

3-Chloro-*N'*-(4-hydroxy-3-nitrobenzylidene)benzohydrazide methanol disolvate

Zhen Zhang

Experimental Center, Linyi University, Linyi Shandong 276005, People's Republic of China

Correspondence e-mail: zhangzhen_lynu@126.com

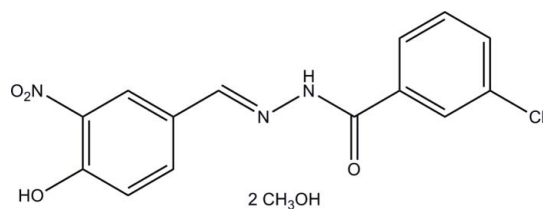
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.067; wR factor = 0.208; data-to-parameter ratio = 15.2.

In the title compound, $\text{C}_{14}\text{H}_{10}\text{ClN}_3\text{O}_4 \cdot 2\text{CH}_3\text{O}$, the main molecule is in an *E* configuration with respect to the methyldene unit. The dihedral angle between the mean planes of the two benzene rings is $1.9(3)^\circ$. In the crystal, intermolecular $\text{N}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{O}$ and bifurcated $\text{O}-\text{H} \cdots (\text{O}, \text{O})$ hydrogen bonds link the components into sheets parallel to (100). An intramolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bond is also present.

Related literature

For the biological applications of hydrazone compounds, see: Ajani *et al.* (2010); Avaji *et al.* (2009); Fan *et al.* (2010); Rasras *et al.* (2010). For related hydrazone structures, see: Zhang (2011a,b); Ahmad *et al.* (2010); Ban (2010); Ji & Lu (2010); Shalash *et al.* (2010).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{10}\text{ClN}_3\text{O}_4 \cdot 2\text{CH}_3\text{O}$
 $M_r = 383.78$
 Monoclinic, $P2_1/c$
 $a = 7.626(2)$ Å
 $b = 18.846(5)$ Å

$c = 12.739(3)$ Å
 $\beta = 94.689(4)^\circ$
 $V = 1824.6(8)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.25$ mm⁻¹
 $T = 298$ K

$0.23 \times 0.23 \times 0.21$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.945$, $T_{\max} = 0.950$

7726 measured reflections
 3695 independent reflections
 1842 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.208$
 $S = 1.06$
 3695 reflections
 243 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.76$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--|----------|--------------|--------------|----------------|
| $\text{O3}-\text{H3A} \cdots \text{O1}$ | 0.82 | 1.96 | 2.618 (4) | 137 |
| $\text{O3}-\text{H3A} \cdots \text{O5}$ | 0.82 | 2.26 | 2.896 (4) | 135 |
| $\text{O6}-\text{H6} \cdots \text{O5}$ | 0.82 | 1.88 | 2.700 (5) | 179 |
| $\text{N3}-\text{H3} \cdots \text{O6}^i$ | 0.89 (1) | 1.99 (2) | 2.834 (4) | 158 (4) |
| $\text{O5}-\text{H5} \cdots \text{O4}^{\text{ii}}$ | 0.82 | 1.96 | 2.779 (4) | 173 |

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5265).

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supplementary materials

Acta Cryst. (2011). E67, o1630 [doi:10.1107/S1600536811021568]

3-Chloro-*N'*-(4-hydroxy-3-nitrobenzylidene)benzohydrazide methanol disolvate

Z. Zhang

Comment

Hydrazone compounds have received much attention due to their potential applications in biological chemistry (Ajani *et al.*, 2010; Avaji *et al.*, 2009; Fan *et al.*, 2010; Rasras *et al.*, 2010). As a continuation of this work on hydrazone compounds the structure of the title compound is reported herein.

The asymmetric unit of title compound, Fig. 1, contains a hydrazone molecule and two methanol molecules. The hydrazone molecule assumes an *E* configuration with respect to the methylidene unit. The dihedral angle between the mean planes of the two benzene rings is 1.9 (3)°. The bond lengths are comparable to those observed in similar hydrazone compounds (Zhang, 2011a,b; Ahmad *et al.*, 2010; Ban, 2010; Ji & Lu, 2010; Shalash *et al.*, 2010). In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds link the components into two dimensional sheets parallel to (100). An intramolecular O—H···O hydrogen bond is also present (Table 1, Fig. 2).

