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1,6-Bis(2-furyl)-2,5-bis(3-formyl-2-hydroxy-5-methylbenzyl)-2,5-diazahexane

Gang-Chun Sun,[a] Yi-Zhi Li,[b] Zhan-Hang He,[c] Zhong-Jun Li,[c] Jian-Qiang Qu,[a] Chang-Rang Liu[c] and Liu-Fang Wang[a]*

[a]National Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, People's Republic of China, [b]Coordi- State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, and [c]Chemical Department of Zhengzhou University, Zhengzhou 730000, People's Republic of China

Correspondence e-mail: xraychem@nju.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.135
Data-to-parameter ratio = 13.5

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

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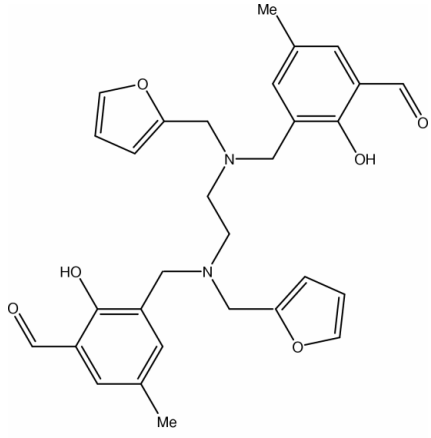
The title compound, $C_{30}H_{32}N_2O_6$, has a center of symmetry. There are two intramolecular O—H···N hydrogen bonds, with an O···N distance of 2.686 (2) Å.

Comment

The chemistry of polynuclear complexes has been stimulated by a desire to mimic the active sites of some metalloenzymes, to search appropriate systems for binding and activating small molecules, and so on (Coughlin & Lippard, 1984). The title compound, (I), was synthesized by the reported method of Shun *et al.* (2001). As there are two different coordination binding sites in the title compound, it should be a good ligand for polynuclear complexes. Similar compounds, such as 1,6-bis(pyridyl)-2,5-bis(2-hydroxy-3-formyl-5-methylbenzyl)-2,5-diazahexane (Fraser *et al.*, 1992), have been reported, but all not of them have been determined crystallographically.



(I)

The title molecule has a center of symmetry at the midpoint of the C6—C6a bond (Fig. 1). Two benzene rings and two furan rings lie on different sides of the main chain C5···C5a. The C—N bond distances are 1.464 (2)–1.484 (2) Å, the C15═O3 and C9—O2 bond distances are 1.224 (3) and 1.352 (2) Å, respectively (Table 1). There are intramolecular O—H···N hydrogen bonds (Table 2). The molecules are stacked along the crystallographic *a* axis (Fig. 2).

Experimental

All commercially available reagents were used as supplied. The title compound was synthesized by the reaction of 1,6-bis(2-furyl)-2,5-diazahexane and 2-hydroxy-3-(chloromethyl)-5-methylbenzaldehyde (molar ratio 1:2.2) in the presence of K_2CO_3 in ethanol solution for 6 h at 313 K (Sun *et al.*, 2001). Colorless crystals suitable for X-ray

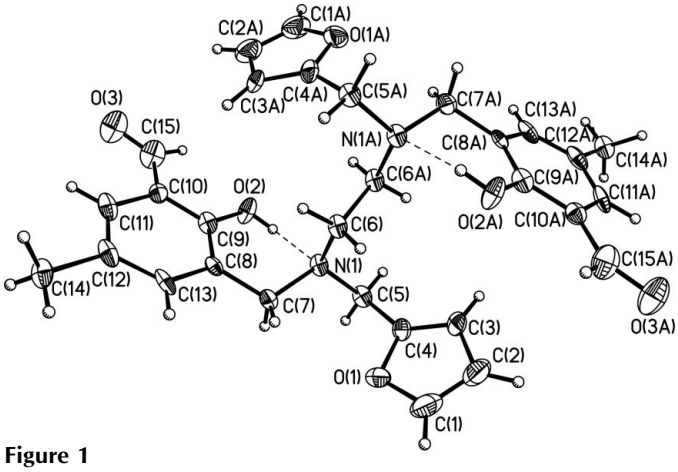


Figure 1
A view of the structure of (I) showing the atom-numbering scheme, with 50% probability displacement ellipsoids.

