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Methyl acetoacetate at 150 K

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 12.7

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

The crystal structure of methyl acetoacetate, $C_5H_8O_3$, at 150 K contains discrete molecules.

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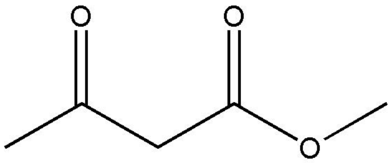
Comment

Many of the esters and ketones used in the flavours and fragrances industry are liquid at room temperature, meaning that 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 ketones are liquid at room temperature. Molecules of methyl acetoacetate, (I), exist as discrete entities in the crystal structure at 150 K, with no strong intermolecular interactions.



(I)

Experimental

A 3 mm column of the title material, which is a liquid at room temperature, was sealed in a 0.3 mm Lindemann tube. The Lindemann tube was not precisely parallel to the φ axis. A single crystal of the compound was grown by keeping the sample under a stream of nitrogen gas (Oxford Cryostream 600) at 180 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 specimen had been obtained, the main data collection was carried out at 150 K. 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 the illuminated volume of the cylindrical sample, which affects the value of $T_{\min}/T_{\max}$.

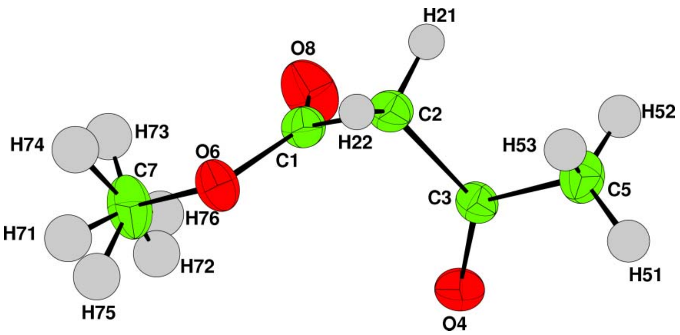


Figure 1
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

Crystal data

$C_5H_8O_3$
$M_r = 116.12$
Monoclinic, $P12_1/c1$
$a = 6.0018$ (2) Å
$b = 8.0384$ (3) Å
$c = 12.4802$ (3) Å
$\beta = 95.5132$ (17)°
$V = 599.32$ (3) Å^3
$Z = 4$
$D_x = 1.287$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1392 reflections
$\theta = 5$–27°
$\mu = 0.11$ mm^{-1}
$T = 150$ K
Cylinder, colourless
0.70 × 0.30 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min} = 0.68$, $T_{\rm max} = 0.95$
2531 measured reflections
1343 independent reflections
1184 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\rm max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.095$
$S = 1.01$
1343 reflections
106 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F) + 0.04 + 0.19P]$ where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.5001 (15)	C3—O4	1.2118 (13)
C1—O6	1.3328 (14)	C3—C5	1.4920 (15)
C1—O8	1.1997 (14)	O6—C7	1.4458 (14)
C2—C3	1.5191 (15)		
C2—C1—O6	111.96 (9)	C2—C3—O4	121.15 (10)
C2—C1—O8	124.51 (11)	C2—C3—C5	115.27 (9)
O6—C1—O8	123.53 (10)	O4—C3—C5	123.58 (10)
C1—C2—C3	112.28 (9)	C1—O6—C7	116.27 (9)

All H atoms were located in a difference map. Alternative positions were clearly visible for the disordered H atoms on C7, whose site occupancy factors were set to 0.5. The H atoms were then repositioned geometrically and refined with soft restraints on the bond lengths and angles to regularize their geometry, with C—H = 0.97–1.01 Å, and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$, after which the restraints were removed.

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*.

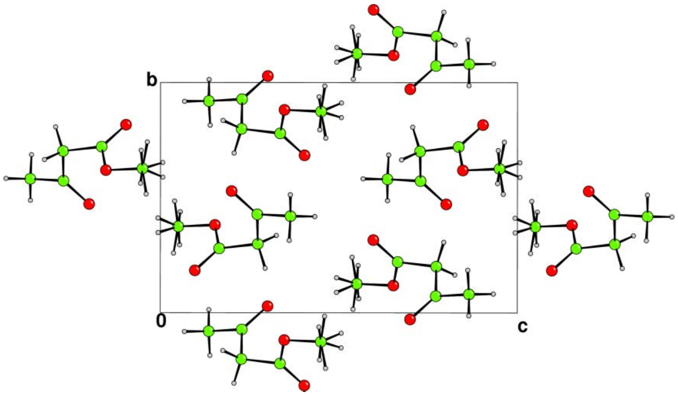


Figure 2
The crystal structure, viewed down the *a* axis.

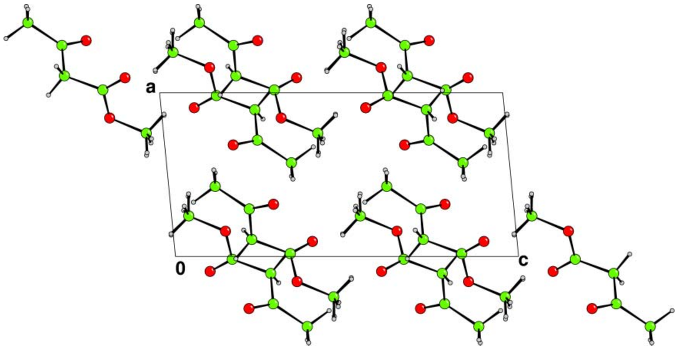


Figure 3
The crystal structure, viewed down the *b* axis.

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