Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Space group revsion of the triclinic polymorph of salicylaldehyde azine

Aamer Saeed,[a]* Michael Bolte[b] and Muhammad Arshad[c]

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, [b]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany, and [c]Chemistry Division, Directorate of Science, PINSTECH, Nilore, Islamabad, Pakistan
Correspondence e-mail: aamersaeed@yahoo.com

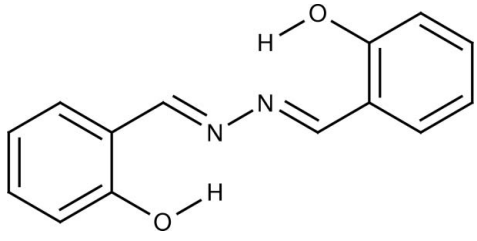
Received 25 November 2011; accepted 20 December 2011

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.116; data-to-parameter ratio = 12.7.

The structure of the title compound, $C_{14}H_{12}N_2O_2$ {systematic name: 2,2′-[hydrazinediylidenebis(methanylylidene)]diphenol}, has already been determined in the triclinic space group $P\overline{1}$ with Z = 4 [El-Medani, Aboaly, Abdalla & Ramadan (2004). *Spectrosc. Lett.* **37**, 619–632]. However, the correct space group should be $P2_1/c$ with Z = 4. This structure is a new polymorph of the already known monoclinic polymorph of salicyladehyde azine, which crystallizes in space group $P2_1/n$ with Z = 2. The benzene rings form a dihedral angle of 46.12 (9)°. Two intramolucular O—H···N hydrogen bonds occur.

Related literature

For the structure of salicylaldehyde azine in $P\overline{1}$ with Z=4, see El-Medani *et al.* (2004). For the other monoclinic polymorph of salicyladehyde azine, see for example Xue *et al.* (1994).



Experimental

Crystal data

$C_{14}H_{12}N_2O_2$
M_r = 240.26
Monoclinic, $P2_1/c$
a = 16.3621 (11) Å
b = 5.9180 (4) Å
c = 13.1706 (9) Å
β = 113.639 (5)°
V = 1168.31 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 K
0.28 × 0.19 × 0.12 mm

Data collection

Stoe IPDS II two-circle diffractometer
14742 measured reflections
2189 independent reflections
1977 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.116
S = 1.17
2189 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.95 (3)	1.81 (3)	2.6454 (19)	145 (2)
O1A—H1A···N1A	0.95 (3)	1.82 (3)	2.6532 (19)	145 (2)

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZJ2044).

References

El-Medani, S. M., Aboaly, M. M., Abdalla, H. H. & Ramadan, R. M. (2004). *Spectrosc. Lett.* **37**, 619–632.
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Xu, X.-X., You, X.-Z., Sun, Z.-F., Wang, X. & Liu, H.-X. (1994). *Acta Cryst.* C**50**, 1169–1171.

supplementary materials

Acta Cryst. (2012). E**68**, o255 [**doi:10.1107/S160053681105478X**]

Space group revsion of the triclinic polymorph of salicylaldehyde azine

A. Saeed, M. Bolte and M. Arshad

Comment

The structure of the title compound, $C_{14}H_{12}N_2O_2$, has already been determined in the triclinic space group $P\overline{1}$ with Z=4 [El-Medani, Aboaly, Abdalla & Ramadan (2004). Spectrosc. Lett. 37, 619–632]. However, the correct space group should be $P2_1/c$ with Z=4. The authors have determined the unit-cell parameters correctly, however, they thought that the structure is triclinic with four molecules in the asymmetric unit, whereas the correct description should be in the monoclinic crystal systems with two half molecules in the asymmetric unit. This structure is a new polymorph of the already known monoclinic polymorph of salicyladehyde azine, which crystallizes in the space group $P2_1/n$ with Z=2.

The title compound crystallizes with two half molecules in the asymmetric unit, both of which are located on a crystallographic centre of inversion. The molecules are essentially planar (r.m.s. deviation for all non H-atoms 0.021 and 0.018 Å, for the two molecules in the asymmetric unit). Bond lengths and angles are in the normal ranges.

