

3,5-Bis(4-methoxyphenyl)-1*H*-1,2,4-triazole monohydrate

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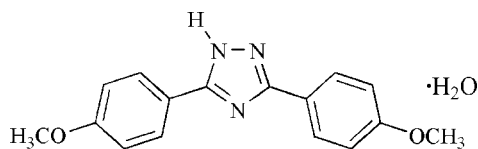
Received 24 April 2009; accepted 5 May 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.052; wR factor = 0.134; data-to-parameter ratio = 13.2.

In the title compound, $\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2 \cdot \text{H}_2\text{O}$, the two benzene rings and the triazole ring lie almost in the same plane, the triazole ring forming dihedral angles of 5.07 (9) and 5.80 (8)° with the benzene rings. In the crystal, there are three relatively strong intermolecular $\text{O}-\text{H} \cdots \text{N}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds, which lead to the formation of a one-dimensional double chain running parallel to the a axis. Weak $\pi-\pi$ interactions between the benzene rings of neighboring chains with a centroid-centroid distance of 3.893 (4) Å result in the formation of layers parallel to the ac plane.

Related literature

For the biological activity and pharmaceutical applications of compounds containing triazole subunits, see: Chai *et al.* (2009); Nadkarni *et al.* (2001); Zhan & Lou (2007). For triazole ring bond-length data, see; Claramunt *et al.* (2001); Zhou *et al.* (2001); John (1998).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2 \cdot \text{H}_2\text{O}$
 $M_r = 299.33$
Triclinic, $P\bar{1}$
 $a = 6.9948$ (18) Å

$b = 11.125$ (3) Å
 $c = 11.184$ (3) Å
 $\alpha = 110.603$ (4)°
 $\beta = 107.932$ (3)°

$\gamma = 95.690$ (4)°
 $V = 753.8$ (3) Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 298$ K
 $0.40 \times 0.20 \times 0.19$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
3854 measured reflections
2651 independent reflections
1993 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.134$
 $S = 1.05$
2651 reflections
201 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H} \cdots A$ | $D-\text{H}$ | $\text{H} \cdots A$ | $D \cdots A$ | $D-\text{H} \cdots A$ |
|---|--------------|---------------------|--------------|-----------------------|
| $\text{O3}-\text{H3A} \cdots \text{N1}$ | 0.97 | 1.96 | 2.902 (2) | 164 |
| $\text{N2}-\text{H2} \cdots \text{O3}^{\text{i}}$ | 0.86 | 1.90 | 2.753 (2) | 170 |
| $\text{O3}-\text{H3B} \cdots \text{N3}^{\text{ii}}$ | 0.96 | 1.97 | 2.885 (2) | 159 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank the National Natural Science Foundation of China (grant Nos. 20871076 and 20671060), the PhD Programs Foundation of the Ministry of Education of China (grant No. 200804450001) and the Shandong Natural Science Foundation (grant No. JQ200803) for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2201).

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supplementary materials

Acta Cryst. (2009). E65, o1260 [doi:10.1107/S160053680901695X]

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Comment

During the past decades, compounds containing triazole subunits have been intensively studied due to their diverse biological activities, such as antibacterial, antitumor, *etc.* and have become a central focus in the study of agricultural and medicinal chemicals (Chai *et al.*, 2009; Nadkarni *et al.*, 2001; Zhan *et al.*, 2007). In a search for more effective antibacterial compounds, we have synthesized the title compound and determined its structure.

The molecular structure of the title compound is shown in Fig. 1. The two benzene rings and the triazole ring almost lie in the same plane. The corresponding dihedral angles of each benzene ring with the triazole ring are 5.07 (9)° (between C2–C7 and N1–N3/C8/C9) and 5.80 (8)° (between N1–N3/C8/C9 and C10–C15), respectively. The bond lengths of the triazole ring are very similar to other 1*H*-1,2,4-triazole derivatives (Claramunt *et al.*, 2001; Zhou *et al.*, 2001). C8–N3 (1.365 (2) Å) and N1–N2 (1.359 (2) Å) are typical for carbon-nitrogen single bonds and nitrogen-nitrogen single bonds, and C8–N1 (1.323 (2) Å) and C9–N3 (1.330 (2) Å) correspond to typical carbon-nitrogen double bonds (John, 1998). C9–N2 (1.333 (2) Å) is a carbon-nitrogen single bond, but the bond length is markedly shorter than usual carbon-nitrogen single bonds and close to a double bond due to its conjugation with the C9–N3 double bond.

