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An Unusual Rearrangement in the Boron-Trifluoride-Promoted Reaction of Tetracyclo[6.3.0.$0^{4,11}$.$0^{5,9}$]undecane-2,7-dione Monoethylene Acetal with Ethyl Diazoacetate

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Abstract. 4,10-Ethylenedioxypentacyclo[6.3.0.$0^{2,6}$.$0^{3,10}$.$0^{5,9}$]undecane-4-oxyacetic acid, $C_{15}H_{18}O_5$, $M_r = 278\cdot31$, triclinic, $P\bar{1}$, $a = 6\cdot705\ (1)$, $b = 7\cdot945\ (1)$, $c = 13\cdot087\ (2)$ Å, $\alpha = 105\cdot73\ (1)$, $\beta = 101\cdot27\ (1)$, $\gamma = 98\cdot73\ (1)°$, $V = 642\cdot4\ (2)$ Å^3, $Z = 2$, $D_x = 1\cdot439$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 1\cdot01$ cm^{-1}, $F(000) = 296$, $T = 295$ K, $R = 0\cdot0470$ for 1968 observed reflections. The cage structure is composed of six fused five-membered rings with three recognizable norbornane moieties, a seven-membered heterocyclic ring in a twist-chair conformation and an extended side chain. The five-membered rings of the norbornane moieties are twisted from the normal envelope conformation toward half-chair. There is an intermolecular hydrogen bond involving the disordered side chain. Molecular-mechanics calculations estimate the angle and torsional strains to be 163·1 and 121·4 kJ mol^{-1}, respectively.

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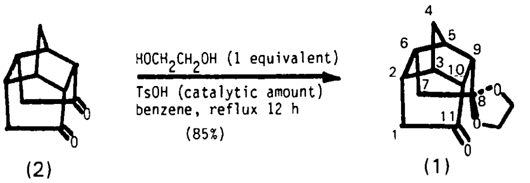
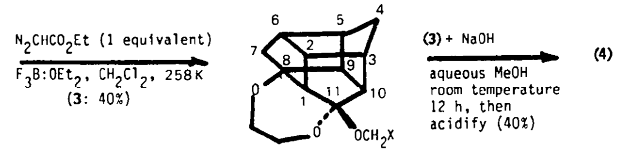
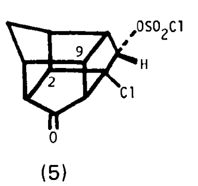
Introduction. As part of a program that involves the synthesis and chemistry of novel, substituted cage compounds (Marchand, 1988), the boron-trifluoride-promoted reaction of tetracyclo-[6.3.0.0^{4,11}.0^{5,9}]undecane-2,7-dione monoethylene acetal (1) with ethyl diazoacetate was studied. The starting material was synthesized by reacting tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecane-2,7-dione (2) (Wenkert & Yoder, 1970; Butler & Munshaw, 1981), with one equivalent of ethylene glycol. The reaction was performed in refluxing benzene in the presence of a catalytic amount of *p*-toluenesulfonic acid. A flask fitted with a Dean–Stark tube was utilized to remove water by continuous azeotropic distillation as the reaction progressed. Reaction of a methylene chloride solution of (1) with ethyl diazoacetate (one equivalent) in methylene chloride at 258 K in the presence of boron trifluoride etherate afforded a single product, (3), m.p. 330–331 K in 40% yield. This compound arises from the formation of a new C(1) to C(8) bond in (1) followed by reaction of the carbonyl. Hydrolysis of ester (3) was carried out by stirring a solution of (3) in excess methanolic sodium hydroxide at room temperature for 12 h. Subsequent acidification of the reaction mixture afforded the corresponding acid, (4), in 84% yield. X-ray diffraction was used to establish unequivocally the structure of (4).



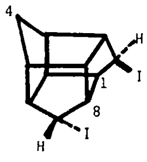
(2) (1)



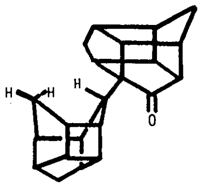
(3) (X = CO$_2$Et)
(4) (X = CO$_2$H)



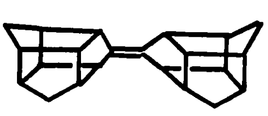
(5)



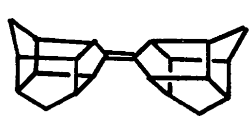
(6)



(7) (mixture of isomers)



(8*a*)



(8*b*)

