

4-Chloro-*N'*-(*Z*)-4-(dimethylamino)-benzylidene]benzohydrazide mono-hydrate

Hoong-Kun Fun,^{a*} P. S. Patil,^b Samuel Robinson Jebas,^{a‡} K. V. Sujith^c and B. Kalluraya^c

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of Studies in Physics, Mangalore University, Mangalagangotri, Mangalore 574 199, India, and ^cDepartment of Studies in Chemistry, Mangalore University, Mangalagangotri, Mangalore 574 199, India
Correspondence e-mail: hkfun@usm.my

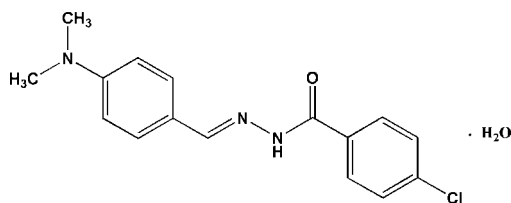
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.114; data-to-parameter ratio = 21.0.

In the title compound, $\text{C}_{16}\text{H}_{16}\text{ClN}_3\text{O}\cdot\text{H}_2\text{O}$, the dihedral angle between the two aromatic rings is $44.58(11)^\circ$. The N atom of the dimethylamino group adopts a pyramidal configuration. In the crystal structure, molecules are linked into a two-dimensional network parallel to the (001) plane by intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{N}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds involving the water molecule and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds. In addition, $\text{C}-\text{H}\cdots\pi$ interactions are observed.

Related literature

For the biological activities of hydrazones, see: Bedia *et al.* (2006); Rollas *et al.* (2002); Terzioglu & Gürsoy (2003); Duraisamy *et al.* (2008); Singh *et al.* (1992); Ergenç & Günay, (1998); Durgun *et al.* (1993). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{16}\text{ClN}_3\text{O}\cdot\text{H}_2\text{O}$

$M_r = 319.78$

[‡] Permanent address: Department of Physics, Karunya University, Karunya Nagar, Coimbatore 641 114, India.

Orthorhombic, $P2_12_12_1$
 $a = 6.4418(1)$ Å
 $b = 6.9344(1)$ Å
 $c = 33.8083(7)$ Å
 $V = 1510.22(4)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 100.0(1)$ K
 $0.32 \times 0.16 \times 0.07$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2005)
 $T_{\min} = 0.921$, $T_{\max} = 0.981$

12336 measured reflections
4311 independent reflections
3301 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.114$
 $S = 1.01$
4311 reflections
205 parameters
2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.34$ e Å⁻³
Absolute structure: Flack (1983),
1724 Friedel pairs
Flack parameter: $-0.14(7)$

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 are the centroids of the C1–C6 and C9–C14 rings, respectively.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{O1W}-\text{H1W1}\cdots\text{N3}^{\text{i}}$ | 0.84 | 2.28 | 3.045 (2) | 152 |
| $\text{N1}-\text{H1N1}\cdots\text{O1W}$ | 0.86 (1) | 2.00 (1) | 2.843 (3) | 169 (2) |
| $\text{O1W}-\text{H2W1}\cdots\text{O1}^{\text{ii}}$ | 0.84 | 2.02 | 2.790 (2) | 152 |
| $\text{O1W}-\text{H2W1}\cdots\text{N2}^{\text{ii}}$ | 0.84 | 2.59 | 3.240 (3) | 135 |
| $\text{C15}-\text{H15C}\cdots\text{Cl1}^{\text{iii}}$ | 0.96 | 2.78 | 3.704 (2) | 163 |
| $\text{C1}-\text{H1}\cdots\text{Cg1}^{\text{iv}}$ | 0.93 | 2.97 | 3.621 (2) | 128 |
| $\text{C4}-\text{H4}\cdots\text{Cg1}^{\text{v}}$ | 0.93 | 2.89 | 3.565 (2) | 130 |
| $\text{C10}-\text{H10}\cdots\text{Cg2}^{\text{vi}}$ | 0.93 | 2.87 | 3.589 (2) | 135 |
| $\text{C13}-\text{H13}\cdots\text{Cg2}^{\text{vii}}$ | 0.93 | 2.81 | 3.497 (3) | 131 |

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x + 1, y, z$; (iii) $-x - \frac{1}{2}, -y + 1, z - \frac{1}{2}$; (iv) $-x - 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (v) $-x, y + \frac{3}{2}, -z + \frac{3}{2}$; (vi) $x + \frac{1}{2}, -y - \frac{1}{2}, -z$; (vii) $x - \frac{1}{2}, -y + \frac{1}{2}, -z$.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2637).

