2-(9-Anthrylmethylideneamino)-4-methylphenol

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The title compound, C_{22}H_{17}NO, is a novel Schiff base synthesized via a condensation reaction between 9-anthrace-necarboxaldehyde and 2-amino-p-cresol. The asymmetric unit contains two independent molecules that are joined by an O···H···OH hydrogen bond. An intramolecular O···H···N hydrogen bond occurs in each molecule. π-stacking about inversion centers was observed between adjacent phenol rings [centroid–centroid distance = 3.850 (2) Å] and adjacent anthracene rings [centroid–centroid distance = 3.843 (2) Å]. The C–N=*=N–C torsion angles between the phenol and anthracene rings are close to 180° with values of 174.06 (15)° and 179.85 (14)°.

Related literature

For related structures, see: De et al. (2008); Ünver et al. (2009). For bond-length data, see: Allen et al. (1987). For background to the use of luminescent metal complexes formed by Schiff bases in light emitting diode collection and solar energy collection, see: Liao et al. (2009); Mak et al. (2009).

Experimental

Crystal data

C_{22}H_{17}NO  
μ = 0.08 mm\(^{-1}\)  
\(\mu = 0.08 mm^{-1}\)  
T = 90 K  
\(T = 90 K\)  
3.70 × 0.15 × 0.05 mm  
\(3.70 \times 0.15 \times 0.05 \text{ mm}\)

Data collection

Nonius KappaCCD diffractometer  
4454 reflections with \(I > 2\sigma(I)\)  
7462 independent reflections  
442 parameters  
7462 independent reflections  
Nonius KappaCCD diffractometer  
with Oxford Cryostream  
35942 measured reflections  
442 parameters  
7462 independent reflections  
442 parameters  
442 parameters

Refinement

R[F^2 > 2σ(F^2)] = 0.054  
H atoms treated by a mixture of independent and constrained refinement  
\(R[F^2 > 2\sigma(F^2)] = 0.054\)  
\(\Deltaρ_{\text{max}} = 0.30 \text{ e Å}^{-3}\)  
\(\Deltaρ_{\text{max}} = 0.30 \text{ e Å}^{-3}\)  
\(\Deltaρ_{\text{max}} = 0.28 \text{ e Å}^{-3}\)  
\(\Deltaρ_{\text{max}} = 0.28 \text{ e Å}^{-3}\)

Table 1

| D–H ··· A | D–H | H ··· A | D ··· A | D–H ··· A |
|---|---|---|---|---|
| O1 – H1O1 ··· N1 | 0.82 (2) | 2.27 (2) | 2.754 (2) | 118.0 (17) |
| O2 – H2O2 ··· O1 | 0.86 (2) | 2.11 (2) | 2.8602 (18) | 144.9 (18) |
| O2 – H2O2 ··· N2 | 0.86 (2) | 2.17 (2) | 2.695 (2) | 119.1 (17) |

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SIR97 (Altomare et al., 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2039).

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2-(9-Anthrylmethylideneamino)-4-methylphenol

Andrés Villalpando, Frank R. Fronczek and Ralph Isovitsch

S1. Comment

Schiff bases can form luminescent metal complexes that are used in research areas that range from light emitting diode construction to solar energy collection (Liao et al., 2009; Mak et al., 2009). Our research explores the synthesis and photophysics of novel anthracenyl Schiff bases and their metal complexes toward the goal of utilizing them in the preparation of light emitting diodes.

The structure of the title compound is shown in Figure 1. The asymmetric unit is comprised of two independent molecules of the title compound joined together by a hydrogen bond of length 2.8602 (18) Å, which is formed from the interaction of the OH groups on the phenol rings. π-stacking about inversion centers was observed between adjacent phenol rings with a centroid-centroid distance of 3.850 Å and between adjacent anthracene rings with a centroid-centroid distance of 3.834 Å.

There is slight variation in the bond lengths and angles of the two independent molecules. The central C—N double bond, C15—N1, has a bond length of 1.280 (2) Å. This bond length is close to the literature value of 1.279 Å for a C(sp²)=N(sp²) bond (Allen et al., 1987). The C—C bond, C1—C15 and C23—C37, that connects the anthracene to the central C—N double bond has bond lengths of 1.477 (2) and 1.470 (2) Å, respectively. The C—N bond, N1—C16 and N2—C38, that connects the phenyl ring to the central C—N double bond has bond lengths of 1.419 (2) and 1.414 (2) Å, respectively. The phenol ring has a C—O bond, O1—C17 and O2—C39, with a bond length of 1.368 (2) and 1.371 (2) Å. The bond angles of the nitrogen and carbon atoms of the central C—N double bond were 118.53 (15); 119.84 (15)° and 123.23 (16); 123.46 (16)°, which indicate the sp² hybridization of these atoms. The observed bond lengths and angles compare well with those found in similar compounds (Ünver et al., 2008; De et al., 2008). The angles between the planes of the anthracene and phenyl rings, C16—N1—C15—C1 and C38—N2—C37—C23, are 174.06 (15) and 179.85 (14)°, respectively.