In the already known monoclinic polymorph there is just one half molecule in the asymmetric unit which is located on a centre of inversion. The dihedral angle between symmetry equivalent molecules is 64.6°. The title compound, on the other hand, crystallizes with two half molecules in the asymmetric unit, which enclose a dihedral angle of 47.4°. The dihedral angle between symmetry equivalent molecules is 67.5°. Thus the difference between the two monoclinic polymorphs is the different mutual orientation of the molecules in the unit cell.

Experimental

Hydrazine hydrate, (1 mmol) dissolved in 5 ml ethanol was added dropwise to a solution of salicylaldehyde, (2.2 mmol) in 10 ml ethanol at room temperature with continuous stirring. The reaction mixture was reflux for 4 h and completion monitored by TLC. The reaction mixture was concentrated and resulted product was separated. Single crystal of the compound, suitable for X-ray crystallography, was grown by slow evaporation from an ethyl acetate-ethanol solution (2:1). as colourless crystals: Anal. calcd. for $C_{14}H_{12}N_2O_2$: C, 44.95; H, C, 69.99; H, 5.03; N, 11.66 98%; found: C, 69.99; H, 5.03; N, 11.66; %.

Refinement

The H atoms were initially located by difference Fourier synthesis. Subsequently, H atoms bonded to C atoms were refined using a riding model, with C—H = 0.95 Å and with U_{iso}(H) = 1.2U_{eq}(C). The H atoms bonded to O were freely refined.

Figures

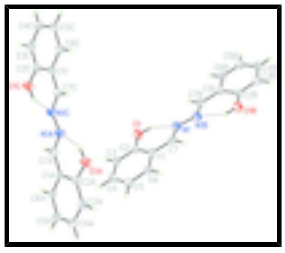

Fig. 1. Molecular structure of title compound showing the two molecules in the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level. The atoms of the second molecule in the asymmetric unit are labelled with suffix A. Symmetry operators: (B): 2 - *x*,1 - *y*,-*z*, (C): 1 - *x*,1 - *y*,-*z*.

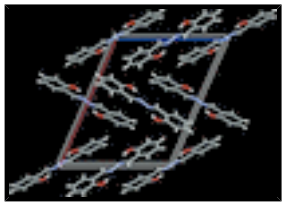

Fig. 2. Packing diagram of the title compound with view along the *b* axis.

2-((1*E*)-{(*E*)-2-[(2-hydroxyphenyl)methylidene]hydrazin-1- ylidene}methyl)phenol

Crystal data

$C_{14}H_{12}N_2O_2$	$F(000) = 504$
$M_r = 240.26$	$D_x = 1.366$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 14708 reflections
$a = 16.3621$ (11) Å	$\theta = 3.4$–$26.2°$
$b = 5.9180$ (4) Å	$\mu = 0.09$ mm^{-1}
$c = 13.1706$ (9) Å	$T = 173$ K
$\beta = 113.639$ (5)°	Plate, light brown
$V = 1168.31$ (14) Å^3	0.28 × 0.19 × 0.12 mm
$Z = 4$	

Data collection

Stoe IPDS II two-circle diffractometer	1977 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.079$
graphite	$\theta_{max} = 25.7°$, $\theta_{min} = 3.4°$
ω scans	$h = -19 \rightarrow 19$
14742 measured reflections	$k = -7 \rightarrow 7$
2189 independent reflections	$l = -15 \rightarrow 15$

