(Declercq, Germain, Van Meerssche, Demuynck, De Clercq & Vandewalle, 1980); crystal structure of psilostachyin-A (Wilzer, Han, Zambrano, Fronczek & Watkins, 1988); crystal structure of confertiflorin (Vargas, Fronczek, Fischer & Hostettmann, 1986); conformational asymmetry parameters (Duax & Norton, 1975).

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1,4-Dibromohomocubane Ethylene Ketal

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Abstract. 1,4-Dibromopentacyclo$[4.3.0.0^{2,5}.0^{3,8}.0^{4,7}]$-nonan-9-one ethylene ketal (1), $C_{11}H_{10}Br_2O_2$, $M_r = 334{\cdot}02$, monoclinic, $P2_1/a$, $a = 12{\cdot}651$ (4), $b = 6{\cdot}197$ (1), $c = 14{\cdot}301$ (3) Å, $\beta = 107{\cdot}15$ (2)°, $V = 1071{\cdot}4$ (4) Å^3, $Z = 4$, $D_x = 2{\cdot}071$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 74{\cdot}7$ cm^{-1}, $F(000) = 648$, $T = 295$ K, $R = 0{\cdot}0664$ for 1365 reflections. The cage structure consists of four four-membered rings fused to a norbornane moiety (two fused five-membered rings) with an ethylene ketal attached to the methylene bridge of the norbornane. Two four-membered rings are planar and two are folded along a diagonal. The five-membered ethylene ketal ring is in an envelope conformation but the flap is not at the spiro fusion center with the cage. Molecular mechanics calculations give $\Delta H_f = 168{\cdot}3$ kJ mol^{-1} and E(strain) $= 619{\cdot}3$ kJ mol^{-1} with major contributions from angle (359·8 kJ mol^{-1}) and torsional strain (272·0 kJ mol^{-1}).

Experimental. The title compound was synthesized by literature procedures (Chapman, Key & Toyne, 1970; Mehta, Srikrishna & Suri, 1980), and recrystallization yielded a colorless, poor-quality crystal (asymmetric peak profiles and backgrounds with some diffraction spots exceeding maximum scan width) of dimensions 0·50 × 0·43 × 0·35 mm. After data collection an attempt was made to cleave the crystal to a smaller size to reduce the absorption correction; however, the crystal shattered into small fragments. All data were collected on a Nicolet $R3M/\mu$ update of a $P2_1$ diffractometer, ω scan technique ($3 \leq 2\theta \leq 50°$), variable scan rate of 4 to 29·3° min^{-1}, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($23{\cdot}16 \leq 2\theta \leq 28{\cdot}84°$);

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

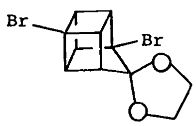
U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
C(1)	0·8677 (7)	0·5104 (15)	0·2434 (6)	0·041 (3)
Br(1)	0·7258 (1)	0·6453 (2)	0·2086 (1)	0·064 (1)
C(2)	0·8724 (8)	0·2754 (15)	0·2819 (7)	0·044 (3)
C(3)	0·9830 (9)	0·1939 (18)	0·2692 (7)	0·048 (4)
C(4)	1·0413 (7)	0·2669 (15)	0·3753 (6)	0·037 (3)
Br(4)	1·1525 (1)	0·1040 (2)	0·4662 (1)	0·059 (1)
C(5)	0·9309 (7)	0·3520 (17)	0·3873 (7)	0·045 (4)
C(6)	0·9507 (8)	0·5724 (15)	0·3410 (7)	0·045 (4)
C(7)	1·0605 (7)	0·4878 (16)	0·3299 (7)	0·043 (3)
C(8)	1·0310 (7)	0·3882 (16)	0·2276 (6)	0·042 (3)
C(9)	0·9319 (7)	0·5132 (15)	0·1668 (6)	0·037 (3)
O(1)	0·8782 (5)	0·4164 (11)	0·0780 (4)	0·047 (2)
O(2)	0·9575 (6)	0·7280 (11)	0·1468 (5)	0·057 (3)
C(10)	0·8440 (9)	0·5836 (19)	0·0065 (8)	0·060 (5)
C(11)	0·9257 (10)	0·7590 (19)	0·0472 (8)	0·061 (5)