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supplementary materials

Acta Cryst. (2008). E64, o1594-o1595 [doi:10.1107/S1600536808022861]

4-Chloro-*N'*-(*Z*)-4-(dimethylamino)benzylidene]benzohydrazide monohydrate

H.-K. Fun, P. S. Patil, S. R. Jebas, K. V. Sujith and B. Kalluraya

Comment

Hydrazone compounds have been demonstrated to possess antimicrobial, anticonvulsant, analgesic, antiinflammatory, antiplatelet, antitubercular, anticancer and antitumoral activities (Bedia *et al.*, 2006; Rollas *et al.*, 2002; Terzioglu & Gürsoy, 2003). They are used as chromogenic receptors to show colorimetric responses and UV-Vis spectral changes in the presence of fluoride ions in organic solvents (Duraismy *et al.*, 2008). Hydrazide-hydrazone compounds are not only intermediates but they are also very effective organic compounds in their own right. When they are used as intermediates, coupling products can be synthesized by using the active hydrogen component of CONHNCH azometine group (Singh *et al.*, 1992). *N*-Alkyl hydrazides can be synthesized by reduction of hydrazones with NaBH₄ (Ergenç & Günay, 1998) and substituted 1,3,4-oxadiazolines can be synthesized when hydrazones are heated in the presence of acetic anhydride (Durgun *et al.*, 1993). Prompted by these reviews, the title compound was synthesized and its crystal structure reported.

The bond lengths and angles in the title molecule (Fig. 1) are found to have normal values (Allen *et al.*, 1987). The dihedral angle between the two benzene rings (C1–C6 and C9–C14) is 44.58 (11)° indicating that the molecule is non-planar. Atom N3 adopts a pyramidal configuration.

The crystal packing (Fig. 2) shows that the molecules are linked into two-dimensional networks parallel to the (001) plane by intermolecular O—H···N, O—H···O and C—H···Cl hydrogen bonds. In addition, the packing is stabilized by C—H···π interactions.

Experimental

The title compound was prepared by Schiff base condensation of 4-chlorophenyl hydrazide (0.01 mol) and 4-(dimethylamino)benzaldehyde (0.01 mol) in ethanol (30 ml) with 3 drops of concentrated H₂SO₄. Excess ethanol was removed from the reaction mixture under reduced pressure. The solid product obtained was filtered, washed with water and dried. Single crystals suitable for X-ray analysis were obtained from an ethanol solution by slow evaporation (yield 64%). Analysis % for C₁₆H₁₆N₃OCl found (calculated): C 63.62 (63.68), H 5.37 (5.3), N 13.88 (13.93).

Refinement

The imino H atom was located in a difference map and refined with a N-H distance restraint of 0.85 (1) Å. The water H atoms were also located in a difference map and allowed to ride on the O atom, with $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{O})$. The remaining H atoms were positioned geometrically [C-H = 0.93-0.96 %Å] and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. A rotating group model was used for the methyl groups.

Figures

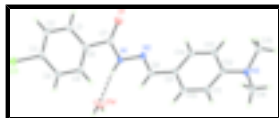


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.



Fig. 2. The crystal packing of the title compound, viewed along the *a* axis.

(I)

Crystal data

$C_{16}H_{16}ClN_3O \cdot H_2O$

$M_r = 319.78$

Orthorhombic, $P2_12_12_1$

Hall symbol: $P\ 2ac\ 2ab$

$a = 6.4418\ (1)\ \text{\AA}$

$b = 6.9344\ (1)\ \text{\AA}$

$c = 33.8083\ (7)\ \text{\AA}$

$V = 1510.22\ (4)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 672$

$D_x = 1.406\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1906 reflections

$\theta = 2.4\text{--}23.3^\circ$

$\mu = 0.26\ \text{mm}^{-1}$

$T = 100.0\ (1)\ \text{K}$

Needle, colourless

$0.32 \times 0.16 \times 0.07\ \text{mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100.0\ (1)\ \text{K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.921$, $T_{\max} = 0.981$

12336 measured reflections

4311 independent reflections

3301 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.056$

$\theta_{\max} = 30.1^\circ$

$\theta_{\min} = 2.4^\circ$

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 6$

$l = -47 \rightarrow 37$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.114$

