

n-Butyl acetate

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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.040
 wR factor = 0.104
Data-to-parameter ratio = 21.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_6\text{H}_{12}\text{O}_2$, was prepared by a modified
zone-refinement method at 150 K and consists of discrete
molecules in van der Waals contact.

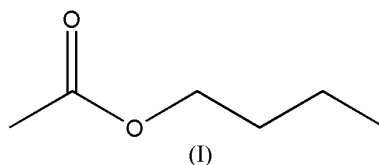
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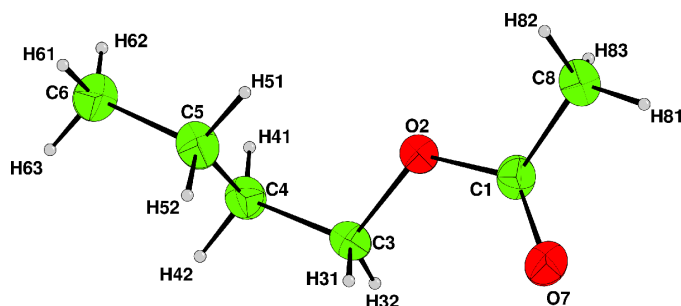
Comment

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. As part of a programme to systematize *in situ* crystal growth from liquids, we have examined a range of commercially available chemicals. Low-molecular-weight organic esters are liquid at room temperature. To date, only the crystal structure of methyl acetate has been determined (Barrow *et al.*, 1981). It was shown that the molecules of methyl acetate exist as discrete entities, without any strong intermolecular contacts. *n*-Butyl acetate, (I), was examined because it has a melting point suitable for our trials. The crystal structure is similar to that of methyl acetate, consisting of discrete molecules in van der Waals contact. The most evident feature is the pairwise parallel butyl residues related by a crystallographic centre of symmetry. The open packing of the structure is reflected in its low density of 1.09 Mg m^{-3} .



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, which was not accurately parallel to the φ axis. A single crystal of the compound was grown by keeping the compound under a cold nitrogen gas stream at 150 K (a little below its melting point), and slowly moving a small

**Figure 1**

The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

liquid zone, created by a micro-heating coil, up and down the sample. Once a suitable approximately single-crystal specimen had been obtained, the main data collection was carried out at this temperature. Because not all the data were collected with the Lindemann tube perpendicular to the X-ray beam, the multi-scan corrections applied by *DENZO/SCALEPACK* (Otwinowski & Minor, 1997) also contain contributions due to changes in illuminated volume of the cylindrical sample; this is reflected in the ratio T_{\min}/T_{\max} .

Crystal data

$C_6H_{12}O_2$	$Z = 2$
$M_r = 116.16$	$D_x = 1.089 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 4.7272 (1) \text{ \AA}$	Cell parameters from 1476 reflections
$b = 7.6955 (3) \text{ \AA}$	$\theta = 5\text{--}27^\circ$
$c = 10.1387 (4) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\alpha = 100.7426 (13)^\circ$	$T = 150 \text{ K}$
$\beta = 96.0038 (15)^\circ$	Cylinder, colourless
$\gamma = 99.3371 (18)^\circ$	$0.80 \times 0.20 \times 0.20 \text{ mm}$
$V = 354.09 (2) \text{ \AA}^3$	

Data collection

Nonius KappaCCD diffractometer	1585 independent reflections
ω scans	1194 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (<i>DENZO/SCALEPACK</i> ; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.035$
$T_{\min} = 0.33$, $T_{\max} = 0.98$	$\theta_{\text{max}} = 27.4^\circ$
5958 measured reflections	$h = -6 \rightarrow 5$
	$k = -9 \rightarrow 9$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F) + 0.045 + 0.061P]$
$wR(F^2) = 0.104$	where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
$S = 0.97$	$(\Delta/\sigma)_{\text{max}} < 0.0001$
1585 reflections	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
73 parameters	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

C1—O2	1.3411 (14)	C3—C4	1.5096 (16)
C1—O7	1.2037 (14)	C4—C5	1.5192 (17)
C1—C8	1.4981 (17)	C5—C6	1.5189 (18)
O2—C3	1.4571 (14)		
O2—C1—O7	123.27 (11)	O2—C3—C4	107.14 (9)
O2—C1—C8	111.16 (10)	C3—C4—C5	113.98 (10)
O7—C1—C8	125.57 (11)	C4—C5—C6	112.59 (11)
C1—O2—C3	115.87 (9)		

All H atoms were located in a difference map, but those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H = 0.97–1.01 \AA), after which they were refined with riding constraints and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

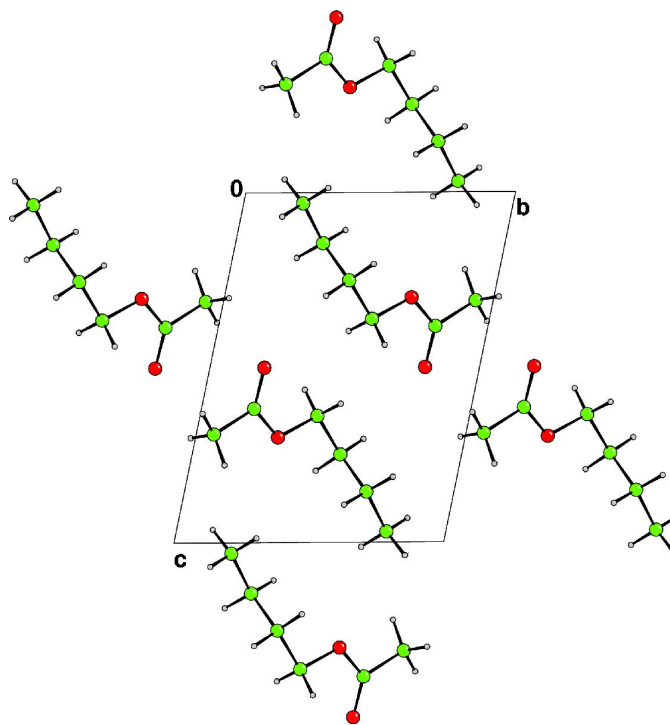


Figure 2

A packing diagram, viewed along the a axis, showing the parallel pairing of the butyl groups.

Data collection: *COLLECT* (Nonius, 1997); 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*.

References

- Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Barrow, M. J., Craddock, S., Ebsworth, E. A. V. & Rankin, D. W. H. (1981). *J. Chem. Soc. Dalton Trans.* **16**, 1988–1993.
- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
- Nonius (1997). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.