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# *N*,*N*′,*N*″,*N*‴-Tetrakis(2-hydroxybenzylidene)biphenyl-3,3′,4,4′-tetramine dimethylformamide solvate

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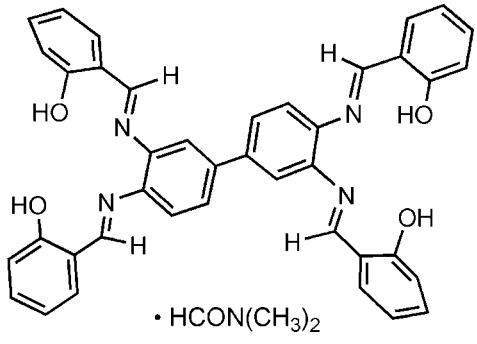
Key indicators: single-crystal X-ray study; $T$ = 187 K; mean $\sigma$(C–C) = 0.004 Å; disorder in solvent or counterion; $R$ factor = 0.066; $wR$ factor = 0.200; data-to-parameter ratio = 12.9.

The title compound, $C_{40}H_{30}N_4O_4\cdot C_3H_7NO$, is an *O*-hydroxy Schiff base. The molecule, which lies on a symmetry centre, exists as an enol–imine tautomer, in which two independent intramolecular O—H···N hydrogen bonds are formed.

## Related literature

For related literature, see: Calligaris *et al.* (1972); Filarowski *et al.* (2003); Kǫsar *et al.* (2005); Maslen & Waters (1975); Stewart & Lingafelter (1959).




## Experimental

### *Crystal data*

$C_{40}H_{30}N_4O_4\cdot C_3H_7NO$
$M_r$ = 703.78
Monoclinic, $P2_1/n$
$a$ = 15.2109 (19) Å
$b$ = 6.3608 (8) Å
$c$ = 20.119 (3) Å
$\beta$ = 93.882 (2)°
$V$ = 1942.1 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 187 (2) K
0.30 × 0.21 × 0.04 mm

### *Data collection*

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.976, $T_{max}$ = 0.997
9558 measured reflections
3426 independent reflections
2490 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.201
$S$ = 1.02
3426 reflections
266 parameters
22 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$A$···N2 | 0.84 | 1.82 | 2.562 (3) | 147 |
| O1—H1···N1 | 0.84 | 1.88 | 2.629 (3) | 147 |

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2088).

## References

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3885 [ **doi:10.1107/S1600536807040883** ]

# *N*,*N*',*N*'',*N*'''-Tetrakis(2-hydroxybenzylidene)biphenyl-3,3',4,4'-tetramine dimethylformamide solvate

**X.-Q. Che**

## Comment

The *O*-Hydroxy Schiff bases derived from the reaction of *o*-hydroxy aldehydes with aniline have been extensively examined (Steward & Lingafelter, 1959; Calligaris *et al.*, 1972; Maslen & Waters, 1975). There are two possible types of intramolecular hydrogen bonds in Schiff bases, namely keto-amine (N—H···O) and enol-imine (N···H—O) tautomeric forms. The present X-ray investigation shows that the title compound, $C_{40}H_{30}N_4O4.C_3H_7NO$, (I), prefers the enol-imine tautomeric form rather than the keto-amine tautomeric form.

The molecule is halved by a centre of symmetry; the asymmetric unit (Fig. 1), presents two strong intramolecular O—H···N hydrogen bond interactions (Table. 1). These O—H···N contacts (2.562 (3)–2.629 (3) Å) satisfy the corresponding distances of strong hydrogen bonds in the literature (Filarowski *et al.*, 2003; K˛osar *et al.*, 2005). Non-H atoms in (I) are not coplanar, the mean deviation of the atoms from the least-squares plane being 0.293 Å. The compound crystallizes with a DMF solvato molecule.

## Experimental

All reagents were purchased (Adrich) and used without further purification. Compound (I) was synthesized by mixing 3,3'-diaminobenzidine (2.14 g, 10 mmol) and salicylaldehyde (4.88 g, 40 mmol) in ethanol (100 ml) at 343 K for 20 min. Then compound (I) (0.631 g, 1 mmol) was dissolved in 20 ml DMF. After heating at 373 K for 5 min, the mixture was allowed to cool and evaporate naturally. Yellow plate-shaped crystals of (I) suitable for single-crystal X-ray diffraction were obtained by evaporating the mixture at room temperature for a period of 4 d. Analysis found: C 73.5, H 5.3, N 9.8%; $C_{43}H_{37}N_5O_5$ requires: C 73.38, H 5.30, N 9.95%.

