

Salicyldoxime-III at 150 K

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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.033
 wR factor = 0.079
Data-to-parameter ratio = 9.7

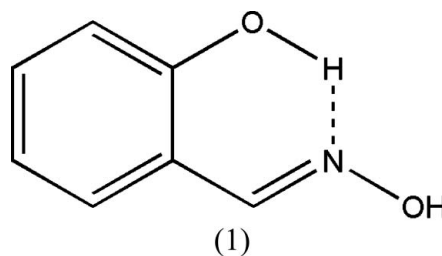
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Salicyldoxime derivatives crystallize in either hydrogen-bonded ring or chain motifs. A polymorph of the parent compound, salicyldoxime, characterized by ring formation, has been known for some time. We now report a new polymorph of salicyldoxime (2-hydroxybenzaldehyde oxime, $\text{C}_7\text{H}_7\text{NO}_2$), which exhibits chain formation and which has two molecules per asymmetric unit. π - π stacking interactions occur between the chains. We refer to this polymorph as salicyldoxime-III.

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Comment

Salicyldoximes bearing branched alkyl chains are used as extractants to effect the separation and concentration operations in the hydrometallurgical recovery of copper, accounting for around 30% of annual production (Kordosky, 2002). The $\text{N}_2\text{O}_2^{2-}$ donor set in bis-salicyldoxime complexes is stabilized by interligand hydrogen bonds, forming a pseudo-macrocylic arrangement (*e.g.* Fig. 1*a*). The high selectivity of salicyldoximes for copper over other metal ions is the result of the compatibility of the size of the cavity at the centre of the pseudo-macrocycle and the ionic radius of Cu^{2+} (Smith *et al.*, 2002).



The crystal structure of the parent compound salicyldoxime, (1), was determined using X-ray diffraction by Pfluger & Harlow (1973) [Cambridge Structural Database (CSD, Version 5.27; Allen, 2002) refcode SALOXM]. We refer to the phase investigated by these workers as salicyldoxime-I. We have recently shown that salicyldoxime-I undergoes a phase transition at 5.3 GPa to a second phase, salicyldoxime-II (Wood *et al.*, 2006).

Salicyldoxime-I crystallizes in space group $P2_1/n$. Pairs of molecules, related by inversion centres, form intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds to produce a dimer (Fig. 1*b*), for which the graph-set descriptor is $R_4^4(10)$ (Bernstein *et al.*, 1995). This dimeric form closely resembles the pseudo-macrocylic arrangement observed in metal complexes, and is only observed in the free ligands in the solid state in salicyldoxime derivatives which carry small substituents [*e.g.* CSD

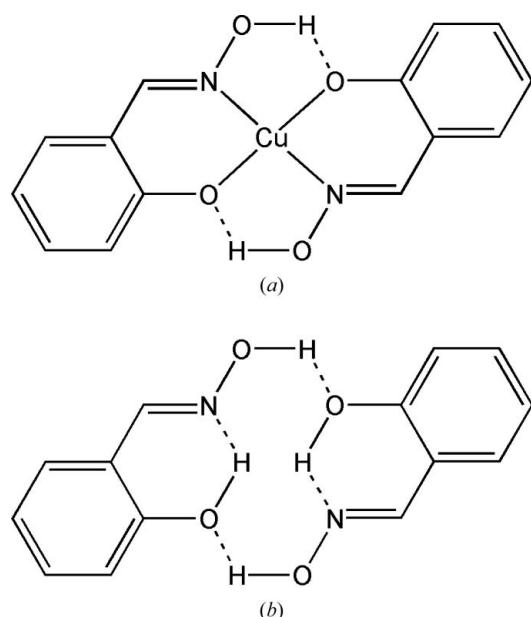


Figure 1
Pseudomacrocycle formation by salicylaldoxime. (a) Salicylaldoxime complexation by copper(II). (b) Hydrogen-bonded dimers formed in the crystal structure of salicylaldoxime-I. Dashed lines indicate hydrogen bonds.