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

C(1)—Br(1)	1·908 (9)	C(1)—C(2)	1·551 (13)
C(1)—C(6)	1·527 (12)	C(1)—C(9)	1·544 (15)
C(2)—C(3)	1·547 (16)	C(2)—C(5)	1·546 (12)
C(3)—C(4)	1·547 (13)	C(3)—C(8)	1·543 (15)
C(4)—Br(4)	1·899 (8)	C(4)—C(5)	1·549 (14)
C(4)—C(7)	1·564 (14)	C(5)—C(6)	1·569 (14)
C(6)—C(7)	1·536 (15)	C(7)—C(8)	1·528 (13)
C(8)—C(9)	1·511 (12)	C(9)—O(1)	1·387 (10)
C(9)—O(2)	1·418 (12)	O(1)—C(10)	1·430 (13)
O(2)—C(11)	1·374 (13)	C(10)—C(11)	1·494 (16)
Br(1)—C(1)—C(2)	116·0 (7)	Br(1)—C(1)—C(6)	118·0 (7)
C(2)—C(1)—C(6)	88·2 (6)	Br(1)—C(1)—C(9)	117·5 (6)
C(2)—C(1)—C(9)	106·8 (8)	C(6)—C(1)—C(9)	106·3 (8)
C(1)—C(2)—C(3)	101·9 (8)	C(1)—C(2)—C(5)	90·9 (7)
C(3)—C(2)—C(5)	91·5 (7)	C(2)—C(3)—C(4)	88·6 (8)
C(2)—C(3)—C(8)	105·2 (8)	C(4)—C(3)—C(8)	91·2 (7)
C(3)—C(4)—Br(4)	124·0 (7)	C(3)—C(4)—C(5)	91·4 (7)
Br(4)—C(4)—C(5)	127·9 (7)	C(3)—C(4)—C(7)	86·3 (7)
Br(4)—C(4)—C(7)	125·1 (6)	C(5)—C(4)—C(7)	90·2 (7)
C(2)—C(5)—C(4)	88·6 (7)	C(2)—C(5)—C(6)	86·8 (7)
C(4)—C(5)—C(6)	89·3 (8)	C(1)—C(6)—C(5)	90·9 (7)
C(1)—C(6)—C(7)	102·7 (8)	C(5)—C(6)—C(7)	90·5 (7)
C(4)—C(7)—C(6)	90·0 (7)	C(4)—C(7)—C(8)	91·1 (7)
C(6)—C(7)—C(8)	105·2 (7)	C(3)—C(8)—C(7)	87·7 (7)
C(3)—C(8)—C(9)	105·5 (7)	C(7)—C(8)—C(9)	105·7 (8)
C(1)—C(9)—C(8)	96·1 (7)	C(1)—C(9)—O(1)	115·3 (8)
C(8)—C(9)—O(1)	113·8 (8)	C(1)—C(9)—O(2)	110·7 (8)
C(8)—C(9)—O(2)	113·4 (7)	O(1)—C(9)—O(2)	107·4 (7)
C(9)—O(1)—C(10)	107·7 (7)	C(9)—O(2)—C(11)	109·0 (7)
O(1)—C(10)—C(11)	102·7 (8)	O(2)—C(11)—C(10)	105·0 (10)

systematic absences ($h0l$, $h = 2n + 1$, $0k0$, $k = 2n + 1$) consistent with space group $P2_1/a$; monitored reflections 601 and 523 showed maximum variations in intensities of ±2%; 2173 reflections collected ($-15 \leq h \leq 15$, $0 \leq k \leq 7$, $0 \leq l \leq 17$), equivalent reflections were averaged (R_{int} = 0·017), 1884 unique, 1365 $\geq 3\sigma(I)$; Lorentz–polarization and a ψ-scan-based absorption correction applied (transmission factors 0·067 to 0·134); structure solved by direct methods, H atoms located in a difference map, H atoms of cage refined with isotropic thermal parameters, H atoms of ketal allowed to ride on attached atom but a single isotropic thermal parameter refined, block-cascade least-squares refinement gave R = 0·0664, wR = 0·0711 for 165 parameters and 1365 reflections (R = 0·0925 and wR = 0·0748 for all 1884 data); S = 1·468, $(\Delta/\sigma)_{max}$ = 0·007, largest peaks in final difference map of −0·75 and +1·00 e Å^{-3} (adjacent to Br atoms); $\sum w(|F_o| - |F_c|)^2$ minimized where $w = [\sigma^2(F_o) + 0{\cdot}00088F_o^2]^{-1}$, $\sigma^2(F_o)$ from counting statistics, isotropic extinction correction applied, $F = F_c/[1{\cdot}0 + 0{\cdot}002(x)F_c^2/\sin(2\theta)]^{0{\cdot}25}$ where x refined to 0·00056 (4) (*SHELXTL;* Nicolet Instrument Corporation, 1986). All computer programs supplied by Nicolet for Desktop 30 Microeclipse and Nova 4/C configuration; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 gives the atomic positional parameters while Table 2 gives a listing of the interatomic distances and valence angles. Fig. 1 is a drawing of the title compound.*



