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3,4-Dimethoxy-*N*-(4-nitrobenzylidene)-aniline

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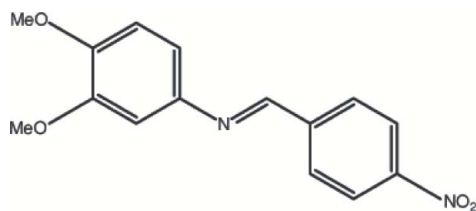
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.120; data-to-parameter ratio = 15.1.

In the title molecule, $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_4$, the dihedral angle between the two benzene rings is 29.52 (8°). The nitro and two methoxy substituents are almost coplanar with their respective benzene rings. The crystal structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For general background, see: Bey & Vevert (1977); Bezas & Zervas (1961); Fleet & Fleming (1969); Lucas *et al.* (1960); Macho *et al.* (2004). For a related structure, see: Akkurt *et al.* (2005).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_4$
 $M_r = 286.28$
 Monoclinic, $P2_1/c$
 $a = 7.9536$ (4) Å

 $b = 8.2258$ (3) Å
 $c = 21.3418$ (10) Å
 $\beta = 96.125$ (4) $^\circ$
 $V = 1388.31$ (11) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 296$ K
 $0.25 \times 0.21 \times 0.18$ mm

Data collection

 Stoe IPDS II diffractometer
 Absorption correction: integration
 (*X-RED32*; Stoe & Cie, 2002)
 $T_{\min} = 0.975$, $T_{\max} = 0.982$

 14598 measured reflections
 2880 independent reflections
 1797 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.120$
 $S = 0.83$
 2880 reflections

 191 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.13$ e Å⁻³
 $\Delta\rho_{\min} = -0.12$ e Å⁻³
Table 1

 Hydrogen-bond geometry (Å, $^\circ$).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C8}-\text{H8C}\cdots\text{O1}^{\text{i}}$ | 0.96 | 2.55 | 3.255 (3) | 130 |
| $\text{C8}-\text{H8C}\cdots\text{O4}^{\text{ii}}$ | 0.96 | 2.56 | 3.405 (3) | 147 |
| $\text{C14}-\text{H14}\cdots\text{O2}^{\text{iii}}$ | 0.93 | 2.56 | 3.246 (2) | 131 |

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2312).

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supplementary materials

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3,4-Dimethoxy-*N*-(4-nitrobenzylidene)aniline

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Comment

Schiff bases belong to a widely used group of organic intermediates which are important for production of certain chemicals, such as pharmaceuticals and rubber additives (Macho *et al.*, 2004), and as amino protective groups in organic synthesis (Bey & Vevert, 1977; Lucas *et al.*, 1960; Fleet & Fleming, 1969; Bezas & Zervas, 1961). As we are interested in Schiff bases, we report here the crystal structure of the title compound, (I).

In (I), Fig. 1, the dihedral angle between the two benzene rings (C1–C6) and (C10–C15) is 29.52 (8)° and the C1–N1–C9–C10 torsion angle is 176.12 (15)°. The nitro and two methoxy substituents are coplanar with their respective benzene rings.

The crystal structure of (I) is stabilized by intermolecular C—H···O interactions, Fig. 2 and Table 1.

Experimental

A mixture of 3,4-dimethoxyaniline (3 mmol) and 4-nitrobenzaldehyde (3 mmol) was refluxed in EtOH for 4 h. After cooling the solution, the formed precipitate was filtered off and washed with ethanol to give pure Schiff base as an orange solid in an 89% yield; m. pt. = 429–431 K. IR (KBr, cm⁻¹): 1600.3 (C?N). ¹H NMR (CDCl₃) δ 3.92, 3.94 (2 x OCH₃, s, 6H), 6.92–8.66 (ArH, m, 7H), 8.90 (HC?N, s, 1H). ¹³C NMR (CDCl₃) δ 55.97, 56.12 (2 OCH₃), 105.63–149.50 (C?C aromatic carbons), 155.03 (C?N).

Refinement

After checking for their presence in the Fourier map, all hydrogen atoms were placed in calculated positions and allowed to ride on their parent atoms with the C—H = 0.93 Å (aromatic) and C—H = 0.96 Å (methyl) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}_{\text{aromatic}})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Figures

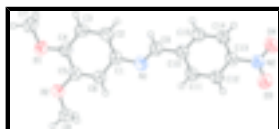


Fig. 1. View of (I) showing with the atom-numbering scheme and 50% probability displacement ellipsoids

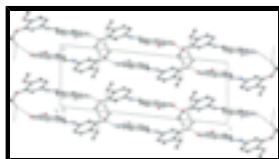


Fig. 2. View down the *b* axis of the packing and C—H···O contacts in the unit cell of (I). H atoms not involved in C—H···O contacts (dashed lines) have been omitted for clarity.