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 atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.5421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.17$	$(\Delta/\sigma)_{max} < 0.001$
2189 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.029 (3)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.87613 (9)	0.1392 (2)	0.06801 (11)	0.0380 (4)
H1	0.9110 (17)	0.204 (4)	0.0324 (19)	0.059 (7)*
N1	0.97379 (9)	0.4503 (2)	0.02477 (11)	0.0281 (3)
C1	0.90680 (10)	0.5083 (3)	0.15398 (13)	0.0253 (4)
C2	0.86776 (11)	0.2920 (3)	0.14005 (13)	0.0274 (4)
C3	0.81945 (12)	0.2302 (3)	0.20203 (14)	0.0317 (4)
H3	0.7920	0.0857	0.1917	0.038*
C4	0.81141 (12)	0.3789 (3)	0.27845 (14)	0.0341 (4)
H4	0.7785	0.3352	0.3205	0.041*
C5	0.85078 (12)	0.5913 (3)	0.29465 (15)	0.0358 (4)
H5	0.8457	0.6917	0.3481	0.043*
C6	0.89718 (11)	0.6543 (3)	0.23210 (14)	0.0312 (4)
H6	0.9233	0.8005	0.2422	0.037*
C7	0.95815 (11)	0.5834 (3)	0.09241 (13)	0.0264 (4)
H7	0.9807	0.7334	0.1021	0.032*
O1A	0.62345 (9)	0.8865 (2)	0.17775 (10)	0.0357 (3)
H1A	0.5876 (18)	0.814 (5)	0.111 (2)	0.066 (8)*
N1A	0.52522 (10)	0.5585 (2)	0.04906 (11)	0.0283 (3)
C1A	0.59119 (10)	0.5328 (3)	0.24640 (13)	0.0248 (4)
C2A	0.63079 (11)	0.7490 (3)	0.26375 (13)	0.0267 (4)
C3A	0.67951 (11)	0.8257 (3)	0.37086 (14)	0.0306 (4)

H3A	0.7069	0.9703	0.3822	0.037*
C4A	0.68836 (12)	0.6931 (3)	0.46105 (14)	0.0331 (4)
H4A	0.7218	0.7473	0.5339	0.040*
C5A	0.64861 (12)	0.4804 (3)	0.44581 (14)	0.0331 (4)
H5A	0.6539	0.3904	0.5079	0.040*
C6A	0.60154 (11)	0.4023 (3)	0.33969 (13)	0.0284 (4)
H6A	0.5754	0.2561	0.3294	0.034*
C7A	0.54094 (11)	0.4405 (3)	0.13719 (13)	0.0263 (4)
H7A	0.5191	0.2900	0.1303	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0503 (8)	0.0324 (7)	0.0411 (7)	−0.0115 (6)	0.0286 (6)	−0.0101 (6)
N1	0.0308 (7)	0.0300 (8)	0.0273 (7)	−0.0024 (6)	0.0155 (6)	0.0022 (6)
C1	0.0227 (8)	0.0290 (9)	0.0242 (8)	−0.0001 (6)	0.0094 (6)	0.0002 (6)
C2	0.0271 (8)	0.0298 (9)	0.0251 (8)	−0.0010 (7)	0.0103 (7)	−0.0018 (7)
C3	0.0291 (9)	0.0329 (9)	0.0348 (9)	−0.0043 (7)	0.0145 (7)	0.0030 (7)
C4	0.0287 (9)	0.0463 (11)	0.0327 (9)	0.0014 (8)	0.0180 (7)	0.0046 (8)
C5	0.0325 (9)	0.0450 (11)	0.0350 (9)	0.0001 (8)	0.0189 (8)	−0.0079 (8)
C6	0.0282 (9)	0.0319 (9)	0.0350 (9)	−0.0018 (7)	0.0141 (7)	−0.0053 (7)
C7	0.0258 (8)	0.0268 (8)	0.0263 (8)	−0.0003 (6)	0.0101 (6)	0.0019 (6)
O1A	0.0482 (8)	0.0300 (7)	0.0301 (7)	−0.0072 (6)	0.0170 (6)	0.0017 (5)
N1A	0.0332 (7)	0.0296 (8)	0.0234 (7)	−0.0005 (6)	0.0125 (6)	−0.0030 (6)
C1A	0.0224 (8)	0.0283 (8)	0.0260 (8)	0.0033 (6)	0.0121 (6)	0.0008 (6)
C2A	0.0277 (8)	0.0276 (8)	0.0282 (8)	0.0017 (7)	0.0146 (7)	0.0015 (7)
C3A	0.0286 (9)	0.0302 (9)	0.0339 (9)	−0.0025 (7)	0.0136 (7)	−0.0039 (7)
C4A	0.0289 (9)	0.0421 (10)	0.0268 (9)	−0.0001 (8)	0.0094 (7)	−0.0030 (7)
C5A	0.0325 (9)	0.0399 (10)	0.0264 (9)	0.0023 (8)	0.0114 (7)	0.0057 (7)
C6A	0.0270 (8)	0.0299 (9)	0.0293 (9)	0.0012 (7)	0.0124 (7)	0.0035 (7)
C7A	0.0273 (8)	0.0273 (8)	0.0274 (8)	0.0012 (7)	0.0142 (7)	−0.0007 (7)