S2. Experimental

Synthetic procedures were carried out using standard techniques. Solvents and reagents were used as received. The melting point was determined in open capillaries and is uncorrected. 1H and 13C NMR spectra were recorded on a JEOL ECX 300 MHz spectrometer using TMS as the internal standard. The IR spectrum was recorded as a KBr disk on a JASCO 460 FTIR. Mass spectrometry was provided by the Washington University Mass Spectrometry Resource with support from the NIH National Center for Research Resources (Grant No. P41RR0954).

The title compound was synthesized using a modification of the method of De et al. (2008). 20 ml of methanol, 9-anthracencarboxaldehyde (0.251 g, 1.22 mmol), and 2-amino-p-cresol (0.124 g, 1.01 mmol), and four drops of acetic acid were added to a 50 ml round bottom flask with a magnetic stir bar. The solution was refluxed for 1.5 hours until it was a bright orange color. The solution was then gravity filtered hot and allowed to slowly cool, yielding 0.185 g (59% yield) of bright orange-yellow needle-like crystals.
MP 170-174° C; IR (KBr disk) 3465, 3356, 3052, 3018, 2860, 1604, 1502 cm−1; 1H NMR (300 MHz, CDCl3) ppm 9.84 (s, 1H), 8.69 (d, 2H), 8.51 (s, 1H), 8.02 (d, 2H), 7.54 (m, 5H), 7.31 (s, 1H), 7.11 (m, 1H), 7.02 (d, 1H), 2.43 (s, 3H); 13C NMR (75 MHz, CDCl3) ppm 157.8, 150.0, 137.9, 131.5, 130.6, 129.1, 129.0, 128.9, 128.1, 127.2, 125.6, 125.2, 118.3, 115.4, 21.1; EI—HR—MS: m/z for [M+H]+ = 312.1373, Calcd. m/z for [M+H]+ = 312.1388.

S3. Refinement

Hydrogen atoms on C were placed in idealized positions with C—H bond distances 0.95 - 0.98 Å and thereafter treated as riding. Displacement parameters for H were assigned as Uiso = 1.2Ueq of the attached atom (1.5 for methyl and OH). A torsional parameter was refined for each methyl group, and OH hydrogen positions were refined.

Figure 1

The asymmetric unit with ellipsoids at the 50% probability level and H atoms having arbitrary radius.

2-(9-Anthrylmethylideneamino)-4-methylphenol

Crystal data

| C22H17NO | Z = 4 |
|----------|------|
| M = 311.37 | F(000) = 656 |
| Triclinic, P1 | D = 1.320 Mg m−3 |
| Hall symbol: -P 1 | Mo Kα radiation, λ = 0.71073 Å |
| a = 8.6037 (15) Å | Cell parameters from 7377 reflections |
| b = 12.839 (3) Å | θ = 2.5–27.8° |
| c = 15.015 (3) Å | μ = 0.08 mm−1 |
| α = 94.508 (9)° | T = 90 K |
| β = 97.164 (11)° | Lath, yellow |
| γ = 106.490 (11)° | 0.37 × 0.15 × 0.05 mm |
| V = 1566.6 (6) Å³ | |

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**Data collection**

Nonius KappaCCD diffractometer with Oxford Cryostream
Radiation source: fine-focus sealed tube
Graphite monochromator
\( \omega \) scans with \( \kappa \) offsets
35942 measured reflections
7462 independent reflections
4454 reflections with \( I > 2\sigma(I) \)

**Refinement**

Refinement on \( F^2 \)
Least-squares matrix: full
\( R[F^2 > 2\sigma(F^2)] = 0.054 \)
\( wR(F^2) = 0.127 \)

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
\( w = 1/[\sigma(F_o^2) + (0.0571P)^2] \)
\( P = (F_o^2 + 2F_c^2)/3 \)
Extinction correction: SHELXL97 (Sheldrick, 2008), \( F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{1/4} \)
Extinction coefficient: 0.0034 (10)