The packing of the molecules in the crystal structure is stabilized through N–H···O, O–H···O and π – π interactions. Water molecules act both as hydrogen-acceptor and as hydrogen-donor which leads to the formation of a one dimensional double chain running parallel to the *a* axis (Fig. 2, Table 1). The ring made up of C10 to C15 (with the centroid Cg1) is parallel to its symmetry related counterpart with a Cg1···Cg1ⁱⁱⁱ distance of 3.893 (4) Å [symmetry code: (iii)-*x*, -*y*, -*z*]. Adjacent chains are linked *via* these intermolecular π – π interactions between the Cg1 rings to form a two-dimensional layer parallel to the *ac* plane (Fig. 3).

Experimental

A mixture of 4-methoxyphenylmethylenemalononitrile (20 mmol), hydrazine dihydrochloride (20 mmol) and hydrazine hydrate (60 mmol) in ethylene glycol (10 ml) was heated to 403 K with stirring for 3–4 h. After cooling to room temperature, the reaction mixture was diluted with water (20 ml). The precipitate was filtered, washed with water, dried and purified by column chromatography on silica gel using CH₂Cl₂ as the eluent to afford a white solid after evaporation of the solvent. The white solid was dissolved in ethanol and colourless crystals of the title compound were obtained on slow evaporation of the solvent at room temperature.

Refinement

Hydrogen atoms attached to carbon were placed in geometrically idealized positions (C_{arene}–H = 0.93 Å, C_{methyl}–H = 0.96 Å) and refined using a riding model with isotropic displacement parameters $U_{\text{iso}} = 1.2$ (1.5 for methyl groups) $U_{\text{eq}}(\text{C})$.

supplementary materials

The H atoms attached to N and O atoms were located by Fourier difference synthesis and refined using a riding model with isotropic displacement parameters of $U_{\text{iso}} = 1.2 U_{\text{eq}}(\text{N})$ and $U_{\text{iso}} = 1.5 U_{\text{eq}}(\text{O})$.

Figures

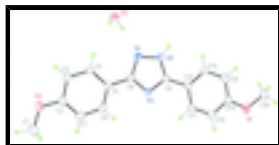


Fig. 1. The molecular structure, with atom labels and 30% probability displacement ellipsoids.

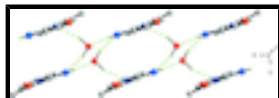


Fig. 2. View of a one dimensional double chain of the title structure. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

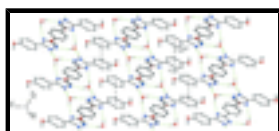


Fig. 3. The crystal packing of the title compound *via* weak π — π interactions. The distance of centroids is 3.893 (4) Å (Dashed lines: hydrogen bonds; broken lines: π — π interactions.) [symmetry code: (iii) $-x, -y, -z$].

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Crystal data

$\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2 \cdot \text{H}_2\text{O}$

$M_r = 299.33$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 6.9948$ (18) Å

$b = 11.125$ (3) Å

$c = 11.184$ (3) Å

$\alpha = 110.603$ (4)°

$\beta = 107.932$ (3)°

$\gamma = 95.690$ (4)°

$V = 753.8$ (3) Å³

$Z = 2$

$F_{000} = 316$

$D_x = 1.319$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1120 reflections

$\theta = 2.2$ – 24.0 °

$\mu = 0.09$ mm⁻¹

$T = 298$ K

Block, colourless

$0.40 \times 0.20 \times 0.19$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ K

phi and ω scans

Absorption correction: none

3854 measured reflections

2651 independent reflections

1993 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\text{max}} = 25.1$ °

$\theta_{\text{min}} = 2.0$ °

$h = -4 \rightarrow 8$

$k = -13 \rightarrow 11$

$l = -12 \rightarrow 13$

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | H-atom parameters constrained |
| $wR(F^2) = 0.134$ | $w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 0.0124P]$ |
| $S = 1.05$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2651 reflections | $(\Delta/\sigma)_{\max} = 0.001$ |
| 201 parameters | $\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$ |
| | Extinction correction: none |

