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Key indicators

Single-crystal X-ray study

 $T = 293\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$ R factor = 0.099 wR factor = 0.312

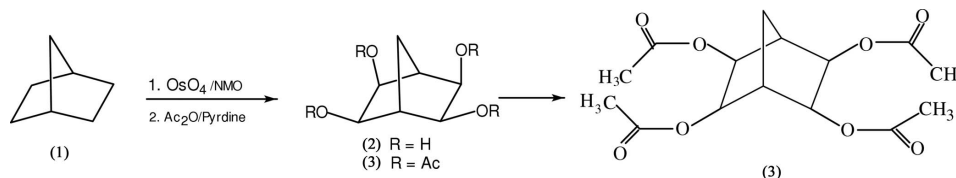
Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.(2*RS*,3*SR*,5*RS*,6*SR*)-Bicyclo[2.2.1]heptane-2,3,5,6-tetrayl tetraacetate

In the title compound, $\text{C}_{15}\text{H}_{20}\text{O}_8$, the bicyclo system has a six-membered ring with a boat conformation. Three types of intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and one intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond are present in the crystal structure.

Comment

The effect of cyclitols on human and plant mitotic cells has been documented (Sable & Katchian, 1967). Norbornanetetrol (2) closely resembles myo-inositol and DL-(1,2,3,4)-cyclohexanetetrol in its effects on these cells. It has been reported that all these compounds induce an increase in chromosome contraction during prophase, and their effect is still visible in anaphase chromatids. Norbornanetetraacetate (3) is derivative of norbornanetetrol (2). The crystal structure of (3) was investigated in order to confirm its formation and understand its reactivity. The molecular structure of (3) is shown in Fig. 1.



Two non-planar five-membered rings constitute the bicyclo system. The $\text{O}=\text{C}$ bond lengths (Table 1) agree with values reported in the literature [1.193 (3) Å in 1-acetyl-4-(*p*-chlorobenzylidene-amino)-3-ethyl-4,5-dihydro-1H-1,2,4-triazol-5-one (Çoruh, Kahveci, Şaşmaz, Ağar, Kim & Erdönmez, 2003) and 1.197 (3) Å in 1-acetyl-4-(*p*-chlorobenzylideneamino)-3-methyl-4,5-dihydro-1H-1,2,4-triazol-5-one (Çoruh, Kahveci, Şaşmaz, Ağar & Kim, 2003)]. The $\text{O}-\text{C}$ bond lengths are within normal ranges. The bicyclo[2.2.1]-heptane system often possesses a twist about the $\text{C}3\cdots\text{C}1$ vector. This twisting can be seen from the $\text{C}3-\text{C}7-\text{C}6-\text{C}2$ and $\text{C}3-\text{C}5-\text{C}4-\text{C}2$ torsion angles of 8.1 (5) and 7.6 (4)°, respectively. The two five-membered rings have envelope conformations, with atom C1 displaced by 0.88 (5) Å from the $\text{C}3/\text{C}7/\text{C}6/\text{C}2$ plane and by 0.86 (5) Å from the $\text{C}3/\text{C}5/\text{C}4/\text{C}2$ plane.

In the bicyclo[2.2.1]heptane fragment of (Fig. 1), the angles between planes *A* ($\text{C}3/\text{C}1/\text{C}2$), *B* ($\text{C}3/\text{C}7/\text{C}6/\text{C}2$) and *C* ($\text{C}3/\text{C}5/\text{C}4/\text{C}2$) are as follows: $A/B = 57.34$ (29)°, $A/C = 54.82$ (27)° and $B/C = 67.95$ (23)°. The $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bond parameters are given in Table 2.