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\( \AA^2 \))**

| Atom | \( x \)       | \( y \)       | \( z \)       | \( U_{eq} \)       |
|------|--------------|--------------|--------------|------------------|
| O1   | 0.30490 (15) | 0.14730 (11) | 0.20434 (9)  | 0.0223 (3)       |
| H10H | 0.261 (2)    | 0.1946 (17)  | 0.2172 (14)  | 0.033*           |
| N1   | 0.13998 (16) | 0.26290 (12) | 0.10202 (10) | 0.0177 (3)       |
| C1   | 0.0939 (2)   | 0.44058 (15) | 0.09855 (11) | 0.0164 (4)       |
| C2   | −0.0671 (2)  | 0.42007 (15) | 0.11964 (11) | 0.0167 (4)       |
| C3   | −0.1820 (2)  | 0.31351 (16) | 0.10892 (12) | 0.0215 (4)       |
| H3   | −0.1516      | 0.2527       | 0.0851       | 0.026*           |
| C4   | −0.3344 (2)  | 0.29700 (16) | 0.13208 (13) | 0.0246 (5)       |
| H4   | −0.4084      | 0.2251       | 0.1244       | 0.029*           |
| C5   | −0.3837 (2)  | 0.38605 (17) | 0.16758 (13) | 0.0249 (5)       |
| H5   | −0.4894      | 0.3733       | 0.1850       | 0.030*           |
| C6   | −0.2806 (2)  | 0.48933 (16) | 0.17677 (12) | 0.0213 (4)       |
| H6   | −0.3157      | 0.5486       | 0.1994       | 0.026*           |
| C7   | −0.1195 (2)  | 0.51040 (15) | 0.15290 (11) | 0.0170 (4)       |
| C8   | −0.0120 (2)  | 0.61631 (15) | 0.16377 (12) | 0.0196 (4)       |
|  | x     | y      | z      | Ueq  |
|---|-------|--------|--------|------|
| H8 | -0.0473 | 0.6754 | 0.1867 | 0.023* |
| C9 | 0.1462 (2) | 0.63784 (15) | 0.14184 (12) | 0.0181 (4) |
| C10 | 0.2576 (2) | 0.74645 (16) | 0.15396 (13) | 0.0237 (4) |
| H10 | 0.2225 | 0.8059 | 0.1763 | 0.028* |
| C11 | 0.4120 (2) | 0.76605 (17) | 0.13417 (14) | 0.0289 (5) |
| H11 | 0.4841 | 0.8386 | 0.1425 | 0.035* |
| C12 | 0.4657 (2) | 0.67730 (16) | 0.10083 (13) | 0.0262 (5) |
| H12 | 0.5740 | 0.6911 | 0.0870 | 0.031* |
| C13 | 0.3642 (2) | 0.57331 (16) | 0.08864 (13) | 0.0222 (4) |
| H13 | 0.4031 | 0.5156 | 0.0665 | 0.027* |
| C14 | 0.2004 (2) | 0.54824 (15) | 0.10823 (12) | 0.0178 (4) |
| C15 | 0.1512 (2) | 0.34955 (15) | 0.06342 (12) | 0.0181 (4) |
| H15 | 0.1987 | 0.3556 | 0.0095 | 0.022* |
| C16 | 0.1843 (2) | 0.17612 (14) | 0.05799 (12) | 0.0165 (4) |
| C17 | 0.2645 (2) | 0.11812 (15) | 0.11268 (12) | 0.0170 (4) |
| C18 | 0.3106 (2) | 0.03190 (15) | 0.07482 (12) | 0.0191 (4) |
| H18 | 0.3682 | -0.0058 | 0.1120 | 0.023* |
| C19 | 0.2723 (2) | 0.00072 (15) | -0.01782 (13) | 0.0218 (4) |
| H19 | 0.3061 | -0.0577 | -0.0435 | 0.026* |
| C20 | 0.1853 (2) | 0.05329 (15) | -0.07398 (12) | 0.0202 (4) |
| C21 | 0.1432 (2) | 0.14129 (16) | -0.03464 (12) | 0.0198 (4) |
| H21 | 0.0850 | 0.1787 | -0.0718 | 0.024* |
| C22 | 0.1364 (2) | 0.01475 (18) | -0.17348 (13) | 0.0316 (5) |
| H22A | 0.0264 | -0.0380 | -0.1843 | 0.047* |
| H22B | 0.2149 | -0.0203 | -0.1943 | 0.047* |
| H22C | 0.1359 | 0.0774 | -0.2067 | 0.047* |
| O2 | 0.53743 (15) | 0.04269 (11) | 0.27603 (8) | 0.0207 (3) |