## Refinement

All H atoms were visible in difference Fourier maps but were placed in calculated positions with C—H= 0.95 Å C–H: 0.98 Å; C—$H_3$ and O—H: 0.84 Å, in all cases with $U_{iso}$(H) = 1.2 $U_{eq}$(C) and 1.5 $U_{eq}$(O). Non-H atoms were refined anisotropically. The maximum positive peak of 0.337 e Å$^{-3}$ in the final difference electron density map was located 1.03 Å from atom C21. The crystal data were collected at 187 K. The shape of some displacement ellipsoids in the DMF solvate suggests some kind of disorder.

**Figures**

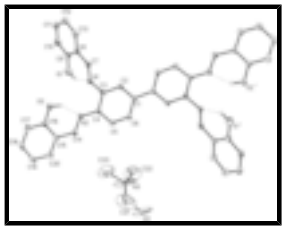

Fig. 1. A view of the molecule of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids. The dashed line indicates the intramolecular hydrogen bonds. Some H atoms are omitted for clarity. Symmetry codes: i 1 − *x*, 2 − *y*, −*z*.

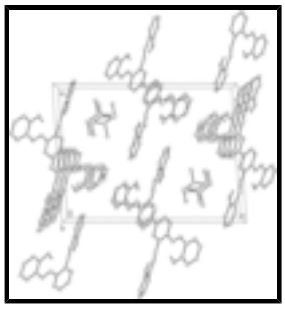

Fig. 2. A packing diagram for (I).

## *N*,*N*',*N*'',*N*'''-Tetrakis(2-hydroxybenzylidene)biphenyl-3,3',4,4'-tetramine dimethylformamide solvate

*Crystal data*

| | |
|---|---|
| $C_{40}H_{30}N_4O_4·C_3H_7NO$ | $F_{000}$ = 740 |
| $M_r$ = 703.78 | $D_x$ = 1.203 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 15.2109 (19) Å | Cell parameters from 864 reflections |
| $b$ = 6.3608 (8) Å | θ = 3.7–22.8º |
| $c$ = 20.119 (3) Å | μ = 0.08 mm$^{-1}$ |
| β = 93.882 (2)º | $T$ = 187 (2) K |
| $V$ = 1942.1 (4) Å$^3$ | Plate, yellow |
| $Z$ = 2 | 0.30 × 0.21 × 0.04 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX II CCD diffractometer | 3426 independent reflections |
| Radiation source: fine-focus sealed tube | 2490 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.033 |
| $T$ = 187(2) K | $\theta_{max}$ = 25.0º |
| φ and ω scans | $\theta_{min}$ = 3.3º |
| Absorption correction: multi-scan (SADABS; Bruker, 2004) | $h$ = −18→11 |
| $T_{min}$ = 0.976, $T_{max}$ = 0.997 | $k$ = −7→7 |
| 9558 measured reflections | $l$ = −23→23 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.066$ | H-atom parameters constrained |
| $wR(F^2) = 0.201$ | $w = 1/[\sigma^2(F_o^2) + (0.1084P)^2 + 1.0323P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.03$ | $(\Delta/\sigma)_{max} = 0.008$ |
| 3426 reflections | $\Delta\rho_{max} = 0.34$ e Å$^{-3}$ |
| 266 parameters | $\Delta\rho_{min} = -0.21$ e Å$^{-3}$ |
| 22 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.48192 (15) | 0.9023 (4) | 0.01409 (12) | 0.0269 (6) | |
| C2 | 0.39813 (16) | 0.8299 (4) | −0.00735 (13) | 0.0307 (6) | |
| H2 | 0.3646 | 0.9065 | −0.0407 | 0.037* | |
| C3 | 0.36209 (16) | 0.6484 (4) | 0.01879 (12) | 0.0288 (6) | |
| C4 | 0.41093 (16) | 0.5308 (4) | 0.06705 (12) | 0.0266 (6) | |
| C5 | 0.49461 (17) | 0.6027 (4) | 0.08812 (14) | 0.0357 (7) | |
| H5 | 0.5288 | 0.5256 | 0.1210 | 0.043* | |
| C6 | 0.52886 (17) | 0.7832 (4) | 0.06225 (14) | 0.0356 (7) | |
| H6 | 0.5863 | 0.8275 | 0.0778 | 0.043* | |
| C7 | 0.21535 (18) | 0.7113 (5) | −0.01503 (13) | 0.0368 (7) | |
| H7 | 0.2263 | 0.8524 | −0.0014 | 0.044* | |
| C8 | 0.12903 (18) | 0.6584 (5) | −0.04537 (14) | 0.0383 (7) | |
| C9 | 0.1089 (2) | 0.4546 (5) | −0.06790 (15) | 0.0437 (7) | |
| C10 | 0.0257 (2) | 0.4126 (5) | −0.09811 (17) | 0.0572 (9) | |
| H10 | 0.0119 | 0.2752 | −0.1141 | 0.069* | |
| C11 | −0.0367 (2) | 0.5690 (6) | −0.10487 (17) | 0.0569 (9) | |
| H11 | −0.0934 | 0.5381 | −0.1253 | 0.068* | |

