	H(12) ⁷⁾	3.55(7)
H(4)	H(5) ⁷⁾	3.36(6)
H(4)	H(12) ⁷⁾	3.51(7)
H(5)	H(14) ⁶⁾	3.13(7)
H(6)	H(11) ¹⁰⁾	2.63(7)
H(6)	H(10) ²⁾	3.40(7)
H(7)	H(16) ³⁾	3.01(7)
H(7)	H(10) ²⁾	3.51(8)
H(8)	H(16) ²⁾	3.41(7)
H(9)	H(10) ²⁾	3.58(6)

O(2)	H(6) ⁹⁾	3.32(5)
O(2)	H(20) ⁸⁾	3.49(4)
O(2)	C(12) ⁸⁾	3.553(8)
O(2)	C(22) ⁹⁾	3.582(6)
O(5)	H(7) ⁴⁾	2.99(5)
O(5)	C(15) ⁴⁾	3.143(7)
O(5)	C(20) ⁴⁾	3.366(7)
O(5)	C(17) ⁹⁾	3.454(7)
N(3)	H(18) ⁸⁾	3.10(4)
N(3)	H(11) ⁸⁾	3.12(4)
N(3)	C(22) ⁹⁾	3.537(6)
C(2)	H(10) ²⁾	3.37(6)
C(5)	H(19) ⁵⁾	3.56(5)
C(6)	H(20) ⁸⁾	3.55(4)
C(8)	H(18) ⁸⁾	3.33(4)
C(9)	H(20) ⁸⁾	3.19(4)
C(9)	C(9) ⁹⁾	3.560(9)
C(12)	H(1) ⁷⁾	3.58(5)
C(13)	H(12) ⁶⁾	3.54(5)
C(14)	H(2) ⁴⁾	2.93(5)
C(14)	H(11) ¹⁰⁾	3.52(4)
C(15)	H(2) ⁴⁾	3.30(5)
C(16)	H(15) ¹²⁾	3.41(5)
C(17)	H(2) ⁴⁾	3.40(5)
C(18)	H(10) ²⁾	3.48(5)
C(20)	H(2) ⁴⁾	2.89(5)
C(20)	H(19) ²⁾	3.28(5)
C(22)	H(14) ⁶⁾	3.21(6)
H(1)	H(19) ⁵⁾	2.79(7)
H(1)	H(20) ⁵⁾	3.41(6)
H(2)	H(20) ⁸⁾	3.58(7)
H(4)	H(18) ⁸⁾	2.99(6)
H(4)	H(17) ⁸⁾	3.43(6)
H(4)	H(14) ⁸⁾	3.57(7)
H(5)	H(5) ⁴⁾	3.14(8)
H(6)	H(16) ¹⁰⁾	3.25(6)
H(6)	H(16) ²⁾	3.60(7)
H(7)	H(9) ⁵⁾	3.28(6)
H(7)	H(19) ⁵⁾	3.59(8)
H(9)	H(16) ²⁾	3.16(6)
H(10)	H(15) ¹²⁾	2.73(8)

H(11) H(15)¹²⁾ 3.32(6) H(13) H(20)⁵⁾ 3.29(6)

Symmetry operations

(1)	$X+1, Y-1, Z$	(2)	$-X+1/2, Y-1/2, -Z+3/2$
(3)	$-X+3/2, Y-1/2, -Z+3/2$	(4)	$-X+1, -Y, -Z+2$
(5)	$X+1, Y, Z$	(6)	$-X+1, -Y+1, -Z+2$
(7)	$X-1, Y, Z$	(8)	$-X, -Y+1, -Z+2$
(9)	$-X, -Y, -Z+2$	(10)	$X, Y-1, Z$
(11)	$X, Y+1, Z$	(12)	$-X+1/2, Y+1/2, -Z+3/2$

X-ray Structure Report for **8bE**

Experimental

Data Collection

A colorless prismatic crystal of $C_{19}H_{18}O_4NCl$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $28.76 < 2\theta < 29.83^\circ$ corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned} a &= 10.499(3) \text{ \AA} \\ b &= 13.601(3) \text{ \AA} & \beta &= 108.39(2)^\circ \\ c &= 13.298(2) \text{ \AA} \\ V &= 1801.9(7) \text{ \AA}^3 \end{aligned}$$

For $Z = 4$ and F.W. = 359.81, the calculated density is 1.33 g/cm³. The systematic absences of:

$$\begin{aligned} h0l: & l \neq 2n \\ 0k0: & k \neq 2n \end{aligned}$$

uniquely determine the space group to be:

$$P2_1/c (\#14)$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 55.0° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.32° with a take-off angle of 6.0° . Scans of $(1.52 + 0.30 \tan \theta)^\circ$ were made at a speed of $16.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum

of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 4197 reflections which were collected, 4032 were unique ($R_{\text{int}} = 0.022$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 2.3 cm⁻¹. Azimuthal scans of several reflections indicated no need for an absorption correction.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement³ was based on 1080 observed reflections ($I > 2.00\sigma(I)$, $2\theta < 55.01$) and 226 variable parameters and converged (largest parameter shift was 0.06 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.072$$

$$R_w = [(\sum w (|F_o| - |F_c|)^2 / \sum w F_o^2)]^{1/2} = 0.078$$

$$R_w = [(\sum w (F_o^2 - F_c^2)^2 / \sum w (F_o^2)^2)]^{1/2} = 0.000 \text{ for all data}$$

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.072 \text{ for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁴ was 2.33. The weighting scheme was based on counting statistics and included a factor ($p = 0.007$) to downweight the intense reflections.

Plots of $\sum w (|F_o| - |F_c|)^2$ versus $|F_o|$, 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 0.29 and -0.29 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the *teXsan*⁹ crystallographic software package of Molecular Structure Corporation.

References

(1) SAPI91: Fan Hai-Fu (1991). Structure Analysis Programs with Intelligent Control, Rigaku Corporation, Tokyo, Japan.

(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: $\sum w (|F_o| - |F_c|)^2$ where
 $w = 1/[\sigma^2(F_o)] = [\sigma_c^2(F_o) + p^2 F_o^2/4]^{-1}$
 $\sigma_c(F_o) = \text{e.s.d. based on counting statistics}$
 $p = \text{p-factor}$

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

$[\sum w (|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$
 where: N_o = number of observations
 N_v = 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) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) 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).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₉ H ₁₈ O ₄ NCl
Formula Weight	359.81
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	25 (28.8 - 29.8 $^{\circ}$)
Omega Scan Peak Width at Half-height	0.32 $^{\circ}$
Lattice Parameters	a = 10.499(3) Å b = 13.601(3) Å c = 13.298(2) Å β = 108.39(2) $^{\circ}$ V = 1801.9(7) Å ³
Space Group	P2 ₁ /c (#14)
Z value	4
D _{calc}	1.326 g/cm ³
F ₀₀₀	752.00
μ (MoK α)	2.34 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.00,
10.00, 40.00)	
Temperature	23.0 $^{\circ}$ C
Collimator Size	1.0 mm

Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm
Scan Type	ω -2 θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	$(1.52 + 0.30 \tan \theta)^\circ$
2 $\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 4197 Unique: 4032 ($R_{\text{int}} = 0.022$)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SAPI91)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o - F_c)^2$
Least Squares Weights	$1/\sigma^2(F_o) = 4F_o^2/\sigma^2(F_o^2)$
p-factor	0.0070
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I > 2.00\sigma(I)$, $2\theta < 55.01^\circ$)	1080
No. Variables	226
Reflection/Parameter Ratio	4.78
Residuals: R; R _w	0.072 ; 0.078
Residuals: R ₁	0.072
No. of Reflections to calc R ₁	1080
Goodness of Fit Indicator	2.33
Max Shift/Error in Final Cycle	0.060
Maximum peak in Final Diff. Map	0.29 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.29 e ⁻ /Å ³

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

atom	x	y	z	B_{eq}
Cl(1)	0.9616(3)	0.0252(3)	0.1355(3)	5.84(10)
O(2)	2.115(1)	0.2238(8)	0.5369(9)	7.0(3)
O(3)	1.6115(7)	-0.0816(5)	0.2553(6)	3.0(2)
O(4)	1.5359(8)	-0.0982(6)	0.3961(6)	4.1(2)
O(15)	2.043(1)	0.2165(9)	0.6713(9)	7.8(3)
N(1)	2.036(1)	0.1960(8)	0.580(1)	5.0(3)
C(4)	1.7242(10)	0.0041(8)	0.4100(8)	2.6(3)
C(5)	1.818(1)	0.0382(9)	0.3640(8)	3.3(3)
C(7)	1.350(1)	-0.0052(9)	0.1699(9)	3.5(3)
C(8)	1.226(1)	0.0365(9)	0.1588(9)	3.8(3)
C(9)	1.3695(10)	-0.1067(8)	0.1754(8)	2.3(3)
C(10)	1.260(1)	-0.1646(8)	0.1672(9)	3.2(3)
C(11)	1.134(1)	-0.126(1)	0.155(1)	4.2(3)
C(12)	1.119(1)	-0.0258(10)	0.1502(9)	3.6(3)
C(14)	1.613(1)	-0.0635(8)	0.3553(8)	2.9(3)
C(16)	1.534(1)	-0.1400(8)	0.0770(9)	3.0(3)
C(17)	1.532(1)	-0.2522(8)	0.2301(9)	3.5(3)
C(18)	1.729(1)	0.0373(9)	0.5100(9)	3.7(3)
C(19)	1.923(1)	0.1297(8)	0.5178(9)	3.1(3)
C(20)	1.919(1)	0.1000(10)	0.419(1)	4.0(3)
C(21)	1.831(1)	0.0995(9)	0.5639(9)	4.0(3)
C(22)	1.674(1)	-0.1765(10)	0.082(1)	4.6(4)
C(23)	1.428(1)	-0.1911(8)	-0.0092(10)	3.5(3)
C(24)	1.5085(10)	-0.1480(7)	0.1866(8)	2.3(3)
C(25)	1.334(1)	-0.150(1)	-0.086(1)	4.9(4)
H(1)	1.8112	0.0166	0.2923	4.0
H(2)	1.4260	0.0364	0.1747	4.2
H(3)	1.2126	0.1076	0.1577	4.4
H(4)	1.2718	-0.2364	0.1701	3.8
H(5)	1.0566	-0.1699	0.1491	4.9
H(6)	1.6610	0.0148	0.5410	4.5
H(7)	1.9854	0.1233	0.3863	4.5
H(8)	1.8363	0.1229	0.6349	5.2
H(9)	1.5301	-0.0711	0.0576	3.7
H(12)	1.4274	-0.2621	-0.0095	4.6
H(13)	1.3254	-0.0799	-0.0928	6.3
H(14)	1.2667	-0.1885	-0.1393	6.3
H(15)	1.5260	-0.2551	0.3006	4.2
H(16)	1.4663	-0.2968	0.1868	4.2
H(17)	1.6189	-0.2753	0.2325	4.2
H(18)	1.6832	-0.2450	0.1001	5.9
H(19)	1.6900	-0.1687	0.0152	5.9

H(20) 1.7425 -0.1413 0.1345 5.9

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	0.041(2)	0.106(3)	0.078(3)	0.013(2)	0.024(2)	-0.002(2)
O(2)	0.083(8)	0.089(9)	0.094(9)	-0.040(7)	0.026(7)	-0.029(7)
O(3)	0.031(4)	0.043(5)	0.039(5)	-0.006(4)	0.011(4)	-0.001(4)
O(4)	0.058(5)	0.065(6)	0.038(5)	-0.015(5)	0.023(4)	0.005(5)
O(15)	0.106(9)	0.105(10)	0.068(7)	-0.042(7)	0.005(7)	-0.021(7)
N(1)	0.065(8)	0.052(8)	0.061(9)	0.003(7)	0.001(7)	0.014(8)
C(4)	0.031(6)	0.030(7)	0.034(6)	0.007(5)	0.004(5)	-0.001(6)
C(5)	0.035(6)	0.054(9)	0.036(8)	0.005(7)	0.008(6)	-0.001(6)
C(7)	0.032(6)	0.040(8)	0.061(8)	-0.007(6)	0.016(6)	-0.004(7)
C(8)	0.047(7)	0.036(8)	0.061(9)	-0.007(7)	0.017(6)	0.001(7)
C(9)	0.038(6)	0.022(7)	0.025(6)	-0.012(5)	0.008(5)	-0.007(5)
C(10)	0.060(8)	0.026(7)	0.040(8)	-0.012(6)	0.021(6)	-0.003(6)
C(11)	0.034(7)	0.08(1)	0.053(9)	-0.007(7)	0.017(6)	-0.005(8)
C(12)	0.033(7)	0.056(9)	0.051(8)	0.010(7)	0.019(6)	0.003(7)
C(14)	0.046(7)	0.038(8)	0.025(7)	0.008(6)	0.009(6)	0.004(6)
C(16)	0.044(7)	0.041(8)	0.033(7)	-0.010(6)	0.016(6)	-0.004(6)
C(17)	0.053(7)	0.031(7)	0.043(8)	0.007(6)	0.007(6)	0.008(6)
C(18)	0.047(7)	0.049(8)	0.046(8)	-0.013(7)	0.018(6)	-0.004(7)
C(19)	0.032(6)	0.025(7)	0.045(8)	-0.010(6)	-0.008(6)	-0.009(6)
C(20)	0.039(7)	0.061(10)	0.054(9)	0.001(7)	0.017(7)	0.014(8)
C(21)	0.068(9)	0.046(9)	0.042(8)	0.006(8)	0.021(7)	-0.002(7)
C(22)	0.055(8)	0.07(1)	0.064(10)	0.007(8)	0.036(7)	-0.013(8)
C(23)	0.049(7)	0.045(8)	0.043(8)	-0.009(7)	0.022(6)	-0.022(7)
C(24)	0.029(6)	0.021(7)	0.040(7)	0.005(5)	0.013(5)	-0.010(6)
C(25)	0.051(8)	0.08(1)	0.051(9)	-0.020(8)	0.007(7)	-0.020(8)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Cl(1)	C(12)	1.75(1)	O(2)	N(1)	1.21(1)
O(3)	C(14)	1.35(1)	O(3)	C(24)	1.48(1)
O(4)	C(14)	1.20(1)	O(15)	N(1)	1.23(1)
N(1)	C(19)	1.51(1)	C(4)	C(5)	1.39(1)
C(4)	C(14)	1.49(1)	C(4)	C(18)	1.39(1)
C(5)	C(20)	1.37(2)	C(5)	H(1)	0.98
C(7)	C(8)	1.39(1)	C(7)	C(9)	1.39(1)
C(7)	H(2)	0.96	C(8)	C(12)	1.38(1)
C(8)	H(3)	0.98	C(9)	C(10)	1.37(1)
C(9)	C(24)	1.53(1)	C(10)	C(11)	1.39(2)
C(10)	H(4)	0.98	C(11)	C(12)	1.37(2)
C(11)	H(5)	0.99	C(16)	C(22)	1.53(1)
C(16)	C(23)	1.49(1)	C(16)	C(24)	1.56(1)
C(16)	H(9)	0.97	C(17)	C(24)	1.52(1)
C(17)	H(15)	0.96	C(17)	H(16)	0.96
C(17)	H(17)	0.95	C(18)	C(21)	1.38(2)
C(18)	H(6)	0.98	C(19)	C(20)	1.36(2)
C(19)	C(21)	1.36(2)	C(20)	H(7)	0.98
C(21)	H(8)	0.98	C(22)	H(18)	0.96
C(22)	H(19)	0.96	C(22)	H(20)	0.96
C(23)	C(25)	1.29(2)	C(23)	H(12)	0.97
C(25)	H(13)	0.97	C(25)	H(14)	0.98