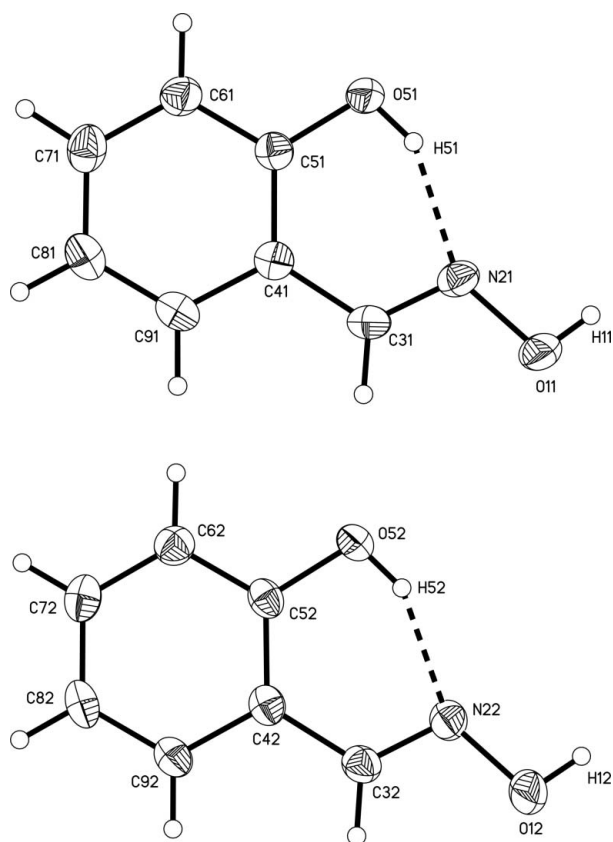


Figure 2
The two molecules comprising the asymmetric unit of salicylaldoxime-III. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as circles of arbitrary radii. Dashed lines indicate hydrogen bonds.

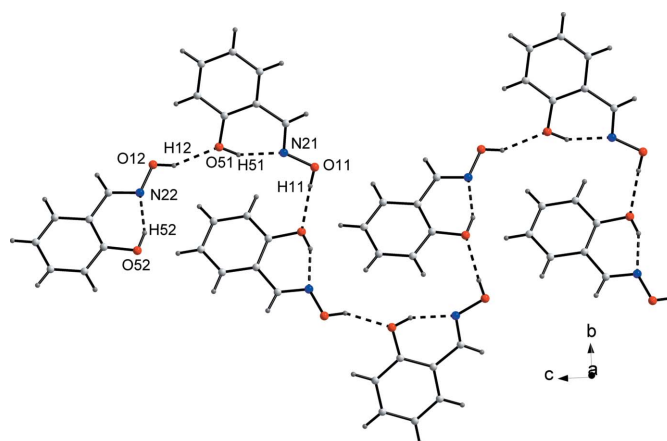


Figure 3
Hydrogen-bonded chains in salicylaldoxime-III. Dashed lines indicate hydrogen bonds.

refcodes ABULIT (Xu *et al.*, 2004) and CLSALX (Simonsen *et al.*, 1961)]. Bulky alkyl substituents lead to hydrogen-bonded chain motifs in preference to rings [e.g. CSD refcodes HEPKET10 (Kozioł & Kosturkiewicz, 1984) and HELBOP (Maurin, 1994)].

We now report the crystal structure of a third polymorph of salicylaldoxime, salicylaldoxime-III, obtained under ambient conditions by recrystallization from a solution of hexane and chloroform. Weissenberg photographs, taken using a crystal of salicylaldoxime obtained from alcohol, were indexed by Merritt & Schroeder (1956) on the basis of an orthorhombic cell with dimensions $a = 12.69$, $b = 13.51$ and $c = 7.98$ Å, although no coordinates were determined. These cell dimensions closely resemble those determined here for salicylaldoxime-III. In the same paper, the authors report a powder pattern, which Pfluger & Harlow (1973) claim actually corresponds to the monoclinic form, salicylaldoxime-I. However, a powder pattern simulated (using *PLATON*; Spek, 2006) on the basis of the structural parameters reported here for phase III more closely resembles the data reported by Merritt & Schroeder (1956) than the pattern calculated for phase I (sourcing coordinates from CSD refcode SALOXM). For example, the first six simulated d spacings for form III are 6.89, 6.36, 5.89, 5.74, 5.04 and 4.61 Å; the corresponding data for phase I are 9.59, 6.54, 6.26, 4.82, 4.71 and 4.50 Å, while the data reported by Merritt & Schroeder are 6.76, 6.32, 5.99, 5.68, 5.10 and 4.58 Å. We therefore disagree with Pfluger & Harlow's conclusion regarding the pattern reported by Merritt & Schroeder.

Salicylaldoxime-III is characterized by the formation of hydrogen-bonded chains rather than hydrogen-bonded rings. There are two molecules in the asymmetric unit of salicylaldoxime-III (Fig. 2), which alternate along a hydrogen-bonded chain formed by intermolecular oximic O—H...O hydrogen bonds (Fig. 3). The chains run along the crystallographic c axis, being generated by a $\cdot 2_1$ operation. Intramolecular phenolic O—H...N hydrogen bonds are also formed (Fig. 3).

