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1,4-Bis(dimorpholinomethyl)benzene

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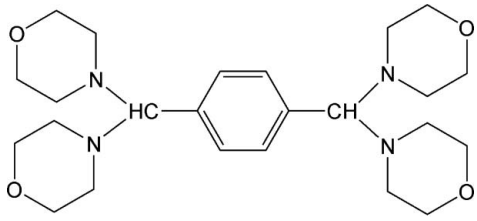
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.132; data-to-parameter ratio = 19.0.

The title compound, $C_{24}H_{38}N_4O_4$, was synthesized by the reaction of terephthalaldehyde and morpholine in ethanol. The N atoms of the four morpholine rings are bonded to benzyl C atoms, forming a centrosymmetric 1,4-bis(dimorpholinomethyl)benzene molecule.

Related literature

For related literature, see: Averkiev *et al.* (2005); Bellon *et al.* (1996); Fujihara *et al.* (2002); Goodman & Jacobsen (2002); Lanman & Myers (2004); Ma *et al.* (2005).



Experimental

Crystal data

$C_{24}H_{38}N_4O_4$
M_r = 446.58
Monoclinic, $P2_1/n$
a = 11.5497 (6) Å
b = 8.6735 (4) Å
c = 12.1645 (6) Å
β = 90.2410 (12)°
V = 1218.58 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.20 × 0.12 × 0.10 mm

Data collection

Rigaku Weissenberg IP diffractometer
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1985)
T_{min} = 0.823, T_{max} = 0.992
2966 measured reflections
2780 independent reflections
1609 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 0.95
2780 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Data collection: *TEXRAY* (Molecular Structure Corporation, 1985); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2163).

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supplementary materials

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1,4-Bis(dimorpholinomethyl)benzene

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Comment

Recently, much attention has been focused on morpholine-containing compounds due to their biological activity as well as chemical and industrial versatility (Bellon *et al.*, 1996; Goodman & Jacobsen, 2002; Lanman & Myers, 2004). Herein, we report the synthesis and crystal structure of a new morpholine-containing compound, 1,4-bis(dimorpholinomethyl)benzene.

As shown in Fig. 1, the nitrogen atoms of four morpholine molecules are bonded to C9 and C9^{i}[symmetry code, (i) 1 − *x*, 1 − *y*, 1 − *z*], respectively. The title molecule possesses a centre of symmetry at the centroid of the benzene ring. The bond lengths and bond angles in the title compound are within normal ranges. The dihedral angle between the plane formed by C9, N1, N2 and the benzene ring plane is 78.87 (9)°. The bond angle of N2—C9—N1 is 107.7 (1)° and the bond length of the backbone C9—C10 is 1.520 (2) Å, which are comparable with those reported in the references (Fujihara *et al.*, 2002; Ma *et al.*, 2005). The morpholine rings adopt the usual chair conformation. They are similar to those of 4,4'-di(morpholin-1-yl)azoxyfurazan (Averkiev *et al.*, 2005) and 2,5-bis-(morpholinomethyl)hydroquinone (Ma *et al.*, 2005). There are no significant contacts among the neighboring molecules, therefore, the molecules pack together only through van der Waals forces in the solid state.

Experimental

All reagents were of AR grade, available commercially and used without further purification. The mixture of terephthalaldehyde (0.67 g, 5 mmol), morpholine (1.74 g, 20 mmol) was heated and refluxed in ethanol (20 ml) for 5 h, and then the resulting solution was cooled to room temperature. After filtration, the filtrate was allowed to stand at room temperature. Upon slow evaporation, colorless block crystals suitable for X-ray diffraction analysis were isolated three days later.

Refinement

All the H atoms on carbon were placed in calculated positions and refined using a riding model with C—H distance in the range 0.93 − 0.98 Å, and U_{iso}(H)= 1.2U_{eq}(C).