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)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|------------|---------------|--------------|----------------------------------|
| C1 | 1.3626 (4) | 0.5268 (3) | 0.8502 (3) | 0.0831 (8) |
| H1A | 1.4171 | 0.4539 | 0.8091 | 0.125* |
| H1B | 1.4672 | 0.5887 | 0.9349 | 0.125* |
| H1C | 1.3199 | 0.5696 | 0.7886 | 0.125* |
| C2 | 1.0219 (3) | 0.3942 (2) | 0.7668 (2) | 0.0493 (5) |
| C3 | 0.8500 (3) | 0.3622 (2) | 0.7965 (2) | 0.0506 (5) |
| H3 | 0.8550 | 0.3978 | 0.8864 | 0.061* |
| C4 | 0.6729 (3) | 0.27787 (19) | 0.6928 (2) | 0.0447 (5) |
| H4 | 0.5585 | 0.2571 | 0.7137 | 0.054* |
| C5 | 0.6599 (3) | 0.22255 (18) | 0.55736 (19) | 0.0394 (5) |
| C6 | 0.8338 (3) | 0.2549 (2) | 0.5307 (2) | 0.0524 (6) |
| H6 | 0.8292 | 0.2193 | 0.4409 | 0.063* |
| C7 | 1.0141 (3) | 0.3388 (2) | 0.6341 (2) | 0.0578 (6) |
| H7 | 1.1300 | 0.3576 | 0.6138 | 0.069* |
| C8 | 0.4703 (3) | 0.13404 (18) | 0.44671 (18) | 0.0368 (4) |
| C9 | 0.2616 (3) | 0.00131 (18) | 0.24816 (19) | 0.0379 (5) |
| C10 | 0.1682 (3) | -0.08505 (18) | 0.10190 (19) | 0.0393 (5) |
| C11 | 0.2847 (3) | -0.0918 (2) | 0.0197 (2) | 0.0519 (6) |

