

4,4'-Dimethyl-2,2'-[(3-azapentane-1,5-diy)]bis(nitrilomethylidene)diphenol

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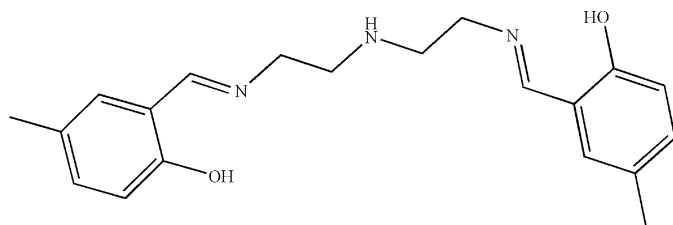
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 9.3.

In the crystal structure of the title Schiff base, $\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_2$, the salicylaldimine groups at each end of the molecule are essentially planar and make a dihedral angle of $84.94(3)^\circ$ with each other. There are strong intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds and a weak intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond.

Related literature

For related literature, see: Rodriguez de Barbarin *et al.* (1994).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_2$
 $M_r = 339.43$
 Orthorhombic, $Pca2_1$

$a = 9.132(4)$ Å
 $b = 5.834(3)$ Å
 $c = 34.365(16)$ Å

$V = 1830.8(15)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.08$ mm⁻¹
 $T = 296(2)$ K
 $0.34 \times 0.32 \times 0.28$ mm

Data collection

Bruker APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.970$, $T_{\max} = 0.981$

14593 measured reflections
 2125 independent reflections
 1867 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.108$
 $S = 1.07$
 2125 reflections
 228 parameters

1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1A}\cdots\text{N1}$	0.82	1.89	2.614 (4)	146
$\text{O2}-\text{H2A}\cdots\text{N2}$	0.82	1.88	2.602 (3)	146
$\text{N3}-\text{H3A}\cdots\text{O1}^1$	0.86	2.54	3.140 (3)	128

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2306).

References

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supplementary materials

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4,4'-Dimethyl-2,2'-[(3-azapentane-1,5-diyl)bis(nitrilomethylidene)]diphenol

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Comment

It was reported that zinc coordinated by phenolate groups in a Schiff-base ligand can act as a nucleophile to catalyze ester hydrolysis (Rodriguez de Barbarin *et al.*, 1994). These results promoted us to investigate linear amine-phenol ligands obtained by reducing Schiff bases, which have greater flexibility, better water solubility and more inertness to hydrolytic decomposition than corresponding Schiff bases. While a number of their complexes with transition metals and main group metals have been reported, the crystal structures of these Schiff-base ligands remain relatively unexplored. So we present here the crystal structure of the title compound, *N,N'*-bis(5-methylsalicylidene)-1,5-diamino-3-azapentane, (I).

The molecular structure of (I) is illustrated in Fig. 1. Compound (I) is a typical salicylaldehyde schiff derivative with normal geometric parameters. The two pendant moieties attached to the ends of the C—C—N—C—C backbone adopt a *cis* conformation. The N3 atom exhibits tetrahedral sp^3 hybridization, whereas the two amide N atoms display planar sp^2 hybridization. The C8—N1 and C13—N2 bonds show the expected double-bond character. In our case, the salicylaldimine moiety is nearly planar. The dihedral angle between the salicylaldimine groups is 84.94 (3)°. The crystal structure of (I) is stabilized by intramolecular O—H \cdots N hydrogen bonds and an intermolecular N—H \cdots O hydrogen bond (Table 1).

Experimental

N-(2-aminoethyl)ethane-1,2-diamine (0.01 mol, 1.03 g) and 2-hydroxy-5-methylbenzaldehyde (0.02 mol, 2.72 g) were dissolved in ethanol and the solution was refluxed for 1 h. After evaporation, a crude product was recrystallized twice from ethanol to give a pure yellow product. Yield: 80.5%. Calcd. for C₂₀H₂₅N₃O₂: C 70.77, H 7.42, N 12.38; Found: C 71.02, H 7.47, N 12.27%.

Refinement

All H atoms were located from a difference Fourier map. Then H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å, O—H = 0.82 Å and N—H = 0.86 Å) and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $1.5U_{\text{eq}}(\text{O})$. In the absence of significant anomalous scattering effects, Friedel pairs have been merged.