The chains interact with each other *via* π - π stacking contacts formed between two symmetry-independent molecules. Within these stacking interactions, the atoms forming the phenyl ring of molecule 2 (based on O12 *etc.*) lie between 3.394 (2) and 3.519 (2) Å from the mean plane of molecule 1 (based on O11). The dihedral angle between the two phenyl planes is 2.69 (5)°.

Experimental

Salicylaldehyde was obtained from Acros. The solid was dissolved in chloroform and enough hexane was added to induce precipitation of a small quality of solid. Chloroform was added to redissolve the precipitated solid, and the solution was filtered into a small beaker through glass wool. Crystals of salicylaldehyde grew on allowing the solution to evaporate over the course of 5 d at room temperature.

Crystal data

$C_7H_7NO_2$	$Z = 8$
$M_r = 137.14$	$D_x = 1.398 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.6691 (2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$b = 12.7162 (3) \text{ \AA}$	$T = 150 \text{ K}$
$c = 13.3652 (3) \text{ \AA}$	Block, colourless
$V = 1303.40 (5) \text{ \AA}^3$	$0.42 \times 0.25 \times 0.18 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	15665 measured reflections
ω scans	1886 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2006)	1618 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.740$, $T_{\max} = 0.980$	$R_{\text{int}} = 0.061$
	$\theta_{\max} = 28.9^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F^2) + (0.04P)^2]$,
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
$wR(F^2) = 0.079$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 0.94$	$\Delta\rho_{\max} = 0.22 \text{ e \AA}^{-3}$
1886 reflections	$\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$
194 parameters	Extinction correction: Larson
H atoms treated by a mixture of independent and constrained refinement	(1970), equation 22
	Extinction coefficient: $2.2 (2) \times 10^2$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O11-H11 \cdots O52 ⁱ	0.81 (2)	2.01 (2)	2.8137 (17)	176 (2)
O12-H12 \cdots O51 ⁱⁱ	0.87 (2)	1.99 (2)	2.7945 (18)	155 (2)
O51-H51 \cdots N21	0.86 (2)	1.85 (2)	2.6384 (18)	152 (2)
O52-H52 \cdots N22	0.85 (2)	1.84 (2)	2.6285 (18)	153.4 (19)

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$.

H atoms on O atoms (H11, H51, H12 and H52) were found in a difference Fourier map and their positions refined, subject to O-H distance restraints of 0.84 (5) Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. The remaining H atoms were positioned geometrically and constrained to ride on their host atoms, with C-H = 0.93-0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data on this light-atom structure were collected with Mo $K\alpha$ radiation, and dispersion effects are negligible. The absolute configuration of the crystal used for data collection has not been determined in this study. Friedel pairs were merged.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT; data reduction: SAINT (Bruker, 2003); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: CRYSTALS (Betteridge *et al.*, 2003); molecular graphics: DIAMOND (Brandenburg, 2006) and XP (Sheldrick, 1997); software used to prepare material for publication: CRYSTALS and PLATON (Spek, 2006).

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supporting information

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2-hydroxybenzaldehyde oxime

Crystal data

$C_7H_7NO_2$

$M_r = 137.14$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.6691$ (2) Å

$b = 12.7162$ (3) Å

$c = 13.3652$ (3) Å

$V = 1303.40$ (5) Å³

$Z = 8$

$F(000) = 576$

$D_x = 1.398$ Mg m⁻³

Melting point = 332–334 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7295 reflections

$\theta = 2$ – 28°

$\mu = 0.10$ mm⁻¹

$T = 150$ K

Block, colourless

$0.42 \times 0.25 \times 0.18$ mm

Data collection

Bruker SMART APEX CCD area-detector
diffractometer

Graphite monochromator

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 2006)

$T_{\min} = 0.740$, $T_{\max} = 0.980$

15665 measured reflections

1886 independent reflections

1618 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$

$\theta_{\max} = 28.9^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -10 \rightarrow 9$

$k = -17 \rightarrow 16$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.079$

$S = 0.94$

1886 reflections

194 parameters

4 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: geom/difmap

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F^2) + (0.04P)^2]$,

where $P = [\max(F_o^2, 0) + 2F_c^2]/3$

$(\Delta/\sigma)_{\max} = 0.000152$

$\Delta\rho_{\max} = 0.22$ e Å⁻³

$\Delta\rho_{\min} = -0.28$ e Å⁻³

Extinction correction: Larson (1970), Equation
22

Extinction coefficient: 220 (20)