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

| | |
|--|--|
| $S = 1.01$ | $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$ |
| 4311 reflections | $\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$ |
| 205 parameters | Extinction correction: none |
| 2 restraints | Absolute structure: Flack (1983), 1724 Friedel pairs |
| Primary atom site location: structure-invariant direct methods | Flack parameter: -0.14 (7) |
| Secondary atom site location: difference Fourier map | |

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|---------------|----------------------------------|
| C11 | 0.39865 (9) | 0.50981 (9) | 0.568340 (15) | 0.02126 (14) |
| O1 | -0.3268 (2) | 0.4990 (2) | 0.42597 (4) | 0.0206 (3) |
| N1 | -0.0370 (3) | 0.4579 (3) | 0.38853 (5) | 0.0146 (4) |
| N2 | -0.1527 (3) | 0.4602 (3) | 0.35420 (5) | 0.0151 (4) |
| N3 | -0.4409 (3) | 0.4624 (3) | 0.17091 (5) | 0.0166 (4) |
| C1 | -0.0827 (4) | 0.4376 (3) | 0.49549 (6) | 0.0154 (5) |
| H1 | -0.2199 | 0.3967 | 0.4971 | 0.018* |
| C2 | 0.0388 (4) | 0.4421 (3) | 0.52925 (6) | 0.0173 (5) |
| H2 | -0.0147 | 0.4008 | 0.5534 | 0.021* |
| C3 | 0.2405 (3) | 0.5086 (4) | 0.52666 (6) | 0.0154 (4) |
| C4 | 0.3243 (4) | 0.5704 (3) | 0.49095 (6) | 0.0157 (5) |
| H4 | 0.4595 | 0.6170 | 0.4897 | 0.019* |
| C5 | 0.2028 (3) | 0.5613 (3) | 0.45730 (7) | 0.0153 (5) |
| H5 | 0.2578 | 0.6005 | 0.4332 | 0.018* |
| C6 | -0.0002 (3) | 0.4943 (4) | 0.45901 (6) | 0.0148 (4) |
| C7 | -0.1372 (3) | 0.4843 (3) | 0.42347 (6) | 0.0136 (4) |
| C8 | -0.0579 (4) | 0.4106 (3) | 0.32251 (6) | 0.0147 (5) |
| H8 | 0.0795 | 0.3699 | 0.3239 | 0.018* |
| C9 | -0.1631 (4) | 0.4172 (3) | 0.28408 (6) | 0.0135 (5) |
| C10 | -0.0582 (4) | 0.3550 (3) | 0.25039 (7) | 0.0158 (5) |
| H10 | 0.0757 | 0.3062 | 0.2527 | 0.019* |
| C11 | -0.1513 (4) | 0.3651 (3) | 0.21331 (6) | 0.0156 (5) |
| H11 | -0.0797 | 0.3200 | 0.1913 | 0.019* |
| C12 | -0.3506 (4) | 0.4420 (3) | 0.20852 (6) | 0.0152 (5) |