3,4-Dimethoxy-*N*-(4-nitrobenzylidene)aniline

Crystal data

| | |
|----------------------------------|---|
| $C_{15}H_{14}N_2O_4$ | $F_{000} = 600$ |
| $M_r = 286.28$ | $D_x = 1.370 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| Hall symbol: -P 2ybc | $\lambda = 0.71073 \text{ \AA}$ |
| $a = 7.9536 (4) \text{ \AA}$ | Cell parameters from 11667 reflections |
| $b = 8.2258 (3) \text{ \AA}$ | $\theta = 1.9\text{--}27.3^\circ$ |
| $c = 21.3418 (10) \text{ \AA}$ | $\mu = 0.10 \text{ mm}^{-1}$ |
| $\beta = 96.125 (4)^\circ$ | $T = 296 \text{ K}$ |
| $V = 1388.31 (11) \text{ \AA}^3$ | Prism, brown |
| $Z = 4$ | $0.25 \times 0.21 \times 0.18 \text{ mm}$ |

Data collection

| | |
|--|--|
| Stoe IPDS II diffractometer | 2880 independent reflections |
| Monochromator: plane graphite | 1797 reflections with $I > 2\sigma(I)$ |
| Detector resolution: $6.67 \text{ pixels mm}^{-1}$ | $R_{\text{int}} = 0.038$ |
| $T = 296 \text{ K}$ | $\theta_{\text{max}} = 26.5^\circ$ |
| ω scans | $\theta_{\text{min}} = 1.9^\circ$ |
| Absorption correction: integration (X-RED32; Stoe & Cie, 2002) | $h = -9 \rightarrow 9$ |
| $T_{\text{min}} = 0.975$, $T_{\text{max}} = 0.982$ | $k = -10 \rightarrow 10$ |
| 14598 measured reflections | $l = -26 \rightarrow 26$ |

Refinement

| | |
|--|---|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | $w = 1/[\sigma^2(F_o^2) + (0.0824P)^2]$ |
| $wR(F^2) = 0.120$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.83$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 2880 reflections | $\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$ |
| 191 parameters | $\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = KFc[1 + 0.001Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Secondary atom site location: difference Fourier map | Extinction coefficient: 0.0071 (16) |

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|--------------|----------------------------------|
| O1 | 0.34730 (16) | -0.17529 (15) | 0.28084 (6) | 0.0633 (4) |
| O2 | 0.14131 (16) | 0.04285 (15) | 0.23416 (6) | 0.0624 (4) |
| O3 | 0.1545 (2) | 1.09341 (17) | 0.58860 (7) | 0.0859 (6) |
| O4 | 0.2133 (2) | 0.96182 (16) | 0.67456 (6) | 0.0775 (6) |
| N1 | 0.2395 (2) | 0.38177 (18) | 0.41916 (7) | 0.0580 (5) |
| N2 | 0.1851 (2) | 0.96644 (18) | 0.61754 (7) | 0.0578 (5) |
| C1 | 0.2679 (2) | 0.2324 (2) | 0.38839 (8) | 0.0524 (6) |
| C2 | 0.3732 (2) | 0.1113 (2) | 0.41434 (8) | 0.0581 (6) |
| C3 | 0.4020 (2) | -0.0275 (2) | 0.37992 (9) | 0.0575 (6) |
| C4 | 0.3240 (2) | -0.0470 (2) | 0.31966 (8) | 0.0509 (5) |
| C5 | 0.2137 (2) | 0.0741 (2) | 0.29350 (8) | 0.0503 (6) |
| C6 | 0.1891 (2) | 0.2128 (2) | 0.32730 (8) | 0.0536 (6) |
| C7 | 0.4578 (3) | -0.3022 (3) | 0.30482 (11) | 0.0780 (8) |
| C8 | 0.0173 (3) | 0.1542 (2) | 0.20751 (9) | 0.0637 (7) |
| C9 | 0.2415 (2) | 0.3850 (2) | 0.47827 (9) | 0.0571 (6) |
| C10 | 0.2237 (2) | 0.5361 (2) | 0.51351 (8) | 0.0520 (6) |
| C11 | 0.1908 (2) | 0.6842 (2) | 0.48291 (8) | 0.0574 (6) |
| C12 | 0.1756 (2) | 0.8243 (2) | 0.51682 (8) | 0.0566 (6) |
| C13 | 0.1945 (2) | 0.8156 (2) | 0.58166 (8) | 0.0492 (5) |
| C14 | 0.2255 (2) | 0.6721 (2) | 0.61381 (8) | 0.0560 (6) |
| C15 | 0.2377 (2) | 0.5328 (2) | 0.57890 (8) | 0.0588 (6) |
| H2 | 0.42540 | 0.12260 | 0.45520 | 0.0700* |
| H3 | 0.47450 | -0.10770 | 0.39770 | 0.0690* |
| H6 | 0.11900 | 0.29450 | 0.30920 | 0.0640* |
| H7A | 0.46300 | -0.38430 | 0.27310 | 0.1170* |
| H7B | 0.41610 | -0.34920 | 0.34130 | 0.1170* |
| H7C | 0.56890 | -0.25870 | 0.31620 | 0.1170* |
| H8A | -0.02420 | 0.11930 | 0.16580 | 0.0960* |
| H8B | 0.06680 | 0.26030 | 0.20550 | 0.0960* |
| H8C | -0.07470 | 0.15840 | 0.23320 | 0.0960* |
| H9 | 0.25480 | 0.28770 | 0.50050 | 0.0690* |
| H11 | 0.17910 | 0.68810 | 0.43910 | 0.0690* |