Experimental. Good quality single crystals of (4), m.p. 423–424 K, were obtained by careful recrystallization from ethyl acetate. A colorless crystal of dimensions 0·30 × 0·38 × 0·43 mm was mounted on a Nicolet *R*3*m*/μ update of a *P*2$_1$ diffractometer; data collected in the ω-scan mode ($3 \leq 2\theta \leq 50°$), variable scan rate of 4 to 29·5° min^{-1}, graphite-monochromated Mo *K*α radiation; lattice parameters from a least-squares refinement of 25 reflections ($26{\cdot}10 \leq 2\theta \leq 29{\cdot}50°$); monitored reflections $41\bar{2}$ and $0\bar{2}\bar{5}$ showed variations in intensities of less than ±2%. Intensity statistics consistent with space group $P\bar{1}$; 2263 independent reflections measured ($-7 \leq h \leq 7$, $-9 \leq k \leq 9$, $0 \leq l \leq 15$), equivalent reflections averaged ($R_{\rm int} = 0{\cdot}028$), 1968 with $I \geq 3\sigma(I)$; Lorentz–polarization corrections applied, ψ-scan-based empirical absorption correction (transmission factors 0·861 to 0·892); structure solved by direct methods and refined by anisotropic block-cascade least-squares techniques, all H atoms located in a difference map and refined with isotropic thermal parameters, O atoms of acid side chain disordered with O(5) and O(5*a*) positions refined to occupancy factors of 0·633 (5) and 0·36 (1), respectively; $R = 0{\cdot}0470$, $wR = 0{\cdot}0692$ for 264 parameters and 1968 reflections ($R = 0{\cdot}0529$ and $wR = 0{\cdot}0704$ for all 2263 independent reflections), $S = 1{\cdot}898$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}009$, largest peaks in a final difference map of $+0{\cdot}39$ and $-0{\cdot}29$ e Å^{-3}; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}00086F_o^2]^{-1}$. *SHELXTL* computer programs (Sheldrick, 1986) 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 contains a listing of atomic positional parameters and Table 2 interatomic distances and bond angles.*

Discussion. A drawing of compound (4) is shown in Fig. 1. The cage structure is composed of six fused five-membered rings, and three norbornane moieties can be recognized in the structure. This strained cage structure is of considerable interest, and X-ray structural data are available on a derivative of D$_3$-trishomocubane (5) (Tolstikov, Lerman, Galin, Struchkov & Andrianov, 1978), an iodo derivative (6) (Marchand, Chou, Ekstrand & van der Helm, 1976), compounds (7*a*) and (7*b*) (Watson, Nagl, Marchand & Deshpande, 1989) and compounds (8*a*) and (8*b*) (Watson, Nagl, Marchand, Deshpande & Reddy, 1988). Compound (6) contains a crystallo-

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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 52043 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table. 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

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

	x	y	z	U_{eq}
C(1)	4096 (3)	660 (2)	7761 (1)	37 (1)
C(2)	4147 (3)	1974 (3)	8907 (2)	51 (1)
C(3)	2957 (4)	3430 (3)	8645 (2)	55 (1)
C(4)	1198 (5)	3403 (4)	9222 (3)	78 (1)
C(5)	595 (4)	1366 (4)	8889 (2)	63 (1)
C(6)	2602 (3)	864 (3)	9360 (2)	61 (1)
C(7)	2333 (3)	−1054 (3)	8688 (2)	56 (1)
C(8)	2061 (3)	−660 (2)	7609 (1)	38 (1)
C(9)	562 (3)	694 (3)	7645 (2)	43 (1)
C(10)	1814 (3)	2380 (2)	7467 (2)	41 (1)
C(11)	3530 (2)	1707 (2)	6978 (1)	34 (1)
O(1)	1535 (2)	−2303 (2)	6740 (1)	43 (1)
C(12)	1221 (4)	−2252 (3)	5633 (2)	51 (1)
C(13)	2938 (3)	−1007 (2)	5458 (2)	47 (1)
O(2)	2728 (2)	787 (2)	5856 (1)	41 (1)
O(3)	5298 (2)	3059 (2)	7112 (1)	41 (1)
C(14)	4817 (3)	4347 (3)	6594 (2)	50 (1)
C(15)	6711 (3)	5673 (3)	6669 (2)	58 (1)
O(4)	8470 (2)	5249 (2)	6882 (1)	66 (1)
O(5)	6527 (4)	6842 (4)	6247 (3)	81 (2)
O(5*A*)	6770 (13)	7315 (9)	7142 (21)	182 (7)

