Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl propionate at 185 K

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Key indicators

Single-crystal X-ray study
T = 185 K
Mean σ(C–C) = 0.011 Å
Disorder in main residue
R factor = 0.062
wR factor = 0.100
Data-to-parameter ratio = 8.9

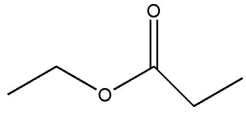
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 February 2005
Accepted 16 February 2005
Online 25 March 2005

The title compound, $C_5H_{10}O_2$, was prepared by a modified zone-refining technique, and has a layered structure with a high degree of disorder within the layers.

Comment

Many of the esters and ketones used in the flavours and fragrances industry are liquid at room temperature; thus, in the past, crystalline derivatives have had to be prepared for X-ray analysis. The examination of this substance is part of a programme to simplify and systematize the study of substances which are liquid at room temperature.



(I)

The diffraction pattern could be indexed as tetragonal with a = 9.52 Å and c = 6.89 Å. If some weak and diffuse reflections in layers perpendicular to c were included in the indexing, this doubled the c axis. The diffraction data were processed on the basis of both cells. In both cases, the space group assignment was uncertain. A clear molecular motif could be obtained by direct methods for a range of space groups, though those for the larger cell could not be refined. Eventually, it was decided to discard the weak layers, and work only with the smaller cell. For this cell, space groups $P4_2bc$ (No. 106) and $P4_2/mbc$ (No. 135) gave plausible but disordered solutions. In both cases, the molecules lie at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, with their molecular mirror plane more or less perpendicular to c. This leads to the molecules lying in sheets perpendicular to c, with an intersheet spacing of 3.45 Å. In space group No. 106, the molecule lies on a twofold rotation axis only, whereas in space group No. 135 it lies on the centre of symmetry at the intersection of a twofold axis and a mirror plane. The structure was refined in both space groups.

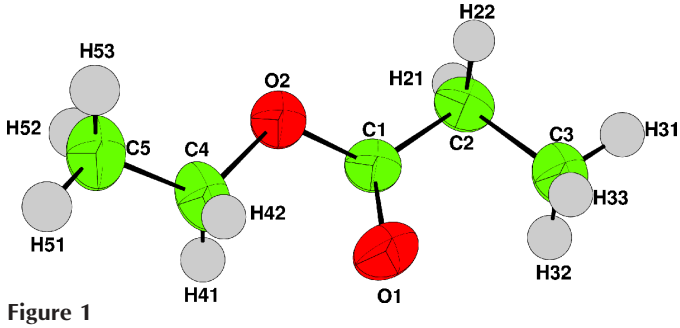


Figure 1
The title molecular structure, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii. The disorder is not shown.

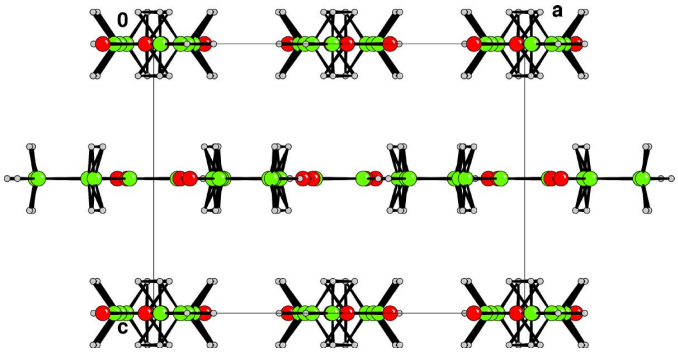


Figure 2
Packing diagram, showing the overall layered structure, viewed along the *b* axis, with disorder included.