supplementary materials

| | | | | |
|------|-------------|---------------|---------------|------------|
| H11 | 0.4199 | -0.0417 | 0.0587 | 0.062* |
| C12 | 0.2030 (3) | -0.1712 (2) | -0.1177 (2) | 0.0598 (6) |
| H12 | 0.2828 | -0.1741 | -0.1711 | 0.072* |
| C13 | 0.0040 (3) | -0.2468 (2) | -0.1776 (2) | 0.0503 (5) |
| C14 | -0.1137 (3) | -0.2420 (2) | -0.0979 (2) | 0.0539 (6) |
| H14 | -0.2481 | -0.2932 | -0.1372 | 0.065* |
| C15 | -0.0313 (3) | -0.1610 (2) | 0.0403 (2) | 0.0499 (5) |
| H15 | -0.1121 | -0.1576 | 0.0932 | 0.060* |
| C16 | -0.2664 (4) | -0.4015 (2) | -0.3827 (2) | 0.0745 (8) |
| H16A | -0.2851 | -0.4629 | -0.3425 | 0.112* |
| H16B | -0.2915 | -0.4491 | -0.4783 | 0.112* |
| H16C | -0.3617 | -0.3455 | -0.3735 | 0.112* |
| N1 | 0.2943 (2) | 0.10899 (16) | 0.46420 (16) | 0.0437 (4) |
| N2 | 0.1636 (2) | 0.02456 (16) | 0.33612 (16) | 0.0424 (4) |
| H2 | 0.0354 | -0.0092 | 0.3148 | 0.051* |
| N3 | 0.4569 (2) | 0.06990 (15) | 0.31449 (15) | 0.0404 (4) |
| O1 | -0.0613 (2) | -0.32325 (16) | -0.31464 (15) | 0.0726 (5) |
| O2 | 1.1911 (2) | 0.48023 (16) | 0.87639 (15) | 0.0693 (5) |
| O3 | 0.2326 (2) | 0.08148 (15) | 0.69915 (14) | 0.0540 (4) |
| H3A | 0.2339 | 0.1008 | 0.6215 | 0.100* |
| H3B | 0.3314 | 0.0294 | 0.7142 | 0.100* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| C1 | 0.0486 (14) | 0.087 (2) | 0.079 (2) | -0.0039 (13) | 0.0166 (13) | 0.0062 (16) |
| C2 | 0.0446 (12) | 0.0498 (13) | 0.0414 (12) | 0.0095 (10) | 0.0117 (10) | 0.0088 (10) |
| C3 | 0.0611 (14) | 0.0521 (13) | 0.0338 (12) | 0.0116 (10) | 0.0194 (10) | 0.0106 (10) |
| C4 | 0.0481 (12) | 0.0474 (12) | 0.0392 (12) | 0.0092 (9) | 0.0215 (9) | 0.0139 (10) |
| C5 | 0.0434 (11) | 0.0407 (11) | 0.0372 (11) | 0.0122 (9) | 0.0182 (9) | 0.0157 (9) |
| C6 | 0.0494 (13) | 0.0623 (14) | 0.0362 (12) | 0.0052 (10) | 0.0205 (10) | 0.0075 (10) |
| C7 | 0.0457 (12) | 0.0662 (15) | 0.0519 (14) | 0.0043 (11) | 0.0238 (11) | 0.0103 (12) |
| C8 | 0.0401 (10) | 0.0403 (11) | 0.0340 (11) | 0.0121 (8) | 0.0173 (8) | 0.0157 (9) |
| C9 | 0.0373 (10) | 0.0439 (11) | 0.0381 (11) | 0.0114 (9) | 0.0175 (9) | 0.0191 (10) |
| C10 | 0.0417 (11) | 0.0419 (11) | 0.0351 (11) | 0.0075 (9) | 0.0147 (9) | 0.0166 (9) |
| C11 | 0.0477 (12) | 0.0585 (14) | 0.0380 (12) | -0.0078 (10) | 0.0166 (10) | 0.0109 (11) |
| C12 | 0.0638 (15) | 0.0636 (15) | 0.0438 (13) | -0.0072 (12) | 0.0258 (11) | 0.0130 (12) |
| C13 | 0.0615 (14) | 0.0427 (12) | 0.0346 (12) | 0.0000 (10) | 0.0095 (10) | 0.0123 (10) |
| C14 | 0.0437 (12) | 0.0563 (14) | 0.0495 (14) | -0.0016 (10) | 0.0101 (10) | 0.0170 (11) |
| C15 | 0.0397 (11) | 0.0608 (14) | 0.0475 (13) | 0.0072 (10) | 0.0173 (10) | 0.0202 (11) |
| C16 | 0.0777 (17) | 0.0606 (16) | 0.0490 (15) | -0.0158 (13) | -0.0073 (12) | 0.0160 (13) |
| N1 | 0.0420 (9) | 0.0526 (10) | 0.0366 (10) | 0.0123 (8) | 0.0178 (8) | 0.0148 (8) |
| N2 | 0.0329 (8) | 0.0539 (10) | 0.0385 (10) | 0.0071 (7) | 0.0147 (7) | 0.0160 (8) |
| N3 | 0.0370 (9) | 0.0472 (10) | 0.0338 (9) | 0.0064 (7) | 0.0148 (7) | 0.0124 (8) |
| O1 | 0.0834 (12) | 0.0678 (11) | 0.0387 (10) | -0.0165 (9) | 0.0129 (8) | 0.0074 (8) |
| O2 | 0.0503 (9) | 0.0764 (12) | 0.0499 (10) | -0.0021 (8) | 0.0085 (7) | 0.0033 (9) |
| O3 | 0.0439 (8) | 0.0746 (10) | 0.0513 (9) | 0.0170 (7) | 0.0265 (7) | 0.0255 (8) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|---------------|-------------|
| C1—O2 | 1.412 (3) | C9—C10 | 1.462 (3) |
| C1—H1A | 0.9600 | C10—C15 | 1.379 (3) |
| C1—H1B | 0.9600 | C10—C11 | 1.393 (3) |
| C1—H1C | 0.9600 | C11—C12 | 1.368 (3) |
| C2—O2 | 1.370 (2) | C11—H11 | 0.9300 |
