J-6630-m1

**X-ray crystallographic data for (±)-25:**

Data Collection:

A clear, colorless crystal of molecular formula $C_{18}H_{20}O_3$ with approximate dimensions of 0.13 x 0.25 x 0.48 mm was obtained by recrystallization from acetone at 23 °C by slow evaporation. It was mounted on a glass fiber and placed in a goniometer head on an Enraf-Nonius CAD4 computer controlled kappa axis diffractometer equipped with a graphite monochromator using MoKa radiation ($\lambda = 0.71073$ Å).

Unit cell parameters and an orientation matrix for data collection were obtained by auto-indexing and least-squares refinement of 22 reflections in the range $2° < \Theta < 14°$. The crystal system was monoclinic with cell constants and calculated volume of: a = 11.388(3) Å, b = 10.536(2) Å, c = 13.154(4) Å, ß = 105.99(2)°, V = 1511(1) Å$^3$. From space group required extinctions and subsequent least squares refinement the space group was determined to be $P2_1/c$ (# 14). A total of 3014 unique reflections were collected at a temperature of 25±1 °C using the omega-2theta scan technique to a maximum $2\Theta$ angle of 50.0°.

Data Reduction:

As a check on crystal and electronic stability two representative reflections were measured every 60 minutes. A linear decay correction was applied to the data. Lorentz, polarization, and no absorption corrections were made.

Structure Solution and Refinement:

The structure was solved with SHELX-86[1] using direct methods which revealed the positions of 21 atoms. HYDRO[2] was employed to locate the remaining hydrogens. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they were bonded. Anomalous dispersion effects were included in $F_c$[3]; the values

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[1] Sheldrick, G. M. "SHELXS-86, Program for Crystal Structure Solution," University of Gottingen, 1986.

[2] Fair, C. K. "MolEN, An Interactive Structure Solution Procedure," Enraf-Nonius, Delft, The Netherlands (1990).

[3] Ibers, J. A.; Hamilton, W. C. *Acta Crystallogr.* **1964**, *17*, 781.

for $\Delta f'$ and $\Delta f''$ were those of Cromer.[4] Only 1620 reflections having $F_o$ greater than 3.0 times $\sigma(F_o)$ were used in the refinements. The final cycle of least-squares included 190 variable parameters and converged with a maximum parameter shift of 0.00 times its esd, and unweighted and weighted agreement factors of 0.0746 and 0.0748, respectively. The standard deviation of an observation of unit weight was 0.53. The largest peak in the final difference Fourier has a height of 0.31 e/Å$^3$ with an estimated error based on $\Delta F$[5] of 0.07; 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.

Scattering factors were taken from Cromer and Waber.[10] All calculations were performed on a MicroVAX computer using MolEN.[8]

Table of Crystallographic Data:

| | |
|---|---|
| Formula | $C_{18}H_{20}O_3$ |
| Formula Weight | 284.36 |
| Crystal dimensions, mm | 0.13 x 0.25 x 0.48 |
| Radiation Wavelength, Å | Mo, 0.71073 |
| Temperature, °C | 25 ± 1 |
| Crystal System | monoclinic |
| Space Group | $P2_1/c$ |
| a, Å | 11.338 (3) |
| b, Å | 10.536 (2) |
| c, Å | 13.154 |
| ß, deg | 105.99 (2) |
| V, Å$^3$ | 1511 (1) |
| Z | 4 |

[4]Cromer, D. T.; Waber, J. T. "International Tables for X-Ray Crystallography," Vol. IV, Knyoch, Birmingham, England (1974), Table 2.3.1.

[5]Cruickshank, D. W. J. *Acta. Crystallogr.* **1949**, 2, 154.

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| | |
|---|---|
| Density, g/cm$^3$ | 1.25 |
| Absorption coeff., μ, cm$^{-1}$ | 0.8 |
| Rel. transmission coeff. | 0.000 - 0.000 |
| Scan type | omega-2theta |
| Scan Rate, deg/min | 5.49 |
| Scan Width, deg | 0.9 + 0.350tanΘ |
| hkl ranges, h | -13 to 13 |
| hkl ranges, k | 0 to 12 |
| hkl ranges, l | -15 to 16 |
| 2Θ range, deg | 2.0 - 50.0 |
| Structure solution | Direct methods |
| No. of unique data | 3014 |
| No. of data used in refinement | 1620 |
| Weighting scheme, w | $4F_o^2/[\sigma(F_o)^2]^2$ |
| No. of parameters refined | 190 |
| R[6] | 0.0746 |
| $R_w$[7] | 0.0748 |
| GOF | 0.5 |
| Largest shift/esd | 0.00 |
| High peak in diff map, e/Å$^3$ | 0.31 (6) |