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| | | | | |
|------|-------------|------------|-------------|------------|
| C13 | -0.4564 (3) | 0.5034 (4) | 0.24292 (6) | 0.0160 (4) |
| H13 | -0.5897 | 0.5538 | 0.2408 | 0.019* |
| C14 | -0.3643 (3) | 0.4893 (3) | 0.27960 (6) | 0.0151 (4) |
| H14 | -0.4376 | 0.5286 | 0.3019 | 0.018* |
| C15 | -0.6672 (4) | 0.4541 (4) | 0.16837 (7) | 0.0205 (5) |
| H15A | -0.7266 | 0.5409 | 0.1874 | 0.031* |
| H15B | -0.7132 | 0.3251 | 0.1738 | 0.031* |
| H15C | -0.7105 | 0.4907 | 0.1423 | 0.031* |
| C16 | -0.3386 (4) | 0.3595 (4) | 0.13815 (7) | 0.0245 (6) |
| H16A | -0.1993 | 0.4065 | 0.1352 | 0.037* |
| H16B | -0.4144 | 0.3811 | 0.1141 | 0.037* |
| H16C | -0.3354 | 0.2239 | 0.1438 | 0.037* |
| H1N1 | 0.0892 (19) | 0.419 (3) | 0.3882 (7) | 0.028 (7)* |
| O1W | 0.3779 (3) | 0.3316 (2) | 0.37632 (5) | 0.0259 (4) |
| H1W1 | 0.4093 | 0.2594 | 0.3573 | 0.039* |
| H2W1 | 0.4830 | 0.3923 | 0.3837 | 0.039* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| C11 | 0.0253 (3) | 0.0248 (3) | 0.0137 (3) | -0.0021 (3) | -0.0051 (2) | 0.0007 (3) |
| O1 | 0.0130 (7) | 0.0297 (9) | 0.0192 (8) | 0.0003 (7) | -0.0006 (6) | -0.0030 (8) |
| N1 | 0.0122 (9) | 0.0197 (11) | 0.0120 (9) | 0.0011 (8) | -0.0031 (7) | -0.0003 (8) |
| N2 | 0.0170 (9) | 0.0155 (10) | 0.0127 (9) | -0.0013 (7) | -0.0028 (7) | -0.0004 (7) |
| N3 | 0.0203 (10) | 0.0189 (11) | 0.0107 (9) | 0.0014 (8) | -0.0012 (7) | -0.0007 (8) |
| C1 | 0.0170 (11) | 0.0118 (11) | 0.0174 (11) | 0.0005 (8) | 0.0039 (9) | -0.0007 (8) |
| C2 | 0.0236 (12) | 0.0168 (12) | 0.0117 (11) | -0.0016 (9) | 0.0041 (9) | -0.0012 (9) |
| C3 | 0.0221 (11) | 0.0160 (11) | 0.0081 (10) | 0.0016 (10) | -0.0016 (8) | -0.0010 (10) |
| C4 | 0.0149 (11) | 0.0166 (12) | 0.0155 (12) | -0.0004 (8) | 0.0005 (9) | -0.0013 (9) |
| C5 | 0.0193 (12) | 0.0157 (12) | 0.0110 (11) | 0.0009 (8) | 0.0017 (9) | -0.0003 (9) |
| C6 | 0.0151 (10) | 0.0148 (11) | 0.0144 (10) | 0.0012 (9) | -0.0001 (8) | -0.0042 (10) |
| C7 | 0.0170 (10) | 0.0132 (11) | 0.0108 (10) | -0.0012 (9) | 0.0007 (8) | 0.0003 (9) |
| C8 | 0.0124 (10) | 0.0162 (11) | 0.0155 (12) | -0.0015 (9) | -0.0007 (9) | 0.0006 (9) |
| C9 | 0.0157 (11) | 0.0128 (11) | 0.0121 (11) | -0.0013 (8) | -0.0013 (9) | 0.0004 (8) |
| C10 | 0.0131 (11) | 0.0179 (12) | 0.0164 (12) | -0.0009 (9) | 0.0025 (9) | -0.0008 (9) |
| C11 | 0.0166 (11) | 0.0191 (12) | 0.0111 (11) | -0.0002 (9) | 0.0025 (9) | -0.0030 (9) |
| C12 | 0.0192 (11) | 0.0127 (11) | 0.0136 (11) | -0.0016 (9) | -0.0013 (9) | 0.0021 (8) |
| C13 | 0.0145 (10) | 0.0169 (11) | 0.0167 (11) | 0.0024 (10) | 0.0000 (8) | -0.0003 (10) |
| C14 | 0.0196 (10) | 0.0142 (11) | 0.0116 (10) | 0.0002 (10) | 0.0022 (8) | -0.0025 (9) |
| C15 | 0.0207 (12) | 0.0222 (14) | 0.0186 (12) | 0.0021 (10) | -0.0054 (9) | 0.0008 (10) |
| C16 | 0.0297 (14) | 0.0304 (15) | 0.0134 (12) | 0.0070 (11) | -0.0010 (10) | -0.0015 (10) |
| O1W | 0.0162 (9) | 0.0304 (10) | 0.0310 (10) | 0.0004 (7) | -0.0004 (8) | -0.0137 (8) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------|-----------|--------|-----------|
| C11—C3 | 1.739 (2) | C8—C9 | 1.466 (3) |
| O1—C7 | 1.229 (2) | C8—H8 | 0.93 |
| N1—C7 | 1.358 (3) | C9—C10 | 1.393 (3) |
| N1—N2 | 1.379 (2) | C9—C14 | 1.397 (3) |

