Comment
The compound 1,10,13,19-tetraazatricycl[8.6.6.03'8]-docosa-3,5,7,14,20-dione was synthesized during our study of the transition metal complexes of cross-bridged tetraazamacrocycles (Hubin et al., 1998). Initially, our research centred on ethylene cross-bridged tetraazamacrocycles (Weisman et al., 1996), but it has recently expanded to include macrocycles bridged by other groups and macrocycles containing amide groups. Recent work by others (Denat et al., 1997) describes bis-amide macrocycles and corresponding reduced tetramine species that have been cross-bridged by five-atom groups derived from m-xylene and 2,6-dimethylpyridine bis-electrophiles. Although no crystal structures of these molecules were reported, we considered that a four-atom bridged analogue would be more effective as a rigid ligand. 1,10,13,19-Tetraazatricycl[8.6.6.03'8]docosa-3,5,7,triene-14,20-dione was synthesized as the monohydrate, (1), by the high-dilution reaction of o,a'-dibromo-o-xylene and trans-diprotected 5,12-dioxycyclam (Tomalia & Wilson, 1985) and its crystal structure determined.

No other cross-bridged 5,12-dioxocyclams have been structurally characterized to date, although we have recently determined the crystal structure of unbridged unsubstituted 1,4,8,11-tetraazacyclotetradecane-5,12-dione, (2), and its NiII complex (Hubin et al., 1999). The structures of several bis-linked substituted 5,12-dioxocyclam ligands and their NiII complexes have been determined (Dumas et al., 1995); these could also be regarded as bridged dioxocyclams.

In (1), the dioxocyclam has a U-shaped conformation, with the C==O groups pointing outwards and with the o-xyllyl bridge spanning the base of the U (Fig. 1). Compared with the unbridged species, this molecule has a clearly defined cavity in which the nitrogen donors appear to be well oriented for complexation. The bridges in the linked dioxocyclams characterized by Dumas et al. (1995) are much longer than that in (1) and the macrocycles have extended rather than U-shaped conformations, similar to those in the unsubstituted complex.

The asymmetric unit of (1) contains one water molecule, which is hydrogen bonded to one of the C==O groups [O11...O001 2.852(2) Å] and to a symmetry-
Fig. 1. View of the molecule of (1) showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as circles of arbitrary radii.

related water molecule [O001...O001] 2.991 (3) Å; symmetry code: (i) 2 - x, - y, 1 - z. Since the second hydrogen bond is across an inversion centre, there must be disorder in the water-molecule orientations.

Experimental

Compound (1) was obtained by the substitution of α,α'-di-bromo-α-xylene for its m-xylene analogue in the literature cross-bridging reaction of (2) (Denat et al., 1997). All other steps were followed according to the literature procedure; yields were 70–80%. X-ray quality crystals of (1) were obtained from the slow evaporation of a propan-2-ol solution.

Crystal data

C_{18}H_{26}N_4O_2.H_2O

M_r = 348.44

Monoclinic

P2_1/n

a = 9.9190 (10) Å

b = 12.898 (2) Å

c = 14.045 (2) Å

β = 101.983 (5)°

V = 1757.7 (4) Å^3

Z = 4

D_x = 1.317 Mg m^-3

D_m not measured

Data collection

Siemens SMART diffractometer

ω scans

Absorption correction:
multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.77, T_{max} = 0.96

10 113 measured reflections
4114 independent reflections

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 5802 reflections

θ = 3–20°

μ = 0.091 mm^-1

T = 220 (2) K

Block

0.5 × 0.5 × 0.4 mm

Colourless

Refinement

Refinement on F^2

R(F^2 > 2σ(F^2)) = 0.047

wR(F^2) = 0.137

S = 1.061

4114 reflections

234 parameters

H atoms riding

w = 1/σ^2(F^2) + (0.0708P)^2 + 0.3237P

where P = (F^2 + 2F_c^2)/3

(A/σ')max = 0.014

Apmax = 0.655 e Å^-3

Apmin = -0.338 e Å^-3

Extinction correction: none

Scattering factors from International Tables for Crystallography (Vol. C)

Compound (1) contains one lattice water molecule but its H atoms could not be located. The remaining H atoms were added at calculated positions and refined using a riding model, with isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1263). Services for accessing these data are described at the back of the journal.

References