supplementary materials

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|-----|---------|---------|---------|---------|
| H12 | 0.15290 | 0.92300 | 0.49640 | 0.0680* |
| H14 | 0.23770 | 0.66920 | 0.65760 | 0.0670* |
| H15 | 0.25570 | 0.43380 | 0.59960 | 0.0710* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| O1 | 0.0634 (8) | 0.0591 (7) | 0.0660 (8) | 0.0120 (6) | 0.0008 (6) | -0.0126 (6) |
| O2 | 0.0720 (8) | 0.0637 (8) | 0.0490 (7) | 0.0131 (6) | -0.0053 (6) | -0.0096 (6) |
| O3 | 0.1359 (14) | 0.0530 (8) | 0.0664 (9) | 0.0118 (8) | -0.0004 (9) | 0.0023 (7) |
| O4 | 0.1171 (12) | 0.0690 (9) | 0.0466 (8) | -0.0019 (8) | 0.0094 (8) | -0.0067 (7) |
| N1 | 0.0677 (10) | 0.0568 (9) | 0.0489 (9) | -0.0007 (7) | 0.0037 (7) | -0.0073 (7) |
| N2 | 0.0698 (10) | 0.0533 (9) | 0.0501 (9) | -0.0024 (7) | 0.0059 (7) | -0.0002 (7) |
| C1 | 0.0570 (10) | 0.0523 (10) | 0.0483 (9) | -0.0023 (8) | 0.0075 (8) | -0.0046 (8) |
| C2 | 0.0580 (10) | 0.0687 (12) | 0.0467 (10) | 0.0004 (9) | 0.0009 (8) | -0.0034 (9) |
| C3 | 0.0532 (10) | 0.0606 (11) | 0.0581 (11) | 0.0081 (8) | 0.0033 (8) | 0.0016 (9) |
| C4 | 0.0490 (9) | 0.0505 (9) | 0.0538 (10) | 0.0010 (8) | 0.0081 (8) | -0.0052 (8) |
| C5 | 0.0511 (10) | 0.0546 (10) | 0.0451 (9) | -0.0018 (8) | 0.0045 (7) | -0.0038 (8) |
| C6 | 0.0606 (11) | 0.0525 (10) | 0.0475 (9) | 0.0049 (8) | 0.0055 (8) | -0.0008 (8) |
| C7 | 0.0742 (14) | 0.0677 (13) | 0.0899 (16) | 0.0252 (11) | -0.0007 (12) | -0.0115 (11) |
| C8 | 0.0767 (13) | 0.0642 (12) | 0.0482 (10) | 0.0096 (9) | -0.0024 (9) | 0.0049 (9) |
| C9 | 0.0669 (11) | 0.0538 (10) | 0.0508 (10) | -0.0017 (9) | 0.0076 (8) | -0.0026 (8) |
| C10 | 0.0577 (10) | 0.0530 (10) | 0.0455 (9) | -0.0036 (8) | 0.0063 (8) | -0.0023 (8) |
| C11 | 0.0716 (12) | 0.0605 (11) | 0.0393 (9) | -0.0030 (9) | 0.0021 (8) | 0.0017 (8) |
| C12 | 0.0707 (12) | 0.0508 (10) | 0.0472 (10) | -0.0016 (8) | 0.0019 (8) | 0.0042 (8) |
| C13 | 0.0545 (10) | 0.0501 (9) | 0.0430 (9) | -0.0058 (7) | 0.0053 (7) | -0.0017 (7) |
| C14 | 0.0718 (12) | 0.0566 (10) | 0.0399 (9) | -0.0004 (9) | 0.0073 (8) | 0.0030 (8) |
| C15 | 0.0786 (13) | 0.0503 (10) | 0.0478 (10) | 0.0033 (9) | 0.0081 (9) | 0.0049 (8) |