| H20H | 0.488 (2) | 0.0923 (17) | 0.2760 (13) | 0.031* |
| N2 | 0.52578 (16) | 0.20557 (12) | 0.39729 (10) | 0.0168 (3) |
| C23 | 0.48926 (19) | 0.38573 (14) | 0.40197 (11) | 0.0142 (4) |
| C24 | 0.3154 (2) | 0.35684 (14) | 0.38026 (11) | 0.0150 (4) |
| C25 | 0.2085 (2) | 0.25083 (15) | 0.38727 (11) | 0.0175 (4) |
| H25 | 0.2540 | 0.1949 | 0.4049 | 0.021* |
| C26 | 0.0420 (2) | 0.22873 (15) | 0.36905 (12) | 0.0197 (4) |
| H26 | -0.0263 | 0.1578 | 0.3746 | 0.024* |
| C27 | -0.0304 (2) | 0.30931 (16) | 0.34210 (12) | 0.0205 (4) |
| H27 | -0.1463 | 0.2922 | 0.3290 | 0.025* |
| C28 | 0.0661 (2) | 0.41141 (16) | 0.33494 (12) | 0.0195 (4) |
| H28 | 0.0167 | 0.4656 | 0.3175 | 0.023* |
| C29 | 0.2409 (2) | 0.43844 (15) | 0.35330 (11) | 0.0161 (4) |
| C30 | 0.3395 (2) | 0.54295 (15) | 0.34506 (11) | 0.0172 (4) |
| H30 | 0.2891 | 0.5956 | 0.3249 | 0.021* |
| C31 | 0.5098 (2) | 0.57245 (14) | 0.36561 (11) | 0.0167 (4) |
| C32 | 0.6089 (2) | 0.67977 (15) | 0.35648 (13) | 0.0229 (4) |
| H32 | 0.5579 | 0.7320 | 0.3359 | 0.027* |
| C33 | 0.7750 (2) | 0.70842 (15) | 0.37681 (13) | 0.0236 (4) |
| H33 | 0.8392 | 0.7804 | 0.3709 | 0.028* |
| C34 | 0.8524 (2) | 0.63117 (15) | 0.40679 (12) | 0.0211 (4) |
| Atom | U<sup>11</sup> | U<sup>22</sup> | U<sup>33</sup> | U<sup>12</sup> | U<sup>13</sup> | U<sup>23</sup> |
|------|---------------|---------------|---------------|--------------|--------------|--------------|
| O1   | 0.0282 (7)    | 0.0235 (8)    | 0.0179 (7)    | 0.0142 (6)   | −0.0002 (6)  | 0.0010 (6)   |
| N1   | 0.0183 (8)    | 0.0189 (9)    | 0.0177 (8)    | 0.0092 (6)   | 0.0015 (6)   | 0.0003 (7)   |
| C1   | 0.0197 (9)    | 0.0186 (10)   | 0.0134 (9)    | 0.0097 (8)   | 0.0013 (7)   | 0.0030 (8)   |
| C2   | 0.0191 (9)    | 0.0201 (10)   | 0.0128 (9)    | 0.0099 (8)   | −0.0006 (7)  | 0.0029 (8)   |
| C3   | 0.0212 (10)   | 0.0212 (11)   | 0.0224 (10)   | 0.0094 (8)   | 0.0001 (8)   | −0.0016 (8)  |
| C4   | 0.0196 (9)    | 0.0238 (11)   | 0.0290 (11)   | 0.0055 (8)   | 0.0021 (8)   | 0.0014 (9)   |
| C5   | 0.0202 (10)   | 0.0313 (12)   | 0.0253 (11)   | 0.0110 (9)   | 0.0043 (8)   | 0.0018 (9)   |
| C6   | 0.0217 (10)   | 0.0283 (12)   | 0.0184 (10)   | 0.0151 (9)   | 0.0032 (8)   | 0.0009 (8)   |
| C7   | 0.0195 (9)    | 0.0232 (11)   | 0.0116 (9)    | 0.0121 (8)   | 0.0008 (7)   | 0.0036 (8)   |
| C8   | 0.0262 (10)   | 0.0200 (11)   | 0.0173 (10)   | 0.0150 (8)   | 0.0018 (8)   | 0.0028 (8)   |
| C9   | 0.0221 (9)    | 0.0204 (11)   | 0.0139 (9)    | 0.0098 (8)   | 0.0010 (8)   | 0.0042 (8)   |
| C10  | 0.0311 (11)   | 0.0165 (10)   | 0.0264 (11)   | 0.0118 (8)   | 0.0034 (9)   | 0.0034 (9)   |
| C11  | 0.0289 (11)   | 0.0217 (12)   | 0.0369 (13)   | 0.0070 (9)   | 0.0053 (9)   | 0.0087 (10)  |
| C12  | 0.0225 (10)   | 0.0264 (12)   | 0.0333 (12)   | 0.0099 (9)   | 0.0077 (9)   | 0.0095 (10)  |