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

C(1)—C(2)	1·569 (2)	C(1)—C(8)	1·532 (2)
C(1)—C(11)	1·514 (3)	C(2)—C(3)	1·575 (3)
C(2)—C(6)	1·564 (3)	C(3)—C(4)	1·521 (4)
C(3)—C(10)	1·519 (3)	C(4)—C(5)	1·524 (4)
C(5)—C(6)	1·524 (4)	C(5)—C(9)	1·565 (3)
C(6)—C(7)	1·502 (3)	C(7)—C(8)	1·511 (3)
C(8)—C(9)	1·578 (3)	C(8)—O(1)	1·421 (2)
C(9)—C(10)	1·568 (3)	C(10)—C(11)	1·519 (3)
C(11)—O(2)	1·403 (2)	C(11)—O(3)	1·421 (2)
O(1)—C(12)	1·436 (3)	C(12)—C(13)	1·496 (3)
C(13)—O(2)	1·418 (2)	O(3)—C(14)	1·415 (3)
C(14)—C(15)	1·490 (3)	C(15)—O(4)	1·275 (3)
C(15)—O(5)	1·212 (4)		
C(2)—C(1)—C(8)	98·0 (1)	C(2)—C(1)—C(11)	103·6 (1)
C(8)—C(1)—C(11)	104·3 (1)	C(1)—C(2)—C(3)	105·2 (2)
C(1)—C(2)—C(6)	104·0 (1)	C(3)—C(2)—C(6)	103·3 (2)
C(2)—C(3)—C(4)	106·0 (2)	C(2)—C(3)—C(10)	97.8 (2)
C(4)—C(3)—C(10)	101·9 (2)	C(3)—C(4)—C(5)	94·4 (2)
C(4)—C(5)—C(6)	103·1 (2)	C(4)—C(5)—C(9)	105·5 (2)
C(6)—C(5)—C(9)	98·9 (2)	C(2)—C(6)—C(5)	99·2 (2)
C(2)—C(6)—C(7)	105·4 (2)	C(5)—C(6)—C(7)	103·6 (2)
C(6)—C(7)—C(8)	94·4 (2)	C(1)—C(8)—C(7)	103·3 (1)
C(1)—C(8)—C(9)	98·1 (1)	C(7)—C(8)—C(9)	106·2 (2)
C(1)—C(8)—O(i)	118·5 (2)	C(7)—C(8)—O(1)	109·0 (2)
C(9)—C(8)—O(1)	119·8 (1)	C(5)—C(9)—C(8)	102·7 (2)
C(5)—C(9)—C(10)	102·8 (1)	C(8)—C(9)—C(10)	105·3 (1)
C(3)—C(10)—C(9)	100·0 (2)	C(3)—C(10)—C(11)	103·0 (1)
C(9)—C(10)—C(11)	104·3 (1)	C(1)—C(11)—C(10)	95·6 (2)
C(1)—C(11)—O(2)	119·2 (1)	C(10)—C(11)—O(2)	109·7 (1)
C(1)—C(11)—O(3)	108·5 (1)	C(10)—C(11)—O(3)	114·8 (1)
O(2)—C(11)—O(3)	108·7 (1)	C(8)—O(1)—C(12)	118·8 (2)
O(1)—C(12)—C(13)	113·7 (1)	C(12)—C(13)—O(2)	110·0 (2)
C(11)—O(2)—C(13)	118·4 (1)	C(11)—O(3)—C(14)	112·9 (1)
O(3)—C(14)—C(15)	112·2 (2)	C(14)—C(15)—O(4)	117·7 (2)
C(14)—C(15)—O(5)	119·2 (2)	O(4)—C(15)—O(5)	120·0 (2)

graphic twofold axis passing through C(4) and the C(1)—C(8) bond. In compound (5) there is an approximate threefold axis passing through C(2) and C(9) in spite of the differences in hybridization and substitution. In norbornane structures, *e.g.* C(2)C(3)C(10)C(9)C(5)C(6)C(4), the ethane bonds of the C(2)—C(6) and C(9)—C(10) type are usually significantly longer than the methano bridge and the connecting bonds, *e.g.* C(3)—C(4) and C(2)—C(3) types, respectively. In the cage structure (4) there are three recognizable norbornane moieties which share nine common bonds. Six of these involve ethane-type bonds of one norbornane and connecting bonds of another, and these six bonds average 1·570 (5) Å. Three pure methano-type bonds average 1·513 (8) Å while three shared connecting-bond types, *e.g.* C(3)—C(10) types, average 1·525 (6) Å. These data are consistent with the distances reported for compounds (5), (6), (7*a*), (7*b*), (8*a*), (8*b*) and norbornane (Dallinga & Toneman, 1968). In norbornane and simply substituted norbornane derivatives, the five-membered rings exhbit ideal envelope conformations; however, the fusion of a norbornyl moiety to the cage structure (4) leads to a considerable twist toward half-chair (Toromanoff, 1980). Torsion angles of the type C(3)C(2)C(6)C(5) range from 15·9 (2) to 20·3 (2)° compared with the value of 0° for an ideal envelope. The pseudorotation phase angles (Altona, Geise & Romers, 1968) range from −8 to +7° for the six five-membered rings.