Table 4. Bond Angles($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(14)	O(3)	C(24)	119.8(8)	O(2)	N(1)	O(15)	124(1)
O(2)	N(1)	C(19)	117(1)	O(15)	N(1)	C(19)	117(1)
C(5)	C(4)	C(14)	123.2(10)	C(5)	C(4)	C(18)	119(1)
C(14)	C(4)	C(18)	116.9(10)	C(4)	C(5)	C(20)	120.4(10)
C(4)	C(5)	H(1)	118.8	C(20)	C(5)	H(1)	120.8
C(8)	C(7)	C(9)	121.7(10)	C(8)	C(7)	H(2)	119.8
C(9)	C(7)	H(2)	118.4	C(7)	C(8)	C(12)	117(1)
C(7)	C(8)	H(3)	121.9	C(12)	C(8)	H(3)	120.2
C(7)	C(9)	C(10)	117(1)	C(7)	C(9)	C(24)	119.2(9)
C(10)	C(9)	C(24)	123.3(10)	C(9)	C(10)	C(11)	122(1)
C(9)	C(10)	H(4)	118.5	C(11)	C(10)	H(4)	118.9
C(10)	C(11)	C(12)	117(1)	C(10)	C(11)	H(5)	120.8
C(12)	C(11)	H(5)	121.4	Cl(1)	C(12)	C(8)	118.7(10)

C1(1)	C(12)	C(11)	119.0(9)	C(8)	C(12)	C(11)	122(1)
O(3)	C(14)	O(4)	125(1)	O(3)	C(14)	C(4)	110.9(9)
O(4)	C(14)	C(4)	124.0(10)	C(22)	C(16)	C(23)	110.6(10)
C(22)	C(16)	C(24)	112.4(9)	C(22)	C(16)	H(9)	106.3
C(23)	C(16)	C(24)	112.3(9)	C(23)	C(16)	H(9)	106.7
C(24)	C(16)	H(9)	108.1	C(24)	C(17)	H(15)	110.8
C(24)	C(17)	H(16)	110.9	C(24)	C(17)	H(17)	111.2
H(15)	C(17)	H(16)	107.3	H(15)	C(17)	H(17)	108.4
H(16)	C(17)	H(17)	108.2	C(4)	C(18)	C(21)	118.7(10)
C(4)	C(18)	H(6)	119.6	C(21)	C(18)	H(6)	121.6
N(1)	C(19)	C(20)	118(1)	N(1)	C(19)	C(21)	118(1)
C(20)	C(19)	C(21)	122(1)	C(5)	C(20)	C(19)	118(1)
C(5)	C(20)	H(7)	119.9	C(19)	C(20)	H(7)	121.4
C(18)	C(21)	C(19)	119(1)	C(18)	C(21)	H(8)	120.0
C(19)	C(21)	H(8)	120.1	C(16)	C(22)	H(18)	110.3
C(16)	C(22)	H(19)	112.0	C(16)	C(22)	H(20)	111.1
H(18)	C(22)	H(19)	107.8	H(18)	C(22)	H(20)	107.6
H(19)	C(22)	H(20)	107.7	C(16)	C(23)	C(25)	126(1)
C(16)	C(23)	H(12)	118.1	C(25)	C(23)	H(12)	114.9
O(3)	C(24)	C(9)	109.0(8)	O(3)	C(24)	C(16)	103.1(8)
O(3)	C(24)	C(17)	109.4(8)	C(9)	C(24)	C(16)	108.8(8)
C(9)	C(24)	C(17)	114.7(9)	C(16)	C(24)	C(17)	111.3(8)
C(23)	C(25)	H(13)	121.9	C(23)	C(25)	H(14)	122.9
H(13)	C(25)	H(14)	115.2				

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C1(1)	C(12)	C(8)	C(7)	-179.7(9)	C1(1)	C(12)	C(11)	C(10)	-179.7(9)
O(2)	N(1)	C(19)	C(20)	3(1)	O(2)	N(1)	C(19)	C(21)	-178(1)
O(3)	C(14)	C(4)	C(5)	-3(1)	O(3)	C(14)	C(4)	C(18)	173.4(9)
O(3)	C(24)	C(9)	C(7)	36(1)	O(3)	C(24)	C(9)	C(10)	-
				145.6(10)					
O(3)	C(24)	C(16)	C(22)	62(1)	O(3)	C(24)	C(16)	C(23)	-172.2(9)
O(4)	C(14)	O(3)	C(24)	0(1)	O(4)	C(14)	C(4)	C(5)	175(1)
O(4)	C(14)	C(4)	C(18)	-7(1)	O(15)	N(1)	C(19)	C(20)	-175(1)
O(15)	N(1)	C(19)	C(21)	2(1)	N(1)	C(19)	C(20)	C(5)	178(1)
N(1)	C(19)	C(21)	C(18)	-178(1)	C(4)	C(5)	C(20)	C(19)	-1(1)
C(4)	C(14)	O(3)	C(24)	179.6(8)	C(4)	C(18)	C(21)	C(19)	1(1)
C(5)	C(4)	C(18)	C(21)	-3(1)	C(5)	C(20)	C(19)	C(21)	0(1)
C(7)	C(8)	C(12)	C(11)	1(1)	C(7)	C(9)	C(10)	C(11)	0(1)
C(7)	C(9)	C(24)	C(16)	-75(1)	C(7)	C(9)	C(24)	C(17)	159(1)

C(8)	C(7)	C(9)	C(10)	1(1)	C(8)	C(7)	C(9)	C(24)	179(1)
C(8)	C(12)	C(11)	C(10)	-1(1)	C(9)	C(7)	C(8)	C(12)	-1(1)
C(9)	C(10)	C(11)	C(12)	0(1)	C(9)	C(24)	O(3)	C(14)	59(1)
C(9)	C(24)	C(16)	C(22)	177.9(9)	C(9)	C(24)	C(16)	C(23)	-56(1)
C(10)	C(9)	C(24)	C(16)	102(1)	C(10)	C(9)	C(24)	C(17)	-22(1)
C(11)	C(10)	C(9)	C(24)	-178(1)	C(14)	O(3)	C(24)	C(16)	174.5(8)
C(14)	O(3)	C(24)	C(17)	-67(1)	C(14)	C(4)	C(5)	C(20)	-179(1)
C(14)	C(4)	C(18)	C(21)	179(1)	C(17)	C(24)	C(16)	C(22)	-54(1)
C(17)	C(24)	C(16)	C(23)	70(1)	C(18)	C(4)	C(5)	C(20)	3(1)
C(18)	C(21)	C(19)	C(20)	0(1)	C(22)	C(16)	C(23)	C(25)	-126(1)
C(24)	C(16)	C(23)	C(25)	107(1)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
Cl(1)	H(13) ¹⁾	2.98	Cl(1)	H(1) ²⁾	2.99
Cl(1)	H(20) ²⁾	3.22	Cl(1)	H(14) ¹⁾	3.28
Cl(1)	C(25) ¹⁾	3.42(1)	Cl(1)	H(7) ²⁾	3.53
O(2)	H(19) ³⁾	2.77	O(2)	H(3) ⁴⁾	2.80
O(2)	H(5) ⁵⁾	2.94	O(2)	H(18) ³⁾	3.23
O(2)	C(22) ³⁾	3.38(2)	O(2)	C(4) ⁶⁾	3.49(1)
O(2)	H(17) ⁶⁾	3.50	O(2)	C(14) ⁶⁾	3.52(2)
O(2)	H(20) ³⁾	3.60	O(3)	H(13) ⁷⁾	3.29
O(4)	H(6) ⁸⁾	2.71	O(4)	H(12) ⁹⁾	2.72
O(4)	H(18) ⁹⁾	3.41	O(4)	C(23) ⁹⁾	3.46(1)
O(4)	C(18) ⁸⁾	3.48(1)	O(15)	H(5) ⁸⁾	2.96
O(15)	H(20) ⁶⁾	3.02	O(15)	H(3) ⁴⁾	3.02
O(15)	H(14) ⁵⁾	3.39	O(15)	H(17) ⁶⁾	3.47
O(15)	H(18) ⁶⁾	3.49	O(15)	H(1) ⁶⁾	3.49
O(15)	C(11) ⁸⁾	3.60(2)	N(1)	H(3) ⁴⁾	3.23
N(1)	H(5) ⁵⁾	3.42	N(1)	H(1) ⁶⁾	3.48
N(1)	C(5) ⁶⁾	3.51(2)	C(4)	H(16) ⁵⁾	3.37
C(5)	H(4) ⁵⁾	3.20	C(5)	H(13) ⁷⁾	3.49
C(5)	H(14) ⁷⁾	3.50	C(5)	C(19) ⁶⁾	3.52(1)
C(7)	H(19) ⁷⁾	3.34	C(7)	H(17) ⁵⁾	3.36
C(8)	H(17) ⁵⁾	3.13	C(8)	H(19) ⁷⁾	3.27
C(10)	H(8) ⁸⁾	3.15	C(10)	H(14) ⁹⁾	3.24
C(11)	H(8) ⁸⁾	2.71	C(12)	H(8) ⁸⁾	3.05
C(16)	H(13) ⁷⁾	3.31	C(16)	H(9) ⁷⁾	3.34

C(17)	H(2) ¹⁰	3.12	C(17)	H(3) ¹⁰	3.24
C(18)	H(12) ⁵	3.18	C(18)	H(16) ⁵	3.57
C(19)	H(4) ⁵	3.24	C(19)	C(20) ⁶	3.51(2)
C(19)	H(5) ⁵	3.56	C(20)	H(4) ⁵	2.98
C(20)	H(5) ⁵	3.29	C(20)	C(20) ⁶	3.55(2)
C(21)	H(12) ⁵	3.19	C(21)	H(14) ⁵	3.32
C(21)	H(7) ⁶	3.54	C(22)	H(13) ⁷	3.49
C(23)	H(15) ¹¹	3.10	C(25)	H(15) ¹¹	3.15
C(25)	H(1) ⁷	3.25	C(25)	H(9) ⁷	3.30
C(25)	H(4) ¹¹	3.47	C(25)	H(2) ⁷	3.47
C(25)	H(8) ¹⁰	3.52	H(1)	H(13) ⁷	2.73
H(1)	H(14) ⁷	3.04	H(1)	H(4) ⁵	3.55
H(2)	H(15) ⁵	2.88	H(2)	H(16) ⁵	2.92
H(2)	H(17) ⁵	2.94	H(2)	H(19) ⁷	3.03
H(2)	H(13) ⁷	3.18	H(2)	H(9) ⁷	3.30
H(3)	H(17) ⁵	2.48	H(3)	H(19) ⁷	2.92
H(3)	H(15) ⁵	3.22	H(3)	H(16) ⁵	3.60
H(4)	H(14) ⁹	2.75	H(4)	H(7) ¹⁰	3.20
H(4)	H(8) ⁸	3.50	H(5)	H(8) ⁸	2.81
H(5)	H(7) ¹⁰	2.86	H(5)	H(20) ²	3.26
H(5)	H(14) ⁹	3.55	H(6)	H(12) ⁵	3.16
H(6)	H(6) ⁸	3.23	H(8)	H(14) ⁵	2.79
H(8)	H(12) ⁵	3.16	H(9)	H(9) ⁷	2.42
H(9)	H(13) ⁷	2.51	H(12)	H(15) ¹¹	3.03
H(13)	H(20) ⁷	3.10	H(13)	H(19) ⁷	3.55
H(14)	H(15) ¹¹	3.16	H(14)	H(16) ¹¹	3.58
H(15)	H(19) ⁹	3.01			

Symmetry operations

(1)	-X+2,-Y,-Z	(2)	X-1,Y,Z
(3)	-X+4,Y+1/2,-Z+1/2	(4)	X+1,-Y+1/2,Z+1/2
(5)	-X+3,Y+1/2,-Z+1/2	(6)	-X+4,-Y,-Z+1
(7)	-X+3,-Y,-Z	(8)	-X+3,-Y,-Z+1
(9)	X,-Y-1/2,Z+1/2	(10)	-X+3,Y-1/2,-Z+1/2
(11)	X,-Y-1/2,Z-1/2		

X-ray Structure Report for **7fE**

Experimental

Data Collection

A colorless prismatic crystal of $C_{23}H_{21}O_4N$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $20.98 < 2\theta < 26.49^\circ$ corresponded to a primitive triclinic cell with dimensions:

$$\begin{array}{ll} a = 11.656(2) \text{ \AA} & \alpha = 92.12(2)^\circ \\ b = 12.003(2) \text{ \AA} & \beta = 101.38(2)^\circ \\ c = 7.257(3) \text{ \AA} & \gamma = 91.17(2)^\circ \\ V = 994.2(4) \text{ \AA}^3 \end{array}$$

For $Z = 2$ and F.W. = 375.42, the calculated density is 1.25 g/cm³. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 45.0° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.40° with a take-off angle of 6.0° . Scans of $(1.31 + 0.30 \tan \theta)^\circ$ were made at a speed of $8.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background

counting time was 2:1. The diameter of the incident beam collimator was 6.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 2270 reflections which were collected, 2117 were unique ($R_{\text{int}} = 0.115$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.9 cm⁻¹. Azimuthal scans of several reflections indicated no need for an absorption correction. A correction for secondary extinction was applied (coefficient = 1.01850e-07).

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ was based on 2117 all reflections ($2\theta < 44.96$) and 338 variable parameters and converged (largest parameter shift was 0.17 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (F_o^2 - F_c^2) / \Sigma F_o^2 = 0.205$$

$$R_w = [(\Sigma w (F_o^2 - F_c^2)^2 / \Sigma w (F_o^2)^2)]^{1/2} = 0.178$$

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.047 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁴ was 1.01. The weighting scheme was based on counting statistics and included a factor ($p = 0.050$) to downweight the intense reflections. Plots of $\Sigma w (F_o^2 - F_c^2)^2$ versus F_o^2 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 0.30 and -0.28 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; the values for Δf' and Δf'' were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the texsan⁹ crystallographic software package of Molecular Structure Corporation.

References

(1) SAPI91: Fan Hai-Fu (1991). Structure Analysis Programs with Intelligent Control, Rigaku Corporation, Tokyo, Japan.