| | | | | | |
|---|---|---|---|---|---|
| C12 | −0.0181 (2) | 0.7696 (6) | −0.08249 (18) | 0.0560 (9) | |
| H12 | −0.0616 | 0.8769 | −0.0871 | 0.067* | |
| C13 | 0.06497 (19) | 0.8126 (5) | −0.05303 (16) | 0.0474 (8) | |
| H13 | 0.0782 | 0.9511 | −0.0378 | 0.057* | |
| C14 | 0.40784 (17) | 0.2289 (4) | 0.13573 (13) | 0.0330 (6) | |
| H14 | 0.4662 | 0.2592 | 0.1529 | 0.040* | |
| C15 | 0.36358 (17) | 0.0459 (4) | 0.15970 (12) | 0.0308 (6) | |
| C16 | 0.27688 (17) | −0.0025 (4) | 0.13528 (13) | 0.0325 (6) | |
| C17 | 0.2362 (2) | −0.1822 (4) | 0.15791 (15) | 0.0413 (7) | |
| H17 | 0.1780 | −0.2159 | 0.1412 | 0.050* | |
| C18 | 0.2793 (2) | −0.3114 (4) | 0.20413 (14) | 0.0428 (8) | |
| H18 | 0.2510 | −0.4345 | 0.2187 | 0.051* | |
| C19 | 0.3637 (2) | −0.2632 (5) | 0.22955 (15) | 0.0456 (8) | |
| H19 | 0.3930 | −0.3514 | 0.2621 | 0.055* | |
| C20 | 0.4049 (2) | −0.0865 (5) | 0.20737 (14) | 0.0422 (7) | |
| H20 | 0.4628 | −0.0540 | 0.2249 | 0.051* | |
| C21 | 0.7766 (7) | 0.589 (3) | 0.3218 (5) | 0.228 (9) | 0.50 |
| H21 | 0.7556 | 0.5590 | 0.3642 | 0.273* | 0.50 |
| C22 | 0.7287 (11) | 0.555 (3) | 0.1895 (4) | 0.237 (10) | 0.50 |
| H22A | 0.7875 | 0.5964 | 0.1772 | 0.284* | 0.50 |
| H22B | 0.6840 | 0.6357 | 0.1628 | 0.284* | 0.50 |
| H22C | 0.7199 | 0.4049 | 0.1811 | 0.284* | 0.50 |
| C23 | 0.6224 (7) | 0.556 (3) | 0.2722 (6) | 0.183 (7) | 0.50 |
| H23A | 0.6135 | 0.5440 | 0.3199 | 0.219* | 0.50 |
| H23B | 0.6050 | 0.4243 | 0.2498 | 0.219* | 0.50 |
| H23C | 0.5863 | 0.6716 | 0.2531 | 0.219* | 0.50 |
| N1 | 0.27785 (14) | 0.5760 (3) | −0.00565 (11) | 0.0347 (6) | |
| N2 | 0.37056 (13) | 0.3513 (3) | 0.09206 (10) | 0.0287 (5) | |
| N3 | 0.7204 (5) | 0.601 (3) | 0.2628 (3) | 0.253 (10) | 0.50 |
| O1 | 0.16868 (15) | 0.2970 (3) | −0.06137 (13) | 0.0576 (7) | |
| H1 | 0.2155 | 0.3424 | −0.0420 | 0.086* | |
| O2 | 0.23246 (12) | 0.1201 (3) | 0.08988 (11) | 0.0465 (6) | |
| H2A | 0.2659 | 0.2155 | 0.0775 | 0.070* | |
| O3 | 0.8612 (5) | 0.622 (2) | 0.3127 (4) | 0.182 (6) | 0.50 |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0278 (13) | 0.0250 (13) | 0.0286 (13) | −0.0023 (11) | 0.0066 (10) | −0.0032 (10) |
| C2 | 0.0317 (14) | 0.0255 (14) | 0.0345 (14) | −0.0033 (11) | −0.0001 (11) | 0.0041 (11) |
| C3 | 0.0314 (13) | 0.0243 (13) | 0.0307 (13) | −0.0059 (11) | 0.0017 (11) | −0.0013 (11) |
| C4 | 0.0281 (13) | 0.0226 (13) | 0.0298 (13) | −0.0025 (10) | 0.0073 (10) | −0.0010 (10) |
| C5 | 0.0327 (14) | 0.0343 (15) | 0.0393 (15) | −0.0036 (12) | −0.0026 (11) | 0.0092 (12) |
| C6 | 0.0275 (13) | 0.0340 (15) | 0.0447 (16) | −0.0095 (12) | −0.0028 (12) | 0.0072 (13) |
| C7 | 0.0381 (15) | 0.0325 (15) | 0.0392 (16) | −0.0121 (13) | −0.0018 (12) | 0.0045 (12) |
| C8 | 0.0343 (15) | 0.0402 (16) | 0.0394 (15) | −0.0124 (13) | −0.0035 (12) | 0.0104 (13) |
| C9 | 0.0447 (17) | 0.0413 (17) | 0.0434 (17) | −0.0120 (14) | −0.0089 (13) | 0.0120 (14) |