Table of Positional Parameters:

| Atom | x | y | z | B (A$^2$) |
|---|---|---|---|---|
| O1 | 0.9197 (3) | 0.2000 (3) | 0.8090 (3) | 3.34 (7) |
| O2 | 0.9412 (3) | 0.0445 (3) | 1.0958 (3) | 3.25 (7) |
| O3 | 0.7900 (4) | 0.0569 (4) | 0.7260 (3) | 4.39 (9) |

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[6] $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$

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

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| | | | | |
|---|---|---|---|---|
| C1 | 0.9862 (4) | 0.2590 (5) | 0.9019 (4) | 2.7 (1) |
| C2 | 1.0660 (5) | 0.3526 (5) | 0.8857 (4) | 3.5 (1) |
| C3 | 1.1398 (5) | 0.4141 (5) | 0.9719 (4) | 3.6 (1) |
| C4 | 1.1337 (5) | 0.3852 (5) | 1.0714 (4) | 4.0 (1) |
| C5 | 1.0531 (5) | 0.2931 (5) | 1.0870 (4) | 3.4 (1) |
| C6 | 0.9785 (4) | 0.2287 (4) | 1.0013 (4) | 2.49 (9) |
| C7 | 0.8856 (4) | 0.1336 (5) | 1.0152 (4) | 2.6 (1) |
| C8 | 0.8070 (4) | 0.0775 (5) | 0.9097 (4) | 2.8 (1) |
| C9 | 0.8336 (4) | 0.1051 (5) | 0.8093 (4) | 3.0 (1) |
| C10 | 0.8434 (5) | -0.0085 (6) | 1.1312 (4) | 4.1 (1) |
| C11 | 0.7485 (5) | 0.0958 (5) | 1.1244 (4) | 3.5 (1) |
| C12 | 0.7669 (4) | 0.1840 (5) | 1.0372 (4) | 2.7 (1) |
| C13 | 0.6991 (4) | 0.1533 (5) | 0.9243 (4) | 2.9 (1) |
| C14 | 0.5990 (5) | 0.1899 (5) | 0.8550 (4) | 3.5 (1) |
| C15 | 0.4983 (5) | 0.2776 (5) | 0.8691 (5) | 4.0 (1) |
| C16 | 0.5333 (7) | 0.4124 (6) | 0.8501 (8) | 9.2 (3) |
| C17 | 0.3793 (6) | 0.2441 (7) | 0.7891 (6) | 6.9 (2) |
| C18 | 0.4787 (6) | 0.2657 (8) | 0.9782 (6) | 7.6 (2) |
| H2 | 1.068 | 0.375 | 0.816 | 6.0* |
| H3 | 1.196 | 0.477 | 0.962 | 6.0* |
| H4 | 1.185 | 0.429 | 1.131 | 6.0* |
| H5 | 1.050 | 0.273 | 1.157 | 6.0* |
| H8 | 0.807 | -0.077 | 1.087 | 6.0* |
| H10B | 0.807 | -0.012 | 0.900 | 6.0* |
| H10A | 0.874 | -0.038 | 1.202 | 6.0* |
| H11B | 0.667 | 0.063 | 1.106 | 6.0* |
| H11A | 0.764 | 0.139 | 1.190 | 6.0* |

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| | | | | |
|---|---|---|---|---|
| H12 | 0.753 | 0.266 | 1.061 | 6.0* |
| H14 | 0.588 | 0.157 | 0.786 | 6.0* |
| H16A | 0.472 | 0.470 | 0.858 | 10.0* |
| H16B | 0.610 | 0.433 | 0.899 | 10.0* |
| H16C | 0.541 | 0.418 | 0.780 | 10.0* |
| H17A | 0.315 | 0.297 | 0.797 | 10.0* |
| H17B | 0.390 | 0.254 | 0.720 | 10.0* |
| H17C | 0.360 | 0.158 | 0.799 | 10.0* |
| H18A | 0.415 | 0.321 | 0.985 | 10.0* |
| H18B | 0.458 | 0.181 | 0.990 | 10.0* |
| H18C | 0.553 | 0.288 | 1.029 | 10.0* |

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)*[a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos\gamma)*B(1,2) + ac(\cos\beta)*B(1,3) + bc(\cos\alpha)*B(2,3)]$.