Geometric parameters (Å, °)

O1—C2	1.357 (2)	O1A—C2A	1.360 (2)
O1—H1	0.95 (3)	O1A—H1A	0.95 (3)
N1—C7	1.289 (2)	N1A—C7A	1.289 (2)
N1—N1^{i}	1.398 (3)	N1A—N1A^{ii}	1.405 (3)
C1—C6	1.400 (2)	C1A—C6A	1.403 (2)
C1—C2	1.409 (2)	C1A—C2A	1.411 (2)
C1—C7	1.452 (2)	C1A—C7A	1.447 (2)
C2—C3	1.394 (2)	C2A—C3A	1.389 (2)
C3—C4	1.382 (3)	C3A—C4A	1.382 (2)
C3—H3	0.9500	C3A—H3A	0.9500
C4—C5	1.390 (3)	C4A—C5A	1.394 (3)
C4—H4	0.9500	C4A—H4A	0.9500
C5—C6	1.377 (2)	C5A—C6A	1.377 (2)
C5—H5	0.9500	C5A—H5A	0.9500

C6—H6	0.9500	C6A—H6A	0.9500
C7—H7	0.9500	C7A—H7A	0.9500
C2—O1—H1	108.9 (15)	C2A—O1A—H1A	108.8 (16)
C7—N1—N1^{i}	113.21 (17)	C7A—N1A—N1A^{ii}	113.13 (17)
C6—C1—C2	118.53 (15)	C6A—C1A—C2A	118.12 (15)
C6—C1—C7	118.83 (15)	C6A—C1A—C7A	118.90 (15)
C2—C1—C7	122.62 (15)	C2A—C1A—C7A	122.99 (15)
O1—C2—C3	118.33 (16)	O1A—C2A—C3A	118.27 (15)
O1—C2—C1	121.93 (15)	O1A—C2A—C1A	121.73 (15)
C3—C2—C1	119.74 (15)	C3A—C2A—C1A	119.99 (15)
C4—C3—C2	120.09 (17)	C4A—C3A—C2A	120.49 (16)
C4—C3—H3	120.0	C4A—C3A—H3A	119.8
C2—C3—H3	120.0	C2A—C3A—H3A	119.8
C3—C4—C5	120.94 (16)	C3A—C4A—C5A	120.45 (16)
C3—C4—H4	119.5	C3A—C4A—H4A	119.8
C5—C4—H4	119.5	C5A—C4A—H4A	119.8
C6—C5—C4	119.08 (17)	C6A—C5A—C4A	119.22 (16)
C6—C5—H5	120.5	C6A—C5A—H5A	120.4
C4—C5—H5	120.5	C4A—C5A—H5A	120.4
C5—C6—C1	121.60 (17)	C5A—C6A—C1A	121.71 (16)
C5—C6—H6	119.2	C5A—C6A—H6A	119.1
C1—C6—H6	119.2	C1A—C6A—H6A	119.1
N1—C7—C1	121.24 (15)	N1A—C7A—C1A	121.26 (15)
N1—C7—H7	119.4	N1A—C7A—H7A	119.4
C1—C7—H7	119.4	C1A—C7A—H7A	119.4
C6—C1—C2—O1	−178.31 (15)	C6A—C1A—C2A—O1A	−179.65 (15)
C7—C1—C2—O1	0.3 (2)	C7A—C1A—C2A—O1A	0.5 (2)
C6—C1—C2—C3	1.2 (2)	C6A—C1A—C2A—C3A	1.0 (2)
C7—C1—C2—C3	179.82 (15)	C7A—C1A—C2A—C3A	−178.84 (15)
O1—C2—C3—C4	178.26 (15)	O1A—C2A—C3A—C4A	179.50 (16)
C1—C2—C3—C4	−1.3 (3)	C1A—C2A—C3A—C4A	−1.1 (3)
C2—C3—C4—C5	0.2 (3)	C2A—C3A—C4A—C5A	0.1 (3)
C3—C4—C5—C6	1.0 (3)	C3A—C4A—C5A—C6A	1.0 (3)
C4—C5—C6—C1	−1.0 (3)	C4A—C5A—C6A—C1A	−1.2 (3)
C2—C1—C6—C5	−0.1 (3)	C2A—C1A—C6A—C5A	0.2 (2)
C7—C1—C6—C5	−178.71 (16)	C7A—C1A—C6A—C5A	179.97 (15)
N1^{i}—N1—C7—C1	−178.38 (16)	N1A^{ii}—N1A—C7A—C1A	−179.39 (16)
C6—C1—C7—N1	175.52 (15)	C6A—C1A—C7A—N1A	176.00 (15)
C2—C1—C7—N1	−3.1 (2)	C2A—C1A—C7A—N1A	−4.2 (2)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*; (ii) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.95 (3)	1.81 (3)	2.6454 (19)	145 (2)
O1A—H1A···N1A	0.95 (3)	1.82 (3)	2.6532 (19)	145 (2)