Figures

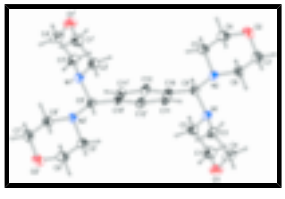

Fig. 1. The molecular structure of (I) with the atom-numbering scheme and 50% probability displacement ellipsoids [symmetry code:(i) 1 − *x*, 1 − *y*, 1 − *z*].

1,4-Bis(dimorpholinomethyl)benzene

Crystal data

$C_{24}H_{38}N_4O_4$	F_{000} = 484
M_r = 446.58	D_x = 1.217 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2780 reflections
a = 11.5497 (6) Å	θ = 2.4–27.5º
b = 8.6735 (4) Å	μ = 0.08 mm^{-1}
c = 12.1645 (6) Å	T = 293 (2) K
β = 90.2410 (12)º	Block, colourless
V = 1218.58 (10) Å^3	0.20 × 0.12 × 0.10 mm
Z = 2	

Data collection

Rigaku Weissenberg IP diffractometer	2780 independent reflections
Radiation source: fine-focus sealed tube	1609 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 293(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (TEXRAY; Molecular Structure Corporation, 1985)	h = −14→0
T_{min} = 0.823, T_{max} = 0.992	k = −11→0
2966 measured reflections	l = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.132	$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.95	$(\Delta/\sigma)_{max} < 0.001$
2780 reflections	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
146 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.55070 (11)	0.42236 (14)	0.20137 (9)	0.0428 (3)
N2	0.45661 (10)	0.20450 (13)	0.27757 (9)	0.0398 (3)
O1	0.74303 (13)	0.56750 (17)	0.10348 (11)	0.0763 (4)
O2	0.35081 (12)	−0.08940 (14)	0.24153 (11)	0.0708 (4)
C1	0.6295 (2)	0.6191 (2)	0.08464 (15)	0.0702 (6)
H1A	0.5993	0.5696	0.0190	0.084*
H1B	0.6309	0.7294	0.0714	0.084*
C2	0.54923 (17)	0.58595 (18)	0.17956 (13)	0.0551 (5)
H2A	0.5745	0.6421	0.2443	0.066*
H2B	0.4712	0.6188	0.1613	0.066*
C3	0.66818 (15)	0.3723 (2)	0.22554 (14)	0.0576 (5)
H3A	0.6692	0.2625	0.2405	0.069*
H3B	0.6975	0.4259	0.2899	0.069*
C4	0.74258 (17)	0.4077 (2)	0.12769 (16)	0.0731 (6)
H4A	0.8212	0.3737	0.1423	0.088*
H4B	0.7139	0.3512	0.0644	0.088*
C5	0.36981 (15)	0.1420 (2)	0.35187 (13)	0.0523 (4)
H5A	0.2937	0.1787	0.3304	0.063*
H5B	0.3853	0.1771	0.4262	0.063*
C6	0.37208 (18)	−0.0308 (2)	0.34842 (14)	0.0629 (5)
H6A	0.4471	−0.0667	0.3737	0.075*
H6B	0.3140	−0.0708	0.3982	0.075*
C7	0.43089 (19)	−0.0256 (2)	0.16656 (16)	0.0711 (6)
H7A	0.4122	−0.0614	0.0931	0.085*
H7B	0.5079	−0.0623	0.1848	0.085*
C8	0.43114 (16)	0.14682 (19)	0.16718 (13)	0.0550 (5)
H8A	0.4890	0.1845	0.1162	0.066*
H8B	0.3561	0.1847	0.1433	0.066*
C9	0.46178 (13)	0.37317 (16)	0.28013 (11)	0.0404 (4)
H9	0.3870	0.4125	0.2540	0.048*
C10	0.48108 (13)	0.43400 (17)	0.39599 (11)	0.0396 (4)
C11	0.56048 (13)	0.37049 (17)	0.46801 (11)	0.0454 (4)
H11	0.6019	0.2832	0.4475	0.054*