Experimental

A methanol solution (50 ml) of 3-chlorobenzohydrazide (0.01 mol) and 4-hydroxy-3-nitrobenzaldehyde (0.01 mol) was stirred at room temperature for 30 min to give a yellow solution. Yellow block-shaped single crystals suitable for X-ray diffraction were formed by slow evaporation of the solution in air.

Refinement

The amino atom, H3, was in from a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. The remaining H atoms were positioned geometrically and refined using the riding-model approximation, with C—H = 0.93–0.96 Å, O—H = 0.82 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O})$.

Figures

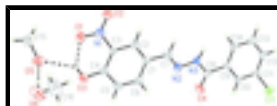


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids at the 30% probability level. Hydrogen bonds are shown as dashed lines.



Fig. 2. The packing of the title compound. Hydrogen bonds are shown as dashed lines. Only H atoms involved in hydrogen bonds are shown.

3-Chloro-*N'*-(4-hydroxy-3-nitrobenzylidene)benzohydrazide methanol disolvate

Crystal data

C₁₄H₁₀ClN₃O₄·2CH₄O

$F(000) = 800$

supplementary materials

$M_r = 383.78$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.626$ (2) Å

$b = 18.846$ (5) Å

$c = 12.739$ (3) Å

$\beta = 94.689$ (4)°

$V = 1824.6$ (8) Å³

$Z = 4$

$D_x = 1.397$ Mg m⁻³

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

Cell parameters from 954 reflections

$\theta = 2.5$ – 24.5 °

$\mu = 0.25$ mm⁻¹

$T = 298$ K

Block, yellow

$0.23 \times 0.23 \times 0.21$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.945$, $T_{\max} = 0.950$

7726 measured reflections

3695 independent reflections

1842 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 26.5$ °, $\theta_{\min} = 2.7$ °

$h = -9 \rightarrow 9$

$k = -22 \rightarrow 23$

$l = -15 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.208$

$S = 1.06$

3695 reflections

243 parameters

1 restraint

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H atoms treated by a mixture of independent and
constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2]$