| C10 | 0.058 (2) | 0.050 (2) | 0.061 (2) | −0.0285 (18) | −0.0165 (16) | 0.0119 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.0399 (18) | 0.064 (2) | 0.064 (2) | −0.0195 (17) | −0.0152 (15) | 0.0195 (18) |
| C12 | 0.0387 (17) | 0.060 (2) | 0.068 (2) | −0.0083 (16) | −0.0056 (15) | 0.0176 (18) |
| C13 | 0.0381 (16) | 0.0464 (18) | 0.057 (2) | −0.0076 (15) | −0.0029 (14) | 0.0071 (15) |
| C14 | 0.0320 (14) | 0.0311 (14) | 0.0357 (14) | −0.0051 (12) | 0.0010 (11) | 0.0022 (12) |
| C15 | 0.0375 (14) | 0.0261 (13) | 0.0294 (13) | −0.0015 (11) | 0.0077 (11) | 0.0009 (11) |
| C16 | 0.0391 (15) | 0.0269 (14) | 0.0322 (14) | −0.0030 (12) | 0.0069 (11) | −0.0009 (11) |
| C17 | 0.0448 (17) | 0.0365 (16) | 0.0437 (16) | −0.0122 (14) | 0.0103 (13) | −0.0018 (13) |
| C18 | 0.064 (2) | 0.0257 (14) | 0.0411 (16) | −0.0081 (14) | 0.0218 (15) | 0.0007 (12) |
| C19 | 0.064 (2) | 0.0335 (16) | 0.0405 (16) | 0.0023 (15) | 0.0102 (15) | 0.0123 (13) |
| C20 | 0.0430 (16) | 0.0407 (17) | 0.0426 (16) | −0.0001 (14) | 0.0010 (13) | 0.0077 (13) |
| C21 | 0.30 (2) | 0.33 (2) | 0.044 (6) | 0.02 (2) | −0.022 (11) | 0.011 (10) |
| C22 | 0.200 (15) | 0.46 (3) | 0.049 (6) | 0.008 (18) | 0.023 (8) | 0.047 (12) |
| C23 | 0.105 (9) | 0.36 (2) | 0.086 (8) | 0.054 (12) | 0.013 (7) | 0.007 (12) |
| N1 | 0.0331 (12) | 0.0332 (13) | 0.0369 (12) | −0.0110 (11) | −0.0053 (9) | 0.0070 (10) |
| N2 | 0.0307 (11) | 0.0237 (11) | 0.0321 (12) | −0.0026 (9) | 0.0059 (9) | 0.0012 (9) |
| N3 | 0.119 (8) | 0.60 (3) | 0.032 (4) | 0.202 (14) | −0.029 (4) | −0.015 (9) |
| O1 | 0.0574 (14) | 0.0385 (12) | 0.0737 (17) | −0.0128 (11) | −0.0191 (12) | 0.0003 (11) |
| O2 | 0.0399 (11) | 0.0408 (12) | 0.0574 (13) | −0.0139 (9) | −0.0071 (10) | 0.0143 (10) |
| O3 | 0.068 (4) | 0.384 (16) | 0.087 (5) | 0.092 (8) | −0.037 (4) | −0.018 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.389 (4) | C14—C15 | 1.444 (3) |
| C1—C2 | 1.396 (3) | C14—H14 | 0.9500 |
| C1—C1$^{i}$ | 1.487 (5) | C15—C20 | 1.394 (4) |
| C2—C3 | 1.396 (3) | C15—C16 | 1.410 (4) |
| C2—H2 | 0.9500 | C16—O2 | 1.347 (3) |
| C3—C4 | 1.399 (4) | C16—C17 | 1.391 (4) |
| C3—N1 | 1.418 (3) | C17—C18 | 1.374 (4) |
| C4—C5 | 1.391 (4) | C17—H17 | 0.9500 |
| C4—N2 | 1.406 (3) | C18—C19 | 1.384 (5) |
| C5—C6 | 1.378 (4) | C18—H18 | 0.9500 |
| C5—H5 | 0.9500 | C19—C20 | 1.375 (4) |
| C6—H6 | 0.9500 | C19—H19 | 0.9500 |
| C7—N1 | 1.287 (4) | C20—H20 | 0.9500 |
| C7—C8 | 1.449 (4) | C21—O3 | 1.328 (8) |
| C7—H7 | 0.9500 | C21—N3 | 1.418 (7) |
| C8—C13 | 1.384 (4) | C21—H21 | 0.9500 |
| C8—C9 | 1.401 (4) | C22—N3 | 1.515 (7) |
| C9—O1 | 1.354 (4) | C22—H22A | 0.9800 |
| C9—C10 | 1.392 (4) | C22—H22B | 0.9800 |
| C10—C11 | 1.375 (5) | C22—H22C | 0.9800 |
| C10—H10 | 0.9500 | C23—N3 | 1.542 (8) |
| C11—C12 | 1.376 (5) | C23—H23A | 0.9800 |
| C11—H11 | 0.9500 | C23—H23B | 0.9800 |
| C12—C13 | 1.386 (4) | C23—H23C | 0.9800 |
| C12—H12 | 0.9500 | O1—H1 | 0.8400 |
| C13—H13 | 0.9500 | O2—H2A | 0.8400 |
| C14—N2 | 1.277 (3) | | |

