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Tris(2-succinimidoethyl)amine hydrate (1/0.075)

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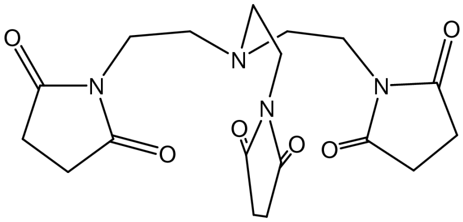
Key indicators

Single-crystal X-ray study
T = 210 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.071
wR factor = 0.144
Data-to-parameter ratio = 13.1

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

The title compound, $C_{18}H_{24}N_4O_6\cdot0.075H_2O$, (I), was prepared as part of a research programme to prepare and identify new tripodal ligands which exhibit instability at low pH, thereby releasing coordinated metal cations under such conditions. Each molecule lies on a crystallographic threefold axis with the three arms arranged in the form of a claw. Angles within the five-membered succinimide ring, where the r.m.s. deviation of the five atoms from the least-squares ring plane is only 0.004 Å, vary from 104.8 (3)° at a methylene C atom to 112.7 (3)° at the formally sp^2 N atom. The structure contains a partially occupied disordered water molecule, the source of which is presumably the water produced as a by-product of the synthetic reaction.



(I)

Experimental

Tris(2-aminoethylamine) (0.146 g, 1.00 mmol) and succinic anhydride (0.300 g, 3.00 mmol) were heated under reflux in benzene (70 ml) for 12 h. Upon cooling, colourless hexagonal crystals of the title compound were formed.

Crystal data

$C_{18}H_{24}N_4O_6\cdot0.075H_2O$
$M_r = 393.67$
Trigonal, $R\overline{3}$
$a = 13.200\ (2)$ Å
$c = 18.874\ (5)$ Å
$V = 2848.0\ (10)$ Å^3
$Z = 6$
$D_x = 1.376$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 29 reflections
$\theta = 25.1$–$29.6°$
$\mu = 0.11$ mm^{-1}
$T = 210\ (2)$ K
Hexagonal plate, colourless
0.39 × 0.32 × 0.06 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
ω/θ scans
1343 measured reflections
1128 independent reflections
738 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.137$

$\theta_{\max} = 25.0°$
$h = -15 \rightarrow 15$
$k = -7 \rightarrow 15$
$l = 0 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity decay: none

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.144$
$S = 1.28$
1128 reflections
86 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.023P)^2 + 9.981P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

At convergence of a refinement model containing no hydration, a peak of 0.66 e Å^{-3} was observed, approximately three times the size of other peaks and troughs in the difference Fourier synthesis. This feature was modelled isotropically as the O atom of a partially occupied water molecule. However, its occupancy of the $\overline{3}$ site at $(\frac{2}{3},\frac{1}{3},\frac{1}{3})$ refined to only 0.15 (2), corresponding to a 0.075-hydrate. Water H atoms were not located; methylene H atoms were introduced at geometrically calculated positions and allowed to ride 0.98 Å from their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *STADI*4 (Stoe & Cie, 1996); cell refinement: *STADI*4; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL*97 and *PLATON*.

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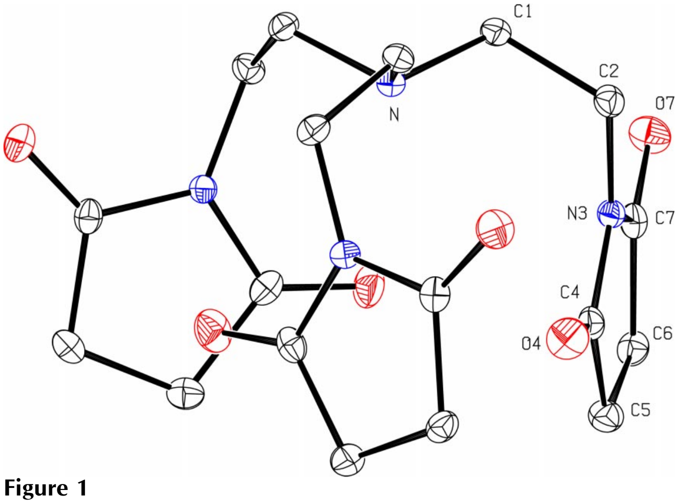

Figure 1
A view of the title compound showing the atom-numbering scheme and displacement ellipsoids drawn at the 20% probability level. For clarity, the partially occupied water molecule and all H atoms have been omitted.

References

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
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