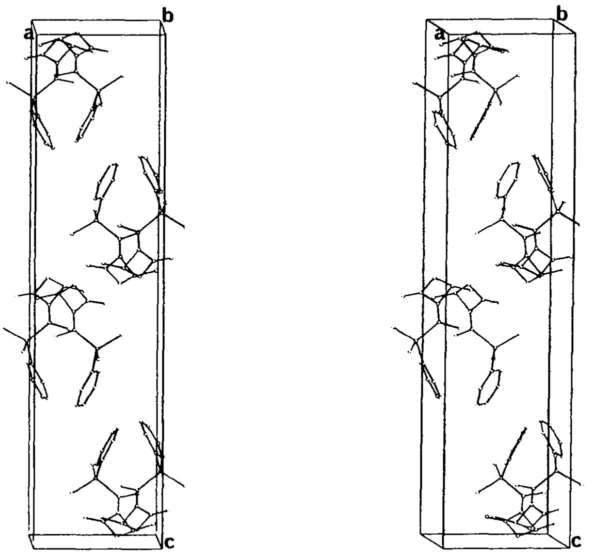

Fig. 2. Stereoscopic drawing showing the packing of molecules in the unit cell. H atoms are removed for clarity.

Related literature. α-Alkylidene-β-lactams have recently assumed importance as β-lactamase inhibitors (Chen, Chang, Hedberg, Guarino, Welch, Kiessling, Retsema, Haskell, Anderson, Manousos & Barrett, 1978). β-Silylenones are valuable synthetic intermediates for the synthesis of α,β-unsaturated ketones (Fleming & Perry, 1981).

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Acta Cryst. (1988). C**44**, 393–395

7-Chloro-6-oxobicyclo[3.2.0]hept-2-en-5-yl 2,4,6-Trimethylbenzoate

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(*Received* 29 *June* 1987; *accepted* 7 *October* 1987)

Abstract. $C_{17}H_{17}ClO_3$ (1), $M_r = 304{\cdot}78$, monoclinic, $P2_1/n$, $a = 11{\cdot}795\,(2)$, $b = 7{\cdot}649\,(1)$, $c = 17{\cdot}827\,(2)$ Å, $\beta = 104{\cdot}57\,(1)^\circ$, $V = 1556{\cdot}6\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}300$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}49$ cm^{-1}, $F(000) = 640$, $T = 293$ K, $R = 0{\cdot}0521$ for 1883 unique reflections. The cyclobutane ring is folded by 3·6 (3)° along C(1)—C(6). A least-squares plane (r.m.s.d. 0·012 Å) through this ring makes an angle of 64·5 (6)° with a plane through the cyclopentene ring (r.m.s.d. 0·012 Å). Torsion angles around the five-membered ring range from −3·0 (6) to 3·2 (4)°. The plane of the phenyl ring (r.m.s.d. 0·003 Å) and the C(8)C(9)O(2)O(3) plane (r.m.s.d. 0·003 Å) make an angle of 81·4 (6)°.

Experimental. Colorless, transparent crystals of (1) from chloroform, 0·68 × 0·53 × 0·18 mm; Nicolet $R3m/\mu$ update of $P2_1$ diffractometer; data collected Wyckoff mode ($3 \leq 2\theta \leq 50^\circ$; 2θ fixed, ω varied), scan rate 4 – 29·3° min^{-1}, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($25{\cdot}01 \leq 2\theta \leq 40{\cdot}42^\circ$), angles measured by a centering routine; systematic absences ($h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$) consistent with space group $P2_1/n$, Laue symmetry $2/m$, monitored reflections ($12\bar{9}$ and $13\bar{1}$) showed only

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Table 1. *Atomic coordinates* ($\times10^4$) *and isotropic thermal parameters* (Å$^2\times10^3$)

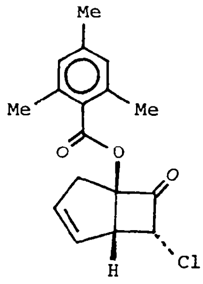
	x	*y*	*z*	*U**
Cl	−6278 (1)	415 (1)	8127 (1)	82 (1)
C(1)	−3852 (2)	−375 (4)	8431 (2)	55 (1)
C(2)	−4164 (3)	−1855 (4)	8892 (2)	70 (1)
C(3)	−3852 (3)	−1572 (4)	9634 (2)	72 (1)
C(4)	−3211 (3)	88 (4)	9852 (2)	65 (1)
C(5)	−3222 (2)	933 (3)	9067 (2)	48 (1)
C(6)	−4236 (2)	2222 (4)	8764 (2)	52 (1)
C(7)	−4821 (2)	977 (4)	8118 (2)	60 (1)
O(1)	−4497 (2)	3586 (3)	8992 (1)	81 (1)
O(2)	−2063 (1)	1573 (2)	9111 (1)	53 (1)
O(3)	−2717 (2)	2842 (3)	7962 (1)	76 (1)
C(8)	−1926 (2)	2576 (4)	8518 (2)	53 (1)
C(9)	−722 (2)	3290 (4)	8656 (1)	47 (1)
C(10)	173 (2)	2286 (4)	8494 (2)	53 (1)
C(11)	1272 (2)	3028 (4)	8604 (2)	55 (1)
C(12)	1509 (2)	4718 (4)	8864 (2)	52 (1)
C(13)	602 (3)	5670 (4)	9023 (2)	54 (1)
C(14)	−518 (2)	5003 (4)	8919 (1)	51 (1)
C(15)	−43 (3)	447 (4)	8184 (2)	86 (2)
C(16)	2706 (3)	5490 (4)	8961 (2)	76 (1)
C(17)	−1490 (3)	6115 (4)	9063 (2)	72 (1)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and valence angles* (°)