| | | | |
|---|---|---|---|
| C6—C1—C2 | 116.8 (2) | C15—C14—H14 | 119.2 |
| C6—C1—C1$^{i}$ | 122.4 (3) | C20—C15—C16 | 118.4 (2) |
| C2—C1—C1$^{i}$ | 120.8 (3) | C20—C15—C14 | 121.2 (2) |
| C1—C2—C3 | 122.0 (2) | C16—C15—C14 | 120.4 (2) |
| C1—C2—H2 | 119.0 | O2—C16—C17 | 118.9 (2) |
| C3—C2—H2 | 119.0 | O2—C16—C15 | 121.6 (2) |
| C2—C3—C4 | 120.0 (2) | C17—C16—C15 | 119.5 (3) |
| C2—C3—N1 | 120.4 (2) | C18—C17—C16 | 120.7 (3) |
| C4—C3—N1 | 119.5 (2) | C18—C17—H17 | 119.7 |
| C5—C4—C3 | 117.9 (2) | C16—C17—H17 | 119.7 |
| C5—C4—N2 | 124.8 (2) | C17—C18—C19 | 120.4 (3) |
| C3—C4—N2 | 117.3 (2) | C17—C18—H18 | 119.8 |
| C6—C5—C4 | 121.4 (3) | C19—C18—H18 | 119.8 |
| C6—C5—H5 | 119.3 | C20—C19—C18 | 119.5 (3) |
| C4—C5—H5 | 119.3 | C20—C19—H19 | 120.2 |
| C5—C6—C1 | 121.9 (2) | C18—C19—H19 | 120.2 |
| C5—C6—H6 | 119.0 | C19—C20—C15 | 121.5 (3) |
| C1—C6—H6 | 119.0 | C19—C20—H20 | 119.2 |
| N1—C7—C8 | 123.1 (3) | C15—C20—H20 | 119.2 |
| N1—C7—H7 | 118.5 | O3—C21—N3 | 114.2 (9) |
| C8—C7—H7 | 118.5 | O3—C21—H21 | 122.9 |
| C13—C8—C9 | 118.9 (3) | N3—C21—H21 | 122.9 |
| C13—C8—C7 | 119.6 (3) | N3—C22—H22A | 109.5 |
| C9—C8—C7 | 121.5 (3) | N3—C22—H22B | 109.5 |
| O1—C9—C10 | 119.0 (3) | H22A—C22—H22B | 109.5 |
| O1—C9—C8 | 121.5 (3) | N3—C22—H22C | 109.5 |
| C10—C9—C8 | 119.4 (3) | H22A—C22—H22C | 109.5 |
| C11—C10—C9 | 120.3 (3) | H22B—C22—H22C | 109.5 |
| C11—C10—H10 | 119.8 | N3—C23—H23A | 109.5 |
| C9—C10—H10 | 119.8 | N3—C23—H23B | 109.5 |
| C10—C11—C12 | 120.9 (3) | H23A—C23—H23B | 109.5 |
| C10—C11—H11 | 119.5 | N3—C23—H23C | 109.5 |
| C12—C11—H11 | 119.5 | H23A—C23—H23C | 109.5 |
| C11—C12—C13 | 118.9 (3) | H23B—C23—H23C | 109.5 |
| C11—C12—H12 | 120.5 | C7—N1—C3 | 118.4 (2) |
| C13—C12—H12 | 120.5 | C14—N2—C4 | 123.8 (2) |
| C8—C13—C12 | 121.5 (3) | C21—N3—C22 | 136.0 (11) |
| C8—C13—H13 | 119.3 | C21—N3—C23 | 114.8 (9) |
| C12—C13—H13 | 119.3 | C22—N3—C23 | 103.3 (10) |
| N2—C14—C15 | 121.7 (2) | C9—O1—H1 | 109.5 |
| N2—C14—H14 | 119.2 | C16—O2—H2A | 109.5 |
| C6—C1—C2—C3 | 1.0 (4) | C7—C8—C13—C12 | 179.2 (3) |
| C1$^{i}$—C1—C2—C3 | −179.2 (3) | C11—C12—C13—C8 | −0.4 (5) |
| C1—C2—C3—C4 | −1.3 (4) | N2—C14—C15—C20 | 180.0 (3) |
| C1—C2—C3—N1 | −178.1 (2) | N2—C14—C15—C16 | −0.2 (4) |
| C2—C3—C4—C5 | 0.9 (4) | C20—C15—C16—O2 | 179.0 (2) |
| N1—C3—C4—C5 | 177.7 (2) | C14—C15—C16—O2 | −0.8 (4) |
| C2—C3—C4—N2 | 179.2 (2) | C20—C15—C16—C17 | −1.7 (4) |