(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: $\sum w(F_o^2 - F_c^2)^2$ where
 $w = 1/[\sigma^2(F_o^2)] = [\sigma_c^2(F_o^2) + (p \cdot (\text{Max}(F_o^2, 0) + 2F_c^2)/3)^2]^{-1}$
 $\sigma_c(F_o^2) = \text{e.s.d. based on counting statistics}$
 $p = \text{p-factor}$

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

$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$
 where: N_o = number of observations
 N_v = 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) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).
- (7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (8) 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).
- (9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₃ H ₂₁ O ₄ N
Formula Weight	375.42
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	25 (21.0 - 26.5 $^{\circ}$)
Omega Scan Peak Width at Half-height	0.40 $^{\circ}$
Lattice Parameters	a = 11.656(2) Å b = 12.003(2) Å c = 7.257(3) Å α = 92.12(2) $^{\circ}$ β = 101.38(2) $^{\circ}$ γ = 91.17(2) $^{\circ}$ V = 994.2(4) Å ³
Space Group	P-1 (#2)
Z value	2
D _{calc}	1.254 g/cm ³
F ₀₀₀	396.00
μ (MoK α)	0.86 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.69,
12.30, 44.72)	

Temperature	23.0 °C
Collimator Size	6.0 mm
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm
Scan Type	ω -2 θ
Scan Rate	8.0°/min (in ω) (up to 5 scans)
Scan Width	$(1.31 + 0.30 \tan \theta)^\circ$
2 $\theta_{\max}$	45.0°
No. of Reflections Measured	Total: 2270 Unique: 2117 ($R_{\text{int}} = 0.115$)
Corrections	Lorentz-polarization Secondary Extinction (coefficient: 1.01850e-07)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SAPI91)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/\sigma^2(F_o^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Reflections (All, $2\sigma < 44.96^\circ$)	2117
No. Variables	338
Reflection/Parameter Ratio	6.26
Residuals: R; Rw	0.205 ; 0.178
Residuals: R1	0.047
No. of Reflections to calc R1	590
Goodness of Fit Indicator	1.01
Max Shift/Error in Final Cycle	0.166
Maximum peak in Final Diff. Map	0.30 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.28 e ⁻ /Å ³

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

atom	x	y	z	B_{eq}
O(1)	0.7479(6)	0.2086(5)	0.9070(10)	4.3(2)
O(2)	0.6321(6)	0.1708(6)	1.113(1)	5.8(3)
O(3)	0.1827(7)	0.0569(8)	0.354(1)	9.2(3)
O(4)	0.2959(7)	0.0931(8)	0.162(1)	8.2(3)
N(1)	0.276(1)	0.0832(9)	0.318(2)	5.8(3)
C(2)	0.842(2)	0.451(2)	1.471(2)	8.3(6)
C(3)	0.773(2)	0.530(1)	1.391(3)	8.4(7)
C(4)	0.716(1)	0.526(1)	1.197(2)	5.5(5)
C(8)	0.694(1)	0.4388(10)	0.887(2)	4.7(4)
C(9)	0.7429(10)	0.434(1)	1.082(2)	4.4(4)
C(10)	0.814(1)	0.348(1)	1.165(2)	4.8(4)
C(11)	0.5527(9)	0.1503(8)	0.784(2)	3.2(3)
C(12)	0.448(1)	0.1074(9)	0.813(2)	4.3(4)
C(13)	0.3578(10)	0.0849(9)	0.657(2)	4.8(4)
C(14)	0.375(1)	0.1083(9)	0.481(2)	4.4(4)
C(15)	0.480(1)	0.1488(9)	0.453(2)	4.6(4)
C(16)	0.571(1)	0.1717(9)	0.607(2)	4.2(4)
C(17)	1.058(2)	0.168(1)	0.761(3)	10.7(6)
C(18)	0.961(1)	0.195(1)	0.796(2)	6.7(4)
C(19)	0.9406(10)	0.287(1)	0.933(2)	5.0(4)
C(20)	0.8507(9)	0.2489(9)	1.053(2)	4.2(3)
C(21)	0.647(1)	0.1761(9)	0.953(2)	4.6(4)
C(23)	0.600(1)	0.611(2)	0.939(3)	8.9(7)
C(24)	0.897(1)	0.149(1)	1.168(2)	6.0(4)
C(25)	0.641(2)	0.610(1)	1.113(3)	8.9(7)
C(26)	1.050(1)	0.340(1)	1.060(2)	7.7(5)
C(27)	0.623(1)	0.524(1)	0.814(2)	7.6(6)
C(28)	0.859(1)	0.361(1)	1.355(3)	7.4(6)
H(1)	0.892(10)	0.466(9)	1.64(2)	13.4(2)
H(2)	0.750(8)	0.606(8)	1.47(1)	7.3(3)
H(3)	0.708(7)	0.378(6)	0.82(1)	2.1(4)
H(4)	0.438(5)	0.101(5)	0.936(8)	-0.1(5)
H(5)	0.285(5)	0.066(5)	0.649(9)	0.5(5)
H(6)	0.482(10)	0.156(9)	0.31(2)	10.0(2)
H(7)	0.641(6)	0.198(6)	0.56(1)	2.6(5)
H(8)	0.89(1)	0.15(1)	0.75(2)	21.0(1)
H(9)	0.539(6)	0.651(6)	0.85(1)	2.5(4)
H(10)	0.643(7)	0.671(6)	1.26(1)	5.4(4)
H(11)	0.610(6)	0.500(6)	0.681(9)	1.2(5)
H(12)	0.912(6)	0.309(6)	1.39(1)	1.2(4)
H(13)	0.908(6)	0.354(6)	0.875(10)	2.4(4)
H(14)	0.847(8)	0.135(9)	1.24(1)	6.7(3)

H(15)	0.895(9)	0.092(9)	1.09(1)	7.1(2)
H(16)	0.976(8)	0.170(9)	1.27(1)	8.9(3)
H(17)	1.12(1)	0.29(1)	1.15(2)	16.2(2)
H(18)	1.101(7)	0.358(7)	0.99(1)	3.6(4)
H(19)	1.03(1)	0.38(1)	1.13(2)	15.0(1)
H(20)	1.083(9)	0.245(8)	0.80(1)	7.2(3)
H(21)	1.070(10)	0.115(9)	0.65(1)	8.7(2)

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.045(5)	0.054(5)	0.067(6)	-0.004(4)	0.020(5)	0.003(4)
O(2)	0.070(6)	0.090(7)	0.066(7)	-0.012(4)	0.022(5)	0.017(5)
O(3)	0.048(6)	0.177(10)	0.114(9)	-0.018(6)	-0.001(6)	-0.009(6)
O(4)	0.087(8)	0.163(10)	0.057(7)	-0.002(6)	0.000(6)	0.003(7)
N(1)	0.060(8)	0.085(9)	0.068(9)	0.007(7)	-0.006(10)	0.001(8)
C(2)	0.18(2)	0.08(1)	0.06(1)	-0.04(1)	0.04(1)	-0.03(1)
C(3)	0.15(2)	0.07(1)	0.12(2)	-0.02(1)	0.08(2)	-0.01(1)
C(4)	0.07(1)	0.06(1)	0.08(1)	0.005(9)	0.044(10)	0.00(1)
C(8)	0.051(8)	0.044(9)	0.09(1)	0.019(8)	0.027(8)	0.024(9)
C(9)	0.067(9)	0.07(1)	0.035(9)	-0.005(8)	0.023(8)	0.022(9)
C(10)	0.066(9)	0.049(9)	0.07(1)	-0.004(8)	0.022(8)	0.022(9)
C(11)	0.033(8)	0.044(7)	0.046(9)	0.001(6)	0.009(8)	0.003(6)
C(12)	0.069(9)	0.054(9)	0.043(9)	0.003(7)	0.023(9)	-0.006(7)
C(13)	0.035(8)	0.063(9)	0.08(1)	-0.010(7)	0.02(1)	-0.015(8)
C(14)	0.048(9)	0.041(8)	0.08(1)	0.010(7)	0.032(9)	-0.004(8)
C(15)	0.060(9)	0.044(8)	0.08(1)	0.020(7)	0.02(1)	0.003(8)
C(16)	0.041(8)	0.071(9)	0.053(10)	-0.007(7)	0.024(8)	0.005(7)
C(17)	0.14(1)	0.10(1)	0.19(2)	0.01(1)	0.08(1)	-0.04(1)
C(18)	0.06(1)	0.09(1)	0.13(1)	0.002(8)	0.059(9)	
	0.007(10)					
C(19)	0.045(8)	0.07(1)	0.08(1)	0.008(8)	0.018(8)	0.000(9)
C(20)	0.044(8)	0.042(8)	0.066(9)	0.011(7)	-0.009(7)	0.004(8)
C(21)	0.062(10)	0.043(8)	0.07(1)	-0.005(7)	0.015(10)	0.009(8)
C(23)	0.07(1)	0.12(2)	0.16(2)	0.03(1)	0.04(1)	0.08(2)
C(24)	0.066(9)	0.07(1)	0.08(1)	-0.01(1)	-0.020(9)	0.036(9)
C(25)	0.11(2)	0.06(1)	0.20(2)	0.03(1)	0.09(2)	0.03(2)
C(26)	0.05(1)	0.14(2)	0.10(1)	-0.024(9)	0.010(8)	-0.01(1)
C(27)	0.09(1)	0.09(1)	0.13(2)	-0.01(1)	0.06(1)	0.01(1)
C(28)	0.10(1)	0.10(2)	0.08(1)	-0.003(10)	0.01(1)	0.01(1)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(20)	1.49(1)	O(1)	C(21)	1.34(1)
O(2)	C(21)	1.21(1)	O(3)	N(1)	1.20(1)
O(4)	N(1)	1.21(1)	N(1)	C(14)	1.50(1)
C(2)	C(3)	1.34(2)	C(2)	C(28)	1.39(2)
C(2)	H(1)	1.3(1)	C(3)	C(4)	1.43(2)
C(3)	H(2)	1.11(9)	C(4)	C(9)	1.43(1)
C(4)	C(25)	1.42(2)	C(8)	C(9)	1.42(1)
C(8)	C(27)	1.38(2)	C(8)	H(3)	0.92(7)
C(9)	C(10)	1.42(1)	C(10)	C(20)	1.53(1)
C(10)	C(28)	1.38(2)	C(11)	C(12)	1.37(1)
C(11)	C(16)	1.38(1)	C(11)	C(21)	1.49(1)
C(12)	C(13)	1.40(1)	C(12)	H(4)	0.93(6)
C(13)	C(14)	1.37(1)	C(13)	H(5)	0.86(6)
C(14)	C(15)	1.36(1)	C(15)	C(16)	1.40(1)
C(15)	H(6)	1.0(1)	C(16)	H(7)	1.00(7)
C(17)	C(18)	1.26(2)	C(17)	H(20)	0.97(10)
C(17)	H(21)	1.01(10)	C(18)	C(19)	1.51(2)
C(18)	H(8)	1.0(2)	C(19)	C(20)	1.56(1)
C(19)	C(26)	1.53(2)	C(19)	H(13)	0.97(6)
C(20)	C(24)	1.53(2)	C(23)	C(25)	1.26(2)
C(23)	C(27)	1.42(2)	C(23)	H(9)	0.99(6)
C(24)	H(14)	0.86(8)	C(24)	H(15)	0.89(10)
C(24)	H(16)	1.08(9)	C(25)	H(10)	1.26(8)
C(26)	H(17)	1.1(1)	C(26)	H(18)	0.90(7)
C(26)	H(19)	0.8(1)	C(27)	H(11)	0.98(6)
C(28)	H(12)	0.90(6)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
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C(20)	O(1)	C(21)	121.6(9)	O(3)	N(1)	O(4)	125(1)
O(3)	N(1)	C(14)	117(1)	O(4)	N(1)	C(14)	116(1)
C(3)	C(2)	C(28)	116(1)	C(3)	C(2)	H(1)	118(5)
C(28)	C(2)	H(1)	125(5)	C(2)	C(3)	C(4)	123(1)
C(2)	C(3)	H(2)	124(5)	C(4)	C(3)	H(2)	111(5)
C(3)	C(4)	C(9)	116(1)	C(3)	C(4)	C(25)	123(1)
C(9)	C(4)	C(25)	119(1)	C(9)	C(8)	C(27)	122(1)
C(9)	C(8)	H(3)	114(5)	C(27)	C(8)	H(3)	122(5)
C(4)	C(9)	C(8)	115(1)	C(4)	C(9)	C(10)	120(1)
C(8)	C(9)	C(10)	124(1)	C(9)	C(10)	C(20)	123(1)
C(9)	C(10)	C(28)	116(1)	C(20)	C(10)	C(28)	119(1)
C(12)	C(11)	C(16)	121(1)	C(12)	C(11)	C(21)	117(1)
C(16)	C(11)	C(21)	120(1)	C(11)	C(12)	C(13)	118(1)
C(11)	C(12)	H(4)	117(4)	C(13)	C(12)	H(4)	123(4)
C(12)	C(13)	C(14)	119(1)	C(12)	C(13)	H(5)	131(5)
C(14)	C(13)	H(5)	108(4)	N(1)	C(14)	C(13)	117(1)
N(1)	C(14)	C(15)	120(1)	C(13)	C(14)	C(15)	121(1)
C(14)	C(15)	C(16)	119(1)	C(14)	C(15)	H(6)	112(6)
C(16)	C(15)	H(6)	127(6)	C(11)	C(16)	C(15)	118(1)
C(11)	C(16)	H(7)	133(4)	C(15)	C(16)	H(7)	107(4)
C(18)	C(17)	H(20)	85(6)	C(18)	C(17)	H(21)	125(7)
H(20)	C(17)	H(21)	133(9)	C(17)	C(18)	C(19)	126(1)
C(17)	C(18)	H(8)	120(11)	C(19)	C(18)	H(8)	112(10)
C(18)	C(19)	C(20)	111.2(10)	C(18)	C(19)	C(26)	116(1)
C(18)	C(19)	H(13)	114(4)	C(20)	C(19)	C(26)	110(1)
C(20)	C(19)	H(13)	104(4)	C(26)	C(19)	H(13)	98(4)
O(1)	C(20)	C(10)	108.7(9)	O(1)	C(20)	C(19)	103.1(9)
O(1)	C(20)	C(24)	107.6(9)	C(10)	C(20)	C(19)	111.0(9)
C(10)	C(20)	C(24)	115(1)	C(19)	C(20)	C(24)	110.2(10)
O(1)	C(21)	O(2)	124(1)	O(1)	C(21)	C(11)	112(1)
O(2)	C(21)	C(11)	123(1)	C(25)	C(23)	C(27)	121(2)
C(25)	C(23)	H(9)	136(5)	C(27)	C(23)	H(9)	100(5)
C(20)	C(24)	H(14)	106(7)	C(20)	C(24)	H(15)	105(7)
C(20)	C(24)	H(16)	112(5)	H(14)	C(24)	H(15)	108(10)
H(14)	C(24)	H(16)	103(8)	H(15)	C(24)	H(16)	119(9)
C(4)	C(25)	C(23)	123(2)	C(4)	C(25)	H(10)	97(4)
C(23)	C(25)	H(10)	138(4)	C(19)	C(26)	H(17)	122(7)
C(19)	C(26)	H(18)	108(5)	C(19)	C(26)	H(19)	106(12)
H(17)	C(26)	H(18)	90(8)	H(17)	C(26)	H(19)	106(14)
H(18)	C(26)	H(19)	123(14)	C(8)	C(27)	C(23)	118(1)
C(8)	C(27)	H(11)	99(4)	C(23)	C(27)	H(11)	142(4)
C(2)	C(28)	C(10)	126(1)	C(2)	C(28)	H(12)	121(6)
C(10)	C(28)	H(12)	111(6)				