C12	0.42098 (13)	0.56446 (17)	0.42950 (12)	0.0445 (4)
H12A	0.3672	0.6090	0.3821	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0500 (8)	0.0404 (7)	0.0380 (6)	−0.0053 (6)	0.0085 (6)	0.0027 (5)
N2	0.0494 (8)	0.0355 (7)	0.0345 (6)	−0.0040 (6)	0.0089 (5)	0.0005 (5)
O1	0.0728 (9)	0.0909 (11)	0.0652 (8)	−0.0337 (8)	0.0154 (7)	0.0087 (7)
O2	0.0860 (10)	0.0511 (8)	0.0754 (9)	−0.0234 (7)	0.0067 (7)	−0.0023 (6)
C1	0.1010 (16)	0.0583 (12)	0.0514 (10)	−0.0190 (11)	0.0081 (10)	0.0114 (8)
C2	0.0721 (12)	0.0468 (10)	0.0465 (9)	−0.0085 (8)	0.0056 (8)	0.0059 (7)
C3	0.0559 (11)	0.0661 (12)	0.0507 (9)	0.0006 (9)	0.0107 (8)	0.0101 (8)
C4	0.0574 (12)	0.0940 (17)	0.0679 (12)	−0.0048 (11)	0.0199 (9)	0.0106 (11)
C5	0.0597 (11)	0.0496 (10)	0.0476 (9)	−0.0087 (8)	0.0126 (8)	0.0042 (7)
C6	0.0757 (13)	0.0508 (11)	0.0622 (11)	−0.0130 (9)	0.0138 (10)	0.0092 (9)
C7	0.0981 (16)	0.0513 (11)	0.0640 (11)	−0.0132 (11)	0.0128 (11)	−0.0129 (9)
C8	0.0746 (12)	0.0470 (10)	0.0436 (9)	−0.0097 (9)	0.0057 (8)	−0.0024 (7)
C9	0.0439 (9)	0.0395 (9)	0.0378 (7)	0.0025 (7)	0.0059 (6)	0.0010 (6)
C10	0.0477 (8)	0.0379 (8)	0.0333 (7)	−0.0018 (7)	0.0076 (6)	0.0011 (6)
C11	0.0536 (10)	0.0401 (8)	0.0425 (8)	0.0129 (7)	0.0083 (7)	−0.0008 (7)
C12	0.0468 (9)	0.0477 (9)	0.0390 (7)	0.0098 (8)	0.0019 (6)	0.0019 (7)

Geometric parameters (Å, °)

N1—C2	1.4435 (18)	C4—H4B	0.9700
N1—C3	1.453 (2)	C5—C6	1.500 (2)
N1—C9	1.4707 (18)	C5—H5A	0.9700
N2—C5	1.4571 (18)	C5—H5B	0.9700
N2—C8	1.4618 (18)	C6—H6A	0.9700
N2—C9	1.4645 (17)	C6—H6B	0.9700
O1—C1	1.403 (2)	C7—C8	1.495 (2)
O1—C4	1.417 (2)	C7—H7A	0.9700
O2—C7	1.415 (2)	C7—H7B	0.9700
O2—C6	1.417 (2)	C8—H8A	0.9700
C1—C2	1.512 (2)	C8—H8B	0.9700
C1—H1A	0.9700	C9—C10	1.5204 (19)
C1—H1B	0.9700	C9—H9	0.9800
C2—H2A	0.9700	C10—C11	1.380 (2)
C2—H2B	0.9700	C10—C12	1.3895 (19)
C3—C4	1.502 (2)	C11—C12^{i}	1.384 (2)
C3—H3A	0.9700	C11—H11	0.9300
C3—H3B	0.9700	C12—C11^{i}	1.384 (2)
C4—H4A	0.9700	C12—H12A	0.9300
C2—N1—C3	109.93 (13)	C6—C5—H5B	109.7
C2—N1—C9	113.40 (12)	H5A—C5—H5B	108.2
C3—N1—C9	115.81 (12)	O2—C6—C5	112.41 (15)
C5—N2—C8	107.84 (12)	O2—C6—H6A	109.1