(1)

Related literature. The synthesis, chemistry and spectral properties of pentacyclo[4.3.0.0^{2,5}.0^{3,8}.0^{4,9}]nonane derivatives have been discussed by Chapman, Key & Toyne (1970). The synthesis of compound (1) from

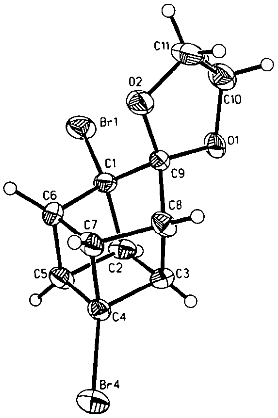


Fig. 1. Compound (1) with thermal ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary size.

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* Lists of H-atom coordinates, anisotropic thermal parameters and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52067 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

snoutanone, the nonan-9-one derivative, has been reported (Mehta, Srikrishna & Suri, 1980). The structures of 5-bromohomocubane-4-carboxyclic acid (Smits, Beurskens, Klunder & Van der Loop, 1986) and homocubane-9-carboxylic acid (Dauben, Schallhorn & Whalen, 1971) have been reported. The molecular mechanics program *MM*2 was used for estimating the heat of formation and strain energies (Allinger & Yu, 1980).

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5-Hydroxy-3,7,4′-trimethoxyflavone

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Abstract. 5-Hydroxy-3,7-dimethoxy-2-(4-methoxyphenyl)chroman-4-one, $C_{18}H_{16}O_6$, $M_r = 328{\cdot}32$, triclinic, $P\bar{1}$, $a = 5{\cdot}2678\,(14)$, $b = 11{\cdot}2895\,(6)$, $c = 12{\cdot}704$ Å, $\alpha = 96{\cdot}29\,(1)$, $\beta = 99{\cdot}62\,(2)$, $\gamma = 94{\cdot}61\,(1)°$, $V = 736{\cdot}60\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}480\,(1)$ g cm^{-3}, monochromatized Mo $K\alpha$ ($\lambda = 0{\cdot}71073$ Å), $\mu = 1{\cdot}045$ cm^{-1}, $F(000) = 688$, $T = 110$ K, final $R = 0{\cdot}037$ for 4284 unique reflections. The 4′-methoxyphenyl ring forms a torsion angle of 27·1 (2)° with the benzopyranone ring. The compound forms a strong intramolecular hydrogen bond.

Experimental. The petroleum ether extract of the leaves of the previously uninvestigated *Haplopappus sonorensis* Compositae yielded the yellow flavonoid which was recrystallized from acetone. Since the positions of the substituents in this biologically active compound cold not easily be determined using spectroscopic methods, a crystal structure investigation was undertaken.

The diffraction data were collected on an Enraf–Nonius CAD-4 diffractometer using $\omega/2\theta$ scans. The crystal was cooled down to 110 ± 0·8 K in a stream of N_2 gas. The cell dimensions were determined from a least-squares fit of 25 reflections, well distributed in reciprocal space, with θ values in the range 16–20°. The maximum scan time was 60 s. Three reflections were measured every 2·8 h to check for decay of the crystal which, however, did not appear to take place. Crystallographic data are given in Table 1. No correction was made for absorption.

The structure was solved using *SHELXS*86 (Sheldrick, 1985), and refined with *SDP* (Enraf–Nonius, 1981). Atomic scattering factors were taken from Cromer & Waber (1974), except that for hydrogen which was taken from Stewart, Davidson & Simpson

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