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Hexacyclo[6.5.1.$0^{1,5}$.$0^{5,12}$.$0^{7,11}$.$0^{9,13}$]tetradecane-4,6,14-trione

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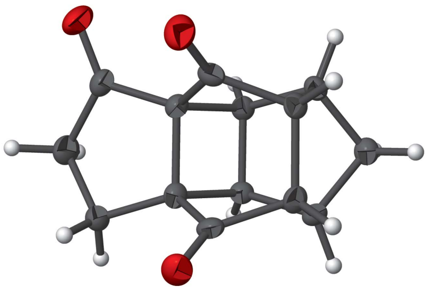
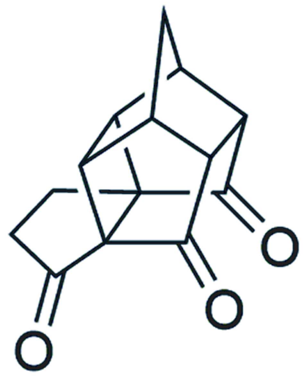
Structural data: full structural data are available from iucrdata.iucr.org

The structure of the title cage compound, $C_{14}H_{12}O_3$, encompasses seven fused rings, *viz*. one four-membered, five five-membered and one six-membered. The four-membered ring is essentially planar, all five-membered rings adopt an envelope conformation and the six-membered ring adopts a boat conformation.

3D view



Chemical scheme



Structure description

As a result of their rigid and strained architectures, polycyclic cage molecules act as a useful scaffold for pharmaceutical applications (Liu *et al.*, 2001; Wilkinson *et al.*, 2014), medicinal chemistry (Wanka *et al.*, 2013; Liu *et al.*, 2011) and energetic materials (Wu *et al.*, 2015; Lal *et al.*, 2014). Some of the oxa-cage systems play an important role in molecular recognition and inclusion phenomena (Marchand *et al.*, 1998). Cage hydrocarbons are useful as core frameworks for photonic/electronic materials (Giacalone & Martín, 2006; Lebedeva *et al.*, 2015) and ligands for organocatalysis (Biegasiewicz *et al.*, 2012).

In view of our research interest in designing various new cage compounds, herein we report the structure and synthesis of the title compound (Fig. 1). The title compound (II) was synthesized (Fig. 2) from inexpensive and commercially available starting materials such as 2,5-dimethoxy benzaldehyde using the Diels–Alder reaction as a key step (Kotha *et al.*, 2017).

The molecular structure of (II) is built up of seven rings: one four-membered, five five-membered and one six-membered rings are fused to a caged carbon framework. The four-membered ring is essentially planar. All five-membered rings adopt an envelope conformation, whereas the six-membered ring is in a boat conformation.