Table 5. Torsion Angles($^{\circ}$)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(20)	C(10)	C(9)	-42(1)	O(1)	C(20)	C(10)	C(28)	143(1)
O(1)	C(20)	C(19)	C(18)	-53(1)	O(1)	C(20)	C(19)	C(26)	175(1)
O(1)	C(21)	C(11)	C(12)	174.8(9)	O(1)	C(21)	C(11)	C(16)	-6(1)
O(2)	C(21)	O(1)	C(20)	-4(1)	O(2)	C(21)	C(11)	C(12)	-6(1)
O(2)	C(21)	C(11)	C(16)	171(1)	O(3)	N(1)	C(14)	C(13)	8(1)
O(3)	N(1)	C(14)	C(15)	-173(1)	O(4)	N(1)	C(14)	C(13)	-172(1)
O(4)	N(1)	C(14)	C(15)	5(1)	N(1)	C(14)	C(13)	C(12)	-179(1)
N(1)	C(14)	C(15)	C(16)	179(1)	C(2)	C(3)	C(4)	C(9)	-3(2)
C(2)	C(3)	C(4)	C(25)	179(1)	C(2)	C(28)	C(10)	C(9)	0(1)
C(2)	C(28)	C(10)	C(20)	173(1)	C(3)	C(2)	C(28)	C(10)	2(2)
C(3)	C(4)	C(9)	C(8)	-174(1)	C(3)	C(4)	C(9)	C(10)	5(1)
C(3)	C(4)	C(25)	C(23)	173(1)	C(4)	C(3)	C(2)	C(28)	0(2)
C(4)	C(9)	C(8)	C(27)	-1(1)	C(4)	C(9)	C(10)	C(20)	-
				177.5(10)	C(4)	C(25)	C(23)	C(27)	2(2)
C(4)	C(9)	C(10)	C(28)	-3(1)	C(8)	C(9)	C(10)	C(20)	2(1)
C(8)	C(9)	C(4)	C(25)	2(1)	C(8)	C(27)	C(23)	C(25)	-1(2)
C(8)	C(9)	C(10)	C(28)	176(1)	C(9)	C(8)	C(27)	C(23)	1(1)
C(9)	C(4)	C(25)	C(23)	-3(2)	C(9)	C(10)	C(20)	C(24)	-
C(9)	C(10)	C(20)	C(19)	70(1)					
				163.5(10)	C(10)	C(9)	C(8)	C(27)	178(1)
C(10)	C(9)	C(4)	C(25)	-177(1)	C(10)	C(20)	C(19)	C(18)	-169(1)
C(10)	C(20)	O(1)	C(21)	-56(1)	C(11)	C(12)	C(13)	C(14)	-1(1)
C(10)	C(20)	C(19)	C(26)	59(1)	C(11)	C(21)	O(1)	C(20)	174.1(8)
C(11)	C(16)	C(15)	C(14)	1(1)	C(12)	C(13)	C(14)	C(15)	2(1)
C(12)	C(11)	C(16)	C(15)	0(1)	C(13)	C(12)	C(11)	C(21)	178(1)
C(13)	C(12)	C(11)	C(16)	0(1)	C(15)	C(16)	C(11)	C(21)	-178(1)
C(13)	C(14)	C(15)	C(16)	-2(1)	C(17)	C(18)	C(19)	C(26)	-9(2)
C(17)	C(18)	C(19)	C(20)	-137(1)	C(19)	C(20)	O(1)	C(21)	-174.8(9)
C(18)	C(19)	C(20)	C(24)	61(1)	C(21)	O(1)	C(20)	C(24)	68(1)
C(19)	C(20)	C(10)	C(28)	-103(1)	C(24)	C(20)	C(19)	C(26)	-69(1)
C(24)	C(20)	C(10)	C(28)	22(1)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(2)	H(6) ¹⁾	2.5(1)	O(2)	H(9) ²⁾	2.99(7)

O(2)	H(7) ¹⁾	3.21(7)
O(2)	H(4) ³⁾	3.34(6)
O(2)	C(12) ³⁾	3.54(1)
O(3)	H(8) ⁴⁾	2.6(2)
O(3)	H(21) ⁶⁾	2.8(1)
O(3)	C(18) ⁴⁾	3.46(2)
O(3)	H(21) ⁴⁾	3.6(1)
O(3)	H(15) ⁵⁾	3.6(1)
O(4)	H(17) ⁵⁾	3.2(1)
O(4)	C(12) ⁷⁾	3.37(1)
O(4)	C(11) ⁴⁾	3.44(1)
N(1)	H(8) ⁴⁾	3.3(2)
C(2)	H(13) ¹⁾	3.16(7)
C(2)	H(11) ¹⁾	3.41(7)
C(2)	H(19) ⁸⁾	3.5(2)
C(3)	H(20) ⁹⁾	3.60(9)
C(4)	H(20) ⁹⁾	3.57(10)
C(8)	H(1) ⁷⁾	3.2(1)
C(9)	H(18) ⁹⁾	3.17(8)
C(11)	H(10) ²⁾	3.14(8)
C(12)	H(4) ³⁾	3.30(6)
C(13)	H(10) ²⁾	2.97(8)
C(13)	H(6) ⁴⁾	3.5(1)
C(14)	H(10) ²⁾	3.23(9)
C(14)	C(15) ⁴⁾	3.55(1)
C(15)	H(10) ²⁾	3.47(9)
C(17)	H(5) ¹¹⁾	3.17(6)
C(17)	H(12) ⁷⁾	3.40(7)
C(18)	H(12) ⁷⁾	3.23(7)
C(19)	H(1) ⁷⁾	3.1(1)
C(21)	H(6) ¹⁾	3.6(1)
C(23)	H(6) ¹⁰⁾	3.4(1)
C(25)	H(18) ⁹⁾	3.24(8)
C(25)	C(27) ²⁾	3.59(2)
C(27)	H(2) ⁷⁾	3.34(9)
C(28)	H(1) ⁸⁾	3.5(1)
H(1)	H(19) ⁸⁾	2.4(2)
H(1)	H(17) ⁸⁾	3.3(2)
H(1)	H(11) ¹⁾	3.4(1)
H(1)	H(12) ⁸⁾	3.5(1)
H(2)	H(11) ¹⁾	2.8(1)

O(2)	C(15) ¹⁾	3.33(1)
O(2)	H(5) ³⁾	3.44(7)
O(2)	C(13) ³⁾	3.54(1)
O(3)	H(16) ⁵⁾	2.76(10)
O(3)	H(17) ⁵⁾	3.2(1)
O(3)	H(15) ⁴⁾	3.5(1)
O(3)	C(24) ⁵⁾	3.56(2)
O(4)	H(4) ⁷⁾	2.56(6)
O(4)	H(15) ⁴⁾	3.3(1)
O(4)	C(21) ⁴⁾	3.42(1)
N(1)	H(17) ⁵⁾	3.2(1)
N(1)	C(16) ⁴⁾	3.58(2)
C(2)	H(3) ¹⁾	3.34(7)
C(2)	H(1) ⁸⁾	3.5(1)
C(3)	H(11) ¹⁾	3.12(7)
C(4)	H(18) ⁹⁾	3.06(7)
C(4)	H(9) ²⁾	3.57(7)
C(8)	H(18) ⁹⁾	3.35(8)
C(9)	H(9) ²⁾	3.54(7)
C(12)	H(10) ²⁾	2.91(8)
C(12)	H(6) ⁴⁾	3.4(1)
C(13)	H(21) ⁶⁾	3.4(1)
C(13)	C(15) ⁴⁾	3.57(1)
C(14)	C(16) ⁴⁾	3.49(1)
C(15)	H(9) ¹⁰⁾	3.32(7)
C(16)	H(10) ²⁾	3.43(9)
C(17)	H(15) ¹²⁾	3.4(1)
C(17)	H(16) ⁷⁾	3.51(9)
C(18)	H(1) ⁷⁾	3.5(1)
C(21)	H(9) ²⁾	3.48(7)
C(23)	H(18) ⁹⁾	3.42(8)
C(24)	H(21) ¹²⁾	3.5(1)
C(25)	H(20) ⁹⁾	3.56(10)
C(26)	H(1) ⁸⁾	3.1(1)
C(27)	H(18) ⁹⁾	3.50(8)
H(1)	H(13) ¹⁾	2.2(1)
H(1)	H(3) ¹⁾	2.9(1)
H(1)	H(18) ⁸⁾	3.4(1)
H(1)	H(18) ¹⁾	3.5(2)
H(1)	H(20) ¹⁾	3.6(2)
H(2)	H(17) ⁸⁾	3.1(2)

H(2)	H(20) ⁹⁾	3.5(1)	H(2)	H(19) ⁸⁾	3.5(2)
H(4)	H(6) ¹⁾	2.7(1)	H(4)	H(4) ³⁾	2.9(1)
H(4)	H(10) ²⁾	3.20(10)	H(4)	H(9) ²⁾	3.28(9)
H(5)	H(21) ⁶⁾	2.6(1)	H(5)	H(14) ³⁾	3.1(1)
H(5)	H(10) ²⁾	3.3(1)	H(5)	H(20) ⁶⁾	3.5(1)
H(6)	H(9) ¹⁰⁾	2.7(1)	H(8)	H(12) ⁷⁾	3.3(2)
H(10)	H(20) ⁹⁾	3.4(1)	H(11)	H(11) ¹⁰⁾	3.3(1)
H(12)	H(20) ¹⁾	3.3(1)	H(12)	H(21) ¹⁾	3.4(1)
H(12)	H(13) ¹⁾	3.5(1)	H(13)	H(19) ⁹⁾	3.2(2)
H(13)	H(18) ⁹⁾	3.6(1)	H(14)	H(21) ¹²⁾	3.2(1)
H(14)	H(21) ¹⁾	3.6(1)	H(15)	H(21) ¹²⁾	3.2(1)
H(16)	H(21) ¹⁾	2.9(1)	H(16)	H(21) ¹²⁾	3.5(2)
H(18)	H(19) ⁹⁾	3.5(2)	H(19)	H(19) ⁹⁾	3.4(3)

Symmetry operations

(1)	X, Y, Z+1	(2)	-X+1, -Y+1, -Z+2
(3)	-X+1, -Y, -Z+2	(4)	-X+1, -Y, -Z+1
(5)	X-1, Y, Z-1	(6)	X-1, Y, Z
(7)	X, Y, Z-1	(8)	-X+2, -Y+1, -Z+3
(9)	-X+2, -Y+1, -Z+2	(10)	-X+1, -Y+1, -Z+1
(11)	X+1, Y, Z	(12)	-X+2, -Y, -Z+2

X-ray Structure Report for **17dE**

Experimental

Data Collection

A colorless prismatic crystal of $C_{26}H_{22}O_8N_2$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $28.41 < 2\theta < 29.98^\circ$ corresponded to a primitive triclinic cell with dimensions:

$$\begin{array}{ll} a = 10.421(2) \text{ \AA} & \alpha = 91.75(1)^\circ \\ b = 12.772(2) \text{ \AA} & \beta = 110.754(9)^\circ \\ c = 10.248(1) \text{ \AA} & \gamma = 106.06(1)^\circ \\ V = 1213.2(3) \text{ \AA}^3 \end{array}$$

For $Z = 2$ and F.W. = 490.47, the calculated density is 1.34 g/cm³. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 55.0° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.33° with a take-off angle of 6.0° . Scans of $(1.63 + 0.30 \tan \theta)^\circ$ were made at a speed of $16.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background

counting time was 2:1. The diameter of the incident beam collimator was 6.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 5835 reflections which were collected, 5587 were unique ($R_{\text{int}} = 0.031$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 1.0 cm⁻¹. Azimuthal scans of several reflections indicated no need for an absorption correction.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ was based on 5587 all reflections ($2\theta < 54.99$) and 414 variable parameters and converged (largest parameter shift was 0.03 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (F_o^2 - F_c^2) / \Sigma F_o^2 = 0.114$$

$$R_w = [(\Sigma w (F_o^2 - F_c^2)^2 / \Sigma w (F_o^2)^2)]^{1/2} = 0.156$$

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.044 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁴ was 0.99. The weighting scheme was based on counting statistics and included a factor ($p = 0.050$) to downweight the intense reflections. Plots of $\Sigma w (F_o^2 - F_c^2)^2$ versus F_o^2 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 0.28 and -0.27 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the teXsan⁹ crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(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: $\sum w(F_o^2 - F_c^2)^2$ where
 $w = 1/[\sigma^2(F_o^2)] = [\sigma_c^2(F_o^2) + (p \cdot (\text{Max}(F_o^2, 0) + 2F_c^2)/3)^2]^{-1}$
 $\sigma_c(F_o^2) = \text{e.s.d. based on counting statistics}$
 $p = \text{p-factor}$

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

$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$
 where: N_o = number of observations
 N_v = 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) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).
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- (8) 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).
- (9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₆ H ₂₂ O ₈ N ₂
Formula Weight	490.47
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	25 (28.4 - 30.0 $^{\circ}$)
Omega Scan Peak Width at Half-height	0.33 $^{\circ}$
Lattice Parameters	a = 10.421(2) Å b = 12.772(2) Å c = 10.248(1) Å α = 91.75(1) $^{\circ}$ β = 110.754(9) $^{\circ}$ γ = 106.06(1) $^{\circ}$ V = 1213.2(3) Å ³
Space Group	P-1 (#2)
Z value	2
D _{calc}	1.343 g/cm ³
F ₀₀₀	512.00
μ (MoK α)	1.01 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.69,
12.30, 44.72)	

Temperature	23.0 °C
Collimator Size	6.0 mm
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm
Scan Type	ω -2 θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	(1.63 + 0.30 tan θ)°
2 $\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 5835 Unique: 5587 ($R_{\text{int}} = 0.031$)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/\sigma^2(F_o^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Reflections (All, $2\sigma < 54.99^\circ$)	5587
No. Variables	414
Reflection/Parameter Ratio	13.50
Residuals: R; R_w	0.114 ; 0.156
Residuals: R_1	0.044
No. of Reflections to calc R_1	1716
Goodness of Fit Indicator	0.99
Max Shift/Error in Final Cycle	0.033
Maximum peak in Final Diff. Map	0.28 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.27 e ⁻ /Å ³

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

atom	x	y	z	B_{eq}
O(1)	0.6704(2)	0.2924(2)	0.3152(2)	4.70(6)
O(2)	0.8746(2)	0.1376(2)	0.5527(2)	5.50(7)
O(4)	1.0191(5)	-0.2247(3)	1.0215(4)	11.4(1)
O(5)	0.6869(3)	0.1966(2)	0.1362(3)	8.72(10)
O(6)	0.6349(3)	0.6366(3)	-0.2287(3)	9.0(1)
O(9)	1.2385(4)	-0.1187(3)	1.1178(3)	9.6(1)
O(10)	0.6802(4)	0.7373(3)	-0.0394(4)	10.3(1)
O(11)	1.1072(3)	0.2047(2)	0.5898(3)	8.45(9)
N(3)	0.6636(4)	0.6502(3)	-0.1042(4)	6.5(1)
N(4)	1.1178(6)	-0.1450(4)	1.0268(4)	7.7(1)
C(1)	0.6462(4)	0.4562(4)	-0.0995(4)	5.6(1)
C(2)	0.9223(5)	-0.0185(3)	0.7287(4)	5.5(1)
C(3)	1.0872(6)	-0.0755(3)	0.9133(4)	5.7(1)
C(5)	1.0115(4)	0.1412(3)	0.6133(4)	5.4(1)
C(6)	0.6481(4)	0.3666(3)	-0.0277(4)	5.4(1)
C(7)	0.6913(4)	0.2105(3)	0.4138(3)	4.62(9)
C(8)	0.6990(4)	0.5702(3)	0.1158(4)	5.12(10)
C(9)	0.6727(3)	0.3774(3)	0.1152(3)	4.31(9)
C(12)	0.8475(4)	0.2153(3)	0.4529(4)	5.3(1)
C(13)	0.7195(5)	0.4615(4)	0.5456(4)	5.5(1)
C(14)	1.0342(4)	0.0612(3)	0.7149(3)	4.80(10)
C(15)	0.6996(4)	0.4793(3)	0.1862(4)	4.73(9)
C(17)	0.5858(5)	0.0976(3)	0.3468(5)	6.2(1)
C(18)	0.5071(4)	0.2405(3)	0.5096(4)	5.5(1)
C(19)	0.4393(5)	0.1946(4)	0.5884(5)	6.4(1)
C(20)	1.1749(5)	0.0700(3)	0.7997(4)	6.2(1)
C(21)	0.8045(7)	0.5672(4)	0.6065(6)	7.3(2)
C(22)	1.2021(6)	0.0016(4)	0.9026(4)	6.5(1)
C(23)	0.9481(5)	-0.0889(3)	0.8286(4)	6.0(1)
C(24)	0.8754(5)	0.3936(4)	0.7297(4)	5.9(1)
C(25)	0.9566(5)	0.5017(6)	0.7922(5)	7.9(2)
C(26)	0.9224(7)	0.5865(5)	0.7277(7)	8.4(2)
C(27)	0.6705(3)	0.5553(3)	-0.0262(4)	4.88(10)
C(28)	0.7555(4)	0.3725(3)	0.6053(4)	4.39(9)
C(29)	0.6759(4)	0.2779(3)	0.1873(4)	5.6(1)
C(30)	0.6637(4)	0.2559(3)	0.5401(3)	4.54(9)
H(1)	0.631(3)	0.453(2)	-0.199(3)	6.0(7)
H(2)	0.824(3)	-0.028(3)	0.671(3)	5.9(7)
H(3)	0.632(3)	0.294(3)	-0.074(3)	6.1(7)
H(4)	0.713(4)	0.641(3)	0.161(3)	6.9(7)
H(5)	0.632(3)	0.451(2)	0.465(3)	4.2(6)
H(6)	0.722(3)	0.490(2)	0.287(3)	4.3(6)

