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Four-centre hydrogen bonds: a triethanolamine–triethanolamine oxide complex

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Key indicators

Single-crystal X-ray study
T = 168 K
Mean σ(C–C) = 0.010 Å
R factor = 0.061
wR factor = 0.134
Data-to-parameter ratio = 6.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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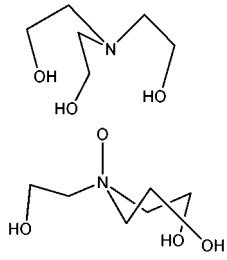
The title complex, $((HOCH_2CH_2)_3N)(HOCH_2CH_2)_3NO)$ or $C_6H_{15}NO_4{\cdot}C_6H_{15}NO_3$, has the amine–oxide O atom trifurcated by a trigonal 'cap' of hydrogen bonds to the hydroxyl H atoms of the triethanolamine lying on the same threefold axis. The amine–oxide hydroxyl H atoms are hydrogen bonded to three adjacent triethanolamine O atoms, completing a three-dimensional polymeric network.

Comment

A few crystals of [triethanolamine][triethanolamine oxide], (I), were found in an vessel used for an attempted metal oxide triethanolamine complex synthesis. The crystal quality was poor, but adequate data were extracted to confirm the structure using the *SMART*/*SAINT* processing system (Siemens, 1996).



(I)

The structure consists of individual molecules with crystallographically imposed threefold symmetry, with the axis passing through the two N atoms and the amine oxide O atom (Fig. 1). The feature of interest concerns the novel trigonal hydrogen bonding 'cap' [O1—H1···O3 O···O 2.705 (7) Å and O—H···O 169°]. There are few classical trifurcated (four-centre) O—H···O acceptors (Desiraju & Steiner, 1999). The more usual arrangement for ethanolamine hydroxyl H atoms, even in trigonal space groups, is for the bonds to be outwards from the central N atom (Parkanyi, *et al.*, 1996; Mootz *et al.*, 1989, 1990). In the triethanolamine structure (Mootz *et al.*, 1989), discrete cage-like dimers are formed in this way, retaining the $\overline{3}$ point symmetry. Many of the reported structures contain singly protonated triethanolamine cations. A further trigonally related set of hydrogen bonds [O2—H2···O1: O···O 2.696 (7) Å and O—H···O 176°; O1 at $-2/3+x$, $-1/3+y$, $-1/3+z$] to the ethanolamine O atoms complete the unique three-dimensional polymeric structure. A survey of the literature (Allen & Kennard, 1993; Cambridge Crystallographic Data Centre, 2002) shows no other triethanolamine oxide compounds, but there are several similar neutral quaternary nitrogen compounds. Many of these

have the oxide oxygen involved in hydrogen bonds, mostly with lattice water molecules: O···H contacts range from 1.70 to 2.06 Å. The mean distances and angles for the 18 relevant hits are O···H 1.90 Å, N—O 1.397 Å, O—H···N 169° and C—N—O 109.5° compared with the mean values here of 1.87 Å, 1.42 (1) Å, 173° and 108.5 (5)°, respectively.

Experimental

The title compound was a by-product from a synthesis of a metal oxide triethanolamine sol using triethanolamine as the solvent and reactant, that had been set aside for six months. Crystals formed in the triethanolamine.

Crystal data

$C_6H_{15}NO_4{\cdot}C_6H_{15}NO_3$
M_r = 314.38
Hexagonal, R_3 (hexagonal axes)
a = 12.065 (5) Å
c = 9.633 (8) Å
V = 1214.4 (13) Å^3
Z = 3
D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 330 reflections
θ = 2.9–21.9°
μ = 0.11 mm^{-1}
T = 168 (2) K
Plate, colourless
0.29 × 0.20 × 0.02 mm

Data collection

Bruker $P4$ diffractometer
ω scans
Absorption correction: none
1170 measured reflections
422 independent reflections
299 reflections with $I > 2\sigma(I)$
R_{int} = 0.211
θ_{max} = 26.4°
$h = -14 \rightarrow 13$
$k = -15 \rightarrow 13$
$l = -8 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.134
S = 1.04
422 reflections
67 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.038P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.016 (5)
Absolute structure: not determined

All H atoms were included in the riding-model approximation, with isotropic displacement parameters constrained to 1.2 times that of the equivalent U_{eq} of their parent atom. Friedel pairs were averaged.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996) and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 in *WinGX* (Farrugia, 1997, 1999); software used to prepare material for publication: *SHELXL*97.

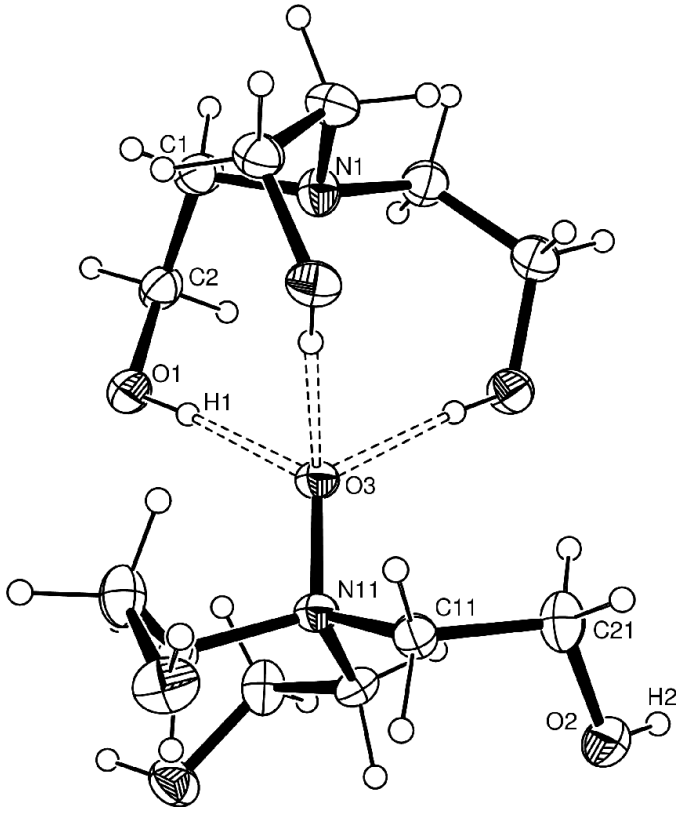


Figure 1
The molecular structure of (I) (Farrugia, 1997, 1999). Displacement ellipsoids are drawn at the 30% probability level. H atoms have arbitrary radii.

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