Crystal structure of 5,12-dimethyl-7,14-diphenyl-1,4,8,11 - tetraazacyclotetradeca-4,11-diene, $C_{24}H_{32}N_4$

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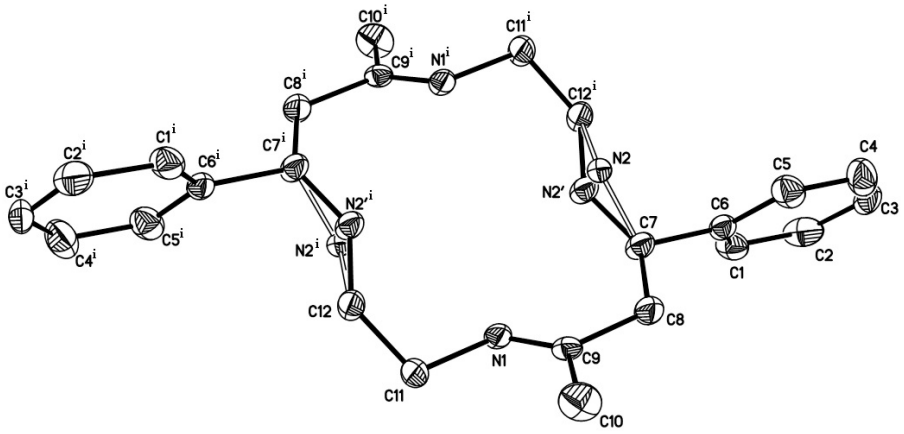

Abstract

$C_{24}H_{32}N_4$, orthorhombic, *Pbca* (no. 61), $a = 9.161(3)$ Å, $b = 8.560(3)$ Å, $c = 27.283(8)$ Å, $V = 2139.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.096$, $wR_{ref}(F^2) = 0.296$, $T = 173$ K.

Source of material

The macrocyclic compound 5,12-dimethyl-7,14-diphenyl-1,4,8,11-tetraazacyclotetradeca-4,11-diene was prepared according to the literature method [1]. The product (10 mg) was dissolved in acetone (10 ml) and crystals of title compound suitable for X-ray diffraction measurement were obtained by slow evaporation of the acetone solvent at room temperature.

Experimental details

H atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). The H atom bound to nitrogen was found in difference Fourier map and refined freely. One of the amine group of the macrocyclic ring (N2, N2') is disordered over two positions. Refinement of the crystal structure yielded an occupancy ratio of the disordered atoms of 0.80(1) : 0.20(1). The large residual values are caused by the disorded N2 atom.

Discussion

In recent years, macrocycles containing nitrogen donor atoms have attracted increasing interest, due to their easy functionalization [2,3].

The 14-membered macrocyclic ring in the title compound shows a symmetric substitution pattern at the C7 and C7^i atoms. The two benzene rings of the title complex are parallel to each other and directed away from the cavity of the 14-membered macrocyclic ring. The amine N2–H and N2^i–H bonds are directed into the cavity of the macrocyclic ring and form an intramolecular interaction with the tertiary amine atoms to stabilize the molecule packing.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.24 × 0.27 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.70 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	54.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9856, 2338
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1318
$N(param)_{refined}$:	136
Programs:	SHELXS-97, SHELXL-97, SHELXTL [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(7)	8*c*	0.0988	–0.0859	0.3969	0.046
H(8A)	8*c*	–0.0775	0.1571	0.3745	0.053
H(8B)	8*c*	0.0827	0.1169	0.3557	0.053
H(3)	8*c*	–0.1410	–0.3664	0.2370	0.078
H(1)	8*c*	0.1301	–0.2914	0.3521	0.055
H(2)	8*c*	0.0710	–0.4303	0.2803	0.074
H(4)	8*c*	–0.2985	–0.1732	0.2672	0.076
H(5)	8*c*	–0.2372	–0.0379	0.3372	0.060
H(10A)	8*c*	–0.0399	0.4169	0.4158	0.087
H(10B)	8*c*	0.0963	0.4092	0.3792	0.087
H(10C)	8*c*	0.1215	0.4489	0.4358	0.087
H(12A)	8*c*	0.2006	0.2452	0.5723	0.048
H(12B)	8*c*	0.0646	0.3305	0.5490	0.048
H(11A)	8*c*	0.3070	0.2191	0.5052	0.065
H(11B)	8*c*	0.2036	0.3621	0.4901	0.065
H(2N)	8*c*	–0.088(5)	–0.027(5)	0.462(2)	0.07(2)

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	8*c*		0.1391(4)	0.1554(3)	0.4613(1)	0.050(2)	0.028(2)	0.030(2)	–0.015(1)	0.005(2)	–0.002(1)
C(6)	8*c*		–0.0475(4)	–0.1493(4)	0.3509(1)	0.041(2)	0.031(2)	0.030(2)	–0.007(2)	0.004(2)	0.000(2)
C(9)	8*c*		0.0809(4)	0.2152(4)	0.4237(1)	0.033(2)	0.025(2)	0.036(2)	–0.006(2)	0.006(2)	0.002(2)
C(7)	8*c*		–0.0067(5)	–0.0557(4)	0.3966(1)	0.053(2)	0.031(2)	0.031(2)	–0.010(2)	0.010(2)	–0.005(2)
C(8)	8*c*		0.0174(5)	0.1120(5)	0.3845(1)	0.057(3)	0.042(2)	0.034(2)	–0.018(2)	0.002(2)	0.001(2)
C(3)	8*c*		–0.1177(7)	–0.3126(7)	0.2664(2)	0.094(4)	0.074(4)	0.026(2)	–0.048(3)	0.002(2)	–0.008(2)
C(1)	8*c*		0.0437(5)	–0.2668(5)	0.3344(2)	0.047(2)	0.037(2)	0.053(3)	–0.007(2)	0.005(2)	–0.010(2)
C(2)	8*c*		0.0087(6)	–0.3495(6)	0.2918(2)	0.071(3)	0.048(3)	0.066(3)	–0.017(2)	0.031(3)	–0.025(2)
C(4)	8*c*		–0.2103(7)	–0.1968(6)	0.2840(2)	0.086(4)	0.051(3)	0.054(3)	–0.023(3)	–0.022(3)	0.007(2)
C(5)	8*c*		–0.1738(5)	–0.1174(5)	0.3255(2)	0.054(3)	0.039(2)	0.056(3)	–0.007(2)	–0.006(2)	0.001(2)
C(10)	8*c*		0.0631(6)	0.3878(5)	0.4126(2)	0.064(3)	0.036(2)	0.074(3)	0.002(2)	–0.004(3)	0.007(2)
C(12)	8*c*		0.1247(5)	0.2347(4)	0.5467(1)	0.061(3)	0.029(2)	0.030(2)	–0.013(2)	–0.001(2)	–0.001(2)
C(11)	8*c*		0.2042(6)	0.2510(5)	0.5003(2)	0.077(3)	0.052(3)	0.034(2)	–0.036(3)	0.000(2)	–0.005(2)
N(2')	8*c*	0.20(1)	–0.041(3)	–0.125(2)	0.4377(6)	0.053(2)	0.031(2)	0.031(2)	–0.010(2)	0.010(2)	–0.005(2)
N(2)	8*c*	0.80	–0.1132(5)	–0.0767(5)	0.4349(1)	0.025(3)	0.023(2)	0.026(2)	0.001(2)	0.001(2)	–0.001(2)

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