H(7)	0.457(4)	0.268(3)	0.426(4)	7.2(7)
H(8)	1.251(4)	0.133(3)	0.798(3)	6.1(7)
H(9)	0.784(4)	0.622(3)	0.560(4)	6.4(7)
H(10)	1.299(4)	0.013(3)	0.961(4)	7.7(7)
H(11)	0.873(4)	-0.146(3)	0.841(4)	8.0(7)
H(12)	0.894(4)	0.332(3)	0.763(4)	7.3(7)
H(13)	1.041(4)	0.510(3)	0.880(4)	9.1(7)
H(14)	0.981(5)	0.660(4)	0.772(5)	11.1(6)
H(15)	0.686(3)	0.206(2)	0.602(3)	4.6(6)
H(16)	0.915(3)	0.291(3)	0.502(3)	6.0(7)
H(17)	0.870(3)	0.193(2)	0.373(3)	5.5(7)
H(18)	0.485(4)	0.102(3)	0.308(4)	9.1(7)
H(19)	0.609(4)	0.059(3)	0.282(4)	7.7(7)
H(20)	0.592(5)	0.056(3)	0.426(4)	10.3(6)
H(21)	0.492(4)	0.166(3)	0.669(4)	7.0(7)
H(22)	0.339(4)	0.188(3)	0.566(3)	7.3(7)

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.082(2)	0.062(1)	0.057(1)	0.041(1)	0.037(1)	0.029(1)
O(2)	0.077(2)	0.072(2)	0.086(2)	0.045(1)	0.042(1)	0.047(1)
O(4)	0.213(4)	0.093(3)	0.107(3)	0.049(3)	0.034(3)	0.057(2)
O(5)	0.205(3)	0.094(2)	0.087(2)	0.098(2)	0.078(2)	0.033(2)
O(6)	0.148(3)	0.162(3)	0.083(2)	0.094(2)	0.065(2)	0.076(2)
O(9)	0.164(3)	0.153(3)	0.075(2)	0.102(3)	0.033(2)	0.045(2)
O(10)	0.181(3)	0.083(2)	0.115(3)	0.027(2)	0.051(2)	0.048(2)
O(11)	0.088(2)	0.133(3)	0.147(3)	0.061(2)	0.074(2)	0.092(2)
N(3)	0.082(2)	0.098(3)	0.085(3)	0.036(2)	0.043(2)	0.047(3)
N(4)	0.165(5)	0.087(3)	0.068(3)	0.077(3)	0.044(3)	0.032(3)
C(1)	0.077(3)	0.105(3)	0.053(2)	0.050(3)	0.033(2)	0.035(3)
C(2)	0.086(3)	0.067(3)	0.062(2)	0.038(3)	0.024(2)	0.021(2)
C(3)	0.117(4)	0.062(3)	0.057(2)	0.054(3)	0.035(3)	0.017(2)
C(5)	0.078(3)	0.079(3)	0.083(3)	0.050(2)	0.048(2)	0.041(2)
C(6)	0.087(3)	0.080(3)	0.055(2)	0.042(2)	0.033(2)	0.017(2)
C(7)	0.077(3)	0.053(2)	0.062(2)	0.030(2)	0.038(2)	0.030(2)
C(8)	0.067(2)	0.067(3)	0.066(3)	0.021(2)	0.030(2)	0.024(2)
C(9)	0.064(2)	0.066(2)	0.047(2)	0.033(2)	0.027(2)	0.020(2)
C(12)	0.079(3)	0.078(3)	0.077(3)	0.049(2)	0.045(2)	0.044(2)
C(13)	0.097(3)	0.070(3)	0.061(2)	0.039(3)	0.042(2)	0.023(2)

C(14)	0.079(3)	0.060(2)	0.066(2)	0.040(2)	0.039(2)	0.022(2)
C(15)	0.069(3)	0.075(3)	0.048(2)	0.032(2)	0.029(2)	0.020(2)
C(17)	0.094(3)	0.058(3)	0.091(3)	0.024(3)	0.045(3)	0.013(2)
C(18)	0.069(3)	0.080(3)	0.072(3)	0.031(2)	0.035(2)	0.026(2)
C(19)	0.071(3)	0.086(3)	0.094(3)	0.020(3)	0.043(3)	0.018(3)
C(20)	0.090(3)	0.074(3)	0.095(3)	0.047(3)	0.043(3)	0.033(3)
C(21)	0.146(5)	0.072(4)	0.093(4)	0.037(4)	0.078(4)	0.027(3)
C(22)	0.098(4)	0.095(3)	0.069(3)	0.063(3)	0.024(3)	0.018(3)
C(23)	0.106(4)	0.059(3)	0.071(3)	0.036(3)	0.034(3)	0.024(2)
C(24)	0.060(3)	0.100(4)	0.073(3)	0.027(3)	0.034(2)	0.025(3)
C(25)	0.066(3)	0.135(5)	0.085(4)	0.004(4)	0.033(3)	-0.003(4)
C(26)	0.118(5)	0.097(4)	0.105(5)	0.001(4)	0.071(4)	-0.008(4)
C(27)	0.058(2)	0.083(3)	0.062(2)	0.033(2)	0.032(2)	0.040(2)
C(28)	0.062(2)	0.066(2)	0.056(2)	0.024(2)	0.037(2)	0.023(2)
C(29)	0.101(3)	0.082(3)	0.064(2)	0.058(2)	0.045(2)	0.029(2)
C(30)	0.063(2)	0.066(3)	0.062(2)	0.034(2)	0.033(2)	0.037(2)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(7)	1.482(3)	O(1)	C(29)	1.342(4)
O(2)	C(5)	1.326(4)	O(2)	C(12)	1.456(4)
O(4)	N(4)	1.214(5)	O(5)	C(29)	1.200(4)
O(6)	N(3)	1.198(4)	O(9)	N(4)	1.217(5)
O(10)	N(3)	1.214(4)	O(11)	C(5)	1.202(4)
N(3)	C(27)	1.477(4)	N(4)	C(3)	1.482(5)
C(1)	C(6)	1.380(5)	C(1)	C(27)	1.364(5)
C(1)	H(1)	0.97(3)	C(2)	C(14)	1.372(5)
C(2)	C(23)	1.387(5)	C(2)	H(2)	0.96(3)
C(3)	C(22)	1.365(5)	C(3)	C(23)	1.359(5)
C(5)	C(14)	1.487(4)	C(6)	C(9)	1.390(4)
C(6)	H(3)	0.97(3)	C(7)	C(12)	1.514(4)
C(7)	C(17)	1.512(5)	C(7)	C(30)	1.551(4)
C(8)	C(15)	1.386(4)	C(8)	C(27)	1.374(4)
C(8)	H(4)	0.95(3)	C(9)	C(15)	1.378(4)
C(9)	C(29)	1.491(4)	C(12)	H(16)	1.02(3)
C(12)	H(17)	0.98(3)	C(13)	C(21)	1.376(6)

C(13)	C(28)	1.387(4)	C(13)	H(5)	0.96(3)
C(14)	C(20)	1.384(5)	C(15)	H(6)	0.97(3)
C(17)	H(18)	1.00(4)	C(17)	H(19)	0.95(4)
C(17)	H(20)	0.97(4)	C(18)	C(19)	1.303(5)
C(18)	C(30)	1.503(4)	C(18)	H(7)	0.97(3)
C(19)	H(21)	0.96(3)	C(19)	H(22)	0.97(3)
C(20)	C(22)	1.391(5)	C(20)	H(8)	0.97(3)
C(21)	C(26)	1.358(7)	C(21)	H(9)	0.89(3)
C(22)	H(10)	0.94(3)	C(23)	H(11)	0.96(4)
C(24)	C(25)	1.396(6)	C(24)	C(28)	1.387(5)
C(24)	H(12)	0.91(3)	C(25)	C(26)	1.351(7)
C(25)	H(13)	0.99(4)	C(26)	H(14)	0.96(5)
C(28)	C(30)	1.509(4)	C(30)	H(15)	0.93(3)

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(7)	O(1)	C(29)	121.7(2)	C(5)	O(2)	C(12)	115.1(3)
O(6)	N(3)	O(10)	123.2(4)	O(6)	N(3)	C(27)	118.8(4)
O(10)	N(3)	C(27)	117.9(4)	O(4)	N(4)	O(9)	124.7(4)
O(4)	N(4)	C(3)	116.9(5)	O(9)	N(4)	C(3)	118.4(5)
C(6)	C(1)	C(27)	118.3(3)	C(6)	C(1)	H(1)	123(1)
C(27)	C(1)	H(1)	117(1)	C(14)	C(2)	C(23)	120.9(4)
C(14)	C(2)	H(2)	121(1)	C(23)	C(2)	H(2)	117(1)
N(4)	C(3)	C(22)	117.4(4)	N(4)	C(3)	C(23)	119.2(4)
C(22)	C(3)	C(23)	123.3(4)	O(2)	C(5)	O(11)	122.9(3)
O(2)	C(5)	C(14)	113.2(3)	O(11)	C(5)	C(14)	123.8(4)
C(1)	C(6)	C(9)	120.6(4)	C(1)	C(6)	H(3)	121(1)
C(9)	C(6)	H(3)	117(1)	O(1)	C(7)	C(12)	104.5(2)
O(1)	C(7)	C(17)	111.3(3)	O(1)	C(7)	C(30)	103.8(2)
C(12)	C(7)	C(17)	113.5(3)	C(12)	C(7)	C(30)	112.1(3)
C(17)	C(7)	C(30)	111.1(3)	C(15)	C(8)	C(27)	118.0(4)
C(15)	C(8)	H(4)	122(2)	C(27)	C(8)	H(4)	119(2)
C(6)	C(9)	C(15)	119.4(3)	C(6)	C(9)	C(29)	118.0(3)
C(15)	C(9)	C(29)	122.5(3)	O(2)	C(12)	C(7)	106.6(3)
O(2)	C(12)	H(16)	107(1)	O(2)	C(12)	H(17)	106(1)
C(7)	C(12)	H(16)	111(1)	C(7)	C(12)	H(17)	114(1)
H(16)	C(12)	H(17)	109(2)	C(21)	C(13)	C(28)	120.2(4)
C(21)	C(13)	H(5)	118(1)	C(28)	C(13)	H(5)	120(1)
C(2)	C(14)	C(5)	122.7(3)	C(2)	C(14)	C(20)	119.7(3)
C(5)	C(14)	C(20)	117.6(4)	C(8)	C(15)	C(9)	120.6(3)
C(8)	C(15)	H(6)	118(1)	C(9)	C(15)	H(6)	121(1)

C(7)	C(17)	H(18)	110(2)	C(7)	C(17)	H(19)	114(2)
C(7)	C(17)	H(20)	103(2)	H(18)	C(17)	H(19)	113(3)
H(18)	C(17)	H(20)	106(3)	H(19)	C(17)	H(20)	107(3)
C(19)	C(18)	C(30)	124.2(4)	C(19)	C(18)	H(7)	120(2)
C(30)	C(18)	H(7)	115(2)	C(18)	C(19)	H(21)	117(2)
C(18)	C(19)	H(22)	121(2)	H(21)	C(19)	H(22)	120(2)
C(14)	C(20)	C(22)	120.0(4)	C(14)	C(20)	H(8)	118(2)
C(22)	C(20)	H(8)	120(2)	C(13)	C(21)	C(26)	120.9(5)
C(13)	C(21)	H(9)	117(2)	C(26)	C(21)	H(9)	121(2)
C(3)	C(22)	C(20)	118.2(4)	C(3)	C(22)	H(10)	124(2)
C(20)	C(22)	H(10)	117(2)	C(2)	C(23)	C(3)	117.9(4)
C(2)	C(23)	H(11)	123(2)	C(3)	C(23)	H(11)	118(2)
C(25)	C(24)	C(28)	120.4(4)	C(25)	C(24)	H(12)	125(2)
C(28)	C(24)	H(12)	114(2)	C(24)	C(25)	C(26)	119.9(5)
C(24)	C(25)	H(13)	115(2)	C(26)	C(25)	H(13)	124(2)
C(21)	C(26)	C(25)	120.3(5)	C(21)	C(26)	H(14)	121(2)
C(25)	C(26)	H(14)	118(2)	N(3)	C(27)	C(1)	118.2(3)
N(3)	C(27)	C(8)	118.8(4)	C(1)	C(27)	C(8)	123.0(3)
C(13)	C(28)	C(24)	118.1(4)	C(13)	C(28)	C(30)	121.1(4)
C(24)	C(28)	C(30)	120.8(3)	O(1)	C(29)	O(5)	125.1(3)
O(1)	C(29)	C(9)	112.2(3)	O(5)	C(29)	C(9)	122.6(3)
C(7)	C(30)	C(18)	113.5(3)	C(7)	C(30)	C(28)	114.7(3)
C(7)	C(30)	H(15)	100(1)	C(18)	C(30)	C(28)	111.4(3)
C(18)	C(30)	H(15)	104(1)	C(28)	C(30)	H(15)	111(1)

Table 5. Torsion Angles($^{\circ}$)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(7)	C(12)	O(2)	178.8(3)	O(1)	C(7)	C(30)	C(18)	74.4(3)
O(1)	C(7)	C(30)	C(28)	-55.2(3)	O(1)	C(29)	C(9)	C(6)	-167.0(3)
O(1)	C(29)	C(9)	C(15)	16.0(5)	O(2)	C(5)	C(14)	C(2)	8.7(5)
O(2)	C(5)	C(14)	C(20)	-170.2(3)	O(2)	C(12)	C(7)	C(17)	-59.8(4)
O(2)	C(12)	C(7)	C(30)	67.0(4)	O(4)	N(4)	C(3)	C(22)	-168.2(4)
O(4)	N(4)	C(3)	C(23)	13.7(5)	O(5)	C(29)	O(1)	C(7)	5.8(6)
O(5)	C(29)	C(9)	C(6)	15.2(6)	O(5)	C(29)	C(9)	C(15)	-161.8(4)
O(6)	N(3)	C(27)	C(1)	1.1(5)	O(6)	N(3)	C(27)	C(8)	-178.0(3)
O(9)	N(4)	C(3)	C(22)	11.9(5)	O(9)	N(4)	C(3)	C(23)	-166.2(4)
O(10)	N(3)	C(27)	C(1)	177.4(4)	O(10)	N(3)	C(27)	C(8)	-1.7(5)
O(11)	C(5)	O(2)	C(12)	1.0(5)	O(11)	C(5)	C(14)	C(2)	-172.7(4)
O(11)	C(5)	C(14)	C(20)	8.5(5)	N(3)	C(27)	C(1)	C(6)	-177.9(3)
N(3)	C(27)	C(8)	C(15)	177.6(3)	N(4)	C(3)	C(22)	C(20)	-177.9(3)
N(4)	C(3)	C(23)	C(2)	176.4(3)	C(1)	C(6)	C(9)	C(15)	-1.9(5)