Figures

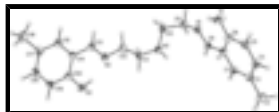


Fig. 1. The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

4,4'-Dimethyl-2,2'-[(3-azapentane-1,5-diyl)bis(nitrilomethylidyne)]diphenol

Crystal data

$C_{20}H_{25}N_3O_2$	$F_{000} = 728$
$M_r = 339.43$	$D_x = 1.231 \text{ Mg m}^{-3}$
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation
Hall symbol: P 2c -2ac	$\lambda = 0.71073 \text{ \AA}$
$a = 9.132 (4) \text{ \AA}$	Cell parameters from 2105 reflections
$b = 5.834 (3) \text{ \AA}$	$\theta = 1.0\text{--}27.5^\circ$
$c = 34.365 (16) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$V = 1830.8 (15) \text{ \AA}^3$	$T = 296 (2) \text{ K}$
$Z = 4$	Block, yellow
	$0.34 \times 0.32 \times 0.28 \text{ mm}$

Data collection

Bruker APEXII area-detector diffractometer	2125 independent reflections
Radiation source: fine-focus sealed tube	1867 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.031$
$T = 296(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{\text{min}} = 0.970$, $T_{\text{max}} = 0.981$	$k = -7 \rightarrow 7$
14593 measured reflections	$l = -44 \rightarrow 44$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.2052P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
2125 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
228 parameters	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.8321 (3)	1.4932 (4)	0.36346 (6)	0.0766 (7)
H1A	0.8862	1.4181	0.3493	0.115*
O2	0.7661 (2)	0.0213 (3)	0.18913 (5)	0.0594 (5)
H2A	0.8305	0.0960	0.1999	0.089*
N1	1.0105 (2)	1.1600 (4)	0.34511 (6)	0.0506 (5)
N2	0.9202 (2)	0.3855 (3)	0.20445 (5)	0.0479 (4)
N3	1.0062 (2)	0.7202 (3)	0.29474 (6)	0.0520 (5)
H3A	1.0210	0.6583	0.3171	0.062*
C1	0.8251 (3)	1.0370 (4)	0.43648 (7)	0.0455 (5)
H1C	0.8686	0.8960	0.4416	0.055*
C2	0.6782 (4)	0.9888 (6)	0.49830 (9)	0.0762 (9)
H2B	0.7544	0.8830	0.5051	0.114*
H2C	0.5895	0.9059	0.4931	0.114*
H2D	0.6622	1.0933	0.5195	0.114*
C3	0.7226 (3)	1.1209 (4)	0.46259 (7)	0.0495 (6)
C4	0.6605 (3)	1.3338 (5)	0.45429 (7)	0.0545 (6)
H4A	0.5923	1.3947	0.4715	0.065*
C5	0.6967 (3)	1.4566 (4)	0.42153 (8)	0.0563 (6)
H5A	0.6526	1.5977	0.4170	0.068*
C6	0.7992 (3)	1.3710 (4)	0.39506 (7)	0.0487 (5)
C7	0.8657 (2)	1.1551 (4)	0.40291 (6)	0.0399 (5)
C8	0.9752 (2)	1.0605 (4)	0.37676 (7)	0.0437 (5)
H8A	1.0208	0.9233	0.3833	0.052*
C9	1.1263 (3)	1.0645 (5)	0.32037 (7)	0.0584 (7)
H9A	1.1836	0.9560	0.3354	0.070*
H9B	1.1911	1.1871	0.3122	0.070*
C10	1.0648 (3)	0.9444 (4)	0.28455 (7)	0.0532 (6)
H10A	0.9879	1.0376	0.2732	0.064*
H10B	1.1416	0.9266	0.2653	0.064*
C11	0.9210 (3)	0.6085 (4)	0.26450 (6)	0.0472 (5)
H11A	0.8477	0.7152	0.2550	0.057*
H11B	0.8698	0.4788	0.2758	0.057*
C12	1.0123 (3)	0.5243 (5)	0.23003 (7)	0.0521 (6)