Table of General Displacement Parameters — U

| Atom | U(1,1) | U(2,2) | U(3,3) | U(1,2) | U(1,3) | U(2,3) |
|---|---|---|---|---|---|---|
| O1 | 0.054 (2) | 0.039 (2) | 0.033 (1) | -0.008 (2) | 0.011 (1) | 0.001 (2) |
| O2 | 0.042 (2) | 0.038 (2) | 0.044 (2) | 0.006 (2) | 0.013 (1) | 0.014 (2) |
| O3 | 0.066 (2) | 0.054 (2) | 0.045 (2) | -0.007 (2) | 0.012 (2) | -0.017 (2) |
| C1 | 0.036 (2) | 0.029 (2) | 0.038 (2) | 0.005 (2) | 0.009 (2) | -0.001 (2) |
| C2 | 0.051 (3) | 0.037 (3) | 0.046 (3) | 0.001 (2) | 0.017 (2) | 0.007 (2) |

| | | | | | |
|---|---|---|---|---|---|
| C3 | 0.047 (3) | 0.034 (3) | 0.062 (3) | -0.006 (2) | 0.024 (2) |
| 0.003 (3) | | | | | |
| C4 | 0.045 (3) | 0.050 (3) | 0.052 (3) | -0.014 (3) | 0.008 (2) |
| -0.007 (3) | | | | | |
| C5 | 0.044 (3) | 0.043 (3) | 0.040 (2) | -0.001 (2) | 0.010 (2) |
| 0.002 (2) | | | | | |
| C6 | 0.034 (2) | 0.025 (2) | 0.034 (2) | 0.004 (2) | 0.008 (2) |
| 0.004 (2) | | | | | |
| C7 | 0.032 (2) | 0.029 (2) | 0.037 (2) | 0.002 (2) | 0.008 (2) |
| 0.004 (2) | | | | | |
| C8 | 0.039 (2) | 0.023 (2) | 0.044 (2) | 0.003 (2) | 0.012 (2) |
| -0.004 (2) | | | | | |
| C9 | 0.037 (2) | 0.032 (3) | 0.042 (2) | 0.009 (2) | 0.009 (2) |
| -0.003 (2) | | | | | |
| C10 | 0.059 (3) | 0.048 (3) | 0.058 (3) | 0.005 (3) | 0.032 (2) |
| 0.011 (3) | | | | | |
| C11 | 0.050 (3) | 0.043 (3) | 0.042 (2) | -0.001 (3) | 0.018 (2) |
| 0.003 (2) | | | | | |
| C12 | 0.036 (2) | 0.027 (2) | 0.041 (2) | 0.000 (2) | 0.015 (2) |
| -0.000 (2) | | | | | |
| C13 | 0.034 (2) | 0.026 (2) | 0.048 (3) | -0.001 (2) | 0.008 (2) |
| -0.007 (3) | | | | | |
| C14 | 0.042 (3) | 0.036 (3) | 0.051 (3) | -0.001 (2) | 0.008 (2) |
| -0.007 (3) | | | | | |
| C15 | 0.035 (3) | 0.043 (3) | 0.066 (3) | 0.006 (3) | 0.000 (2) |
| -0.005 (3) | | | | | |

| | | | | | |
|---|---|---|---|---|---|
| C16 | 0.085 (5) | 0.037 (4) | 0.231 (9) | 0.015 (4) | 0.052 (5) |
| -0.008 (5) | | | | | |
| C17 | 0.049 (3) | 0.080 (5) | 0.122 (6) | 0.015 (4) | 0.002 (4) |
| -0.009 (5) | | | | | |
| C18 | 0.083 (4) | 0.104 (5) | 0.109 (5) | 0.046 (4) | 0.042 (3) |
| -0.001 (5) | | | | | |

The form of the anisotropic displacement parameter is: $\exp[-2\pi^2\{h^2a^2U(1,1) + k^2b^2U(2,2) + l^2c^2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)\}]$, where a, b, and c are reciprocal lattice constants.