C(1)	C(6)	C(9)	C(29)	-179.0(3)	C(1)	C(27)	C(8)	C(15)	-1.5(5)
C(2)	C(14)	C(20)	C(22)	-2.9(5)	C(2)	C(23)	C(3)	C(22)	-1.6(6)
C(3)	C(22)	C(20)	C(14)	2.2(6)	C(3)	C(23)	C(2)	C(14)	0.9(5)
C(5)	O(2)	C(12)	C(7)	-171.4(3)	C(5)	C(14)	C(2)	C(23)	-177.5(3)
C(5)	C(14)	C(20)	C(22)	176.0(3)	C(6)	C(1)	C(27)	C(8)	1.1(5)
C(6)	C(9)	C(15)	C(8)	1.5(5)	C(7)	O(1)	C(29)	C(9)	-171.9(3)
C(7)	C(30)	C(18)	C(19)	127.8(4)	C(7)	C(30)	C(28)	C(13)	81.0(3)
C(7)	C(30)	C(28)	C(24)	-101.4(3)	C(8)	C(15)	C(9)	C(29)	178.4(3)
C(9)	C(6)	C(1)	C(27)	0.6(5)	C(9)	C(15)	C(8)	C(27)	0.2(5)
C(12)	O(2)	C(5)	C(14)	179.6(3)	C(12)	C(7)	O(1)	C(29)	66.8(4)
C(12)	C(7)	C(30)	C(18)	-173.4(3)	C(12)	C(7)	C(30)	C(28)	57.0(3)
C(13)	C(21)	C(26)	C(25)	-1.3(7)	C(13)	C(28)	C(24)	C(25)	0.1(5)
C(13)	C(28)	C(30)	C(18)	-49.6(4)	C(17)	C(7)	O(1)	C(29)	-56.0(4)
C(17)	C(7)	C(30)	C(18)	-45.2(4)	C(17)	C(7)	C(30)	C(28)	-174.8(3)
C(18)	C(30)	C(28)	C(24)	128.0(3)	C(19)	C(18)	C(30)	C(28)	-101.0(4)
C(20)	C(14)	C(2)	C(23)	1.4(5)	C(20)	C(22)	C(3)	C(23)	0.1(6)
C(21)	C(13)	C(28)	C(24)	2.4(5)	C(21)	C(13)	C(28)	C(30)	-180.0(3)
C(21)	C(26)	C(25)	C(24)	3.9(7)	C(25)	C(24)	C(28)	C(30)	-177.5(3)
C(26)	C(21)	C(13)	C(28)	-1.9(6)	C(26)	C(25)	C(24)	C(28)	-3.3(6)
C(29)	O(1)	C(7)	C(30)	-175.5(3)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(6) ¹⁾	3.373(3)	O(4)	H(12) ²⁾	2.65(4)
O(4)	H(14) ³⁾	2.76(5)	O(4)	O(10) ⁴⁾	3.251(6)
O(4)	N(3) ⁴⁾	3.340(6)	O(4)	C(26) ³⁾	3.458(7)
O(4)	C(24) ²⁾	3.468(5)	O(5)	H(10) ⁵⁾	2.89(4)
O(5)	C(22) ⁵⁾	3.129(4)	O(5)	O(9) ⁵⁾	3.174(4)
O(5)	H(14) ⁶⁾	3.21(5)	O(5)	N(4) ⁵⁾	3.232(5)
O(5)	C(3) ⁵⁾	3.317(5)	O(6)	H(7) ¹⁾	2.43(3)
O(6)	H(5) ¹⁾	2.86(3)	O(6)	H(11) ⁷⁾	3.03(4)
O(6)	C(21) ⁸⁾	3.106(5)	O(6)	H(9) ⁸⁾	3.11(3)
O(6)	C(18) ¹⁾	3.389(5)	O(6)	C(26) ⁸⁾	3.403(6)
O(6)	H(6) ¹⁾	3.44(3)	O(6)	H(14) ⁸⁾	3.53(5)
O(6)	C(15) ¹⁾	3.577(4)	O(9)	H(3) ⁵⁾	3.01(3)
O(9)	H(15) ²⁾	3.04(3)	O(9)	H(21) ²⁾	3.11(3)
O(9)	H(2) ²⁾	3.17(3)	O(9)	H(12) ²⁾	3.18(4)
O(9)	H(18) ⁹⁾	3.23(4)	O(9)	C(2) ²⁾	3.417(5)
O(10)	H(8) ⁶⁾	2.68(3)	O(10)	H(11) ⁷⁾	2.82(4)

O(10)	C(20) ⁶⁾	3.053(6)	O(10)	H(10) ⁶⁾	3.19(4)
O(10)	C(22) ⁶⁾	3.290(6)	O(10)	C(9) ¹⁾	3.347(5)
O(10)	C(29) ¹⁾	3.421(5)	O(11)	H(22) ¹⁰⁾	2.57(4)
O(11)	H(4) ⁶⁾	2.86(3)	O(11)	H(9) ⁶⁾	2.93(4)
O(11)	C(19) ¹⁰⁾	3.504(5)	N(3)	H(11) ⁷⁾	3.11(4)
N(3)	C(9) ¹⁾	3.385(4)	N(3)	H(7) ¹⁾	3.40(3)
N(3)	C(15) ¹⁾	3.460(5)	N(4)	H(14) ³⁾	3.17(5)
N(4)	H(12) ²⁾	3.27(4)	N(4)	C(2) ²⁾	3.423(5)
N(4)	H(2) ²⁾	3.52(3)	N(4)	H(3) ⁵⁾	3.54(3)
C(1)	H(13) ⁶⁾	3.14(4)	C(1)	H(4) ¹⁾	3.42(3)
C(1)	C(8) ¹⁾	3.462(5)	C(2)	H(22) ¹¹⁾	3.43(3)
C(3)	H(14) ³⁾	3.35(5)	C(3)	H(17) ⁵⁾	3.48(3)
C(3)	C(23) ²⁾	3.509(5)	C(3)	H(11) ²⁾	3.56(4)
C(5)	H(4) ⁶⁾	3.44(3)	C(5)	H(22) ¹⁰⁾	3.50(4)
C(6)	H(13) ⁶⁾	2.97(4)	C(6)	H(4) ¹⁾	3.49(3)
C(6)	H(21) ⁸⁾	3.54(3)	C(8)	H(13) ⁶⁾	3.14(4)
C(8)	C(27) ¹⁾	3.498(5)	C(9)	H(13) ⁶⁾	2.92(4)
C(9)	C(25) ⁶⁾	3.496(6)	C(9)	H(14) ⁶⁾	3.55(5)
C(12)	H(14) ⁶⁾	3.55(5)	C(13)	H(1) ¹²⁾	3.06(3)
C(15)	H(13) ⁶⁾	2.99(4)	C(15)	C(25) ⁶⁾	3.449(6)
C(15)	C(27) ¹⁾	3.507(4)	C(17)	H(21) ¹¹⁾	3.22(4)
C(18)	H(2) ¹¹⁾	3.56(3)	C(18)	H(1) ¹²⁾	3.58(3)
C(19)	H(20) ¹¹⁾	3.12(4)	C(19)	H(2) ¹¹⁾	3.22(3)
C(19)	H(3) ¹²⁾	3.32(3)	C(19)	H(8) ¹³⁾	3.37(3)
C(19)	H(19) ¹¹⁾	3.52(3)	C(19)	H(1) ¹²⁾	3.53(3)
C(20)	H(19) ⁵⁾	3.44(4)	C(20)	H(4) ⁶⁾	3.51(3)
C(20)	H(22) ¹⁰⁾	3.55(3)	C(20)	H(17) ⁵⁾	3.58(3)
C(21)	H(1) ¹²⁾	3.26(3)	C(21)	H(16) ⁶⁾	3.54(3)
C(22)	H(19) ⁵⁾	3.38(4)	C(22)	H(17) ⁵⁾	3.43(3)
C(22)	H(11) ²⁾	3.58(4)	C(23)	H(14) ³⁾	3.37(5)
C(24)	H(1) ¹²⁾	3.16(3)	C(25)	H(1) ¹²⁾	3.31(3)
C(25)	H(13) ¹⁴⁾	3.35(4)	C(26)	H(1) ¹²⁾	3.39(3)
C(26)	H(17) ⁶⁾	3.48(3)	C(26)	H(16) ⁶⁾	3.53(3)
C(27)	H(13) ⁶⁾	3.20(4)	C(28)	H(1) ¹²⁾	3.04(3)
C(29)	H(14) ⁶⁾	3.31(5)	H(1)	H(5) ⁸⁾	3.45(4)
H(1)	H(21) ⁸⁾	3.58(5)	H(2)	H(22) ¹¹⁾	2.80(5)
H(2)	H(18) ¹¹⁾	3.19(5)	H(2)	H(7) ¹¹⁾	3.43(5)
H(3)	H(21) ⁸⁾	2.73(5)	H(3)	H(13) ⁶⁾	3.46(5)
H(4)	H(8) ⁶⁾	2.80(5)	H(5)	H(5) ¹⁵⁾	3.59(5)
H(6)	H(13) ⁶⁾	3.47(5)	H(7)	H(9) ¹⁵⁾	3.24(5)
H(7)	H(11) ¹¹⁾	3.44(5)	H(8)	H(22) ¹⁰⁾	2.87(5)

H(8)	H(21) ¹⁰⁾	3.17(5)	H(8)	H(19) ⁵⁾	3.40(5)
H(9)	H(22) ¹⁵⁾	3.15(5)	H(9)	H(16) ⁶⁾	3.32(4)
H(9)	H(17) ⁶⁾	3.54(4)	H(10)	H(19) ⁵⁾	3.17(5)
H(10)	H(18) ⁹⁾	3.37(5)	H(10)	H(19) ⁹⁾	3.59(5)
H(11)	H(14) ³⁾	3.15(6)	H(13)	H(13) ¹⁴⁾	2.86(8)
H(14)	H(17) ⁶⁾	2.89(5)	H(14)	H(16) ⁶⁾	3.37(5)
H(18)	H(21) ¹¹⁾	3.50(5)	H(19)	H(21) ¹¹⁾	2.90(5)
H(20)	H(21) ¹¹⁾	2.76(6)	H(20)	H(20) ¹¹⁾	2.95(8)
H(20)	H(22) ¹¹⁾	3.39(5)			

Symmetry operations

(1)	-X+1,-Y+1,-Z	(2)	-X+2,-Y,-Z+2
(3)	X,Y-1,Z	(4)	X,Y-1,Z+1
(5)	-X+2,-Y,-Z+1	(6)	-X+2,-Y+1,-Z+1
(7)	X,Y+1,Z-1	(8)	X,Y,Z-1
(9)	X+1,Y,Z+1	(10)	X+1,Y,Z
(11)	-X+1,-Y,-Z+1	(12)	X,Y,Z+1
(13)	X-1,Y,Z	(14)	-X+2,-Y+1,-Z+2
(15)	-X+1,-Y+1,-Z+1		

X-ray Structure Report for 17e

Experimental

Data Collection

A colorless prism crystal of $C_{17}H_{18}O_2$ having approximate dimensions of 0.25 x 0.25 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-K α radiation.

Indexing was performed from 3 oscillations which were exposed for 5.0 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to an I-centered tetragonal cell (laue class: 4/m) with dimensions:

$$\begin{aligned}a &= 23.703(2) \text{ \AA} \\c &= 10.2725(7) \text{ \AA} \\V &= 5771.2(7) \text{ \AA}^3\end{aligned}$$

For $Z = 16$ and F.W. = 254.33, the calculated density is 1.17 g/cm³. Based on the systematic absences of:

$$\begin{aligned}hkl: & h+k+l \neq 2n \\hk0: & h \neq 2n \\00l: & l \neq 4n\end{aligned}$$

and the successful solution and refinement of the structure, the space group was determined to be:

$$I4_1/a \text{ (#88)}$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ to a maximum 2θ value of 54.9° . A total of 57 images, corresponding to 228.0° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 2.50 minutes per degree. The camera radius

was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 15500 reflections which were collected, 3314 were unique ($R_{\text{int}} = 0.030$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.8 cm^{-1} . A symmetry-related absorption correction using the program ABSCOR¹ was applied which resulted in transmission factors ranging from 0.55 to 0.99. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods² and expanded using Fourier techniques³. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement⁴ was based on 1191 observed reflections ($I > 1.00\sigma(I)$, $2\theta < 54.94$) and 244 variable parameters and converged (largest parameter shift was 0.19 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum (F_o^2 - F_c^2) / \sum F_o^2 = 0.068$$

$$R_w = [(\sum w (F_o^2 - F_c^2)^2 / \sum w (F_o^2)^2)]^{1/2} = 0.078$$

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.036 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁵ was 0.66. The weighting scheme was based on counting statistics and included a factor ($p = 0.050$) to downweight the intense reflections. Plots of $\sum w (F_o^2 - F_c^2)^2$ versus F_o^2 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 0.15 and $-0.17 \text{ e}^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁶. Anomalous dispersion effects were included in Fcalc⁷; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁸. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁹. All calculations were performed using the teXsan¹⁰ crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(3) 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.

(4) Least-Squares:

Function minimized: $\sum w(F_o^2 - F_c^2)^2$ where
 $w = 1/[\sigma^2(F_o^2)] = [\sigma_c^2(F_o^2) + (p \cdot (\text{Max}(F_o^2, 0) + 2F_c^2)/3)^2]^{-1}$
 $\sigma_c(F_o^2) = \text{e.s.d. based on counting statistics}$
 $p = \text{p-factor}$

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

$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$
 where: N_o = number of observations
 N_v = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray

Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) 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).

(10) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₇ H ₁₈ O ₂
Formula Weight	254.33
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.25 X 0.25 X 0.10 mm
Crystal System	tetragonal
Lattice Type	I-centered
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	25365 (3.4 - 55.0 $^{\circ}$)
Indexing Images	3 oscillations at 5.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 23.703(2) Å c = 10.2725(7) Å V = 5771.2(7) Å ³
Space Group	I4 ₁ /a (#88)
Z value	16
D _{calc}	1.171 g/cm ³
F ₀₀₀	2176.00
μ (MoK α)	0.75 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Temperature	23.0 $^{\circ}$ C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.3 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	57 exposures at 2.5 minutes per
degree	

Oscillation Range ($\phi=0.0^\circ, \chi=45.0^\circ$)	ω 130.0 - 194.0 $^\circ$ with 4.0 $^\circ$ step
Oscillation Range ($\phi=180.0^\circ, \chi=45.0^\circ$)	ω 0.0 - 164.0 $^\circ$ with 4.0 $^\circ$ step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9 $^\circ$
No. of Reflections Measured	Total: 15500
	Unique: 3314 ($R_{\text{int}} = 0.030$)
Corrections	Lorentz-polarization
	Absorption
	(trans. factors: 0.5541 - 0.9925)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/\sigma^2(F_o^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I > 1.00\sigma(I)$, $2\theta < 54.94^\circ$)	1191
No. Variables	244
Reflection/Parameter Ratio	4.88
Residuals: R; Rw	0.068 ; 0.078
Residuals: R1	0.036
No. of Reflections to calc R1	830
Goodness of Fit Indicator	0.66
Max Shift/Error in Final Cycle	0.193
Maximum peak in Final Diff. Map	0.15 e $^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	-0.17 e $^-/\text{\AA}^3$