| | | | |
|---|---|---|---|
| N1—C3—C4—N2 | −4.0 (3) | C14—C15—C16—C17 | 178.5 (2) |
| C3—C4—C5—C6 | −0.3 (4) | O2—C16—C17—C18 | 179.9 (3) |
| N2—C4—C5—C6 | −178.4 (2) | C15—C16—C17—C18 | 0.6 (4) |
| C4—C5—C6—C1 | 0.0 (4) | C16—C17—C18—C19 | 0.8 (4) |
| C2—C1—C6—C5 | −0.3 (4) | C17—C18—C19—C20 | −1.1 (4) |
| C1$^{i}$—C1—C6—C5 | 179.9 (3) | C18—C19—C20—C15 | 0.0 (4) |
| N1—C7—C8—C13 | 179.3 (3) | C16—C15—C20—C19 | 1.4 (4) |
| N1—C7—C8—C9 | −1.4 (4) | C14—C15—C20—C19 | −178.8 (3) |
| C13—C8—C9—O1 | −179.6 (3) | C8—C7—N1—C3 | 174.3 (2) |
| C7—C8—C9—O1 | 1.1 (4) | C2—C3—N1—C7 | −44.2 (4) |
| C13—C8—C9—C10 | 0.8 (4) | C4—C3—N1—C7 | 139.0 (3) |
| C7—C8—C9—C10 | −178.5 (3) | C15—C14—N2—C4 | −179.8 (2) |
| O1—C9—C10—C11 | 179.4 (3) | C5—C4—N2—C14 | −2.5 (4) |
| C8—C9—C10—C11 | −1.0 (5) | C3—C4—N2—C14 | 179.4 (2) |
| C9—C10—C11—C12 | 0.5 (5) | O3—C21—N3—C22 | 31 (3) |
| C10—C11—C12—C13 | 0.2 (5) | O3—C21—N3—C23 | 178.4 (16) |
| C9—C8—C13—C12 | −0.1 (5) | | |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2A···N2 | 0.84 | 1.82 | 2.562 (3) | 147 |
| O1—H1···N1 | 0.84 | 1.88 | 2.629 (3) | 147 |