The acid side chain is in an extended conformation, and the C(11)O(3)C(14)C(15)O(4)O(5) grouping is planar, r.m.s.d. 0·08 Å. The two O atoms are disordered by a rotation about the C(14)—C(15) bond. The C(15)—O(5) distance is 1·212 (4) Å, which is reasonable for a C=O functionality. The occupancy factor refined to about 0·67. The C(15)—O(4) and C(15)—O(5*a*) distances refined to 1·275 (3) and 1·274 (8) Å, respectively. The O(5*a*) occupancy factor refined to about 0·33. The O(5) and O(5*a*) sites can be resolved, but peaks in the O(4) direction cannot. The acidic H atom of O(4) forms a hydrogen bond with O(1) of an adjacent molecule, O(4)···O(1)(1+*x*,1+*y*,*z*) = 2·678 (3), H(4)···O(1) = 1·67 (3) Å and O(4)—H(4)···O(1) = 172 (3)°. The

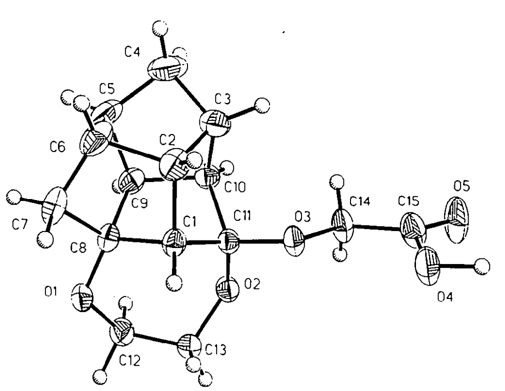

Fig. 1. Drawing of compound (4) with thermal ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary size.

seven-membered heterocyclic ring adopts a twist-chair conformation (Toromanoff, 1980).

All internal bond angles in the cage structure are less than 109°, and eight angles are less than 100°. A molecular-mechanics calculation (Allinger & Yu, 1980) with the acid side chain at C(11) replaced with an OH group gave a heat of formation of $-397·4$ kJ mol^{-1} and a strain energy of 281·6 kJ mol^{-1} with 163·1 kJ mol^{-1} partitioned to angle strain and 121·4 kJ mol^{-1} to torsional strain. The cage structure contains a number of eclipsed bonds which leads to the large torsional contribution. In general, the bond-length pattern resembles that found experimentally; however, the differences between the calculated bond types are not as great as those observed, *i.e.* ethane bonds average 1·557 (5) Å, methano bonds 1·533 (5) Å and the pure connecting bonds 1·540 (4) Å. Librational correction of X-ray bond lengths leads to poorer agreement between calculated and observed values.

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Structure of 3-(4-Hydroxy-2-methylphenyl)-3-phenylphthalide

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Abstract. $C_{21}H_{16}O_3$, $M_r = 316·36$, monoclinic, $P2_1/c$, $a = 7·4826$ (8), $b = 19·446$ (2), $c = 11·119$ (2) Å, $\beta = 102·041$ (8)°, $V = 1582·3$ (4) Å^3, $Z = 4$, $D_x = 1·328$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·72$ mm^{-1}, $F(000) = 664$, $T = 293$ K, final $R = 0·040$ for 2894 unique observed reflections. The overall conformation of the title molecule is very similar to the conformation of its isomer, 3-(2-hydroxy-4-methylphenyl)-3-phenylphthalide [Skrzat, Roszak & Engelen (1988). *Acta Cryst.* C**44**, 537–540]. The tetrahedral geometry of the central C atom is significantly distorted: valence angles range from 102·6 (1) to 116·2 (1)°. The orientation of the substituted phenyl ring relative to the other parts of the molecule is slightly different in the two isomers but their *ortho* substituents are similarly oriented. The packing is determined by an intermolecular hydrogen bond between the hydroxyl and carbonyl groups with O···O = 2·789 (2) Å and ∠O—H···O = 176 (2)°.

Introduction. The present paper is a continuation of our X-ray crystallographic studies of 3,3-diarylphthalides undertaken to confirm their chemical structure and to obtain more information on relationships between their structures and reactivity. The title compound (II) is one of the isomers obtained in the condensation of 2-benzoylbenzoic acid with *m*-cresol. Previously it was reported (Dutt, 1940; Rumiński, 1973) that only isomer (II) was obtained in this reaction. Isomer (I) [3-(2-hydroxy-4-

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