| C2—C7 | 1.373 (3) | C12—C13 | 1.375 (3) |
| C2—C3 | 1.389 (3) | C12—H12 | 0.9300 |
| C3—C4 | 1.370 (3) | C13—O1 | 1.362 (2) |
| C3—H3 | 0.9300 | C13—C14 | 1.380 (3) |
| C4—C5 | 1.390 (3) | C14—C15 | 1.379 (3) |
| C4—H4 | 0.9300 | C14—H14 | 0.9300 |
| C5—C6 | 1.381 (3) | C15—H15 | 0.9300 |
| C5—C8 | 1.461 (3) | C16—O1 | 1.418 (3) |
| C6—C7 | 1.381 (3) | C16—H16A | 0.9600 |
| C6—H6 | 0.9300 | C16—H16B | 0.9600 |
| C7—H7 | 0.9300 | C16—H16C | 0.9600 |
| C8—N1 | 1.323 (2) | N1—N2 | 1.359 (2) |
| C8—N3 | 1.365 (2) | N2—H2 | 0.8600 |
| C9—N3 | 1.330 (2) | O3—H3A | 0.9678 |
| C9—N2 | 1.333 (2) | O3—H3B | 0.9583 |
| O2—C1—H1A | 109.5 | C15—C10—C9 | 123.15 (18) |
| O2—C1—H1B | 109.5 | C11—C10—C9 | 119.01 (17) |
| H1A—C1—H1B | 109.5 | C12—C11—C10 | 120.85 (18) |
| O2—C1—H1C | 109.5 | C12—C11—H11 | 119.6 |
| H1A—C1—H1C | 109.5 | C10—C11—H11 | 119.6 |
| H1B—C1—H1C | 109.5 | C11—C12—C13 | 120.7 (2) |
| O2—C2—C7 | 124.66 (19) | C11—C12—H12 | 119.7 |
| O2—C2—C3 | 115.73 (19) | C13—C12—H12 | 119.7 |
| C7—C2—C3 | 119.61 (19) | O1—C13—C12 | 115.9 (2) |
| C4—C3—C2 | 119.73 (19) | O1—C13—C14 | 124.71 (19) |
| C4—C3—H3 | 120.1 | C12—C13—C14 | 119.4 (2) |
| C2—C3—H3 | 120.1 | C15—C14—C13 | 119.77 (19) |
| C3—C4—C5 | 121.72 (19) | C15—C14—H14 | 120.1 |
| C3—C4—H4 | 119.1 | C13—C14—H14 | 120.1 |
| C5—C4—H4 | 119.1 | C10—C15—C14 | 121.47 (19) |
| C6—C5—C4 | 117.40 (18) | C10—C15—H15 | 119.3 |
| C6—C5—C8 | 120.93 (17) | C14—C15—H15 | 119.3 |
| C4—C5—C8 | 121.67 (17) | O1—C16—H16A | 109.5 |
| C7—C6—C5 | 121.7 (2) | O1—C16—H16B | 109.5 |
| C7—C6—H6 | 119.2 | H16A—C16—H16B | 109.5 |
| C5—C6—H6 | 119.2 | O1—C16—H16C | 109.5 |
| C2—C7—C6 | 119.84 (19) | H16A—C16—H16C | 109.5 |
| C2—C7—H7 | 120.1 | H16B—C16—H16C | 109.5 |
| C6—C7—H7 | 120.1 | C8—N1—N2 | 102.97 (15) |
| N1—C8—N3 | 113.34 (16) | C9—N2—N1 | 110.53 (15) |
| N1—C8—C5 | 123.47 (16) | C9—N2—H2 | 124.7 |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|--------------|
| N3—C8—C5 | 123.19 (16) | N1—N2—H2 | 124.7 |
| N3—C9—N2 | 109.15 (17) | C9—N3—C8 | 104.00 (15) |
| N3—C9—C10 | 125.62 (17) | C13—O1—C16 | 118.50 (18) |
| N2—C9—C10 | 125.23 (17) | C2—O2—C1 | 118.09 (18) |
| C15—C10—C11 | 117.84 (18) | H3A—O3—H3B | 107.8 |
| O2—C2—C3—C4 | 179.32 (17) | C11—C12—C13—O1 | -179.37 (19) |
| C7—C2—C3—C4 | -1.4 (3) | C11—C12—C13—C14 | 0.1 (3) |
| C2—C3—C4—C5 | 0.1 (3) | O1—C13—C14—C15 | 179.8 (2) |
| C3—C4—C5—C6 | 0.5 (3) | C12—C13—C14—C15 | 0.4 (3) |
| C3—C4—C5—C8 | -179.20 (17) | C11—C10—C15—C14 | 0.4 (3) |
| C4—C5—C6—C7 | 0.1 (3) | C9—C10—C15—C14 | -179.59 (18) |
| C8—C5—C6—C7 | 179.80 (18) | C13—C14—C15—C10 | -0.7 (3) |
| O2—C2—C7—C6 | -178.80 (19) | N3—C8—N1—N2 | 0.0 (2) |
| C3—C2—C7—C6 | 2.0 (3) | C5—C8—N1—N2 | 179.44 (16) |
| C5—C6—C7—C2 | -1.3 (3) | N3—C9—N2—N1 | -0.6 (2) |
| C6—C5—C8—N1 | -173.76 (19) | C10—C9—N2—N1 | 179.86 (16) |
| C4—C5—C8—N1 | 6.0 (3) | C8—N1—N2—C9 | 0.35 (19) |
| C6—C5—C8—N3 | 5.6 (3) | N2—C9—N3—C8 | 0.6 (2) |
| C4—C5—C8—N3 | -174.69 (17) | C10—C9—N3—C8 | -179.88 (17) |
| N3—C9—C10—C15 | 175.33 (18) | N1—C8—N3—C9 | -0.4 (2) |
| N2—C9—C10—C15 | -5.2 (3) | C5—C8—N3—C9 | -179.81 (16) |
| N3—C9—C10—C11 | -4.6 (3) | C12—C13—O1—C16 | -178.8 (2) |
| N2—C9—C10—C11 | 174.80 (18) | C14—C13—O1—C16 | 1.8 (3) |
| C15—C10—C11—C12 | 0.1 (3) | C7—C2—O2—C1 | 7.2 (3) |
| C9—C10—C11—C12 | -179.88 (19) | C3—C2—O2—C1 | -173.6 (2) |
| C10—C11—C12—C13 | -0.4 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| O3—H3A...N1 | 0.97 | 1.96 | 2.902 (2) | 164 |
| N2—H2...O3 ⁱ | 0.86 | 1.90 | 2.753 (2) | 170 |
| O3—H3B...N3 ⁱⁱ | 0.96 | 1.97 | 2.885 (2) | 159 |