Fig. 1

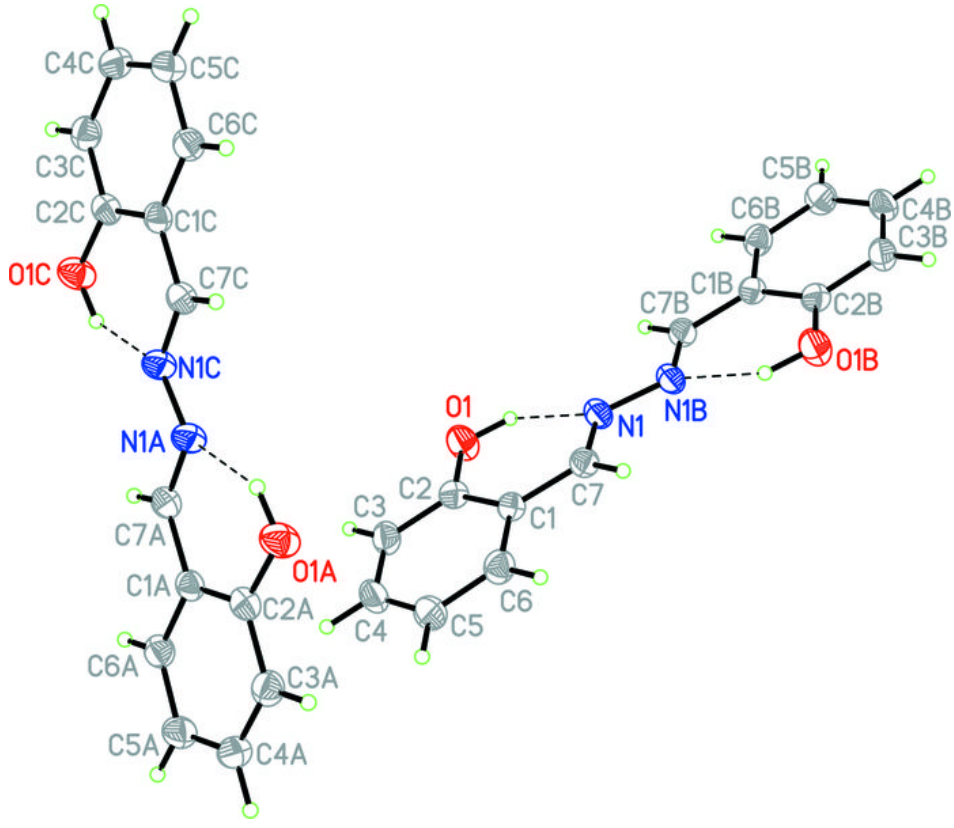
C4C
C5C
C6C
C3C
C2C
C1C
O1C
C7C
N1C
N1A
C7A
O1A
C1A
C2A
C6A
C3A
C5A
C4A
O1
C2
C3
C1
C7
C4
C5
C6
N1
N1B
C5B
C6B
C4B
C3B
C1B
C7B
C2B
O1B

Fig. 2