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Cyclohexanone at 150 K

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.119
Data-to-parameter ratio = 12.1

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

The structure of cyclohexanone, $C_6H_{10}O$, at 150 K is that of discrete molecules, with no strong intermolecular interactions.

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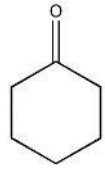
Comment

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. The molecules of cyclohexanone, (I), exist in the crystal structure at 150 K as discrete entities, 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 compound under a cold nitrogen gas stream (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 of 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}.

Crystal data

$C_6H_{10}O$
M_r = 98.14
Orthorhombic, $P2_12_12_1$
a = 5.3736 (2) Å
b = 7.0394 (3) Å
c = 15.1910 (7) Å
V = 574.63 (4) Å^3
Z = 4
D_x = 1.134 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 784 reflections
θ = 5–27°
μ = 0.08 mm^{-1}
T = 150 K
Cylinder, colourless
0.70 × 0.30 × 0.30 mm

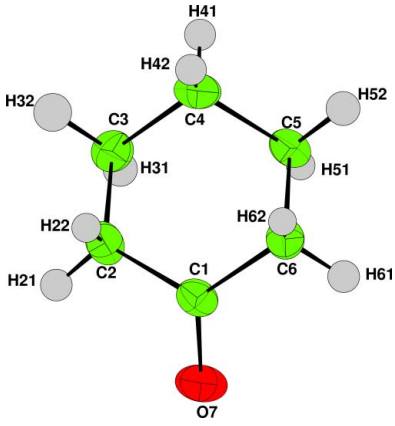


Figure 1
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

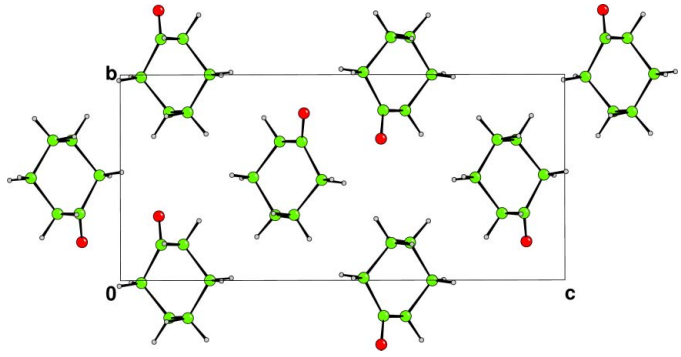


Figure 2
The crystal structure, viewed down the *a* axis.

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.74, T_{max} = 0.98
9235 measured reflections
775 independent reflections
693 reflections with $I > 2\sigma(I)$
R_{int} = 0.085
θ_{max} = 27.4°
$h = -6 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.119
S = 1.02
774 reflections
64 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.08 + 0.07P]$, where $P = (\max(F_o^2, 0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.009
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.501 (2)	C3—C4	1.520 (3)
C1—C6	1.513 (2)	C4—C5	1.523 (3)
C1—O7	1.213 (2)	C5—C6	1.533 (2)
C2—C3	1.532 (3)		
C2—C1—C6	115.45 (14)	C2—C3—C4	111.63 (15)
C2—C1—O7	122.61 (15)	C3—C4—C5	110.85 (16)
C6—C1—O7	121.93 (15)	C4—C5—C6	111.04 (15)
C1—C2—C3	112.29 (15)	C5—C6—C1	111.65 (13)

All H atoms were located in a difference map and 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, and U_{iso}(H) = 1.2U_{eq}(C)], after which they were refined with riding constraints. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

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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supporting information

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$D_x = 1.134$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 784 reflections
$\theta = 5$–27°
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Cylinder, colourless
0.70 × 0.30 × 0.30 mm