| C13  | 0.0240 (10)   | 0.0228 (11)   | 0.0248 (11)   | 0.0136 (8)   | 0.0051 (8)   | 0.0062 (9)   |
| C14  | 0.0217 (9)    | 0.0201 (11)   | 0.0138 (9)    | 0.0103 (8)   | 0.0006 (7)   | 0.0038 (8)   |
| C15  | 0.0162 (9)    | 0.0228 (11)   | 0.0162 (10)   | 0.0077 (8)   | 0.0013 (7)   | 0.0021 (8)   |
| C16  | 0.0151 (8)    | 0.0163 (10)   | 0.0194 (10)   | 0.0056 (7)   | 0.0054 (7)   | 0.0016 (8)   |
| C17  | 0.0163 (9)    | 0.0159 (10)   | 0.0176 (10)   | 0.0033 (7)   | 0.0029 (8)   | 0.0000 (8)   |
| C18  | 0.0176 (9)    | 0.0169 (10)   | 0.0232 (11)   | 0.0067 (8)   | 0.0002 (8)   | 0.0030 (8)   |
| C19  | 0.0168 (9)    | 0.0189 (11)   | 0.0287 (11)   | 0.0045 (8)   | 0.0057 (8)   | −0.0043 (9)  |
| C20  | 0.0148 (9)    | 0.0242 (11)   | 0.0191 (10)   | 0.0030 (8)   | 0.0034 (8)   | −0.0034 (8)  |
| C21  | 0.0166 (9)    | 0.0244 (11)   | 0.0188 (10)   | 0.0060 (8)   | 0.0027 (8)   | 0.0052 (8)   |
| C22  | 0.0275 (11)   | 0.0411 (14)   | 0.0234 (11)   | 0.0081 (10)  | 0.0041 (9)   | −0.0059 (10) |
| O2   | 0.0236 (7)    | 0.0198 (8)    | 0.0208 (7)    | 0.0118 (6)   | −0.0003 (6)  | 0.0000 (6)   |
|    |    |    |    |    |    |
|----|----|----|----|----|----|
| N2 | 0.0180 (8) | 0.0160 (9) | 0.0193 (8) | 0.0086 (6) | 0.0051 (6) |
| C23| 0.0185 (9) | 0.0141 (10) | 0.0117 (9) | 0.0076 (7) | 0.0028 (7) |
| C24| 0.0202 (9) | 0.0162 (10) | 0.0101 (9) | 0.0083 (7) | 0.0025 (7) |
| C25| 0.0201 (9) | 0.0177 (10) | 0.0179 (10) | 0.0092 (8) | 0.0052 (8) |
| C26| 0.0195 (9) | 0.0170 (10) | 0.0214 (10) | 0.0032 (8) | 0.0048 (8) |
| C27| 0.0148 (9) | 0.0281 (12) | 0.0193 (10) | 0.0085 (8) | 0.0007 (8) |
| C28| 0.0213 (9) | 0.0255 (11) | 0.0157 (10) | 0.0142 (8) | 0.0006 (8) |
| C29| 0.0210 (9) | 0.0204 (10) | 0.0095 (9) | 0.0107 (8) | 0.0021 (7) |
| C30| 0.0250 (10) | 0.0163 (10) | 0.0150 (9) | 0.0127 (8) | 0.0043 (8) |
| C31| 0.0243 (9) | 0.0143 (10) | 0.0139 (9) | 0.0079 (8) | 0.0060 (8) |
| C32| 0.0314 (11) | 0.0156 (10) | 0.0249 (11) | 0.0096 (8) | 0.0087 (9) |
| C33| 0.0300 (11) | 0.0115 (10) | 0.0286 (11) | 0.0031 (8) | 0.0100 (9) |
| C34| 0.0199 (9) | 0.0191 (11) | 0.0218 (10) | 0.0027 (8) | 0.0053 (8) |
| C35| 0.0209 (9) | 0.0195 (10) | 0.0176 (10) | 0.0084 (8) | 0.0048 (8) |
| C36| 0.0208 (9) | 0.0131 (10) | 0.0120 (9) | 0.0056 (7) | 0.0042 (7) |
| C37| 0.0154 (9) | 0.0185 (10) | 0.0145 (9) | 0.0067 (7) | 0.0028 (7) |
| C38| 0.0128 (8) | 0.0126 (9) | 0.0201 (10) | 0.0048 (7) | 0.0060 (7) |
| C39| 0.0124 (8) | 0.0190 (10) | 0.0149 (9) | 0.0028 (7) | 0.0023 (7) |
| C40| 0.0161 (9) | 0.0135 (10) | 0.0216 (10) | 0.0058 (7) | 0.0071 (8) |
| C41| 0.0136 (8) | 0.0162 (10) | 0.0254 (10) | 0.0064 (7) | 0.0071 (8) |
| C42| 0.0127 (8) | 0.0193 (10) | 0.0209 (10) | 0.0039 (7) | 0.0059 (7) |
| C43| 0.0157 (9) | 0.0134 (10) | 0.0190 (10) | 0.0037 (7) | 0.0043 (7) |
| C44| 0.0210 (9) | 0.0244 (11) | 0.0224 (11) | 0.0087 (8) | 0.0040 (8) |