determination were obtained by slowly evaporating the ethanol solution at room temperature for about three weeks. Elemental analysis (%) for $C_{30}H_{32}N_2O_6$, found: C 69.59, H 6.45, N 5.44; calculated C 69.78, H 6.20, N 5.43. ^{1}H NMR ($CDCl_3$), δ: 2.26 (*s*, 6H), 2.71 (*s*, 4H), 3.66 (*s*, 4H), 3.69 (*s*, 4H), 6.17 (*d*, 2H), 6.31 (*s*, 2H), 7.27 (*d*, 2H), 7.37 (*s*, 4H), 10.17 (*s*, 2H), 11.25 (*br*, 2H).

Crystal data

$C_{30}H_{32}N_2O_6$	D_x = 1.296 Mg m^{-3}
M_r = 516.58	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1184 reflections
a = 5.306 (1) Å	θ = 1.1–25.0°
b = 16.625 (4) Å	μ = 0.09 mm^{-1}
c = 15.020 (1) Å	T = 293 (2) K
β = 92.06 (1)°	Prismatic, colorless
V = 1324.1 (4) Å^3	0.30 × 0.20 × 0.20 mm
Z = 2	

Data collection

Bruker CCD area-detector diffractometer	2336 independent reflections
φ and ω scans	1988 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	R_{int} = 0.047
T_{min} = 0.979, T_{max} = 0.982	θ_{max} = 25.0°
3224 measured reflections	$h = -6 \rightarrow 6$
	$k = 0 \rightarrow 19$
	$l = 0 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.25P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.135	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
2336 reflections	
173 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å).

C5—N1	1.464 (2)	C7—N1	1.477 (2)
C6—N1	1.484 (2)	C9—O2	1.352 (2)
C6—C6^i	1.515 (4)	C15—O3	1.224 (3)

Symmetry code: (i) $1-x, -y, 2-z$.

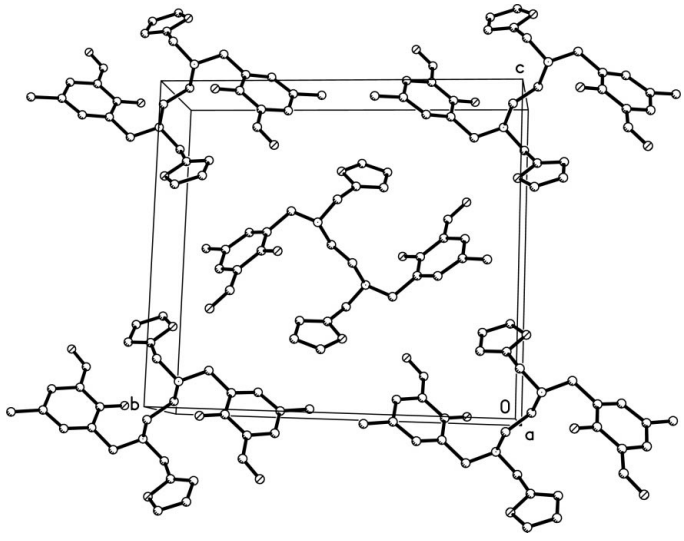


Figure 2
The crystal packing in the *bc* plane.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*A*···N1	0.82	1.99	2.686 (2)	142

All H atoms were placed in geometrically calculated positions with C—H = 0.93–0.97 Å, and refined as riding atoms with U_{iso}(H) = 1.2–1.5U_{eq}(parent atom).

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

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References

Bruker (1997). *SMART, SAINT, XPREP* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Coughlin, P. K. & Lippard, S. J. (1984). *J. Am. Chem. Soc.* **106**, 2328–2336.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fraser, C., Johnston, L., Rheingold, A. L., Haggerty, B. S., Williams, G. K., Whelan, J. & Bosnich, B. (1992). *Inorg. Chem.* **31**, 1835–1844.
Sheldrick, G. M. (1997). *SADABS*, *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Sun, G.-C., He, Z.-H., Li, Z.-J., Yuan, X.-D., Yang, Z.-J., Wang, G.-X., Wang, L.-F. & Liu, C.-R. (2001). *Molecules*, **6**, 1001–1005.