**Fig. 1**

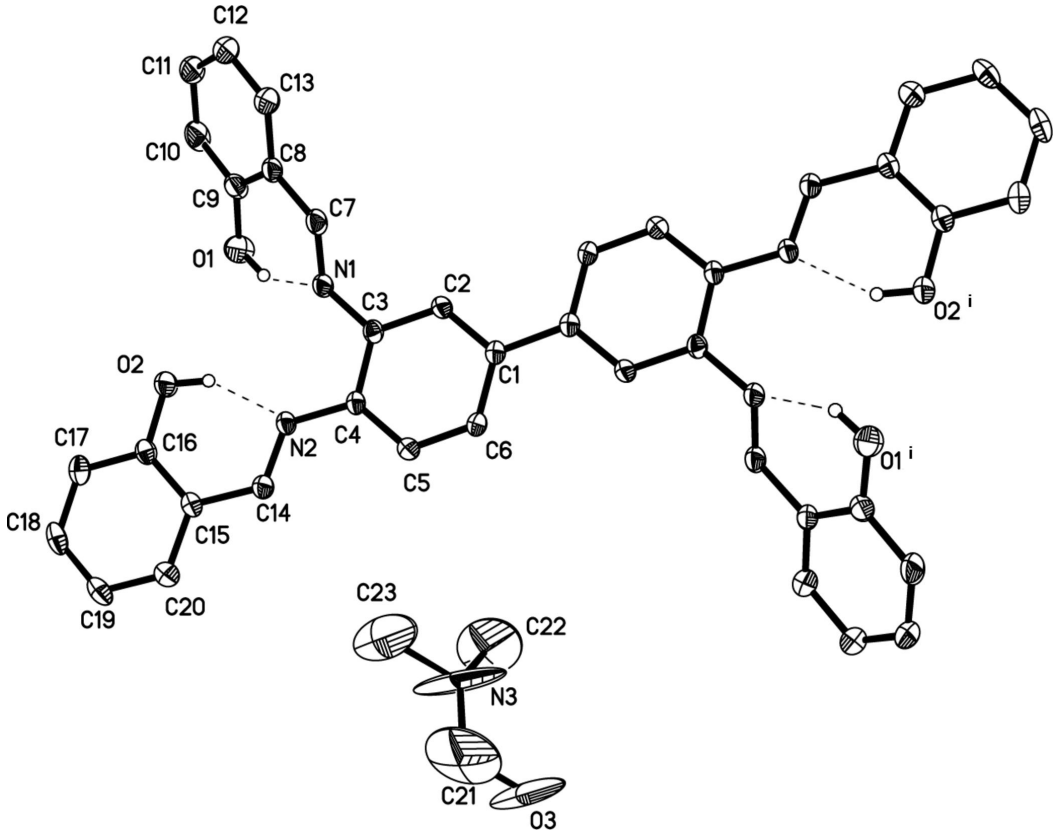
C12
C11
C13
C10
C8
C9
C7
O1
N1
C3
C2
O2 i
O2
C1
C17
C16
N2
C4
C6
O1 i
C5
C18
C14
C15
C23
C22
C20
C19
N3
C21
O3

**Fig. 2**