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trans-(3*S*,5*S*,10*R*,12*R*)-1,8-Bis(2-cyanoethyl)-*C*-*meso*-3,5,7,7,10,12,14,14-octamethyl-1,4,8,11-tetraazacyclotetradecane

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.143
Data-to-parameter ratio = 26.7

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

The structure determination of the tetraaza macrocyclic title compound, $C_{24}H_{46}N_6$, shows that its molecule represents a *meso*-isomer; it occupies a special position about the crystallographic inversion centre. The pendent ethylcyano arms are oriented above and below the N_4 plane and are directed away from the central cavity.

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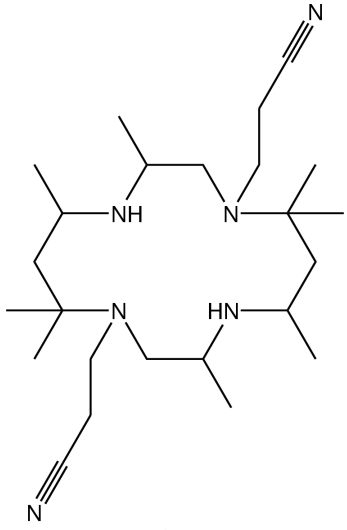
Comment

The 14-membered macrocyclic ring in the title compound, (I) (Fig. 1 and Table 1) reveals a symmetric substitution pattern at the N atoms. The molecule represents a *meso* isomer and is disposed about a crystallographic inversion centre: both chiral C3 and C5 atoms of the reference atom list have an *S* configuration, whereas their symmetry related counterparts, in positions 10 and 12 of the macrocycle, have *R* configurations. The amine N4—H bonds are directed into the cavity formed by the macrocyclic ring and hence contribute to the stabilization of the macrocycle by forming an intramolecular interaction with the tertiary amine atom on the 8 position of the macrocycle [atom $N1^i$ in crystallographic notation; symmetry code: (i) $1-x$, $1-y$, $-z$]. The geometric parameters associated with this interaction are N4—H4n···$N1^i$ 2.17 Å and N4···$N1^i$ 2.947 (2) Å, and the angle subtended at H4n is 140°. The ethylcyano groups are disposed above and below the N_4 plane, and are directed away from the central cavity so that the N1—C10—C11—C12 torsion angle is 175.94 (11)°.



(I)

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The investigation of the complexation potential of (I) with various metal ions is underway.

Experimental

The reduction of 3*S*,10*R*-*C*-*meso*-3,5,7,7,10,12,14,14-octamethyl-1,4,8,11-tetraazacyclotetradeca-4,11-diene (Curtis *et al.*, 1969) with $NaBH_4$ yielded an isomeric mixture of saturated macrocycles, Me_8[14]anes, which have been resolved into three distinct isomers (Bembi *et al.*, 1989). One of the isomers, L_B [according to the nomenclature of Bembi *et al.* (1989)], on reflux with excess $CH_2{=}CH{-}CN$ for 12 h, gave white crystals upon evaporation. These have been characterized as dicyanoethyl-Me_8[14]ane on the basis of elemental analysis, IR, ^{1}H and ^{13}C NMR, and mass spectral data (Hay & Bembi, 1982; Bembi *et al.*, 1989). Crystals for the X-ray analysis were grown by slow cooling (overnight) of a heated acrylonitrile solution of the compound.

Crystal data

$C_{24}H_{46}N_6$	D_x = 1.107 Mg m^{-3}
M_r = 418.67	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 23 reflections
a = 7.974 (3) Å	θ = 7.4–10.4°
b = 17.685 (4) Å	μ = 0.07 mm^{-1}
c = 9.007 (6) Å	T = 173 K
β = 98.74 (4)°	Block, colourless
V = 1255.4 (10) Å^3	0.48 × 0.44 × 0.40 mm
Z = 2	

Data collection

Rigaku AFC-7*R* diffractometer	$h = 0 \rightarrow 11$
ω–2θ scans	$k = 0 \rightarrow 24$
4008 measured reflections	$l = -12 \rightarrow 12$
3652 independent reflections	3 standard reflections every 400 reflections intensity decay: 1.6%
2496 reflections with $I > 2\sigma(I)$	
R_{int} = 0.013	
θ_{max} = 30.0°	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.2245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.045	
$wR(F^2)$ = 0.143	
S = 1.04	$(\Delta/\sigma)_{max}$ < 0.001
3652 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
137 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.4719 (15)	N4—C3	1.4665 (16)
N1—C7^{i}	1.5010 (15)	N4—C5	1.4718 (15)
N1—C10	1.4693 (16)	N12—C12	1.139 (2)
C10—N1—C2	111.56 (10)	C3—N4—C5	115.61 (9)
C10—N1—C7^{i}	114.06 (9)	N12—C12—C11	177.59 (15)
C2—N1—C7^{i}	113.22 (9)		

Symmetry code: (i) $1 - x, 1 - y, -z$.

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement as riding with one common displacement parameter for all methyl and one displacement parameter for all methylene H atoms. The N-bound H atom was located from a difference map but not refined.

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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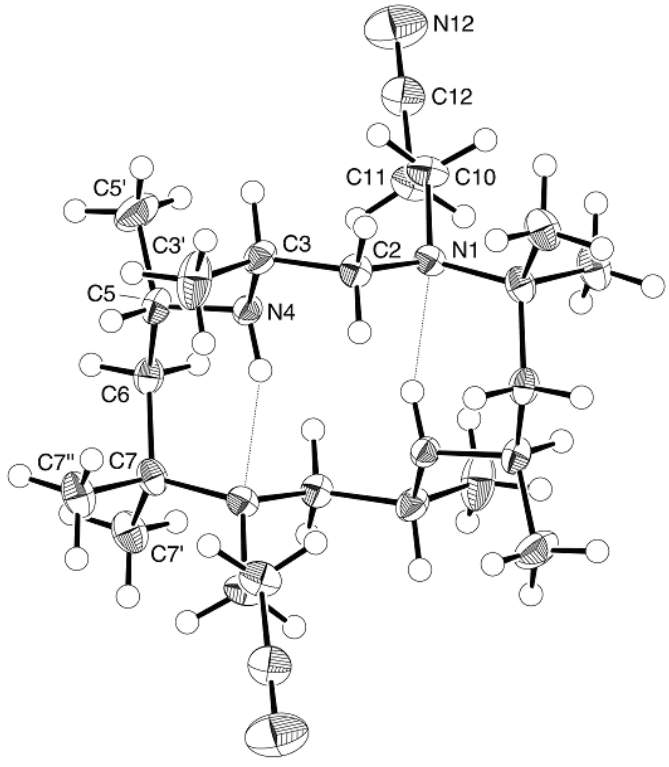

Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).