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

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

$\Delta\rho_{\max} = 0.76$ e Å⁻³

$\Delta\rho_{\min} = -0.26$ e Å⁻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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|--------------|----------------------------------|
| Cl1 | 1.06078 (17) | -0.35391 (5) | 0.03312 (9) | 0.0708 (4) |
| N1 | 0.4832 (5) | 0.32118 (17) | -0.0141 (3) | 0.0546 (9) |
| N2 | 0.7478 (4) | 0.02147 (15) | 0.0491 (2) | 0.0526 (9) |
| N3 | 0.7737 (5) | -0.03895 (16) | -0.0091 (2) | 0.0518 (9) |
| O1 | 0.4617 (5) | 0.37833 (14) | 0.0290 (2) | 0.0781 (10) |
| O2 | 0.4421 (4) | 0.31188 (15) | -0.1072 (2) | 0.0768 (10) |
| O3 | 0.5851 (4) | 0.32945 (13) | 0.21283 (19) | 0.0652 (9) |
| H3A | 0.5220 | 0.3575 | 0.1780 | 0.098* |
| O4 | 0.8426 (5) | -0.10265 (14) | 0.1374 (2) | 0.0773 (10) |
| O5 | 0.3664 (4) | 0.45501 (16) | 0.2154 (2) | 0.0744 (9) |
| H5 | 0.3000 | 0.4414 | 0.2587 | 0.112* |
| O6 | 0.6850 (6) | 0.5111 (2) | 0.2745 (3) | 0.1082 (14) |
| H6 | 0.5888 | 0.4937 | 0.2563 | 0.162* |
| C1 | 0.6684 (5) | 0.14309 (18) | 0.0505 (3) | 0.0474 (10) |
| C2 | 0.5915 (5) | 0.19991 (18) | -0.0036 (3) | 0.0452 (9) |
| H2 | 0.5575 | 0.1957 | -0.0751 | 0.054* |
| C3 | 0.5644 (5) | 0.26316 (18) | 0.0480 (3) | 0.0437 (9) |
| C4 | 0.6108 (5) | 0.27168 (19) | 0.1552 (3) | 0.0482 (10) |
| C5 | 0.6913 (6) | 0.2144 (2) | 0.2073 (3) | 0.0566 (11) |
| H5A | 0.7265 | 0.2186 | 0.2787 | 0.068* |
| C6 | 0.7208 (6) | 0.1523 (2) | 0.1581 (3) | 0.0559 (11) |
| H6A | 0.7765 | 0.1153 | 0.1959 | 0.067* |
| C7 | 0.6967 (5) | 0.07624 (19) | -0.0032 (3) | 0.0530 (10) |
| H7 | 0.6772 | 0.0736 | -0.0761 | 0.064* |
| C8 | 0.8224 (5) | -0.09925 (19) | 0.0410 (3) | 0.0495 (10) |
| C9 | 0.8502 (5) | -0.16287 (17) | -0.0252 (3) | 0.0402 (9) |
| C10 | 0.9332 (5) | -0.22067 (18) | 0.0246 (3) | 0.0459 (9) |
| H10 | 0.9724 | -0.2180 | 0.0956 | 0.055* |
| C11 | 0.9576 (5) | -0.28200 (18) | -0.0311 (3) | 0.0452 (9) |
| C12 | 0.9009 (5) | -0.2878 (2) | -0.1355 (3) | 0.0543 (11) |
| H12 | 0.9173 | -0.3296 | -0.1722 | 0.065* |
| C13 | 0.8189 (6) | -0.2303 (2) | -0.1848 (3) | 0.0564 (11) |
| H13 | 0.7805 | -0.2333 | -0.2558 | 0.068* |
| C14 | 0.7929 (5) | -0.16866 (19) | -0.1309 (3) | 0.0498 (10) |
| H14 | 0.7365 | -0.1305 | -0.1656 | 0.060* |
| H3 | 0.771 (5) | -0.0374 (19) | -0.0792 (9) | 0.060* |
| C15 | 0.2719 (8) | 0.4968 (2) | 0.1385 (4) | 0.0930 (17) |
| H15A | 0.2107 | 0.5338 | 0.1724 | 0.139* |
| H15B | 0.1885 | 0.4676 | 0.0979 | 0.139* |
| H15C | 0.3520 | 0.5175 | 0.0929 | 0.139* |
| C16 | 0.7983 (9) | 0.5025 (3) | 0.1930 (5) | 0.132 (2) |
| H16A | 0.7729 | 0.5382 | 0.1402 | 0.198* |
| H16B | 0.7808 | 0.4563 | 0.1620 | 0.198* |
| H16C | 0.9183 | 0.5071 | 0.2215 | 0.198* |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.0871 (10) | 0.0429 (6) | 0.0794 (8) | 0.0123 (6) | -0.0122 (6) | 0.0061 (5) |
| N1 | 0.064 (2) | 0.048 (2) | 0.051 (2) | 0.0092 (17) | 0.0017 (17) | 0.0028 (16) |
| N2 | 0.058 (2) | 0.0397 (17) | 0.0606 (19) | 0.0051 (16) | 0.0061 (16) | -0.0027 (15) |
| N3 | 0.064 (2) | 0.0435 (18) | 0.0480 (17) | 0.0113 (16) | 0.0056 (17) | -0.0019 (15) |
| O1 | 0.124 (3) | 0.0458 (16) | 0.0628 (18) | 0.0307 (18) | -0.0022 (18) | -0.0022 (14) |
| O2 | 0.104 (3) | 0.070 (2) | 0.0520 (18) | 0.0192 (18) | -0.0196 (17) | -0.0038 (14) |
| O3 | 0.095 (3) | 0.0486 (16) | 0.0492 (15) | 0.0114 (16) | -0.0097 (15) | -0.0068 (13) |
| O4 | 0.127 (3) | 0.0578 (17) | 0.0454 (16) | 0.0261 (18) | -0.0019 (17) | -0.0041 (13) |
| O5 | 0.086 (3) | 0.077 (2) | 0.0608 (19) | 0.0012 (19) | 0.0064 (17) | 0.0053 (15) |
| O6 | 0.140 (4) | 0.105 (3) | 0.078 (2) | -0.042 (3) | 0.000 (2) | -0.014 (2) |
| C1 | 0.051 (3) | 0.039 (2) | 0.052 (2) | 0.0008 (19) | 0.0063 (19) | 0.0016 (17) |
| C2 | 0.045 (2) | 0.044 (2) | 0.047 (2) | -0.0023 (18) | 0.0038 (17) | -0.0009 (16) |
| C3 | 0.049 (3) | 0.0349 (19) | 0.047 (2) | 0.0026 (18) | 0.0043 (18) | 0.0056 (15) |
| C4 | 0.057 (3) | 0.042 (2) | 0.044 (2) | -0.0030 (19) | -0.0013 (19) | -0.0009 (17) |
| C5 | 0.070 (3) | 0.054 (2) | 0.043 (2) | 0.003 (2) | -0.007 (2) | 0.0006 (18) |
| C6 | 0.064 (3) | 0.047 (2) | 0.055 (2) | 0.006 (2) | -0.002 (2) | 0.0099 (18) |
| C7 | 0.060 (3) | 0.045 (2) | 0.054 (2) | 0.001 (2) | 0.007 (2) | -0.0011 (18) |
| C8 | 0.057 (3) | 0.042 (2) | 0.049 (2) | 0.0069 (19) | 0.0000 (19) | -0.0014 (17) |
| C9 | 0.039 (2) | 0.040 (2) | 0.0413 (19) | 0.0031 (17) | 0.0015 (16) | -0.0003 (15) |
| C10 | 0.049 (3) | 0.044 (2) | 0.044 (2) | 0.0033 (19) | 0.0000 (18) | -0.0002 (16) |
| C11 | 0.044 (2) | 0.0346 (19) | 0.057 (2) | -0.0009 (17) | 0.0000 (18) | 0.0045 (16) |
| C12 | 0.059 (3) | 0.047 (2) | 0.056 (2) | 0.003 (2) | -0.003 (2) | -0.0118 (18) |
| C13 | 0.062 (3) | 0.059 (3) | 0.047 (2) | 0.004 (2) | -0.006 (2) | -0.0042 (19) |
| C14 | 0.051 (3) | 0.047 (2) | 0.050 (2) | 0.0079 (19) | -0.0027 (19) | 0.0044 (17) |
| C15 | 0.113 (5) | 0.074 (3) | 0.092 (3) | 0.034 (3) | 0.012 (3) | 0.018 (3) |
| C16 | 0.136 (6) | 0.136 (6) | 0.133 (5) | -0.021 (5) | 0.061 (5) | -0.028 (5) |