Geometric parameters (Å, º)

| Bond | Distance (Å) | Bond | Distance (Å) |
|------|-------------|------|-------------|
| O1—C17 | 1.371 (2) | O2—C39 | 1.368 (2) |
| O1—H10H | 0.82 (2) | O2—H20H | 0.86 (2) |
| N1—C15 | 1.280 (2) | N2—C37 | 1.279 (2) |
| N1—C16 | 1.419 (2) | N2—C38 | 1.414 (2) |
| C1—C14 | 1.410 (2) | C33—C36 | 1.417 (2) |
| C1—C2 | 1.416 (2) | C23—C24 | 1.423 (2) |
| C1—C15 | 1.477 (2) | C23—C37 | 1.470 (2) |
| C2—C3 | 1.426 (2) | C24—C25 | 1.430 (2) |
| C2—C7 | 1.435 (2) | C24—C29 | 1.434 (2) |
| C3—C4 | 1.361 (2) | C25—C26 | 1.366 (2) |
| C3—H3 | 0.9500 | C25—H25 | 0.9500 |
| C4—C5 | 1.417 (3) | C26—C27 | 1.411 (3) |
| C4—H4 | 0.9500 | C26—H26 | 0.9500 |
| C5—C6 | 1.356 (3) | C27—C28 | 1.359 (3) |
| C5—H5 | 0.9500 | C27—H27 | 0.9500 |
| C6—C7 | 1.431 (2) | C28—C29 | 1.429 (2) |
| C6—H6 | 0.9500 | C28—H28 | 0.9500 |
| C7—C8 | 1.395 (2) | C29—C30 | 1.393 (2) |
| C8—C9 | 1.396 (2) | C30—C31 | 1.393 (2) |
| C8—H8 | 0.9500 | C30—H30 | 0.9500 |
| C9—C10 | 1.433 (3) | C31—C32 | 1.427 (2) |
| C9—C14 | 1.438 (2) | C31—C36 | 1.437 (2) |
C10—C11 1.355 (3) C32—C33 1.359 (3)
C10—H10 0.9500 C32—H32 0.9500
C11—C12 1.425 (3) C33—C34 1.414 (3)
C11—H11 0.9500 C33—H33 0.9500
C12—C13 1.356 (3) C34—C35 1.358 (2)
C12—H12 0.9500 C34—H34 0.9500
C13—C14 1.427 (2) C35—C36 1.434 (2)
C13—H13 0.9500 C35—H35 0.9500
C15—H15 0.9500 C37—H37 0.9500
C16—C21 1.396 (2) C38—C43 1.393 (2)
C16—C17 1.397 (2) C38—C39 1.397 (2)
C17—C18 1.383 (2) C39—C40 1.382 (2)
C18—C19 1.388 (3) C40—C41 1.389 (2)
C18—H18 0.9500 C40—H40 0.9500
C19—C20 1.395 (3) C41—C42 1.393 (2)
C19—H19 0.9500 C41—H41 0.9500
C20—C21 1.392 (2) C42—C43 1.388 (2)
C20—C22 1.504 (3) C42—C44 1.509 (2)
C21—H21 0.9500 C43—H43 0.9500
C22—H22A 0.9800 C44—H44A 0.9800
C22—H22B 0.9800 C44—H44B 0.9800
C22—H22C 0.9800 C44—H44C 0.9800