C5—N2—C9	112.73 (12)	C5—C6—H6A	109.1
C8—N2—C9	111.68 (12)	O2—C6—H6B	109.1
C1—O1—C4	109.98 (14)	C5—C6—H6B	109.1
C7—O2—C6	109.88 (13)	H6A—C6—H6B	107.9
O1—C1—C2	112.97 (15)	O2—C7—C8	112.92 (16)
O1—C1—H1A	109.0	O2—C7—H7A	109.0
C2—C1—H1A	109.0	C8—C7—H7A	109.0
O1—C1—H1B	109.0	O2—C7—H7B	109.0
C2—C1—H1B	109.0	C8—C7—H7B	109.0
H1A—C1—H1B	107.8	H7A—C7—H7B	107.8
N1—C2—C1	108.70 (15)	N2—C8—C7	110.32 (14)
N1—C2—H2A	109.9	N2—C8—H8A	109.6
C1—C2—H2A	109.9	C7—C8—H8A	109.6
N1—C2—H2B	109.9	N2—C8—H8B	109.6
C1—C2—H2B	109.9	C7—C8—H8B	109.6
H2A—C2—H2B	108.3	H8A—C8—H8B	108.1
N1—C3—C4	108.38 (14)	N2—C9—N1	107.73 (11)
N1—C3—H3A	110.0	N2—C9—C10	111.85 (12)
C4—C3—H3A	110.0	N1—C9—C10	113.78 (12)
N1—C3—H3B	110.0	N2—C9—H9	107.8
C4—C3—H3B	110.0	N1—C9—H9	107.8
H3A—C3—H3B	108.4	C10—C9—H9	107.8
O1—C4—C3	111.52 (16)	C11—C10—C12	118.04 (13)
O1—C4—H4A	109.3	C11—C10—C9	122.97 (14)
C3—C4—H4A	109.3	C12—C10—C9	118.89 (13)
O1—C4—H4B	109.3	$C10—C11—C12^{i}$	120.58 (14)
C3—C4—H4B	109.3	C10—C11—H11	119.7
H4A—C4—H4B	108.0	$C12^{i}—C11—H11$	119.7
N2—C5—C6	110.00 (14)	$C11^{i}—C12—C10$	121.37 (14)
N2—C5—H5A	109.7	$C11^{i}—C12—H12A$	119.3
C6—C5—H5A	109.7	C10—C12—H12A	119.3
N2—C5—H5B	109.7		
C4—O1—C1—C2	56.2 (2)	C5—N2—C9—N1	−179.27 (12)
C3—N1—C2—C1	57.91 (17)	C8—N2—C9—N1	−57.68 (16)
C9—N1—C2—C1	−170.66 (14)	C5—N2—C9—C10	54.98 (17)
O1—C1—C2—N1	−56.7 (2)	C8—N2—C9—C10	176.58 (12)
C2—N1—C3—C4	−59.91 (18)	C2—N1—C9—N2	168.49 (12)
C9—N1—C3—C4	169.95 (14)	C3—N1—C9—N2	−63.05 (16)
C1—O1—C4—C3	−57.6 (2)	C2—N1—C9—C10	−66.91 (17)
N1—C3—C4—O1	59.7 (2)	C3—N1—C9—C10	61.55 (17)
C8—N2—C5—C6	58.37 (18)	N2—C9—C10—C11	43.93 (19)
C9—N2—C5—C6	−177.88 (14)	N1—C9—C10—C11	−78.42 (18)
C7—O2—C6—C5	55.6 (2)	N2—C9—C10—C12	−139.67 (14)
N2—C5—C6—O2	−58.9 (2)	N1—C9—C10—C12	97.98 (16)
C6—O2—C7—C8	−55.1 (2)	$C12—C10—C11—C12^{i}$	0.1 (2)
C5—N2—C8—C7	−57.8 (2)	$C9—C10—C11—C12^{i}$	176.50 (14)
C9—N2—C8—C7	177.87 (15)	$C11—C10—C12—C11^{i}$	−0.1 (2)

O2—C7—C8—N2	57.5 (2)	C9—C10—C12—C11^{i}	−176.65 (14)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1