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H12A	1.0510	0.6543	0.2158	0.063*
H12B	1.0940	0.4335	0.2394	0.063*
C13	0.8784 (3)	0.4705 (4)	0.17204 (7)	0.0441 (5)
H13A	0.9125	0.6143	0.1647	0.053*
C14	0.7784 (2)	0.3473 (3)	0.14626 (6)	0.0412 (5)
C15	0.7306 (3)	0.4500 (4)	0.11167 (7)	0.0466 (5)
H15A	0.7683	0.5925	0.1048	0.056*
C16	0.5766 (4)	0.4651 (6)	0.05050 (9)	0.0746 (8)
H16A	0.4734	0.4399	0.0474	0.112*
H16B	0.6279	0.4036	0.0285	0.112*
H16C	0.5955	0.6266	0.0524	0.112*
C17	0.6291 (3)	0.3464 (4)	0.08737 (7)	0.0519 (6)
C18	0.5756 (3)	0.1310 (4)	0.09836 (8)	0.0562 (6)
H18A	0.5074	0.0582	0.0825	0.067*
C19	0.6215 (3)	0.0237 (4)	0.13212 (8)	0.0534 (6)
H19A	0.5838	-0.1193	0.1387	0.064*
C20	0.7242 (3)	0.1288 (4)	0.15635 (7)	0.0448 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0892 (16)	0.0680 (11)	0.0725 (13)	0.0077 (11)	0.0161 (12)	0.0338 (10)
O2	0.0733 (12)	0.0477 (9)	0.0574 (10)	0.0009 (8)	0.0025 (9)	0.0103 (8)
N1	0.0448 (10)	0.0629 (12)	0.0440 (10)	-0.0085 (9)	0.0049 (8)	-0.0061 (9)
N2	0.0467 (10)	0.0535 (11)	0.0434 (10)	0.0017 (9)	0.0034 (9)	-0.0054 (9)
N3	0.0551 (11)	0.0628 (12)	0.0381 (9)	-0.0069 (10)	-0.0017 (9)	0.0057 (9)
C1	0.0484 (13)	0.0446 (11)	0.0435 (12)	0.0005 (9)	-0.0054 (10)	0.0057 (9)
C2	0.091 (2)	0.085 (2)	0.0526 (15)	-0.0057 (18)	0.0175 (17)	0.0134 (15)
C3	0.0516 (14)	0.0570 (13)	0.0400 (12)	-0.0081 (11)	0.0041 (10)	0.0013 (10)
C4	0.0518 (14)	0.0562 (14)	0.0554 (14)	-0.0016 (11)	0.0111 (11)	-0.0116 (11)
C5	0.0554 (15)	0.0471 (12)	0.0664 (16)	0.0058 (11)	0.0051 (13)	0.0010 (11)
C6	0.0491 (13)	0.0460 (12)	0.0511 (12)	-0.0032 (10)	-0.0004 (10)	0.0066 (10)
C7	0.0378 (11)	0.0429 (10)	0.0391 (11)	-0.0038 (8)	-0.0007 (9)	-0.0001 (8)
C8	0.0377 (11)	0.0502 (12)	0.0432 (11)	-0.0037 (9)	-0.0034 (9)	-0.0039 (9)
C9	0.0437 (13)	0.0815 (17)	0.0501 (13)	-0.0115 (13)	0.0066 (11)	-0.0145 (13)
C10	0.0551 (14)	0.0640 (14)	0.0405 (12)	-0.0100 (11)	0.0078 (11)	-0.0025 (11)
C11	0.0450 (12)	0.0540 (12)	0.0427 (12)	-0.0070 (10)	0.0053 (10)	-0.0001 (10)
C12	0.0410 (11)	0.0667 (14)	0.0487 (13)	0.0014 (11)	0.0006 (10)	-0.0066 (11)
C13	0.0424 (11)	0.0437 (11)	0.0463 (12)	-0.0010 (9)	0.0062 (9)	-0.0030 (9)
C14	0.0428 (11)	0.0384 (10)	0.0422 (11)	0.0013 (9)	0.0085 (9)	-0.0041 (9)
C15	0.0522 (13)	0.0417 (11)	0.0460 (12)	-0.0025 (10)	0.0059 (10)	-0.0008 (9)
C16	0.089 (2)	0.083 (2)	0.0516 (15)	-0.0001 (18)	-0.0152 (15)	0.0008 (14)
C17	0.0567 (15)	0.0551 (13)	0.0438 (12)	0.0038 (11)	0.0019 (11)	-0.0057 (10)
C18	0.0580 (15)	0.0545 (14)	0.0559 (15)	-0.0049 (11)	0.0013 (12)	-0.0167 (12)
C19	0.0575 (15)	0.0396 (11)	0.0632 (15)	-0.0071 (10)	0.0107 (12)	-0.0086 (11)
C20	0.0494 (12)	0.0386 (11)	0.0463 (12)	0.0021 (9)	0.0108 (10)	-0.0029 (9)

Geometric parameters (Å, °)