Cl–C(7)	1·776 (3)	C(1)–C(2)	1·498 (5)
C(1)–C(5)	1·554 (4)	C(1)–C(7)	1·538 (4)
C(2)–C(3)	1·298 (5)	C(3)–C(4)	1·479 (5)
C(4)–C(5)	1·537 (4)	C(5)–C(6)	1·539 (4)
C(5)–O(2)	1·435 (3)	C(6)–C(7)	1·519 (4)
C(6)–O(1)	1·189 (4)	O(2)–C(8)	1·348 (3)
O(3)–C(8)	1·196 (3)	C(8)–C(9)	1·484 (4)
C(9)–C(10)	1·393 (4)	C(9)–C(14)	1·392 (4)
C(10)–C(11)	1·383 (4)	C(10)–C(15)	1·509 (4)
C(11)–C(12)	1·378 (4)	C(12)–C(13)	1·382 (4)
C(12)–C(16)	1·499 (4)	C(13)–C(14)	1·384 (4)
C(14)–C(17)	1·501 (4)		
C(2)–C(1)–C(5)	103·0 (2)	C(2)–C(1)–C(7)	116·4 (3)
C(5)–C(1)–C(7)	90·4 (2)	C(1)–C(2)–C(3)	112·7 (3)
C(2)–C(3)–C(4)	114·0 (3)	C(3)–C(4)–C(5)	103·6 (2)
C(1)–C(5)–C(4)	106·6 (2)	C(1)–C(5)–C(6)	88·2 (2)
C(4)–C(5)–C(6)	114·6 (2)	C(1)–C(5)–O(2)	122·2 (2)
C(4)–C(5)–O(2)	107·7 (2)	C(6)–C(5)–O(2)	116·5 (2)
C(5)–C(6)–C(7)	91·7 (2)	C(5)–C(6)–O(1)	133·4 (2)
C(7)–C(6)–O(1)	134·6 (2)	Cl–C(7)–C(1)	117·8 (2)
Cl–C(7)–C(6)	113·6 (2)	C(1)–C(7)–C(6)	89·5 (2)
C(5)–O(2)–C(8)	116·9 (2)	O(2)–C(8)–O(3)	121·7 (3)
O(2)–C(8)–C(9)	112·3 (2)	O(3)–C(8)–C(9)	126·1 (3)
C(8)–C(9)–C(10)	120·4 (2)	C(8)–C(9)–C(14)	118·6 (2)
C(10)–C(9)–C(14)	120·9 (2)	C(9)–C(10)–C(11)	118·4 (3)
C(9)–C(10)–C(15)	121·5 (3)	C(11)–C(10)–C(15)	120·1 (3)
C(10)–C(11)–C(12)	122·5 (3)	C(11)–C(12)–C(13)	117·5 (3)
C(11)–C(12)–C(16)	120·9 (3)	C(13)–C(12)–C(16)	121·6 (3)
C(12)–C(13)–C(14)	122·7 (3)	C(9)–C(14)–C(13)	118·1 (3)
C(9)–C(14)–C(17)	121·1 (2)	C(13)–C(14)–C(17)	120·8 (3)

statistical variations in intensities; 2725 independent reflections measured ($0 \le h \le 14$, $0 \le k \le 9$, $-20 \le l \le 20$); $1883 \ge 3\sigma(I)$; Lorentz–polarization corrections, ψ-scan empirical absorption correction (transmission factors 0·854 to 0·924, $R_{\rm merge} = 0{\cdot}019$); structure solved by direct methods, block-cascade least-squares refinement with H atoms refined with single isotropic temperature factor; final $R = 0{\cdot}0521$, $wR = 0{\cdot}0488$ for 243 parameters and 1883 reflections, $R = 0{\cdot}0819$ for all 2725 reflections, $S = 1{\cdot}529$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}027$, $(\Delta/\sigma)_{\rm av} = 0{\cdot}003$; largest peaks in the final difference map of $+0{\cdot}30$ and $-0{\cdot}28$ e Å^{-3}, $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}000249F_o^2]^{-1}$; extinction correction $F^* = F_c[1{\cdot}0 + 0{\cdot}002gF_c^2/\sin(2\theta)]^{0{\cdot}25}$ applied, *g* refined to negligible value of 0·0007 (2). All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Microeclipse and Nova 4/C configurations; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Fig. 1 is a drawing of the title compound (1). Table 1 lists atomic positional parameters, while Table 2 gives interatomic distances and valence angles.†