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

| | | | |
|---------|-----------|---------|-----------|
| O1—C4 | 1.366 (2) | C11—C12 | 1.373 (2) |
| O1—C7 | 1.424 (3) | C12—C13 | 1.378 (2) |
| O2—C5 | 1.359 (2) | C13—C14 | 1.374 (2) |
| O2—C8 | 1.420 (2) | C14—C15 | 1.376 (2) |
| O3—N2 | 1.225 (2) | C2—H2 | 0.9300 |
| O4—N2 | 1.214 (2) | C3—H3 | 0.9300 |
| N1—C1 | 1.423 (2) | C6—H6 | 0.9300 |
| N1—C9 | 1.260 (2) | C7—H7A | 0.9600 |
| N2—C13 | 1.464 (2) | C7—H7B | 0.9600 |
| C1—C2 | 1.379 (2) | C7—H7C | 0.9600 |
| C1—C6 | 1.394 (2) | C8—H8A | 0.9600 |
| C2—C3 | 1.390 (2) | C8—H8B | 0.9600 |
| C3—C4 | 1.376 (3) | C8—H8C | 0.9600 |
| C4—C5 | 1.403 (2) | C9—H9 | 0.9300 |
| C5—C6 | 1.375 (2) | C11—H11 | 0.9300 |
| C9—C10 | 1.467 (2) | C12—H12 | 0.9300 |
| C10—C11 | 1.394 (2) | C14—H14 | 0.9300 |
| C10—C15 | 1.388 (2) | C15—H15 | 0.9300 |