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

atom	x	y	z	B_{eq}
O(1)	0.44883(9)	0.18867(9)	0.4300(2)	4.94(6)
O(2)	0.36301(9)	0.21782(9)	0.2384(2)	5.02(6)
C(1)	0.3982(1)	0.1569(1)	0.4066(4)	4.57(10)
C(2)	0.3799(1)	0.1614(1)	0.2652(3)	3.97(9)
C(3)	0.3264(1)	0.1249(1)	0.2408(4)	4.78(10)
C(4)	0.3041(2)	0.1365(2)	0.1049(5)	7.0(1)
C(5)	0.2887(2)	0.0997(3)	0.0231(6)	9.2(2)
C(6)	0.4276(1)	0.1425(1)	0.1762(3)	4.11(8)
C(7)	0.4520(2)	0.0896(2)	0.1916(4)	5.3(1)
C(8)	0.4954(2)	0.0719(2)	0.1098(5)	6.8(1)
C(9)	0.5161(2)	0.1075(2)	0.0156(5)	7.5(2)
C(10)	0.4928(2)	0.1592(2)	0.0016(5)	8.1(2)
C(11)	0.4492(2)	0.1770(2)	0.0793(4)	6.2(1)
C(12)	0.2815(1)	0.1316(1)	0.3442(4)	4.81(10)
C(13)	0.2797(2)	0.0941(2)	0.4467(5)	6.7(1)
C(14)	0.2398(2)	0.0997(2)	0.5431(5)	8.4(2)
C(15)	0.2008(2)	0.1423(2)	0.5394(5)	7.4(1)
C(16)	0.2018(2)	0.1797(2)	0.4395(5)	6.4(1)
C(17)	0.2417(2)	0.1748(2)	0.3417(5)	5.5(1)
H(1)	0.407(1)	0.118(1)	0.444(4)	7.6(4)
H(2)	0.369(1)	0.172(1)	0.467(3)	3.5(5)
H(3)	0.342(1)	0.086(1)	0.249(3)	3.9(5)
H(4)	0.304(2)	0.174(2)	0.057(5)	13.3(2)
H(5)	0.300(3)	0.064(2)	0.038(7)	17.2(2)
H(6)	0.275(2)	0.110(2)	-0.055(5)	12.5(2)
H(7)	0.437(1)	0.063(1)	0.256(3)	5.9(4)
H(8)	0.509(2)	0.037(1)	0.122(4)	8.3(3)
H(9)	0.549(2)	0.092(2)	-0.033(4)	9.8(3)
H(10)	0.509(2)	0.187(2)	-0.060(4)	9.7(3)
H(11)	0.431(1)	0.214(1)	0.066(3)	7.4(4)
H(12)	0.306(1)	0.063(2)	0.460(4)	8.9(4)
H(13)	0.242(2)	0.074(2)	0.611(4)	9.3(3)
H(14)	0.172(2)	0.146(2)	0.613(4)	11.5(2)
H(15)	0.175(1)	0.212(1)	0.446(4)	7.8(4)
H(16)	0.244(1)	0.200(1)	0.267(3)	4.9(4)
H(17)	0.441(2)	0.224(2)	0.446(5)	12.4(2)
H(18)	0.389(1)	0.240(1)	0.258(3)	4.5(4)

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.047(1)	0.060(1)	0.081(2)	0.005(1)	-0.015(1)	-0.011(1)
O(2)	0.057(1)	0.043(1)	0.091(2)	-0.003(1)	-0.018(1)	0.005(1)
C(1)	0.048(2)	0.060(2)	0.066(3)	0.000(2)	0.001(2)	-0.002(2)
C(2)	0.048(2)	0.043(2)	0.060(3)	-0.001(2)	-0.009(2)	0.006(2)
C(3)	0.056(2)	0.051(2)	0.075(3)	-0.009(2)	-0.010(2)	0.001(2)
C(4)	0.064(3)	0.109(4)	0.092(4)	-0.024(3)	-0.017(3)	-0.021(3)
C(5)	0.080(3)	0.165(6)	0.106(5)	0.022(4)	-0.003(3)	-0.051(5)
C(6)	0.055(2)	0.051(2)	0.050(2)	-0.006(2)	-0.006(2)	0.001(2)
C(7)	0.066(3)	0.059(2)	0.076(3)	0.002(2)	0.007(2)	0.002(2)
C(8)	0.088(3)	0.077(3)	0.093(4)	0.019(3)	-0.001(3)	-0.021(3)
C(9)	0.082(3)	0.127(5)	0.076(4)	0.007(3)	0.015(3)	-0.016(4)
C(10)	0.111(4)	0.114(4)	0.083(4)	-0.001(3)	0.031(3)	0.018(3)
C(11)	0.085(3)	0.080(3)	0.071(3)	0.002(2)	0.008(3)	0.012(3)
C(12)	0.048(2)	0.055(2)	0.081(3)	-0.011(2)	-0.003(2)	0.002(2)
C(13)	0.069(3)	0.066(3)	0.119(4)	0.009(2)	0.019(3)	0.035(3)
C(14)	0.082(3)	0.105(4)	0.130(5)	0.012(3)	0.033(3)	0.057(3)
C(15)	0.068(3)	0.098(3)	0.115(5)	0.006(3)	0.024(3)	0.018(3)
C(16)	0.058(3)	0.074(3)	0.110(4)	0.009(2)	0.004(3)	-0.001(3)
C(17)	0.061(2)	0.060(3)	0.089(3)	-0.004(2)	-0.013(3)	0.013(2)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(4)	1.422(3)	O(2)	C(2)	1.438(4)
C(1)	C(4)	1.522(4)	C(1)	C(9)	1.386(4)
C(1)	C(11)	1.389(4)	C(2)	C(4)	1.518(5)
C(3)	C(4)	1.557(4)	C(3)	C(5)	1.511(5)
C(3)	C(8)	1.517(6)	C(5)	C(6)	1.378(5)
C(5)	C(7)	1.391(4)	C(6)	C(13)	1.376(6)
C(7)	C(12)	1.385(5)	C(8)	C(16)	1.266(6)
C(9)	C(17)	1.373(6)	C(10)	C(11)	1.393(5)
C(10)	C(14)	1.375(6)	C(12)	C(15)	1.356(6)
C(13)	C(15)	1.369(5)	C(14)	C(17)	1.352(6)

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	H(16)	0.84(3)	O(2)	H(14)	0.87(5)
C(2)	H(6)	1.00(3)	C(2)	H(13)	1.02(3)
C(3)	H(18)	0.99(3)	C(6)	H(12)	0.99(4)
C(7)	H(10)	0.97(3)	C(8)	H(9)	1.03(5)
C(9)	H(4)	0.98(3)	C(10)	H(3)	0.89(3)
C(11)	H(5)	0.98(3)	C(12)	H(11)	1.00(3)
C(13)	H(15)	0.93(4)	C(14)	H(2)	1.00(4)
C(15)	H(17)	1.02(4)	C(16)	H(7)	0.90(5)
C(16)	H(8)	0.89(6)	C(17)	H(1)	0.99(4)

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(4)	C(1)	C(9)	122.0(3)	C(4)	C(1)	C(11)	120.5(3)
C(9)	C(1)	C(11)	117.5(3)	O(2)	C(2)	C(4)	111.1(3)
C(4)	C(3)	C(5)	113.7(3)	C(4)	C(3)	C(8)	109.3(3)
C(5)	C(3)	C(8)	112.5(3)	O(1)	C(4)	C(1)	111.8(3)
O(1)	C(4)	C(2)	109.4(3)	O(1)	C(4)	C(3)	105.1(3)
C(1)	C(4)	C(2)	110.0(3)	C(1)	C(4)	C(3)	110.2(3)
C(2)	C(4)	C(3)	110.3(3)	C(3)	C(5)	C(6)	119.4(4)
C(3)	C(5)	C(7)	122.7(4)	C(6)	C(5)	C(7)	117.9(4)
C(5)	C(6)	C(13)	120.6(4)	C(5)	C(7)	C(12)	120.8(4)
C(3)	C(8)	C(16)	125.9(5)	C(1)	C(9)	C(17)	120.9(4)
C(11)	C(10)	C(14)	120.2(4)	C(1)	C(11)	C(10)	120.7(4)
C(7)	C(12)	C(15)	120.4(4)	C(6)	C(13)	C(15)	121.1(4)
C(10)	C(14)	C(17)	119.0(5)	C(12)	C(15)	C(13)	119.3(4)
C(9)	C(17)	C(14)	121.7(5)				

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(4)	O(1)	H(16)	109(1)	C(2)	O(2)	H(14)	110(2)

O(2)	C(2)	H(6)	106(1)	O(2)	C(2)	H(13)	103(1)
C(4)	C(2)	H(6)	111(1)	C(4)	C(2)	H(13)	119(2)
H(6)	C(2)	H(13)	104(2)	C(4)	C(3)	H(18)	100(1)
C(5)	C(3)	H(18)	107(1)	C(8)	C(3)	H(18)	112(1)
C(5)	C(6)	H(12)	124(2)	C(13)	C(6)	H(12)	114(2)
C(5)	C(7)	H(10)	115(1)	C(12)	C(7)	H(10)	123(1)
C(3)	C(8)	H(9)	127(2)	C(16)	C(8)	H(9)	106(3)
C(1)	C(9)	H(4)	118(2)	C(17)	C(9)	H(4)	120(2)
C(11)	C(10)	H(3)	117(2)	C(14)	C(10)	H(3)	122(2)
C(1)	C(11)	H(5)	120(1)	C(10)	C(11)	H(5)	118(1)
C(7)	C(12)	H(11)	123(2)	C(15)	C(12)	H(11)	115(2)
C(6)	C(13)	H(15)	115(2)	C(15)	C(13)	H(15)	123(2)
C(10)	C(14)	H(2)	113(2)	C(17)	C(14)	H(2)	127(2)
C(12)	C(15)	H(17)	120(2)	C(13)	C(15)	H(17)	119(2)
C(8)	C(16)	H(7)	120(3)	C(8)	C(16)	H(8)	116(4)
H(7)	C(16)	H(8)	120(5)	C(9)	C(17)	H(1)	116(2)
C(14)	C(17)	H(1)	121(2)				

Table 7. Torsion Angles($^{\circ}$)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(4)	C(1)	C(9)	-2.0(4)	O(1)	C(4)	C(1)	C(11)	176.7(3)
O(1)	C(4)	C(2)	O(2)	-67.0(3)	O(1)	C(4)	C(3)	C(5)	-72.5(4)
O(1)	C(4)	C(3)	C(8)	54.2(4)	O(2)	C(2)	C(4)	C(1)	56.2(3)
O(2)	C(2)	C(4)	C(3)	177.9(3)	C(1)	C(4)	C(3)	C(5)	166.9(3)
C(1)	C(4)	C(3)	C(8)	-66.3(4)	C(1)	C(9)	C(17)	C(14)	0.5(7)
C(1)	C(11)	C(10)	C(14)	2.4(6)	C(2)	C(4)	C(1)	C(9)	-123.7(4)
C(2)	C(4)	C(1)	C(11)	55.0(4)	C(2)	C(4)	C(3)	C(5)	45.3(4)
C(2)	C(4)	C(3)	C(8)	172.0(3)	C(3)	C(4)	C(1)	C(9)	114.5(4)
C(3)	C(4)	C(1)	C(11)	-66.8(4)	C(3)	C(5)	C(6)	C(13)	179.1(4)
C(3)	C(5)	C(7)	C(12)	-179.2(4)	C(4)	C(1)	C(9)	C(17)	179.0(4)
C(4)	C(1)	C(11)	C(10)	179.6(3)	C(4)	C(3)	C(5)	C(6)	-94.5(4)
C(4)	C(3)	C(5)	C(7)	84.8(4)	C(4)	C(3)	C(8)	C(16)	134.0(5)
C(5)	C(3)	C(8)	C(16)	-98.5(5)	C(5)	C(6)	C(13)	C(15)	0.3(8)
C(5)	C(7)	C(12)	C(15)	-0.2(6)	C(6)	C(5)	C(3)	C(8)	140.5(4)
C(6)	C(5)	C(7)	C(12)	0.1(5)	C(6)	C(13)	C(15)	C(12)	-0.4(8)
C(7)	C(5)	C(3)	C(8)	-40.3(5)	C(7)	C(5)	C(6)	C(13)	-0.2(6)
C(7)	C(12)	C(15)	C(13)	0.3(7)	C(9)	C(1)	C(11)	C(10)	-1.7(6)
C(9)	C(17)	C(14)	C(10)	0.2(8)	C(11)	C(1)	C(9)	C(17)	0.2(6)
C(11)	C(10)	C(14)	C(17)	-1.7(7)					

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(2) ¹⁾	2.797(3)	O(1)	C(12) ²⁾	3.406(5)
O(1)	C(2) ¹⁾	3.512(4)	O(2)	O(2) ¹⁾	2.906(4)
O(2)	O(2) ³⁾	2.906(4)	C(13)	C(16) ⁴⁾	3.489(7)

Symmetry operations

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

X-ray Structure Report for **19**

Experimental

Data Collection

A colorless prism crystal of $C_{17}H_{22}O_2$ having approximate dimensions of 0.35 x 0.30 x 0.30 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-K α radiation.

Indexing was performed from 3 oscillations which were exposed for 2.0 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned}a &= 16.063(2) \text{ \AA} \\b &= 16.375(2) \text{ \AA} \quad \beta = 96.211(4)^\circ \\c &= 5.6664(9) \text{ \AA} \\V &= 1481.7(3) \text{ \AA}^3\end{aligned}$$

For $Z = 4$ and F.W. = 258.36, the calculated density is 1.16 g/cm³. The systematic absences of:

$$\begin{aligned}h0l: & h \neq 2n \\0k0: & k \neq 2n\end{aligned}$$

uniquely determine the space group to be:

$$P2_1/a \text{ (#14)}$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ to a maximum 2θ value of 55.0° . A total of 44 images, corresponding to 220.0° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 1.00 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 7439 reflections which were collected, 3395 were unique ($R_{\text{int}} = 0.017$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm^{-1} . A symmetry-related absorption correction using the program ABSCOR¹ was applied which resulted in transmission factors ranging from 0.77 to 0.98. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods² and expanded using Fourier techniques³. The non-hydrogen atoms were refined anisotropically. The final cycle of full-matrix least-squares refinement⁴ was based on 2209 observed reflections ($I > 2.20\sigma(I)$, $2\theta < 54.96$) and 226 variable parameters and converged (largest parameter shift was 0.64 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (F_o^2 - F_c^2) / \Sigma F_o^2 = 0.181$$

$$R_w = [(\Sigma w (F_o^2 - F_c^2)^2 / \Sigma w (F_o^2)^2)]^{1/2} = 0.146$$

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.065 \quad \text{for } I > 2.2\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁵ was 2.01. The weighting scheme was based on counting statistics and included a factor ($p = 0.050$) to downweight the intense reflections. Plots of $\Sigma w (F_o^2 - F_c^2)^2$ versus F_o^2 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 0.25 and $-0.39 \text{ e}^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁶.

Anomalous dispersion effects were included in F_{calc}^7 ; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁸. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁹. All calculations were performed using the teXsan^{10} crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(3) 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.