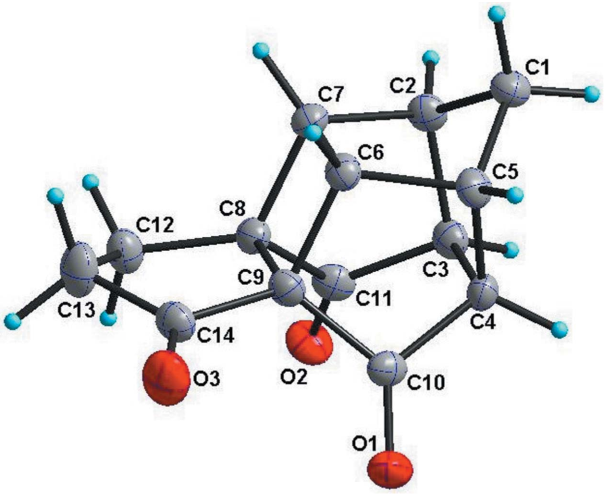


Figure 1
The molecular structure of the title compound (II), with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The title cage compound can be prepared *via* a Diels–Alder reaction of cyclopentadiene with 2,3-dihydro-1*H*-indene-1,4,7-trione followed by [2 + 2] photocycloaddition. To begin with, Diels–Alder adduct (I) (100 mg, 0.43 mmol) was dissolved in dry ethyl acetate (300 ml) and irradiated in a Pyrex immersion well by using 125 W medium pressure UV mercury vapour lamp for 1 h under nitrogen at room temperature. After completion of the reaction (TLC monitoring), the solvent was evaporated under reduced pressure and the crude reaction mixture was purified by silica-gel column chromatography using 40% ethyl acetate in petroleum ether as an eluent to furnish (II) as a colourless crystalline solid (92 mg, 94%). Recrystallization of a [2 + 2] photocycloadduct from a 1:4 mixture of dichloromethane–hexane solvent system delivered orthorhombic crystals of hexacyclic trione (II), m.p. 452–454 K (the melting point was recorded on a veego VMP–CMP melting point apparatus and is uncorrected). IR (neat, cm^{-1}) 2976, 1757, 1740, 1434, 1266, 1139. ^{1}H NMR (500 MHz, CDCl$_3$, p.p.m.): 3.19 (*t*, *J* = 6.2 Hz, 1H), 3.05–3.02 (*m*, 3H), 2.80–2.74 (*m*, 2H), 2.69–2.60 (*m*, 1H), 2.53–2.40 (*m*, 2H), 2.08 (*d*, *J* = 11.5 Hz, 1H), 1.93 (*d*, *J* = 11.4 Hz, 1H), 1.89–1.84 (*m*, 1H). ^{13}C NMR (125 MHz, CDCl$_3$, p.p.m.): 210.7, 209.3, 205.2, 60.0, 56.6, 56.0, 54.7, 43.9, 43.6, 43.3, 42.8, 40.0, 39.9, 20.4; HRMS (ESI, Q-TOF) *m*/*z* calculated for $C_{14}H_{13}O_3$ [*M* + H]$^+$ 229.0859; found: 229.0855.

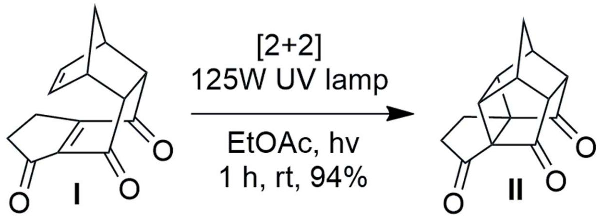


Figure 2
[2 + 2] photocycloaddition of Diels–Alder adduct (I).

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{12}O_3$
M_r	228.24
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	150
a, b, c (Å)	7.4539 (5), 7.8553 (5), 17.2286 (10)
V (Å^3)	1008.78 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.21 × 0.18 × 0.03
Data collection	
Diffractometer	Manufacturer? Model?
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
$T_{\min}$, $T_{\max}$	0.874, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5609, 1769, 1553
$R_{\rm int}$	0.059
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.097, 1.07
No. of reflections	1769
No. of parameters	154
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.27, −0.26
Absolute structure	Flack x determined using 530 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.4 (10)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SUPERFLIP* (Palatinus & Chapuis, 2007; Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012), *SHELXL2018* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{14}H_{12}O_3$
$M_r = 228.24$
Orthorhombic, $P2_12_12_1$
$a = 7.4539$ (5) Å
$b = 7.8553$ (5) Å
$c = 17.2286$ (10) Å
$V = 1008.78$ (11) Å^3
$Z = 4$
$F(000) = 480$
$D_x = 1.503$ Mg m^{-3}
Melting point = 452–454 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3418 reflections
$\theta = 2.3$–30.9°
$\mu = 0.11$ mm^{-1}
$T = 150$ K
Plate, colourless
0.21 × 0.18 × 0.03 mm

Data collection

Rigaku Saturn724+ CCD diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 28.5714 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
$T_{\min} = 0.874$, $T_{\max} = 1.000$
5609 measured reflections
1769 independent reflections
1553 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.059$
$\theta_{\max} = 25.0°$, $\theta_{\min} = 2.4°$
$h = -8 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.097$
$S = 1.07$
1769 reflections
154 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 0.1833P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Absolute structure: Flack x determined using 530 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.4 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were placed in geometrically calculated positions and refined using a riding model with C–H distances of 1.00 Å for all H atoms bound to tertiary C(sp^3) atoms and 0.99 Å for H atoms bound to secondary C(sp^3) atoms. Isotropic displacement parameters for H atoms were calculated as $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Due to the absence of anomalous scatterers, the absolute configuration could not be determined and was arbitrarily set.

