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(1α,3α,5α)-1,3,5-Trimethyl-1,3,5-cyclohexanetricarboxylic Acid Acetonitrile Solvate

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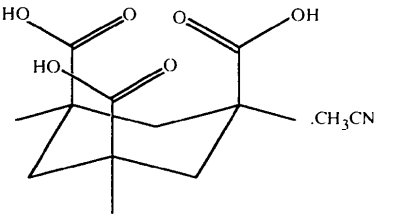
Abstract

In the title compound, $C_{12}H_{18}O_6.CH_3CN$, both components have crystallographically imposed C_3 symmetry. The hydrogen bonds between the three carboxylic acid groups of (1α,3α,5α)-1,3,5-trimethyl-1,3,5-cyclohexanetricarboxylic acid (Kemp's triacid) and three other triacid moieties make a three-dimensional hydrogen-bonding network, producing large intermolecular cavities.

Comment

During a chemical study on the interactions between metal ions and several kinds of (1α,3α,5α)-1,3,5-trimethyl-1,3,5-cyclohexanetricarboxylic acid (Kemp's triacid) (Hirose *et al.*, 1995; Baldwin *et al.*, 1996), the crystal structures of the acid and an adduct were determined. The structure determined for the acid was disordered [trigonal, a = 25.152 (2), c = 12.592 (2) Å, Z = 18, $R\bar{3}$] and is yet to be published. This paper reports the acetonitrile adduct, (I), with the cyclohexane ring having a chair conformation. There is a hydrogen-bonding system between O11 and a neighbouring O10 atom at $(1-x, 1-y, -z)$ [O···O = 2.651 (3) Å and O11—H11···O10 = 175.4 (3)°]. There are no links between the acetonitrile and the triacid.



(I)

For the compound under investigation, each carboxylic acid group of each Kemp's triacid formed intermolecular hydrogen bonds with a centrosymmetrically related neighbour. A three-dimensional interconnecting network (Fig. 2), formed by hydrogen bonds, links neighbouring acid groups to give six-membered sets of Kemp's acids, in alternating hydrogen-bond links above and below the *ab* plane around the lattice points (0,0,0), $(\frac{2}{3},\frac{1}{3},\frac{1}{3})$ and $(\frac{1}{3},\frac{2}{3},\frac{2}{3})$. The acetonitrile lies outside this framework, in a void of 172 Å^3. Corey–Pauling–Kendrew (CPK) space-filling model studies showed that large intermolecular cavities would be formed within the crystal structure if this compound kept its C_3 symmetry. The introduction of acetonitrile is a stabilizing influence, as it would fill this void. The structure of the disodium tetrahydrate has also been determined (Bencini *et al.*, 1994).

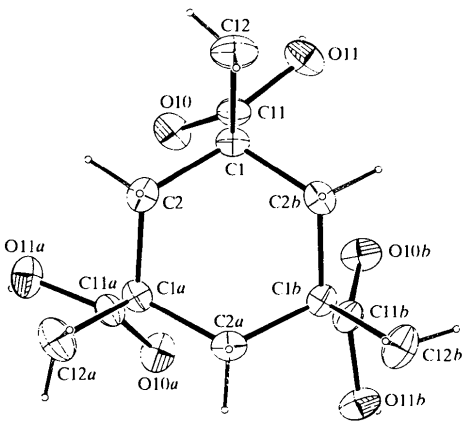


Fig. 1. *PLATON*96 (Spek, 1996) view of the (1α,3α,5α)-1,3,5-trimethyl-1,3,5-cyclohexanetricarboxylic acid of the title compound, with displacement ellipsoids at the 20% probability level. [Symmetry codes: (*a*) $1-y, x-y, z$; (*b*) $1-x+y, 1-x, z$.]

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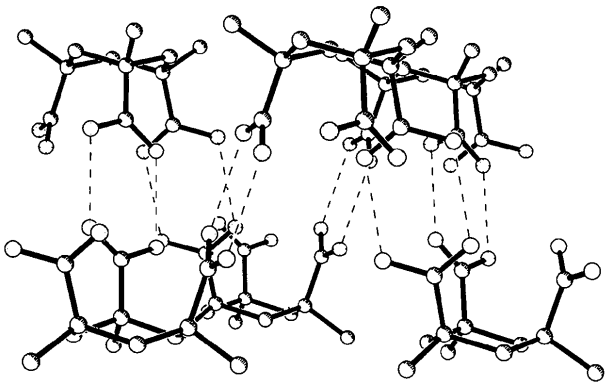

Fig. 2. Part of the three-dimensional interconnecting hydrogen-bonded network of Kemp's acid molecules. The acetonitrile molecules, which lie outside this framework, have been omitted.

Experimental

Crystals of (I) were grown by recrystallization of (1α,3α,5α)-1,3,5-trimethyl-1,3,5-cyclohexanetricarboxylic acid from acetonitrile/water solution (1:1 *v/v*). The unadducted compound was first prepared by Kemp & Petrakis (1981). Its structure was confirmed in a space group without threefold crystallographic symmetry by Rebek *et al.* (1985) [a = 8.471 (2), b = 12.138 (3), c = 12.902 (3) Å, β = 101.8 (2)°, $P2_1/n$] and by Chan *et al.* (1991) [a = 8.465 (2), b = 12.136 (2), c = 12.892 (2) Å, β = 101.78 (1)°, $P2_1/n$].

Crystal data

$C_{12}H_{18}O_6.C_2H_3N$	Mo $K\alpha$ radiation
M_r = 299.32	λ = 0.71073 Å
Trigonal	Cell parameters from 25 reflections
$R\bar{3}$	θ = 10–14°
a = 8.972 (1) Å	μ = 0.098 mm^{-1}
c = 34.15 (2) Å	T = 293 (2) K
V = 2380.7 (14) Å^3	Large block
Z = 6	0.47 × 0.47 × 0.40 mm
D_x = 1.253 Mg m^{-3}	Colourless
D_m not measured	

Data collection

MicroVAX-controlled CAD-4 diffractometer	R_{int} = 0.045
$2\theta/\omega$ scans	θ_{max} = 25°
Absorption correction: none	$h = -10 \rightarrow 10$
3043 measured reflections	$k = -10 \rightarrow 9$
933 independent reflections	$l = -40 \rightarrow 40$
641 reflections with $I > 2\sigma(I)$	3 standard reflections
	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 6.17P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.152	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
S = 1.096	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
933 reflections	
92 parameters	
H atoms refined by a mixture of independent and constrained refinement	Extinction correction: none
	Scattering factors from *International Tables for Crystallography* (Vol. C)

The title structure was solved using direct methods.

Data collection: *SDP* (Frenz, 1985). Cell refinement: *SDP*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *PLATON*96 (Spek, 1996) and *SHELXTL/PC* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1202). Services for accessing these data are described at the back of the journal.

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