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n-Butyl acetate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.104
Data-to-parameter ratio = 21.7

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

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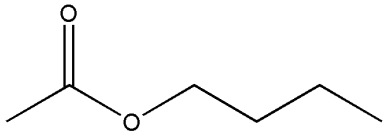
The title compound, $C_6H_{12}O_2$, was prepared by a modified zone-refinement method at 150 K and consists of discrete molecules in van der Waals contact.

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}.



(I)

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

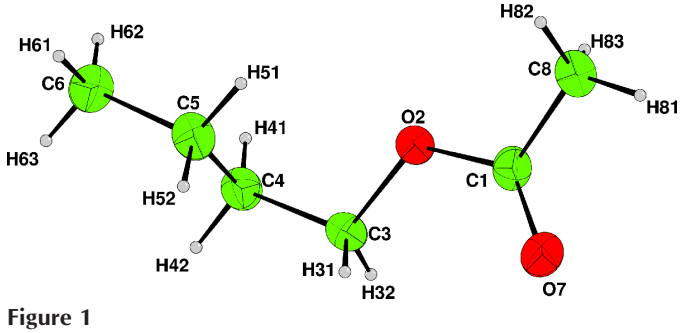


Figure 1
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

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

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

Data collection

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

Refinement

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

Table 1
Selected geometric parameters (Å, °).

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 Å), after which they were refined with riding constraints and with $U_{iso}(H) = 1.2U_{eq}(C)$.

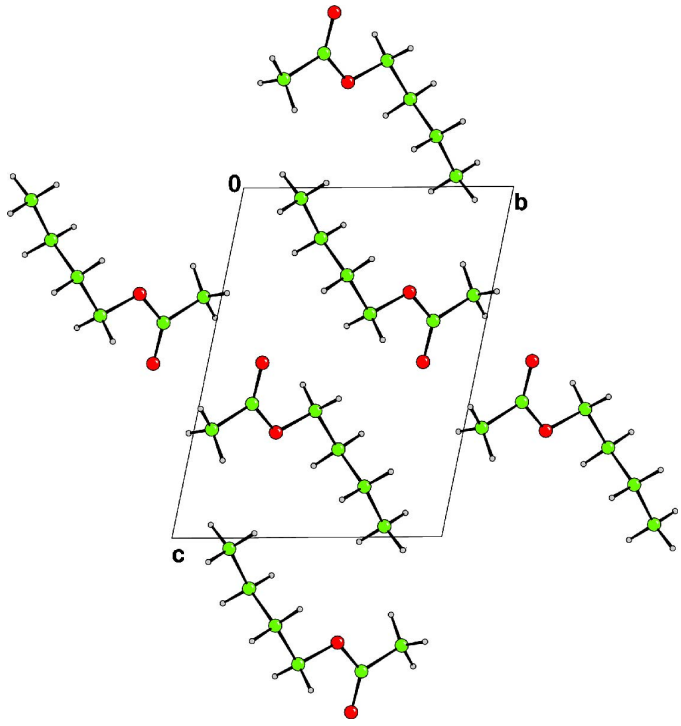


Figure 2
A packing diagram, viewed along the *a* axis, showing the parallel pairing of the butyl groups.

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*.

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$V = 354.09$ (2) Å^3
$Z = 2$
$F(000) = 128$
$D_x = 1.089$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1476 reflections
$\theta = 5$–27°
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Cylinder, colourless
0.80 × 0.20 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)
$T_{min} = 0.33$, $T_{max} = 0.98$
5958 measured reflections
1585 independent reflections
1194 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.4°$, $\theta_{min} = 5.1°$
$h = -6 \rightarrow 5$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.104$
$S = 0.97$
1585 reflections
73 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.045 + 0.061P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.0002$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4329 (2)	0.19532 (15)	0.61673 (12)	0.0319
O2	0.65680 (17)	0.30569 (11)	0.69916 (8)	0.0339
C3	0.8300 (3)	0.43555 (16)	0.63810 (12)	0.0346