C17—O1—H10H 106.8 (15) C39—O2—H20H 105.9 (14)
C15—N1—C16 118.53 (15) C37—N2—C38 119.84 (15)
C14—C1—C2 120.64 (16) C36—C23—C24 120.39 (16)
C14—C1—C15 118.97 (15) C36—C23—C37 117.76 (15)
C2—C1—C15 120.36 (16) C24—C23—C37 121.85 (16)
C1—C2—C3 123.32 (16) C23—C24—C29 123.46 (16)
C1—C2—C7 119.01 (16) C23—C24—C25 119.12 (16)
C3—C2—C7 117.67 (15) C25—C24—C29 117.37 (15)
C4—C3—C2 121.46 (18) C26—C25—C24 121.06 (17)
C4—C3—H3 119.3 C26—C25—H25 119.5
C2—C3—H3 119.3 C24—C25—H25 119.5
C3—C4—C5 120.64 (18) C25—C26—C27 121.23 (17)
C3—C4—H4 119.7 C25—C26—H26 119.4
C5—C4—H4 119.7 C27—C26—H26 119.4
C6—C5—C4 120.15 (17) C28—C27—C26 119.87 (16)
C6—C5—H5 119.9 C28—C27—H27 120.1
C4—C5—H5 119.9 C26—C27—H27 120.1
C5—C6—C7 121.02 (18) C27—C28—C29 120.93 (17)
C5—C6—H6 119.5 C27—C28—H28 119.5
C7—C6—H6 119.5 C29—C28—H28 119.5
C8—C7—C6 121.19 (17) C30—C29—C28 120.78 (16)
C8—C7—C2 119.78 (15) C30—C29—C24 119.70 (15)
C6—C7—C2 119.00 (16) C28—C29—C24 119.53 (16)
C7—C8—C9 121.78 (17) C31—C30—C29 121.87 (16)
C7—C8—H8 119.1 C31—C30—H30 119.1
| Bond                | Angle        | Bond                | Angle        |
|---------------------|--------------|---------------------|--------------|
| C9—C8—H8           | 119.1        | C29—C30—H30        | 119.1        |
| C8—C9—C10          | 121.89 (17)  | C30—C31—C32        | 121.12 (17)  |
| C8—C9—C14          | 119.11 (16)  | C30—C31—C36        | 119.56 (16)  |
| C10—C9—C14         | 118.98 (16)  | C32—C31—C36        | 119.32 (16)  |
| C11—C10—C9         | 121.35 (18)  | C33—C32—C31        | 121.00 (18)  |
| C11—C10—H10        | 119.3        | C33—C32—H32        | 119.5        |
| C9—C10—H10         | 119.3        | C31—C32—H32        | 119.5        |
| C10—C11—C12        | 119.68 (18)  | C32—C33—C34        | 120.05 (17)  |
| C10—C11—H11        | 120.2        | C32—C33—H33        | 120.0        |
| C12—C11—H11        | 120.2        | C34—C33—H33        | 120.0        |
| C13—C12—C11        | 120.09 (17)  | C35—C34—C33        | 120.98 (17)  |
| C13—C12—H12        | 119.6        | C35—C34—H34        | 119.5        |
| C11—C12—H12        | 119.6        | C33—C34—H34        | 119.5        |
| C12—C13—C14        | 121.78 (18)  | C34—C35—C36        | 121.33 (17)  |
| C12—C13—H13        | 119.1        | C34—C35—H35        | 119.3        |
| C14—C13—H13        | 119.1        | C36—C35—H35        | 119.3        |
| C1—C14—C13         | 122.90 (17)  | C23—C36—C35        | 123.36 (16)  |
| C1—C14—C9          | 119.66 (15)  | C23—C36—C31        | 119.28 (15)  |
| C13—C14—C9         | 117.42 (16)  | C35—C36—C31        | 117.32 (16)  |
| N1—C15—C1          | 123.23 (16)  | N2—C37—C23         | 122.99 (16)  |
| N1—C15—H15         | 118.4        | N2—C37—H37         | 118.5        |
| C1—C15—H15         | 118.4        | C23—C37—H37        | 118.5        |
| C21—C16—C17        | 118.71 (16)  | C43—C38—C39        | 119.36 (16)  |
| C21—C16—N1         | 124.14 (16)  | C43—C38—N2         | 125.80 (16)  |