Special details

Experimental. Used Oxford Cryosystems low-temperature device.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O11	0.2811 (2)	0.27564 (10)	0.02463 (9)	0.0368
N21	0.2508 (2)	0.22621 (10)	0.11662 (10)	0.0274
C31	0.1828 (2)	0.13549 (13)	0.10589 (12)	0.0264
C41	0.1437 (2)	0.07010 (13)	0.19232 (12)	0.0242
C51	0.1779 (2)	0.10191 (12)	0.29136 (12)	0.0238
O51	0.24867 (17)	0.19816 (9)	0.31221 (9)	0.0286
C61	0.1421 (2)	0.03505 (14)	0.37079 (12)	0.0291
C71	0.0716 (2)	-0.06313 (14)	0.35366 (14)	0.0322
C81	0.0356 (2)	-0.09565 (14)	0.25657 (15)	0.0313
C91	0.0725 (2)	-0.02997 (13)	0.17736 (13)	0.0285
O12	0.7865 (2)	0.25663 (11)	0.48433 (10)	0.0407
N22	0.7161 (2)	0.16559 (10)	0.44076 (11)	0.0286
C32	0.7209 (2)	0.17066 (12)	0.34528 (13)	0.0275
C42	0.6555 (2)	0.08496 (13)	0.28352 (12)	0.0246
C52	0.5891 (2)	-0.00828 (12)	0.32393 (11)	0.0243
O52	0.58579 (17)	-0.02492 (9)	0.42557 (8)	0.0308
C62	0.5244 (2)	-0.08673 (13)	0.26243 (13)	0.0286
C72	0.5285 (2)	-0.07423 (14)	0.15954 (13)	0.0303
C82	0.5959 (2)	0.01694 (14)	0.11784 (12)	0.0298
C92	0.6577 (2)	0.09598 (13)	0.17902 (12)	0.0275
H11	0.315 (3)	0.3334 (17)	0.0410 (18)	0.0537*
H31	0.1580	0.1096	0.0427	0.0315*
H51	0.270 (3)	0.2251 (17)	0.2545 (15)	0.0427*
H61	0.1667	0.0573	0.4365	0.0331*
H71	0.0482	-0.1061	0.4091	0.0397*
H81	-0.0141	-0.1634	0.2458	0.0370*
H91	0.0544	-0.0523	0.1101	0.0330*
H12	0.768 (3)	0.2501 (18)	0.5479 (17)	0.0593*
H32	0.7654	0.2305	0.3130	0.0324*
H52	0.624 (3)	0.0323 (16)	0.4501 (15)	0.0450*
H62	0.4761	-0.1487	0.2923	0.0336*
H72	0.4791	-0.1260	0.1180	0.0364*
H82	0.5949	0.0260	0.0472	0.0363*
H92	0.7026	0.1581	0.1508	0.0322*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0607 (9)	0.0291 (7)	0.0207 (6)	-0.0041 (7)	0.0001 (6)	0.0029 (5)
N21	0.0348 (8)	0.0267 (7)	0.0206 (6)	0.0048 (6)	-0.0006 (6)	0.0018 (5)
C31	0.0299 (8)	0.0279 (8)	0.0213 (7)	0.0046 (7)	-0.0029 (7)	-0.0036 (7)
C41	0.0215 (7)	0.0248 (8)	0.0264 (8)	0.0037 (7)	-0.0003 (7)	-0.0010 (6)
C51	0.0237 (8)	0.0222 (8)	0.0253 (8)	0.0036 (6)	0.0011 (7)	-0.0017 (6)
O51	0.0410 (7)	0.0236 (5)	0.0213 (5)	-0.0023 (6)	0.0000 (6)	-0.0016 (5)
C61	0.0332 (8)	0.0293 (8)	0.0246 (8)	0.0022 (8)	0.0040 (7)	-0.0017 (7)