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
O1	−0.4454 (3)	−0.5002 (3)	−0.44952 (13)	0.0323 (6)
O2	−0.7382 (3)	−0.2091 (3)	−0.31876 (12)	0.0283 (6)
O3	−0.7593 (4)	−0.5952 (3)	−0.57320 (12)	0.0386 (7)
C1	−0.7649 (5)	−0.7940 (4)	−0.25367 (17)	0.0236 (7)
H1A	−0.678028	−0.808534	−0.210723	0.028*
H1B	−0.857452	−0.884250	−0.251388	0.028*
C2	−0.8456 (4)	−0.6158 (4)	−0.25598 (17)	0.0213 (7)
H2	−0.917858	−0.582697	−0.209453	0.026*
C3	−0.6831 (4)	−0.5005 (4)	−0.27393 (17)	0.0212 (7)
H3	−0.619466	−0.458066	−0.226618	0.025*
C4	−0.5625 (4)	−0.6183 (4)	−0.32811 (17)	0.0201 (7)
H4	−0.438219	−0.636161	−0.307861	0.024*
C5	−0.6744 (4)	−0.7835 (4)	−0.33247 (17)	0.0213 (7)
H5	−0.607082	−0.887681	−0.348789	0.026*
C6	−0.8299 (4)	−0.7293 (4)	−0.38685 (18)	0.0203 (7)
H6	−0.891741	−0.821255	−0.416750	0.024*
C7	−0.9485 (4)	−0.6135 (4)	−0.33487 (17)	0.0189 (7)
H7	−1.080298	−0.637233	−0.333650	0.023*
C8	−0.8843 (4)	−0.4520 (4)	−0.38033 (17)	0.0201 (7)
C9	−0.7637 (4)	−0.5706 (4)	−0.43341 (17)	0.0205 (7)
C10	−0.5679 (4)	−0.5535 (4)	−0.41111 (17)	0.0208 (7)
C11	−0.7643 (4)	−0.3607 (4)	−0.32306 (17)	0.0211 (7)
C12	−1.0053 (4)	−0.3498 (4)	−0.43411 (18)	0.0254 (8)
H12A	−0.956944	−0.233685	−0.441920	0.030*
H12B	−1.127812	−0.340844	−0.412290	0.030*
C13	−1.0076 (5)	−0.4483 (5)	−0.51065 (19)	0.0334 (9)
H13A	−1.109883	−0.528665	−0.511804	0.040*
H13B	−1.019144	−0.368952	−0.555029	0.040*
C14	−0.8314 (4)	−0.5442 (4)	−0.51511 (18)	0.0247 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0259 (13)	0.0392 (15)	0.0317 (14)	−0.0016 (11)	0.0053 (10)	0.0096 (11)
O2	0.0378 (13)	0.0185 (12)	0.0285 (12)	−0.0033 (11)	−0.0014 (11)	−0.0020 (10)
O3	0.0414 (14)	0.0544 (16)	0.0201 (12)	0.0079 (14)	0.0019 (12)	−0.0031 (12)
C1	0.0246 (17)	0.0219 (16)	0.0243 (17)	−0.0021 (15)	0.0004 (14)	0.0028 (14)
C2	0.0229 (16)	0.0238 (16)	0.0173 (16)	0.0012 (16)	0.0031 (13)	−0.0018 (14)
C3	0.0246 (16)	0.0227 (17)	0.0163 (15)	−0.0015 (15)	−0.0035 (13)	−0.0007 (14)
C4	0.0158 (15)	0.0242 (17)	0.0202 (16)	0.0019 (14)	−0.0023 (12)	0.0021 (14)

C5	0.0240 (16)	0.0174 (15)	0.0224 (18)	0.0025 (15)	−0.0013 (14)	0.0008 (13)
C6	0.0219 (16)	0.0191 (15)	0.0198 (17)	−0.0015 (15)	−0.0026 (14)	−0.0009 (13)
C7	0.0169 (15)	0.0194 (16)	0.0203 (16)	−0.0015 (13)	0.0004 (13)	−0.0013 (13)
C8	0.0195 (15)	0.0219 (16)	0.0188 (16)	−0.0019 (14)	−0.0027 (12)	0.0002 (14)
C9	0.0229 (15)	0.0219 (16)	0.0168 (14)	0.0005 (14)	0.0010 (14)	0.0002 (13)
C10	0.0199 (17)	0.0176 (16)	0.0249 (18)	0.0019 (14)	0.0042 (14)	0.0004 (14)
C11	0.0252 (16)	0.0189 (17)	0.0193 (15)	0.0013 (15)	0.0060 (14)	−0.0031 (13)
C12	0.0265 (17)	0.0277 (17)	0.0219 (16)	0.0061 (16)	−0.0012 (14)	0.0011 (15)
C13	0.034 (2)	0.043 (2)	0.0230 (19)	0.0080 (18)	−0.0077 (15)	−0.0020 (17)
C14	0.0284 (16)	0.0279 (17)	0.0177 (17)	−0.0014 (16)	−0.0006 (14)	0.0009 (15)