| | | | |
|--------------------------|-------------|--------------------------|--------|
| O1...O2 | 2.5589 (18) | H2...H9 | 2.2100 |
| O1...C8 ⁱ | 3.255 (3) | H3...C7 | 2.5400 |
| O2...C14 ⁱⁱ | 3.246 (2) | H3...H7B | 2.3400 |
| O2...O1 | 2.5589 (18) | H3...H7C | 2.3300 |
| O3...C12 ⁱⁱⁱ | 3.340 (2) | H6...C8 | 2.5100 |
| O4...C8 ^{iv} | 3.405 (3) | H6...H8B | 2.2300 |
| O1...H8C ⁱ | 2.5500 | H6...H8C | 2.3900 |
| O1...H14 ⁱⁱ | 2.6800 | H7A...C4 ^{xiii} | 3.0500 |
| O2...H14 ⁱⁱ | 2.5600 | H7A...C5 ^{xiii} | 3.0900 |
| O3...H12 | 2.4100 | H7A...O4 ⁱⁱ | 2.8100 |
| O3...H15 ^v | 2.9200 | H7B...C3 | 2.7800 |
| O3...H12 ⁱⁱⁱ | 2.8900 | H7B...H3 | 2.3400 |
| O3...H9 ^v | 2.6500 | H7C...C3 | 2.7600 |
| O4...H14 | 2.4400 | H7C...H3 | 2.3300 |
| O4...H8C ^{iv} | 2.5600 | H7C...H15 ^{xii} | 2.5900 |
| O4...H7A ^{vi} | 2.8100 | H8A...H11 ⁱ | 2.5000 |
| O4...H8B ^{vii} | 2.6800 | H8B...C6 | 2.7000 |
| N2...C3 ^{viii} | 3.317 (2) | H8B...H6 | 2.2300 |
| N1...H11 | 2.6100 | H8B...O4 ^{xiv} | 2.6800 |
| C2...C13 ^{viii} | 3.483 (2) | H8C...C6 | 2.7800 |
| C3...N2 ^{viii} | 3.317 (2) | H8C...H6 | 2.3900 |
| C8...O4 ^{iv} | 3.405 (3) | H8C...O1 ^{ix} | 2.5500 |
| C8...O1 ^{ix} | 3.255 (3) | H8C...C7 ^{ix} | 3.0900 |
| C10...C10 ^{iv} | 3.593 (2) | H8C...O4 ^{iv} | 2.5600 |
| C12...O3 ⁱⁱⁱ | 3.340 (2) | H9...O3 ^x | 2.6500 |
| C13...C2 ^{viii} | 3.483 (2) | H9...C2 | 2.6000 |
| C14...O2 ^{vi} | 3.246 (2) | H9...H2 | 2.2100 |
| C2...H9 | 2.6000 | H9...H15 | 2.4300 |
| C2...H12 ^x | 3.0300 | H11...N1 | 2.6100 |
| C3...H7C | 2.7600 | H11...H8A ^{ix} | 2.5000 |
| C3...H7B | 2.7800 | H12...O3 | 2.4100 |
| C4...H7A ^{xi} | 3.0500 | H12...C2 ^v | 3.0300 |
| C5...H7A ^{xi} | 3.0900 | H12...O3 ⁱⁱⁱ | 2.8900 |
| C6...H8C | 2.7800 | H14...O4 | 2.4400 |
| C6...H8B | 2.7000 | H14...O1 ^{vi} | 2.6800 |
| C7...H15 ^{xii} | 3.0900 | H14...O2 ^{vi} | 2.5600 |
| C7...H3 | 2.5400 | H15...O3 ^x | 2.9200 |
| C7...H8C ⁱ | 3.0900 | H15...H9 | 2.4300 |
| C8...H6 | 2.5100 | H15...C7 ^{xii} | 3.0900 |
| C9...H2 | 2.6800 | H15...H7C ^{xii} | 2.5900 |
| H2...C9 | 2.6800 | | |
| C4—O1—C7 | 117.82 (15) | C1—C2—H2 | 120.00 |