| | | | |
|-------------|-------------|---------------|--------------|
| N1—H1N1 | 0.857 (10) | C10—C11 | 1.391 (3) |
| N2—C8 | 1.280 (3) | C10—H10 | 0.93 |
| N3—C12 | 1.405 (3) | C11—C12 | 1.400 (3) |
| N3—C15 | 1.461 (3) | C11—H11 | 0.93 |
| N3—C16 | 1.473 (3) | C12—C13 | 1.414 (3) |
| C1—C2 | 1.384 (3) | C13—C14 | 1.378 (3) |
| C1—C6 | 1.399 (3) | C13—H13 | 0.93 |
| C1—H1 | 0.93 | C14—H14 | 0.93 |
| C2—C3 | 1.382 (3) | C15—H15A | 0.96 |
| C2—H2 | 0.93 | C15—H15B | 0.96 |
| C3—C4 | 1.390 (3) | C15—H15C | 0.96 |
| C4—C5 | 1.382 (3) | C16—H16A | 0.96 |
| C4—H4 | 0.93 | C16—H16B | 0.96 |
| C5—C6 | 1.389 (3) | C16—H16C | 0.96 |
| C5—H5 | 0.93 | O1W—H1W1 | 0.84 |
| C6—C7 | 1.493 (3) | O1W—H2W1 | 0.83 |
| C7—N1—N2 | 118.25 (17) | C10—C9—C14 | 118.2 (2) |
| C7—N1—H1N1 | 120.4 (17) | C10—C9—C8 | 119.4 (2) |
| N2—N1—H1N1 | 120.3 (17) | C14—C9—C8 | 122.4 (2) |
| C8—N2—N1 | 116.33 (19) | C11—C10—C9 | 120.8 (2) |
| C12—N3—C15 | 117.51 (19) | C11—C10—H10 | 119.6 |
| C12—N3—C16 | 116.49 (19) | C9—C10—H10 | 119.6 |
| C15—N3—C16 | 112.54 (19) | C10—C11—C12 | 121.3 (2) |
| C2—C1—C6 | 120.4 (2) | C10—C11—H11 | 119.4 |
| C2—C1—H1 | 119.8 | C12—C11—H11 | 119.4 |
| C6—C1—H1 | 119.8 | C11—C12—N3 | 121.5 (2) |
| C3—C2—C1 | 119.1 (2) | C11—C12—C13 | 117.5 (2) |
| C3—C2—H2 | 120.4 | N3—C12—C13 | 121.0 (2) |
| C1—C2—H2 | 120.4 | C14—C13—C12 | 120.8 (2) |
| C2—C3—C4 | 121.5 (2) | C14—C13—H13 | 119.6 |
| C2—C3—C11 | 120.08 (17) | C12—C13—H13 | 119.6 |
| C4—C3—C11 | 118.36 (17) | C13—C14—C9 | 121.5 (2) |
| C5—C4—C3 | 118.8 (2) | C13—C14—H14 | 119.3 |
| C5—C4—H4 | 120.6 | C9—C14—H14 | 119.3 |
| C3—C4—H4 | 120.6 | N3—C15—H15A | 109.5 |
| C4—C5—C6 | 120.9 (2) | N3—C15—H15B | 109.5 |
| C4—C5—H5 | 119.5 | H15A—C15—H15B | 109.5 |
| C6—C5—H5 | 119.5 | N3—C15—H15C | 109.5 |
| C5—C6—C1 | 119.2 (2) | H15A—C15—H15C | 109.5 |
| C5—C6—C7 | 122.61 (19) | H15B—C15—H15C | 109.5 |
| C1—C6—C7 | 118.15 (19) | N3—C16—H16A | 109.5 |
| O1—C7—N1 | 122.92 (19) | N3—C16—H16B | 109.5 |
| O1—C7—C6 | 121.92 (18) | H16A—C16—H16B | 109.5 |
| N1—C7—C6 | 115.16 (18) | N3—C16—H16C | 109.5 |
| N2—C8—C9 | 120.9 (2) | H16A—C16—H16C | 109.5 |
| N2—C8—H8 | 119.6 | H16B—C16—H16C | 109.5 |
| C9—C8—H8 | 119.6 | H1W1—O1W—H2W1 | 109.6 |
| C7—N1—N2—C8 | -171.4 (2) | N1—N2—C8—C9 | -176.96 (18) |