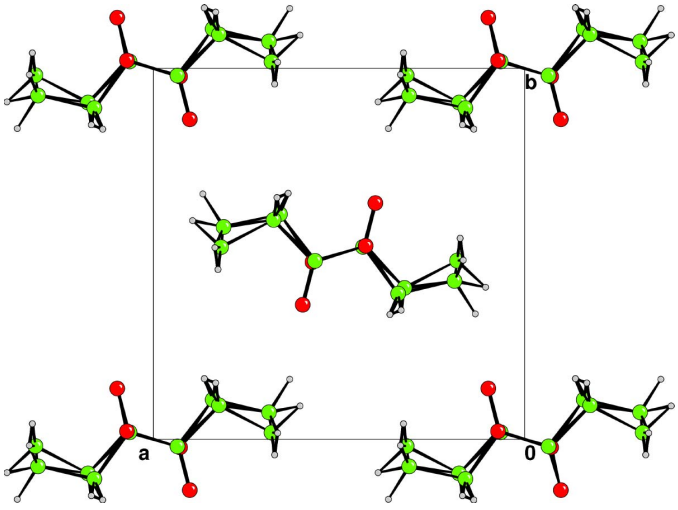


Figure 3
Diagram showing the twofold disordered molecules within one layer, viewed along the *c* axis.

Because both types of disorder lead to interpenetration of the molecule with its image, unrestrained geometric parameters were slightly abnormal. Target values for bond lengths and inter-bond angles were taken from the *MOGUL* database (Bruno *et al.*, 2004), together with standard uncertainties. These were used as restraints for refinement in both space groups.

In space group No. 106, the twofold rotation does not require the molecule to be planar, and the deviations of the non-H atoms from their mean plane are C3 0.08 (2), C2 −0.04 (2), C1 −0.15 (2), O1 −0.04 (2), O2 0.08 (2), C4 0.12 (2), C5 −0.07 (2) Å. The relatively large anisotropic displacement parameters were restrained to satisfy the Hirshfeld (1976) condition for rigid bonds. This refinement concluded satisfactorily with a conventional *R* value of 0.053 for 64 parameters and 383 observations. However, we could see no reason why the disordered molecule should not lie on a centre of symmetry, so the refinement was repeated in space group No. 135. The molecule is not required to be planar, even in this space group, but the atoms lie so close to the mirror that disordered displacements from it could not be modelled. In this space group, the anisotropic displacement parameters are less eccentric than in No. 106, and provide no evidence for believing that the non-H atoms do not actually lie on the mirror plane. It is this refinement which is presented for publication, though we suspect that, in reality, the crystal may suffer from stacking faults and possibly twinning.

Experimental

A 3 mm column of the title material, which is a liquid at room temperature, was sealed in a 0.2 mm Lindemann tube. A single crystal was grown by keeping the compound under a cold nitrogen gas stream at 185 K (not far below its melting point of 198 K) and slowly moving a small liquid zone, created by a micro-heating coil, up and down the sample. Once a suitable approximately single crystal had been obtained, the main data collection was undertaken at this temperature.

Crystal data

$C_5H_{10}O_2$
M_r = 102.14
Tetragonal, $P4_2/mbc$
a = 9.5153 (3) Å
c = 6.8933 (2) Å
V = 624.13 (3) Å^3
Z = 4
D_x = 1.087 Mg m^{-3}
Mo Kα radiation
Cell parameters from 458 reflections
θ = 5–27°
μ = 0.08 mm^{-1}
T = 185 K
Cylinder, colourless
0.50 × 0.1 mm (radius)

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.98, T_{max} = 0.98
719 measured reflections
384 independent reflections
383 reflections with $I > -3\sigma(I)$
R_{int} = 0.013
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.100
S = 0.90
383 reflections
43 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + 0.04 + 0.17P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.168 (16)	C1—C2	1.497 (14)
O2—C1	1.351 (6)	C2—C3	1.519 (10)
O2—C4	1.483 (13)	C4—C5	1.481 (10)
C1—O2—C4	107.6 (6)	O1—C1—C2	119.0 (12)
O2—C1—O1	122.4 (10)	C1—C2—C3	107.8 (9)
O2—C1—C2	118.7 (8)	O2—C4—C5	111.6 (10)

H atoms were positioned geometrically after each cycle, with C—H = 1.0 Å and U_{iso}(H) = 1.2U_{eq}(C). All atoms are disordered, with occupancy factor 0.5.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics:

CAMERON (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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