C4	1.0599 (3)	0.54983 (16)	0.74868 (12)	0.0343
C5	0.9433 (3)	0.67482 (17)	0.85537 (13)	0.0391
C6	1.1774 (3)	0.77932 (18)	0.96950 (14)	0.0473
O7	0.37156 (19)	0.20225 (12)	0.49981 (9)	0.0425
C8	0.2750 (3)	0.06562 (18)	0.68942 (14)	0.0422
H31	0.7054	0.5128	0.6039	0.0396*
H32	0.9191	0.3691	0.5635	0.0386*
H41	1.1621	0.4712	0.7951	0.0409*
H42	1.2056	0.6241	0.7048	0.0407*
H51	0.7923	0.6020	0.8923	0.0463*
H52	0.8546	0.7619	0.8135	0.0456*
H61	1.0958	0.8659	1.0380	0.0562*
H62	1.2725	0.6978	1.0129	0.0569*
H63	1.3283	0.8469	0.9317	0.0556*
H81	0.0981	0.0075	0.6299	0.0492*
H82	0.2414	0.1286	0.7779	0.0492*
H83	0.3985	−0.0178	0.6998	0.0499*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0323 (6)	0.0278 (6)	0.0334 (7)	0.0043 (5)	0.0010 (5)	0.0040 (5)
O2	0.0365 (5)	0.0326 (5)	0.0297 (5)	−0.0025 (4)	0.0006 (3)	0.0092 (4)
C3	0.0359 (6)	0.0347 (6)	0.0330 (6)	−0.0005 (5)	0.0047 (5)	0.0123 (5)
C4	0.0310 (6)	0.0349 (7)	0.0361 (7)	0.0018 (5)	0.0024 (5)	0.0096 (5)
C5	0.0390 (7)	0.0339 (7)	0.0432 (7)	0.0028 (5)	0.0060 (6)	0.0078 (6)
C6	0.0593 (9)	0.0387 (8)	0.0385 (7)	0.0000 (6)	0.0018 (6)	0.0051 (6)
O7	0.0461 (5)	0.0411 (5)	0.0350 (5)	−0.0011 (4)	−0.0061 (4)	0.0092 (4)
C8	0.0460 (7)	0.0353 (7)	0.0410 (7)	−0.0034 (6)	0.0044 (6)	0.0072 (6)

Geometric parameters (Å, º)

C1—O2	1.3411 (14)	C5—C6	1.5189 (18)
C1—O7	1.2037 (14)	C5—H51	0.983
C1—C8	1.4981 (17)	C5—H52	0.983
O2—C3	1.4571 (14)	C6—H61	1.021
C3—C4	1.5096 (16)	C6—H62	0.971
C3—H31	0.984	C6—H63	0.968
C3—H32	1.002	C8—H81	0.967
C4—C5	1.5192 (17)	C8—H82	0.978
C4—H41	0.988	C8—H83	0.948
C4—H42	1.015		
O2—C1—O7	123.27 (11)	C4—C5—H51	108.3
O2—C1—C8	111.16 (10)	C6—C5—H51	109.8
O7—C1—C8	125.57 (11)	C4—C5—H52	109.8
C1—O2—C3	115.87 (9)	C6—C5—H52	108.1
O2—C3—C4	107.14 (9)	H51—C5—H52	108.2

O2—C3—H31	109.0	C5—C6—H61	111.2
C4—C3—H31	109.1	C5—C6—H62	110.7
O2—C3—H32	109.0	H61—C6—H62	111.4
C4—C3—H32	111.0	C5—C6—H63	109.1
H31—C3—H32	111.3	H61—C6—H63	109.3
C3—C4—C5	113.98 (10)	H62—C6—H63	104.9
C3—C4—H41	109.5	C1—C8—H81	106.2
C5—C4—H41	107.8	C1—C8—H82	110.4
C3—C4—H42	108.1	H81—C8—H82	113.2
C5—C4—H42	109.2	C1—C8—H83	105.1
H41—C4—H42	108.1	H81—C8—H83	110.9
C4—C5—C6	112.59 (11)	H82—C8—H83	110.7