Geometric parameters (Å, º)

O1—C10	1.203 (4)	C5—C6	1.550 (4)
O2—C11	1.209 (4)	C6—H6	1.0000
O3—C14	1.204 (4)	C6—C7	1.553 (4)
C1—H1A	0.9900	C6—C9	1.562 (4)
C1—H1B	0.9900	C7—H7	1.0000
C1—C2	1.524 (4)	C7—C8	1.566 (4)
C1—C5	1.518 (4)	C8—C9	1.585 (4)
C2—H2	1.0000	C8—C11	1.513 (4)
C2—C3	1.544 (4)	C8—C12	1.522 (4)
C2—C7	1.561 (4)	C9—C10	1.516 (4)
C3—H3	1.0000	C9—C14	1.509 (4)
C3—C4	1.592 (4)	C12—H12A	0.9900
C3—C11	1.513 (4)	C12—H12B	0.9900
C4—H4	1.0000	C12—C13	1.529 (4)
C4—C5	1.544 (4)	C13—H13A	0.9900
C4—C10	1.518 (4)	C13—H13B	0.9900
C5—H5	1.0000	C13—C14	1.517 (5)
H1A—C1—H1B	110.1	C6—C7—C2	102.4 (2)
C2—C1—H1A	112.6	C6—C7—H7	117.5
C2—C1—H1B	112.6	C6—C7—C8	90.7 (2)
C5—C1—H1A	112.6	C8—C7—H7	117.5
C5—C1—H1B	112.6	C7—C8—C9	89.2 (2)
C5—C1—C2	95.9 (2)	C11—C8—C7	103.8 (2)
C1—C2—H2	115.5	C11—C8—C9	108.6 (2)
C1—C2—C3	103.5 (2)	C11—C8—C12	119.9 (3)
C1—C2—C7	103.2 (2)	C12—C8—C7	123.4 (3)
C3—C2—H2	115.5	C12—C8—C9	107.1 (2)
C3—C2—C7	101.8 (2)	C6—C9—C8	89.7 (2)
C7—C2—H2	115.5	C10—C9—C6	104.1 (2)
C2—C3—H3	113.8	C10—C9—C8	110.4 (2)
C2—C3—C4	102.6 (2)	C14—C9—C6	118.9 (3)
C4—C3—H3	113.8	C14—C9—C8	105.5 (2)
C11—C3—C2	102.9 (2)	C14—C9—C10	123.1 (3)
C11—C3—H3	113.8	O1—C10—C4	128.0 (3)

C11—C3—C4	108.6 (2)	O1—C10—C9	128.5 (3)
C3—C4—H4	113.6	C9—C10—C4	103.5 (2)
C5—C4—C3	102.3 (2)	O2—C11—C3	128.0 (3)
C5—C4—H4	113.6	O2—C11—C8	127.0 (3)
C10—C4—C3	110.0 (2)	C3—C11—C8	104.9 (2)
C10—C4—H4	113.6	C8—C12—H12A	110.7
C10—C4—C5	102.8 (2)	C8—C12—H12B	110.7
C1—C5—C4	104.0 (2)	C8—C12—C13	105.4 (3)
C1—C5—H5	115.5	H12A—C12—H12B	108.8
C1—C5—C6	102.9 (2)	C13—C12—H12A	110.7
C4—C5—H5	115.5	C13—C12—H12B	110.7
C4—C5—C6	101.7 (2)	C12—C13—H13A	110.4
C6—C5—H5	115.5	C12—C13—H13B	110.4
C5—C6—H6	117.2	H13A—C13—H13B	108.6
C5—C6—C7	103.8 (2)	C14—C13—C12	106.6 (3)
C5—C6—C9	107.1 (2)	C14—C13—H13A	110.4
C7—C6—H6	117.2	C14—C13—H13B	110.4
C7—C6—C9	90.5 (2)	O3—C14—C9	125.5 (3)
C9—C6—H6	117.2	O3—C14—C13	126.4 (3)
C2—C7—H7	117.5	C9—C14—C13	108.1 (3)
C2—C7—C8	107.2 (2)		