supplementary materials

| | | | |
|---------------|--------------|-----------------|--------------|
| C5—O2—C8 | 117.30 (13) | C3—C2—H2 | 120.00 |
| C1—N1—C9 | 119.65 (15) | C2—C3—H3 | 120.00 |
| O3—N2—O4 | 122.53 (15) | C4—C3—H3 | 120.00 |
| O3—N2—C13 | 118.52 (15) | C1—C6—H6 | 120.00 |
| O4—N2—C13 | 118.90 (14) | C5—C6—H6 | 120.00 |
| N1—C1—C2 | 123.93 (15) | O1—C7—H7A | 109.00 |
| N1—C1—C6 | 116.99 (15) | O1—C7—H7B | 109.00 |
| C2—C1—C6 | 119.02 (15) | O1—C7—H7C | 109.00 |
| C1—C2—C3 | 120.56 (16) | H7A—C7—H7B | 109.00 |
| C2—C3—C4 | 120.42 (15) | H7A—C7—H7C | 109.00 |
| O1—C4—C3 | 125.44 (15) | H7B—C7—H7C | 109.00 |
| O1—C4—C5 | 115.25 (15) | O2—C8—H8A | 110.00 |
| C3—C4—C5 | 119.30 (15) | O2—C8—H8B | 109.00 |
| O2—C5—C4 | 114.92 (14) | O2—C8—H8C | 109.00 |
| O2—C5—C6 | 125.18 (15) | H8A—C8—H8B | 109.00 |
| C4—C5—C6 | 119.88 (15) | H8A—C8—H8C | 109.00 |
| C1—C6—C5 | 120.77 (15) | H8B—C8—H8C | 109.00 |
| N1—C9—C10 | 122.64 (16) | N1—C9—H9 | 119.00 |
| C9—C10—C11 | 121.59 (16) | C10—C9—H9 | 119.00 |
| C9—C10—C15 | 119.79 (15) | C10—C11—H11 | 120.00 |
| C11—C10—C15 | 118.62 (15) | C12—C11—H11 | 120.00 |
| C10—C11—C12 | 120.62 (16) | C11—C12—H12 | 121.00 |
| C11—C12—C13 | 118.65 (15) | C13—C12—H12 | 121.00 |
| N2—C13—C12 | 118.37 (15) | C13—C14—H14 | 121.00 |
| N2—C13—C14 | 118.90 (15) | C15—C14—H14 | 121.00 |
| C12—C13—C14 | 122.72 (16) | C10—C15—H15 | 119.00 |
| C13—C14—C15 | 117.66 (16) | C14—C15—H15 | 119.00 |
| C10—C15—C14 | 121.69 (15) | | |
| C7—O1—C4—C5 | -179.92 (16) | C3—C4—C5—O2 | -179.20 (15) |
| C7—O1—C4—C3 | 1.8 (2) | O1—C4—C5—C6 | -176.25 (15) |
| C8—O2—C5—C6 | -6.9 (2) | O1—C4—C5—O2 | 2.4 (2) |
| C8—O2—C5—C4 | 174.56 (15) | C3—C4—C5—C6 | 2.2 (2) |
| C1—N1—C9—C10 | 176.12 (15) | C4—C5—C6—C1 | -2.3 (2) |
| C9—N1—C1—C2 | -33.0 (3) | O2—C5—C6—C1 | 179.20 (15) |
| C9—N1—C1—C6 | 149.95 (17) | N1—C9—C10—C11 | 4.9 (3) |
| O3—N2—C13—C12 | 2.7 (2) | N1—C9—C10—C15 | -175.92 (16) |
| O4—N2—C13—C12 | -174.90 (16) | C9—C10—C11—C12 | -179.62 (15) |
| O3—N2—C13—C14 | -178.74 (16) | C15—C10—C11—C12 | 1.2 (2) |
| O4—N2—C13—C14 | 3.6 (2) | C9—C10—C15—C14 | 178.57 (15) |
| N1—C1—C6—C5 | 178.12 (15) | C11—C10—C15—C14 | -2.2 (2) |
| C2—C1—C6—C5 | 0.9 (2) | C10—C11—C12—C13 | 0.4 (2) |
| N1—C1—C2—C3 | -176.33 (16) | C11—C12—C13—N2 | 177.51 (15) |
| C6—C1—C2—C3 | 0.7 (2) | C11—C12—C13—C14 | -1.0 (2) |
| C1—C2—C3—C4 | -0.8 (3) | N2—C13—C14—C15 | -178.48 (15) |
| C2—C3—C4—O1 | 177.65 (15) | C12—C13—C14—C15 | 0.0 (2) |
| C2—C3—C4—C5 | -0.6 (2) | C13—C14—C15—C10 | 1.6 (2) |

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

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> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| C8—H8C···O1 ^{ix} | 0.96 | 2.55 | 3.255 (3) | 130 |
| C8—H8C···O4 ^{iv} | 0.96 | 2.56 | 3.405 (3) | 147 |
| C14—H14···O2 ^{vi} | 0.93 | 2.56 | 3.246 (2) | 131 |

Symmetry codes: (ix) $-x, y+1/2, -z+1/2$; (iv) $-x, -y+1, -z+1$; (vi) $x, -y+1/2, z+1/2$.

Fig. 1

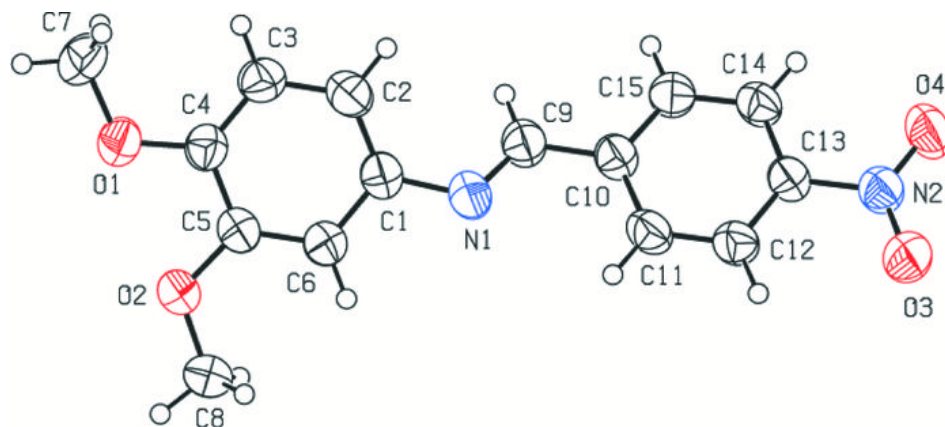


Fig. 2