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

| | | | |
|---------|------------|----------|-----------|
| C11—C11 | 1.738 (3) | C5—C6 | 1.355 (5) |
| N1—O2 | 1.214 (4) | C5—H5A | 0.9300 |
| N1—O1 | 1.226 (4) | C6—H6A | 0.9300 |
| N1—C3 | 1.458 (4) | C7—H7 | 0.9300 |
| N2—C7 | 1.272 (4) | C8—C9 | 1.491 (5) |
| N2—N3 | 1.382 (4) | C9—C14 | 1.386 (5) |
| N3—C8 | 1.341 (4) | C9—C10 | 1.387 (4) |
| N3—H3 | 0.893 (10) | C10—C11 | 1.377 (5) |
| O3—C4 | 1.336 (4) | C10—H10 | 0.9300 |
| O3—H3A | 0.8200 | C11—C12 | 1.369 (5) |
| O4—C8 | 1.227 (4) | C12—C13 | 1.377 (5) |
| O5—C15 | 1.409 (5) | C12—H12 | 0.9300 |
| O5—H5 | 0.8200 | C13—C14 | 1.371 (5) |
| O6—C16 | 1.413 (6) | C13—H13 | 0.9300 |
| O6—H6 | 0.8200 | C14—H14 | 0.9300 |
| C1—C2 | 1.378 (5) | C15—H15A | 0.9600 |