(1)

Related literature. Tropolone synthesis from cyclopentadiene and dichlorocarbene *via* 7,7-dichlorobicyclo[3.2.0]hept-2-en-6-one (Stevens, Reich, Brandt, Fountain & Gaughan, 1965). A study of the hydrolysis (Asao, Machiguchi, Kitamura & Kitahara, 1970) and

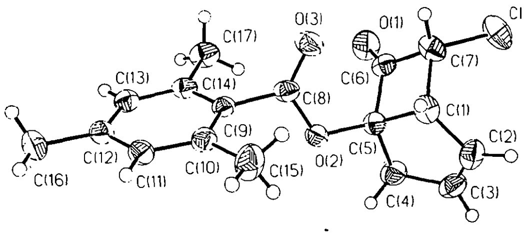


Fig. 1. Drawing of the title compound with thermal ellipsoids drawn at the 35% probability level and H atoms represented by spheres of arbitrary size.

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† Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44441 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

mechanism of hydrolysis (Bartlett & Ando, 1970) of derivatives of bicyclo[3.2.0]hept-2-en-6-one. Related crystal structures (Friedrichsen, Debaerdemaeker, Bottcher, Hahnemann & Schmidt, 1983; Murray-Rust, Murray-Rust & Brown, 1979; Gordon, Pluscek & Ondetti, 1981; Goldstein, Vannes, Houge, Frisque-Hesbain, Wiaux-Zamar, Ghosez, Germain, Declercq, Van Meerssche & Arrieta, 1981).

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SHORT COMMUNICATION

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1988). C**44**, 395–396

On the structure of Ca_8In_3.

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Abstract

The crystal structure of Ca_8In_3, recently described in space group $P1$, has been more satisfactorily refined in $P\bar{1}$. Although the structure is little changed, adding the center of symmetry has eliminated near-singularities in the least-squares matrix, leading to e.s.d.'s that are approximately one-quarter as large as those previously reported.

Recently, the crystal structure of Ca_8In_3 was described (Fornasini, 1987) in the non-centrosymmetric space group $P1$ [$a = 9{\cdot}606\,(2)$, $b = 9{\cdot}717\,(2)$, $c = 9{\cdot}782\,(2)$ Å, $\alpha = 69{\cdot}65\,(2)$, $\beta = 78{\cdot}85\,(2)$, $\gamma = 60{\cdot}34\,(1)°$, $Z = 2$]. The author considered the centrosymmetric space group $P\bar{1}$, but was unable to obtain refinement below an R value of 0·33. Hence, refinement was carried out in space group $P1$ leading to an R of 0·027 for 2789 reflections.

We have experienced no difficulty in carrying out the refinement in space group $P\bar{1}$. The center of symmetry was created by fixing atoms In(3) and In(5) at (0,0,0) and $(0,\frac{1}{2},\frac{1}{2})$ respectively. The other atoms were shifted accordingly (x–0·6668, y–0·1626, z–0·4985). Full-matrix least-squares refinement quickly led to an R of 0·038. With the addition of a secondary-extinction parameter, further refinement resulted in a final R of 0·0276 for the 2789 reflections (recovered

Table 1. *Coordinates and U_{eq} values (all* × 10^4), *space group* $P\bar{1}$

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j |U_{ij}(a_i^*a_j^*)(\mathbf{a}_i.\mathbf{a}_j)|.$$

	x	y	z	U_{eq}(Å^2)
In(3)	0	0	0	154 (1)
In(5)	0	5000	5000	171 (1)
In(1,4)	3352 (0·5)	−1621 (0·5)	4999 (0·4)	160 (1)
In(2,6)	3359 (0·5)	3402 (0·5)	−272 (0·4)	155 (1)
Ca(1,8)	3496 (1)	−2709 (2)	−1078 (1)	179 (2)
Ca(2,6)	2989 (2)	−2 (2)	1098 (1)	182 (2)
Ca(3,4)	3297 (2)	2013 (2)	3493 (1)	181 (2)
Ca(5,10)	220 (2)	981 (2)	−3521 (1)	186 (3)
Ca(7,9)	444 (2)	−3783 (2)	1094 (1)	181 (2)
Ca(11,15)	3332 (2)	2101 (2)	−2980 (1)	252 (3)
Ca(12,16)	1213 (2)	−3469 (2)	−2998 (1)	248 (3)
Ca(13,14)	3965 (2)	−4390 (2)	3301 (1)	216 (2)

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