C1—C2—C3—C4	33.3 (3)	C7—C2—C3—C4	−73.5 (2)
C1—C2—C3—C11	146.1 (2)	C7—C2—C3—C11	39.3 (3)
C1—C2—C7—C6	−33.1 (3)	C7—C6—C9—C8	0.1 (2)
C1—C2—C7—C8	−127.7 (3)	C7—C6—C9—C10	111.1 (2)
C1—C5—C6—C7	33.4 (3)	C7—C6—C9—C14	−107.5 (3)
C1—C5—C6—C9	128.3 (3)	C7—C8—C9—C6	−0.1 (2)
C2—C1—C5—C4	52.9 (3)	C7—C8—C9—C10	−105.1 (3)
C2—C1—C5—C6	−52.8 (3)	C7—C8—C9—C14	119.8 (3)
C2—C3—C4—C5	−0.3 (3)	C7—C8—C11—O2	−150.2 (3)
C2—C3—C4—C10	108.4 (3)	C7—C8—C11—C3	30.7 (3)
C2—C3—C11—O2	136.2 (3)	C7—C8—C12—C13	−80.9 (4)
C2—C3—C11—C8	−44.7 (3)	C8—C9—C10—O1	−116.4 (3)
C2—C7—C8—C9	103.4 (2)	C8—C9—C10—C4	63.1 (3)
C2—C7—C8—C11	−5.6 (3)	C8—C9—C14—O3	169.8 (3)
C2—C7—C8—C12	−146.5 (3)	C8—C9—C14—C13	−12.0 (3)
C3—C2—C7—C6	74.0 (3)	C8—C12—C13—C14	−27.4 (4)
C3—C2—C7—C8	−20.6 (3)	C9—C6—C7—C2	−107.9 (2)
C3—C4—C5—C1	−33.0 (3)	C9—C6—C7—C8	−0.1 (2)
C3—C4—C5—C6	73.6 (3)	C9—C8—C11—O2	115.9 (4)
C3—C4—C10—O1	117.1 (3)	C9—C8—C11—C3	−63.2 (3)
C3—C4—C10—C9	−62.4 (3)	C9—C8—C12—C13	19.8 (3)
C4—C3—C11—O2	−115.5 (4)	C10—C4—C5—C1	−147.2 (2)
C4—C3—C11—C8	63.6 (3)	C10—C4—C5—C6	−40.5 (3)
C4—C5—C6—C7	−74.1 (3)	C10—C9—C14—O3	42.0 (5)
C4—C5—C6—C9	20.8 (3)	C10—C9—C14—C13	−139.8 (3)
C5—C1—C2—C3	−52.9 (3)	C11—C3—C4—C5	−108.8 (3)

C5—C1—C2—C7	52.9 (3)	C11—C3—C4—C10	−0.1 (3)
C5—C4—C10—O1	−134.5 (3)	C11—C8—C9—C6	104.2 (3)
C5—C4—C10—C9	46.0 (3)	C11—C8—C9—C10	−0.8 (3)
C5—C6—C7—C2	−0.1 (3)	C11—C8—C9—C14	−135.9 (3)
C5—C6—C7—C8	107.6 (2)	C11—C8—C12—C13	144.1 (3)
C5—C6—C9—C8	−104.5 (2)	C12—C8—C9—C6	−125.0 (3)
C5—C6—C9—C10	6.5 (3)	C12—C8—C9—C10	130.0 (3)
C5—C6—C9—C14	147.9 (3)	C12—C8—C9—C14	−5.0 (3)
C6—C7—C8—C9	0.1 (2)	C12—C8—C11—O2	−7.6 (5)
C6—C7—C8—C11	−108.9 (2)	C12—C8—C11—C3	173.3 (3)
C6—C7—C8—C12	110.2 (3)	C12—C13—C14—O3	−157.1 (3)
C6—C9—C10—O1	148.6 (3)	C12—C13—C14—C9	24.8 (4)
C6—C9—C10—C4	−31.9 (3)	C14—C9—C10—O1	9.3 (5)
C6—C9—C14—O3	−91.8 (4)	C14—C9—C10—C4	−171.3 (3)
C6—C9—C14—C13	86.4 (3)		