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Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
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$\theta_{max} = 27.4°$, $\theta_{min} = 5.5°$
$h = -6 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.119$
$S = 1.02$
774 reflections
64 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.08 + 0.07P]$, where $P = (\max(F_o^2, 0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.009$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4923 (3)	0.1736 (2)	0.09182 (10)	0.0307
C2	0.2508 (3)	0.1748 (3)	0.14147 (11)	0.0371
C3	0.2225 (4)	0.0010 (3)	0.20128 (12)	0.0402
C4	0.2700 (4)	−0.1828 (3)	0.15156 (12)	0.0415
C5	0.5273 (4)	−0.1814 (3)	0.10946 (12)	0.0374

C6	0.5570 (3)	−0.0119 (2)	0.04697 (11)	0.0335
O7	0.6256 (3)	0.31225 (18)	0.08656 (8)	0.0464
H21	0.2463	0.2905	0.1768	0.0499*
H22	0.1133	0.1791	0.0987	0.0414*
H31	0.3443	0.0116	0.2490	0.0582*
H32	0.0510	−0.0026	0.2272	0.0704*
H41	0.2547	−0.2880	0.1930	0.0476*
H42	0.1459	−0.2002	0.1035	0.0472*
H51	0.6535	−0.1746	0.1558	0.0408*
H52	0.5485	−0.2988	0.0751	0.0569*
H61	0.7281	−0.0098	0.0243	0.0502*
H62	0.4460	−0.0270	−0.0028	0.0385*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0397 (9)	0.0260 (8)	0.0264 (7)	0.0019 (8)	−0.0023 (7)	0.0051 (7)
C2	0.0423 (10)	0.0316 (9)	0.0374 (9)	0.0061 (9)	0.0026 (8)	−0.0016 (8)
C3	0.0448 (10)	0.0381 (10)	0.0377 (9)	−0.0034 (9)	0.0097 (8)	−0.0014 (7)
C4	0.0473 (11)	0.0297 (10)	0.0474 (10)	−0.0084 (9)	0.0056 (9)	0.0014 (8)
C5	0.0415 (9)	0.0250 (9)	0.0458 (9)	0.0020 (8)	0.0010 (8)	0.0010 (8)
C6	0.0348 (9)	0.0322 (9)	0.0337 (8)	0.0020 (8)	0.0033 (7)	−0.0003 (7)
O7	0.0586 (9)	0.0321 (7)	0.0484 (8)	−0.0108 (7)	0.0037 (7)	0.0024 (6)

Geometric parameters (Å, º)

C1—C2	1.501 (2)	C4—C5	1.523 (3)
C1—C6	1.513 (2)	C4—H41	0.976
C1—O7	1.213 (2)	C4—H42	0.997
C2—C3	1.532 (3)	C5—C6	1.533 (2)
C2—H21	0.976	C5—H51	0.979
C2—H22	0.984	C5—H52	0.984
C3—C4	1.520 (3)	C6—H61	0.982
C3—H31	0.980	C6—H62	0.969
C3—H32	1.002		
C2—C1—C6	115.45 (14)	C5—C4—H41	110.615
C2—C1—O7	122.61 (15)	C3—C4—H42	110.906
C6—C1—O7	121.93 (15)	C5—C4—H42	107.479
C1—C2—C3	112.29 (15)	H41—C4—H42	108.838
C1—C2—H21	107.617	C4—C5—C6	111.04 (15)
C3—C2—H21	109.743	C4—C5—H51	109.079
C1—C2—H22	108.516	C6—C5—H51	109.579
C3—C2—H22	109.952	C4—C5—H52	108.806
H21—C2—H22	108.626	C6—C5—H52	108.260
C2—C3—C4	111.63 (15)	H51—C5—H52	110.060
C2—C3—H31	108.170	C5—C6—C1	111.65 (13)
C4—C3—H31	108.700	C5—C6—H61	109.048

C2—C3—H32	110.150	C1—C6—H61	111.075
C4—C3—H32	109.123	C5—C6—H62	109.512
H31—C3—H32	109.015	C1—C6—H62	107.729
C3—C4—C5	110.85 (16)	H61—C6—H62	107.731
C3—C4—H41	108.144		