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Experimental

Compound (3) was prepared according to the literature method (Sable & Katchian, 1967; Shealy & Clayton, 1969; Van Rheezen *et al.*, 1976). A 100 ml three-necked round-bottomed flask, equipped with a magnetic stirrer and a nitrogen inlet, was charged with *N*-methylmorpholine-*N*-oxide (NMO; 1.54 g, 11.40 mmol), water (6 ml) and acetone (4 ml). To this solution were added OsO₄ (*ca* 36 mg, 0.143 mmol) and norbornadiene (1) (500 mg, 5.43 mmol). The resulting mixture was stirred vigorously under nitrogen at room temperature overnight. After 24 h the reaction was complete. Sodium bisulfite (0.5 g) and Florisil (2.0 g) slurried in water (8 ml) were added, the slurry was stirred for 10 min, and the mixture was filtered through a pad of 0.5 g of Celite in a 50 ml sintered-glass funnel. The Celite cake was washed with acetone (4 × 40 ml). The filtrate was neutralized to pH 7 with H₂SO₄. The organic layer was removed *in vacuo*. The pH of the resulting aqueous solution was adjusted to pH 5 with sulfuric acid, and the tetrol (2) was separated from *N*-methylmorpholine bisulfate by extraction with ethyl acetate (4 × 40 ml). The combined ethyl acetate extracts were washed with 10 ml of 25% NaCl solution and two or three times with water, and dried (Na₂SO₄). Evaporation of the solvent gave 650 mg of tetrol (2) (75%). Tetrol (2) was acetylated as described in the literature (Sable & Katchian, 1967). Recrystallization of (3) from methylene chloride–pentane (1:4) gave colourless crystals (m.p. 406–407 K).

Crystal data

C₁₅H₂₀O₈
M_r = 328.31
 Monoclinic, *P*2₁/*c*
a = 13.5589 (15) Å
b = 11.6620 (12) Å
c = 10.7025 (11) Å
 β = 101.758 (2)°
V = 1656.8 (3) Å³
Z = 4

D_x = 1.316 Mg m⁻³
 Mo Kα radiation
 Cell parameters from 1210 reflections
 θ = 1.5–28.0°
 μ = 0.11 mm⁻¹
T = 293 (2) K
 Prism, colourless
 0.35 × 0.34 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 7428 measured reflections
 3216 independent reflections

1210 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.124
 θ_{max} = 28.0°
h = -12 → 17
k = -12 → 15
l = -12 → 12

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.099
wR (*F*²) = 0.312
S = 0.93
 3216 reflections
 208 parameters

H-atom parameters constrained
w = 1/[σ²(*F*_o²) + (0.1627*P*)²]
 where *P* = (*F*_o² + 2*F*_c²)/3
 (Δ/σ)_{max} = 0.021
 Δρ_{max} = 0.30 e Å⁻³
 Δρ_{min} = -0.44 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O8—C9	1.182 (5)	O7—C8	1.197 (6)
C11—O6	1.186 (6)	O5—C10	1.194 (7)
O2—C5—C3	104.4 (4)	O2—C5—C4	111.2 (4)
C2—C6—C7—C3	8.1 (5)	C2—C4—C5—C3	7.6 (4)

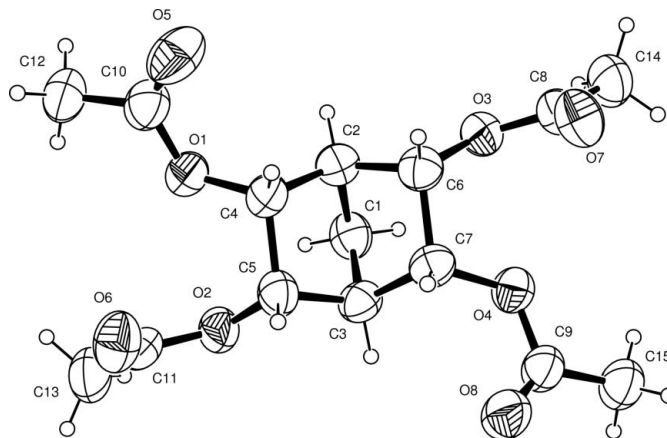


Figure 1

An ORTEP-3 (Farrugia, 1997) drawing of (I), with the atom numbering scheme. Displacement ellipsoids are drawn at the 70% probability level.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C1—H1 <i>B</i> ...O3	0.97	2.51	2.846 (6)	100
C6—H6...O5 ⁱ	0.98	2.51	3.428 (7)	156
C13—H13 <i>B</i> ...O6 ⁱⁱ	0.96	2.51	3.287 (8)	138
C14—H14 <i>B</i> ...O8 ⁱⁱⁱ	0.96	2.59	3.476 (8)	154

Symmetry codes: (i) -*x* + 1, -*y* + 2, -*z*; (ii) -*x* + 1, -*y* + 1, -*z*; (iii) -*x* + 2, *y* + ½, -*z* + ½.

All H atoms were located in a difference synthesis and refined as riding [C—H = 0.96–0.98 Å and *U*_{iso}(H) = 1.2–1.5*U*_{eq}(C)].

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT and SHELXTL (Sheldrick, 1997*b*); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997*a*); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997*a*); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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