| | | | |
|-----------|-----------|---------------|-----------|
| C1—C6 | 1.406 (5) | C15—H15B | 0.9600 |
| C1—C7 | 1.458 (5) | C15—H15C | 0.9600 |
| C2—C3 | 1.385 (5) | C16—H16A | 0.9600 |
| C2—H2 | 0.9300 | C16—H16B | 0.9600 |
| C3—C4 | 1.392 (5) | C16—H16C | 0.9600 |
| C4—C5 | 1.385 (5) | | |
| O2—N1—O1 | 122.1 (3) | O4—C8—C9 | 120.8 (3) |
| O2—N1—C3 | 119.1 (3) | N3—C8—C9 | 117.4 (3) |
| O1—N1—C3 | 118.8 (3) | C14—C9—C10 | 118.6 (3) |
| C7—N2—N3 | 116.0 (3) | C14—C9—C8 | 124.4 (3) |
| C8—N3—N2 | 119.3 (3) | C10—C9—C8 | 117.0 (3) |
| C8—N3—H3 | 119 (2) | C11—C10—C9 | 119.9 (3) |
| N2—N3—H3 | 121 (2) | C11—C10—H10 | 120.0 |
| C4—O3—H3A | 109.5 | C9—C10—H10 | 120.0 |
| C15—O5—H5 | 109.5 | C12—C11—C10 | 121.5 (3) |
| C16—O6—H6 | 109.5 | C12—C11—C11 | 119.3 (3) |
| C2—C1—C6 | 117.8 (3) | C10—C11—C11 | 119.1 (3) |
| C2—C1—C7 | 120.7 (3) | C11—C12—C13 | 118.4 (3) |
| C6—C1—C7 | 121.5 (3) | C11—C12—H12 | 120.8 |
| C1—C2—C3 | 120.4 (3) | C13—C12—H12 | 120.8 |
| C1—C2—H2 | 119.8 | C14—C13—C12 | 121.2 (4) |
| C3—C2—H2 | 119.8 | C14—C13—H13 | 119.4 |
| C2—C3—C4 | 122.0 (3) | C12—C13—H13 | 119.4 |
| C2—C3—N1 | 117.5 (3) | C13—C14—C9 | 120.4 (3) |
| C4—C3—N1 | 120.6 (3) | C13—C14—H14 | 119.8 |
| O3—C4—C5 | 116.8 (3) | C9—C14—H14 | 119.8 |
| O3—C4—C3 | 126.6 (3) | O5—C15—H15A | 109.5 |
| C5—C4—C3 | 116.6 (3) | O5—C15—H15B | 109.5 |
| C6—C5—C4 | 122.4 (4) | H15A—C15—H15B | 109.5 |
| C6—C5—H5A | 118.8 | O5—C15—H15C | 109.5 |
| C4—C5—H5A | 118.8 | H15A—C15—H15C | 109.5 |
| C5—C6—C1 | 120.9 (3) | H15B—C15—H15C | 109.5 |
| C5—C6—H6A | 119.6 | O6—C16—H16A | 109.5 |
| C1—C6—H6A | 119.6 | O6—C16—H16B | 109.5 |
| N2—C7—C1 | 120.4 (3) | H16A—C16—H16B | 109.5 |
| N2—C7—H7 | 119.8 | O6—C16—H16C | 109.5 |
| C1—C7—H7 | 119.8 | H16A—C16—H16C | 109.5 |
| O4—C8—N3 | 121.8 (3) | H16B—C16—H16C | 109.5 |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|----------|-------------|-------------|---------------|
| O3—H3A \cdots O1 | 0.82 | 1.96 | 2.618 (4) | 137 |
| O3—H3A \cdots O5 | 0.82 | 2.26 | 2.896 (4) | 135 |
| O6—H6 \cdots O5 | 0.82 | 1.88 | 2.700 (5) | 179 |
| N3—H3 \cdots O6 ⁱ | 0.89 (1) | 1.99 (2) | 2.834 (4) | 158 (4) |
| O5—H5 \cdots O4 ⁱⁱ | 0.82 | 1.96 | 2.779 (4) | 173 |

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

Fig. 1

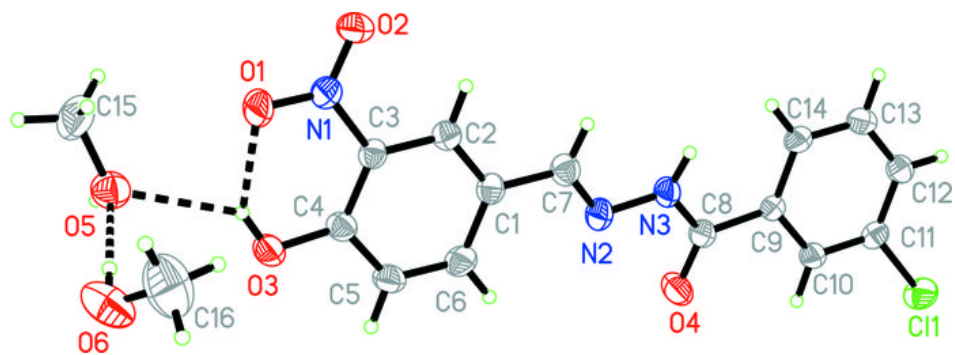


Fig. 2