supplementary materials

| | | | |
|--------------|--------------|-----------------|------------|
| C6—C1—C2—C3 | 1.9 (3) | N2—C8—C9—C10 | -177.5 (2) |
| C1—C2—C3—C4 | -0.2 (4) | N2—C8—C9—C14 | 4.5 (3) |
| C1—C2—C3—C11 | -178.32 (17) | C14—C9—C10—C11 | 0.2 (3) |
| C2—C3—C4—C5 | -1.1 (3) | C8—C9—C10—C11 | -177.9 (2) |
| C11—C3—C4—C5 | 176.95 (17) | C9—C10—C11—C12 | 1.5 (3) |
| C3—C4—C5—C6 | 0.9 (3) | C10—C11—C12—N3 | 176.4 (2) |
| C4—C5—C6—C1 | 0.7 (3) | C10—C11—C12—C13 | -1.9 (3) |
| C4—C5—C6—C7 | 179.3 (2) | C15—N3—C12—C11 | 151.7 (2) |
| C2—C1—C6—C5 | -2.2 (3) | C16—N3—C12—C11 | 13.7 (3) |
| C2—C1—C6—C7 | 179.2 (2) | C15—N3—C12—C13 | -30.0 (3) |
| N2—N1—C7—O1 | 4.3 (3) | C16—N3—C12—C13 | -168.0 (2) |
| N2—N1—C7—C6 | -175.69 (19) | C11—C12—C13—C14 | 0.7 (3) |
| C5—C6—C7—O1 | -151.1 (2) | N3—C12—C13—C14 | -177.7 (2) |
| C1—C6—C7—O1 | 27.4 (3) | C12—C13—C14—C9 | 1.0 (4) |
| C5—C6—C7—N1 | 28.8 (3) | C10—C9—C14—C13 | -1.5 (3) |
| C1—C6—C7—N1 | -152.6 (2) | C8—C9—C14—C13 | 176.6 (2) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------------|----------|-------------|-------------|---------------|
| O1W—H1W1 \cdots N3 ⁱ | 0.84 | 2.28 | 3.045 (2) | 152 |
| N1—H1N1 \cdots O1W | 0.86 (1) | 2.00 (1) | 2.843 (3) | 169 (2) |
| O1W—H2W1 \cdots O1 ⁱⁱ | 0.84 | 2.02 | 2.790 (2) | 152 |
| O1W—H2W1 \cdots N2 ⁱⁱ | 0.84 | 2.59 | 3.240 (3) | 135 |
| C15—H15C \cdots C11 ⁱⁱⁱ | 0.96 | 2.78 | 3.704 (2) | 163 |
| C1—H1 \cdots Cg1 ^{iv} | 0.93 | 2.97 | 3.621 (2) | 128 |
| C4—H4 \cdots Cg1 ^v | 0.93 | 2.89 | 3.565 (2) | 130 |
| C10—H10 \cdots Cg2 ^{vi} | 0.93 | 2.87 | 3.589 (2) | 135 |
| C13—H13 \cdots Cg2 ^{vii} | 0.93 | 2.81 | 3.497 (3) | 131 |

Symmetry codes: (i) $-x, y-1/2, -z+1/2$; (ii) $x+1, y, z$; (iii) $-x-1/2, -y+1, z-1/2$; (iv) $-x-1, y+1/2, -z+3/2$; (v) $-x, y+3/2, -z+3/2$; (vi) $x+1/2, -y-1/2, -z$; (vii) $x-1/2, -y+1/2, -z$.

Fig. 1

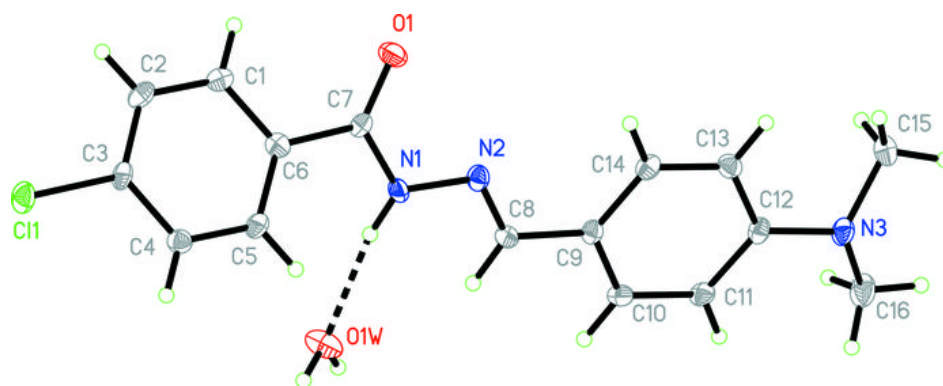


Fig. 2