C71	0.0338 (9)	0.0281 (9)	0.0348 (9)	0.0023 (8)	0.0098 (8)	0.0033 (7)
C81	0.0283 (8)	0.0250 (8)	0.0408 (10)	-0.0019 (7)	0.0048 (8)	-0.0048 (7)
C91	0.0263 (8)	0.0281 (8)	0.0310 (9)	0.0010 (8)	-0.0024 (7)	-0.0070 (7)
O12	0.0677 (10)	0.0285 (6)	0.0259 (6)	-0.0054 (7)	-0.0019 (7)	-0.0051 (6)
N22	0.0384 (8)	0.0223 (6)	0.0252 (7)	0.0037 (7)	-0.0003 (6)	-0.0021 (6)
C32	0.0339 (9)	0.0233 (8)	0.0254 (8)	0.0024 (7)	0.0024 (7)	0.0018 (6)
C42	0.0243 (7)	0.0263 (8)	0.0231 (7)	0.0057 (7)	0.0017 (7)	-0.0012 (6)
C52	0.0250 (8)	0.0276 (8)	0.0204 (7)	0.0056 (7)	0.0023 (7)	0.0005 (7)
O52	0.0442 (7)	0.0275 (6)	0.0208 (6)	-0.0037 (6)	0.0028 (6)	0.0023 (5)
C62	0.0287 (8)	0.0271 (9)	0.0300 (9)	0.0009 (7)	-0.0016 (7)	0.0003 (7)
C72	0.0290 (8)	0.0327 (9)	0.0292 (9)	0.0051 (8)	-0.0049 (8)	-0.0069 (7)
C82	0.0314 (8)	0.0392 (9)	0.0187 (7)	0.0085 (8)	-0.0003 (7)	-0.0018 (7)
C92	0.0305 (8)	0.0290 (8)	0.0229 (8)	0.0043 (8)	0.0021 (7)	0.0030 (7)

Geometric parameters (Å, °)

O11—N21	1.4003 (17)	O12—N22	1.4039 (19)
O11—H11	0.81 (2)	O12—H12	0.86 (2)
N21—C31	1.274 (2)	N22—C32	1.278 (2)
C31—C41	1.454 (2)	C32—C42	1.456 (2)
C31—H31	0.927	C32—H32	0.939
C41—C51	1.409 (2)	C42—C52	1.399 (2)
C41—C91	1.399 (2)	C42—C92	1.404 (2)
C51—O51	1.3677 (19)	C52—O52	1.3751 (19)
C51—C61	1.388 (2)	C52—C62	1.385 (2)
O51—H51	0.86 (2)	O52—H52	0.85 (2)
C61—C71	1.379 (3)	C62—C72	1.385 (2)
C61—H61	0.942	C62—H62	0.958
C71—C81	1.390 (3)	C72—C82	1.386 (2)
C71—H71	0.938	C72—H72	0.941
C81—C91	1.378 (2)	C82—C92	1.380 (2)
C81—H81	0.953	C82—H82	0.951
C91—H91	0.953	C92—H92	0.940
N21—O11—H11	102.9 (17)	N22—O12—H12	105.4 (16)
O11—N21—C31	112.06 (13)	O12—N22—C32	111.20 (14)
N21—C31—C41	120.83 (15)	N22—C32—C42	121.23 (16)
N21—C31—H31	120.6	N22—C32—H32	120.7
C41—C31—H31	118.6	C42—C32—H32	118.1
C31—C41—C51	122.95 (14)	C32—C42—C52	122.73 (14)
C31—C41—C91	119.13 (15)	C32—C42—C92	119.02 (15)
C51—C41—C91	117.91 (15)	C52—C42—C92	118.24 (15)
C41—C51—O51	121.49 (14)	C42—C52—O52	121.24 (14)
C41—C51—C61	120.41 (15)	C42—C52—C62	120.80 (14)
O51—C51—C61	118.10 (14)	O52—C52—C62	117.96 (15)
C51—O51—H51	104.4 (14)	C52—O52—H52	104.1 (14)
C51—C61—C71	120.34 (16)	C52—C62—C72	119.92 (16)
C51—C61—H61	119.3	C52—C62—H62	118.9

C71—C61—H61	120.4	C72—C62—H62	121.2
C61—C71—C81	120.15 (17)	C62—C72—C82	120.24 (16)
C61—C71—H71	118.1	C62—C72—H72	119.8
C81—C71—H71	121.7	C82—C72—H72	119.9
C71—C81—C91	119.74 (16)	C72—C82—C92	119.94 (15)
C71—C81—H81	119.3	C72—C82—H82	119.8
C91—C81—H81	120.9	C92—C82—H82	120.2
C41—C91—C81	121.45 (16)	C42—C92—C82	120.85 (16)
C41—C91—H91	117.5	C42—C92—H92	119.1
C81—C91—H91	121.0	C82—C92—H92	120.0

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O11—H11...O52 ⁱ	0.81 (2)	2.01 (2)	2.8137 (17)	176 (2)
O12—H12...O51 ⁱⁱ	0.87 (2)	1.99 (2)	2.7945 (18)	155 (2)
O51—H51...N21	0.86 (2)	1.85 (2)	2.6384 (18)	152 (2)
O52—H52...N22	0.85 (2)	1.84 (2)	2.6285 (18)	153 (2)

Symmetry codes: (i) $-x+1, y+1/2, -z+1/2$; (ii) $x+1/2, -y+1/2, -z+1$.