(4) Least-Squares:

$$\begin{aligned} \text{Function minimized: } & \sum w(F_o^2 - F_c^2)^2 \text{ where} \\ w = & 1/[\sigma^2(F_o^2)] = [\sigma_c^2(F_o^2) + (p \\ & (\text{Max}(F_o^2, 0) + 2F_c^2)/3)^2]^{-1} \\ \sigma_c(F_o^2) = & \text{e.s.d. based on counting statistics} \\ p = & \text{p-factor} \end{aligned}$$

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

$$\begin{aligned} & [\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2} \\ \text{where: } & N_o = \text{number of observations} \\ & N_v = \text{number of variables} \end{aligned}$$

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

- (7) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).
- (8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (9) 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).
- (10) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₇ H ₂₂ O ₂
Formula Weight	258.36
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.35 X 0.30 X 0.30 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	12047 (4.9 - 54.4 $^{\circ}$)
Indexing Images	3 oscillations at 2.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 16.063(2) Å b = 16.375(2) Å c = 5.6664(9) Å β = 96.211(4) $^{\circ}$ V = 1481.7(3) Å ³
Space Group	P2 ₁ /a (#14)
Z value	4
D _{calc}	1.158 g/cm ³
F ₀₀₀	560.00
μ (MoK α)	0.74 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Temperature	23.0 $^{\circ}$ C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm

Data Images	44 exposures at 1.0 minutes per degree
Oscillation Range ($\phi=0.0^\circ, \chi=45.0^\circ$)	ω 130.0 - 190.0 $^\circ$ with 5.0 $^\circ$ step
Oscillation Range ($\phi=180.0^\circ, \chi=45.0^\circ$)	ω 0.0 - 160.0 $^\circ$ with 5.0 $^\circ$ step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	55.0 $^\circ$
No. of Reflections Measured	Total: 7439 Unique: 3395 ($R_{\text{int}} = 0.017$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.7679 - 0.9781)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/\sigma^2(F_o^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I > 2.20\sigma(I)$, $2\theta < 54.96^\circ$)	2209
No. Variables	226
Reflection/Parameter Ratio	9.77
Residuals: R ; R_w	0.181 ; 0.146
Residuals: R_1	0.065
No. of Reflections to calc R_1	2209
Goodness of Fit Indicator	2.01
Max Shift/Error in Final Cycle	0.641
Maximum peak in Final Diff. Map	0.25 e $^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	-0.39 e $^-/\text{\AA}^3$

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

atom	x	y	z	B_{eq}	
O(1)	0.21529(8)	0.01222(9)	0.3089(2)	3.03(3)	
O(2)	0.13501(9)	-0.00912(10)		-0.0385(3)	3.79(4)
C(1)	0.2095(1)	0.0292(2)	-0.0994(4)	3.85(5)	
C(2)	0.2702(1)	0.0269(1)	0.1295(4)	3.00(4)	
C(3)	0.3349(1)	-0.0443(1)	0.1406(5)	3.65(5)	
C(4)	0.2950(2)	-0.1259(2)	0.0781(6)	4.91(7)	
C(5)	0.3172(2)	-0.1943(2)	0.1871(7)	6.08(9)	
C(6)	0.3848(2)	-0.2006(2)	0.3867(8)	7.5(1)	
C(7)	0.4393(2)	-0.1246(2)	0.4110(9)	6.84(9)	
C(8)	0.3858(2)	-0.0487(2)	0.3813(6)	4.94(6)	
C(9)	0.3151(1)	0.1084(1)	0.1703(4)	3.12(4)	
C(10)	0.3685(2)	0.1359(2)	0.0112(5)	4.26(6)	
C(11)	0.4075(2)	0.2114(2)	0.0404(6)	5.14(7)	
C(12)	0.3933(2)	0.2597(2)	0.2275(6)	5.03(7)	
C(13)	0.3405(2)	0.2340(2)	0.3863(5)	4.83(7)	
C(14)	0.3018(2)	0.1588(2)	0.3580(4)	3.97(6)	
C(15)	0.1302(1)	0.0088(1)	0.2053(4)	3.23(5)	
C(16)	0.0869(2)	-0.0605(2)	0.3147(6)	4.71(7)	
C(17)	0.0874(2)	0.0899(2)	0.2335(6)	4.94(7)	
H(1)	0.195(1)	0.088(1)	-0.154(4)	3.8	
H(2)	0.228(1)	0.000(1)	-0.232(5)	3.8	
H(3)	0.371(1)	-0.030(1)	0.016(4)	3.8	
H(4)	0.255(2)	-0.125(1)	-0.043(5)	3.8	
H(5)	0.290(1)	-0.246(1)	0.137(4)	3.8	
H(6)	0.4208	-0.2464	0.3526	9.0	
H(7)	0.3627	-0.2108	0.5284	9.0	
H(8)	0.477(1)	-0.123(1)	0.271(4)	3.8	
H(9)	0.481(2)	-0.118(1)	0.568(4)	3.8	
H(10)	0.4193	-0.0021	0.4078	6.1	
H(11)	0.3468	-0.0505	0.5023	6.1	
H(12)	0.380(1)	0.102(1)	-0.115(4)	3.8	
H(13)	0.445(1)	0.228(1)	-0.076(5)	3.8	
H(14)	0.414(1)	0.311(1)	0.248(4)	3.8	
H(15)	0.330(1)	0.267(1)	0.515(4)	3.8	
H(16)	0.265(1)	0.141(1)	0.466(4)	3.8	
H(17)	0.087(1)	-0.051(1)	0.477(5)	3.8	
H(18)	0.024(1)	-0.065(1)	0.260(4)	3.8	
H(19)	0.123(1)	-0.112(1)	0.288(4)	3.8	
H(20)	0.119(1)	0.133(2)	0.169(4)	3.8	
H(21)	0.094(1)	0.105(1)	0.398(5)	3.8	
H(22)	0.025(2)	0.085(1)	0.166(4)	3.8	

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.0345(7)	0.0510(9)	0.0296(8)	-0.0031(6)	0.0035(6)	
	0.0022(6)					
O(2)	0.0438(8)	0.064(1)	0.0344(9)	-0.0066(7)	-0.0019(6)	-
	0.0045(7)					
C(1)	0.049(1)	0.068(2)	0.030(1)	-0.004(1)	0.0054(10)	-
	0.001(1)					
C(2)	0.039(1)	0.045(1)	0.030(1)	-0.0014(9)	0.0049(8)	-
	0.0005(9)					
C(3)	0.040(1)	0.042(1)	0.058(2)	0.0008(9)	0.012(1)	-0.006(1)
C(4)	0.057(2)	0.055(2)	0.075(2)	-0.006(1)	0.011(1)	-0.023(2)
C(5)	0.069(2)	0.041(2)	0.124(3)	-0.007(1)	0.026(2)	-0.013(2)
C(6)	0.085(2)	0.054(2)	0.147(4)	0.001(2)	0.016(2)	0.023(2)
C(7)	0.059(2)	0.063(2)	0.134(3)	0.008(1)	-0.004(2)	0.016(2)
C(8)	0.051(1)	0.049(1)	0.083(2)	0.002(1)	-0.012(1)	-0.001(1)
C(9)	0.039(1)	0.044(1)	0.034(1)	0.0014(9)	0.0012(9)	
	0.0049(9)					
C(10)	0.062(1)	0.050(1)	0.054(2)	-0.005(1)	0.021(1)	0.000(1)
C(11)	0.066(2)	0.059(2)	0.073(2)	-0.010(1)	0.023(1)	0.013(1)
C(12)	0.068(2)	0.041(1)	0.081(2)	-0.010(1)	0.006(1)	0.004(1)
C(13)	0.074(2)	0.047(1)	0.063(2)	-0.011(1)	0.011(1)	-0.013(1)
C(14)	0.057(1)	0.051(1)	0.044(1)	-0.004(1)	0.009(1)	-0.004(1)
C(15)	0.037(1)	0.049(1)	0.036(1)	-0.0021(9)	0.0014(9)	-
	0.0018(10)					
C(16)	0.055(1)	0.072(2)	0.052(2)	-0.023(1)	0.008(1)	0.000(1)
C(17)	0.054(2)	0.066(2)	0.067(2)	0.009(1)	0.003(1)	-0.012(1)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
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O(1)	C(2)	1.436(2)	O(1)	C(5)	1.429(2)
O(2)	C(4)	1.426(3)	O(2)	C(5)	1.422(3)
C(1)	C(2)	1.523(3)	C(1)	C(6)	1.385(3)
C(1)	C(7)	1.380(3)	C(2)	C(3)	1.559(3)
C(2)	C(4)	1.537(3)	C(3)	C(11)	1.507(3)
C(3)	C(14)	1.515(4)	C(5)	C(8)	1.500(3)
C(5)	C(12)	1.512(4)	C(6)	C(13)	1.388(4)
C(7)	C(10)	1.382(4)	C(9)	C(10)	1.368(4)
C(9)	C(13)	1.361(4)	C(11)	C(15)	1.310(4)
C(14)	C(16)	1.510(4)	C(15)	C(17)	1.484(5)
C(16)	C(17)	1.519(4)			

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C(3)	H(8)	0.99(2)	C(4)	H(6)	0.96(3)
C(4)	H(7)	1.04(2)	C(6)	H(4)	0.94(2)
C(7)	H(3)	0.94(2)	C(8)	H(13)	1.04(2)
C(8)	H(14)	1.02(2)	C(8)	H(15)	0.93(3)
C(9)	H(2)	0.90(2)	C(10)	H(5)	0.93(2)
C(11)	H(16)	0.89(2)	C(12)	H(10)	0.96(2)
C(12)	H(11)	0.96(3)	C(12)	H(12)	1.04(2)
C(13)	H(1)	0.98(2)	C(14)	H(18)	0.94
C(14)	H(21)	0.98	C(15)	H(17)	0.98(2)
C(16)	H(9)	1.05(2)	C(16)	H(19)	1.05(2)
C(17)	H(20)	0.93	C(17)	H(22)	0.98

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	O(1)	C(5)	110.4(1)	C(4)	O(2)	C(5)	106.2(2)
C(2)	C(1)	C(6)	120.2(2)	C(2)	C(1)	C(7)	122.0(2)
C(6)	C(1)	C(7)	117.8(2)	O(1)	C(2)	C(1)	110.9(2)
O(1)	C(2)	C(3)	107.7(2)	O(1)	C(2)	C(4)	102.7(2)
C(1)	C(2)	C(3)	110.3(2)	C(1)	C(2)	C(4)	110.7(2)
C(3)	C(2)	C(4)	114.3(2)	C(2)	C(3)	C(11)	112.9(2)
C(2)	C(3)	C(14)	111.4(2)	C(11)	C(3)	C(14)	110.1(2)
O(2)	C(4)	C(2)	104.9(2)	O(1)	C(5)	O(2)	104.9(2)

O(1)	C(5)	C(8)	109.0(2)	O(1)	C(5)	C(12)	110.4(2)
O(2)	C(5)	C(8)	108.8(2)	O(2)	C(5)	C(12)	110.9(2)
C(8)	C(5)	C(12)	112.6(2)	C(1)	C(6)	C(13)	121.0(3)
C(1)	C(7)	C(10)	121.1(2)	C(10)	C(9)	C(13)	120.2(3)
C(7)	C(10)	C(9)	120.1(3)	C(3)	C(11)	C(15)	124.1(3)
C(6)	C(13)	C(9)	119.9(3)	C(3)	C(14)	C(16)	112.6(3)
C(11)	C(15)	C(17)	123.9(3)	C(14)	C(16)	C(17)	110.4(2)
C(15)	C(17)	C(16)	112.3(3)				

Table 6. Bond Angles($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(3)	H(8)	104(1)	C(11)	C(3)	H(8)	107(1)
C(14)	C(3)	H(8)	110(1)	O(2)	C(4)	H(6)	107(1)
O(2)	C(4)	H(7)	108(1)	C(2)	C(4)	H(6)	115(1)
C(2)	C(4)	H(7)	112(1)	H(6)	C(4)	H(7)	108(1)
C(1)	C(6)	H(4)	119(1)	C(13)	C(6)	H(4)	119(1)
C(1)	C(7)	H(3)	118(1)	C(10)	C(7)	H(3)	120(1)
C(5)	C(8)	H(13)	105(1)	C(5)	C(8)	H(14)	114(1)
C(5)	C(8)	H(15)	108(1)	H(13)	C(8)	H(14)	116(1)
H(13)	C(8)	H(15)	109(2)	H(14)	C(8)	H(15)	102(1)
C(10)	C(9)	H(2)	116(1)	C(13)	C(9)	H(2)	123(1)
C(7)	C(10)	H(5)	118(1)	C(9)	C(10)	H(5)	121(1)
C(3)	C(11)	H(16)	114(1)	C(15)	C(11)	H(16)	121(1)
C(5)	C(12)	H(10)	109(1)	C(5)	C(12)	H(11)	109(1)
C(5)	C(12)	H(12)	109(1)	H(10)	C(12)	H(11)	100(1)
H(10)	C(12)	H(12)	116(1)	H(11)	C(12)	H(12)	112(1)
C(6)	C(13)	H(1)	117(1)	C(9)	C(13)	H(1)	122(1)
C(3)	C(14)	H(18)	110.5	C(3)	C(14)	H(21)	107.9
C(16)	C(14)	H(18)	110.1	C(16)	C(14)	H(21)	107.3
H(18)	C(14)	H(21)	108.2	C(11)	C(15)	H(17)	120(1)
C(17)	C(15)	H(17)	115(1)	C(14)	C(16)	H(9)	105(1)
C(14)	C(16)	H(19)	107(1)	C(17)	C(16)	H(9)	109(1)
C(17)	C(16)	H(19)	117(1)	H(9)	C(16)	H(19)	105(1)
C(15)	C(17)	H(20)	110.8	C(15)	C(17)	H(22)	107.6
C(16)	C(17)	H(20)	109.9	C(16)	C(17)	H(22)	107.4
H(20)	C(17)	H(22)	108.8				

Table 7. Torsion Angles($^{\circ}$)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(2)	C(1)	C(6)	-177.3(2)	O(1)	C(2)	C(1)	C(7)	-0.2(3)
O(1)	C(2)	C(3)	C(11)	64.5(3)	O(1)	C(2)	C(3)	C(14)	-60.1(2)
O(1)	C(2)	C(4)	O(2)	-18.0(2)	O(1)	C(5)	O(2)	C(4)	-32.9(2)
O(2)	C(4)	C(2)	C(1)	-136.4(2)	O(2)	C(4)	C(2)	C(3)	98.3(2)
O(2)	C(5)	O(1)	C(2)	21.2(2)	C(1)	C(2)	O(1)	C(5)	116.4(2)
C(1)	C(2)	C(3)	C(11)	-174.4(2)	C(1)	C(2)	C(3)	C(14)	61.1(2)
C(1)	C(6)	C(13)	C(9)	-0.2(4)	C(1)	C(7)	C(10)	C(9)	-0.2(4)
C(2)	O(1)	C(5)	C(8)	137.5(2)	C(2)	O(1)	C(5)	C(12)	-98.3(2)
C(2)	C(1)	C(6)	C(13)	177.5(2)	C(2)	C(1)	C(7)	C(10)	-177.3(2)
C(2)	C(3)	C(11)	C(15)	-140.3(3)	C(2)	C(3)	C(14)	C(16)	171.0(2)
C(2)	C(4)	O(2)	C(5)	31.6(2)	C(3)	C(2)	O(1)	C(5)	-122.9(2)
C(3)	C(2)	C(1)	C(6)	63.5(3)	C(3)	C(2)	C(1)	C(7)	-119.4(2)
C(3)	C(11)	C(15)	C(17)	-0.2(5)	C(3)	C(14)	C(16)	C(17)	-60.4(4)
C(4)	O(2)	C(5)	C(8)	-149.3(2)	C(4)	O(2)	C(5)	C(12)	86.3(2)
C(4)	C(2)	O(1)	C(5)	-1.9(2)	C(4)	C(2)	C(1)	C(6)	-64.0(3)
C(4)	C(2)	C(1)	C(7)	113.0(2)	C(4)	C(2)	C(3)	C(11)	-48.9(3)
C(4)	C(2)	C(3)	C(14)	-173.4(2)	C(6)	C(1)	C(7)	C(10)	-0.2(4)
C(6)	C(13)	C(9)	C(10)	-0.2(5)	C(7)	C(1)	C(6)	C(13)	0.3(4)
C(7)	C(10)	C(9)	C(13)	0.3(4)	C(11)	C(3)	C(14)	C(16)	44.9(3)
C(11)	C(15)	C(17)	C(16)	-14.2(5)	C(14)	C(3)	C(11)	C(15)	-15.1(4)
C(14)	C(16)	C(17)	C(15)	43.0(5)					

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	C(4) ¹⁾	3.376(3)			

Symmetry operations

(1) X,Y,Z+1