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

Fig. 1

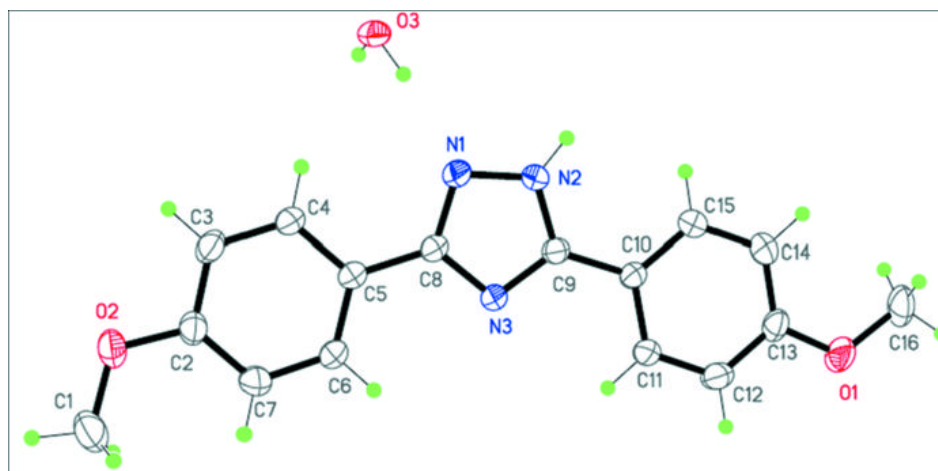


Fig. 2

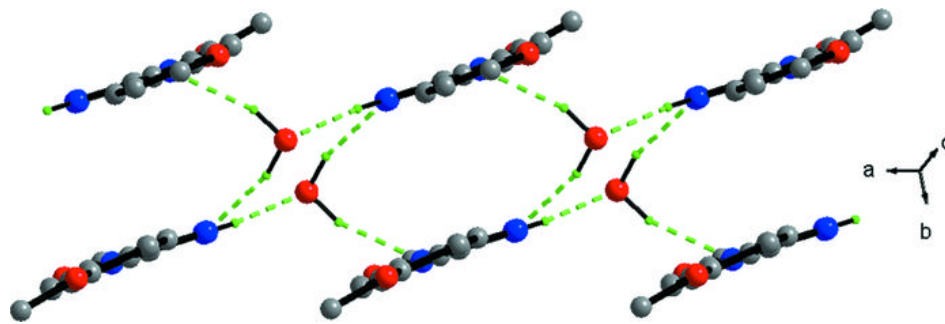


Fig. 3