O1—C6	1.333 (3)	C9—H9A	0.9700
O1—H1A	0.8200	C9—H9B	0.9700
O2—C20	1.345 (3)	C10—H10A	0.9700
O2—H2A	0.8200	C10—H10B	0.9700
N1—C8	1.274 (3)	C11—C12	1.530 (3)
N1—C9	1.467 (3)	C11—H11A	0.9700
N2—C13	1.277 (3)	C11—H11B	0.9700
N2—C12	1.461 (3)	C12—H12A	0.9700
N3—C11	1.453 (3)	C12—H12B	0.9700
N3—C10	1.456 (3)	C13—C14	1.461 (3)
N3—H3A	0.8600	C13—H13A	0.9300
C1—C3	1.386 (3)	C8—H8A	0.9300
C1—C7	1.394 (3)	C14—C15	1.401 (3)
C1—H1C	0.9300	C14—C20	1.411 (3)
C2—C3	1.505 (4)	C15—C17	1.386 (3)
C2—H2B	0.9600	C15—H15A	0.9300
C2—H2C	0.9600	C16—C17	1.521 (4)
C2—H2D	0.9600	C16—H16A	0.9600
C3—C4	1.395 (4)	C16—H16B	0.9600
C4—C5	1.375 (4)	C16—H16C	0.9600
C4—H4A	0.9300	C17—C18	1.400 (4)
C5—C6	1.397 (3)	C18—C19	1.383 (4)
C5—H5A	0.9300	C18—H18A	0.9300
C6—C7	1.424 (3)	C19—C20	1.396 (3)
C7—C8	1.453 (3)	C19—H19A	0.9300
C9—C10	1.524 (3)		
C6—O1—H1A	109.5	N3—C11—C12	113.95 (19)
C20—O2—H2A	109.5	N3—C11—H11A	108.8
C8—N1—C9	120.3 (2)	C12—C11—H11A	108.8
C13—N2—C12	118.8 (2)	N3—C11—H11B	108.8
C11—N3—C10	115.3 (2)	C12—C11—H11B	108.8
C11—N3—H3A	122.3	H11A—C11—H11B	107.7
C10—N3—H3A	122.3	N2—C12—C11	109.28 (19)
C3—C1—C7	122.7 (2)	N2—C12—H12A	109.8
C3—C1—H1C	118.6	C11—C12—H12A	109.8
C7—C1—H1C	118.6	N2—C12—H12B	109.8
C3—C2—H2B	109.5	C11—C12—H12B	109.8
C3—C2—H2C	109.5	H12A—C12—H12B	108.3
H2B—C2—H2C	109.5	N2—C13—C14	121.6 (2)
C3—C2—H2D	109.5	N2—C13—H13A	119.2
H2B—C2—H2D	109.5	C14—C13—H13A	119.2
H2C—C2—H2D	109.5	N1—C8—C7	121.9 (2)
C1—C3—C4	117.2 (2)	N1—C8—H8A	119.0
C1—C3—C2	122.0 (2)	C7—C8—H8A	119.0
C4—C3—C2	120.9 (2)	C15—C14—C20	119.0 (2)
C5—C4—C3	122.3 (2)	C15—C14—C13	119.9 (2)

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C5—C4—H4A	118.9	C20—C14—C13	121.0 (2)
C3—C4—H4A	118.9	C17—C15—C14	122.2 (2)
C4—C5—C6	120.5 (2)	C17—C15—H15A	118.9
C4—C5—H5A	119.7	C14—C15—H15A	118.9
C6—C5—H5A	119.7	C17—C16—H16A	109.5
O1—C6—C5	119.4 (2)	C17—C16—H16B	109.5
O1—C6—C7	122.0 (2)	H16A—C16—H16B	109.5
C5—C6—C7	118.6 (2)	C17—C16—H16C	109.5
C1—C7—C6	118.7 (2)	H16A—C16—H16C	109.5
C1—C7—C8	120.4 (2)	H16B—C16—H16C	109.5
C6—C7—C8	120.8 (2)	C15—C17—C18	117.5 (2)
N1—C9—C10	112.2 (2)	C15—C17—C16	120.9 (2)
N1—C9—H9A	109.2	C18—C17—C16	121.5 (3)
C10—C9—H9A	109.2	C19—C18—C17	121.8 (2)
N1—C9—H9B	109.2	C19—C18—H18A	119.1
C10—C9—H9B	109.2	C17—C18—H18A	119.1
H9A—C9—H9B	107.9	C18—C19—C20	120.3 (2)
N3—C10—C9	110.8 (2)	C18—C19—H19A	119.8
N3—C10—H10A	109.5	C20—C19—H19A	119.8
C9—C10—H10A	109.5	O2—C20—C19	119.1 (2)
N3—C10—H10B	109.5	O2—C20—C14	121.8 (2)
C9—C10—H10B	109.5	C19—C20—C14	119.1 (2)
H10A—C10—H10B	108.1		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots N1	0.82	1.89	2.614 (4)	146
O2—H2A \cdots N2	0.82	1.88	2.602 (3)	146
N3—H3A \cdots O1 ⁱ	0.86	2.54	3.140 (3)	128

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

Fig. 1