| C17—C16—N1         | 117.01 (15)  | C39—C38—N2         | 114.77 (16)  |
| O1—C17—C18         | 118.31 (16)  | O2—C39—C40         | 119.20 (16)  |
| O1—C17—C16         | 121.24 (16)  | O2—C39—C38         | 120.91 (16)  |
| C18—C17—C16        | 120.41 (16)  | C40—C39—C38        | 119.88 (16)  |
| C17—C18—C19        | 119.72 (17)  | C39—C40—C41        | 119.70 (17)  |
| C17—C18—H18        | 120.1        | C39—C40—H40        | 120.2        |
| C19—C18—H18        | 120.1        | C41—C40—H40        | 120.2        |
| C18—C19—C20        | 121.45 (17)  | C40—C41—C42        | 121.63 (17)  |
| C18—C19—H19        | 119.3        | C40—C41—H41        | 119.2        |
| C20—C19—H19        | 119.3        | C42—C41—H41        | 119.2        |
| C21—C20—C19        | 117.80 (17)  | C43—C42—C41        | 117.83 (17)  |
| C21—C20—C22        | 121.23 (18)  | C43—C42—C44        | 121.43 (17)  |
| C19—C20—C22        | 120.97 (17)  | C41—C42—C44        | 120.68 (16)  |
| C20—C21—C16        | 121.79 (17)  | C42—C43—C38        | 121.54 (17)  |
| C20—C21—H21        | 119.1        | C42—C43—H43        | 119.2        |
| C16—C21—H21        | 119.1        | C38—C43—H43        | 119.2        |
| C20—C22—H22A       | 109.5        | C42—C44—H44A       | 109.5        |
| C20—C22—H22B       | 109.5        | C42—C44—H44B       | 109.5        |
| H22A—C22—H22B      | 109.5        | H44A—C44—H44B      | 109.5        |
| C20—C22—H22C       | 109.5        | C42—C44—H44C       | 109.5        |
| H22A—C22—H22C      | 109.5        | H44A—C44—H44C      | 109.5        |
| H22B—C22—H22C      | 109.5        | H44B—C44—H44C      | 109.5        |
| C14—C1—C2—C3       | 178.20 (16)  | C36—C23—C24—C25    | −178.08 (15) |
| Bond | Deviation (°) |
|------|--------------|
| C15—C1—C2—C3 | 0.0 (3) |
| C14—C1—C2—C7 | -1.3 (3) |
| C15—C1—C2—C7 | -179.45 (16) |
| C1—C2—C3—C4 | 178.37 (17) |
| C7—C2—C3—C4 | -2.1 (3) |
| C2—C3—C4—C5 | 0.2 (3) |
| C3—C4—C5—C6 | 1.6 (3) |
| C4—C5—C6—C7 | -1.4 (3) |
| C5—C6—C7—C8 | -178.84 (17) |
| C5—C6—C7—C2 | -0.6 (3) |
| C1—C2—C7—C8 | 0.1 (2) |
| C3—C2—C7—C8 | -179.44 (16) |
| C1—C2—C7—C6 | -178.15 (16) |
| C3—C2—C7—C6 | 2.3 (2) |
| C6—C7—C8—C9 | 179.17 (17) |
| C2—C7—C8—C9 | 1.0 (3) |
| C7—C8—C9—C10 | -179.25 (17) |
| C7—C8—C9—C14 | -0.8 (3) |
| C8—C9—C10—C11 | 178.62 (18) |
| C14—C9—C10—C11 | 0.2 (3) |
| C9—C10—C11—C12 | -0.2 (3) |
| C10—C11—C12—C13 | 0.0 (3) |
| C11—C12—C13—C14 | 0.2 (3) |
| C2—C1—C14—C13 | 179.46 (16) |
| C15—C1—C14—C13 | -2.3 (3) |
| C2—C1—C14—C9 | 1.5 (3) |
| C15—C1—C14—C9 | 179.64 (16) |
| C12—C13—C14—C1 | -178.18 (17) |
| C12—C13—C14—C9 | -0.1 (3) |
| C8—C9—C14—C1 | -0.4 (3) |
| C10—C9—C14—C1 | 178.07 (16) |
| C8—C9—C14—C13 | -178.52 (17) |
| C10—C9—C14—C13 | 0.0 (2) |
| C16—N1—C15—C1 | 174.06 (15) |
| C14—C1—C15—N1 | 130.13 (19) |
| C2—C1—C15—N1 | -51.7 (3) |
| C15—N1—C16—C21 | -41.7 (2) |
| C15—N1—C16—C17 | 142.73 (16) |
| C21—C16—C17—O1 | -178.70 (15) |
| N1—C16—C17—O1 | -2.9 (2) |
| C21—C16—C17—C18 | 3.7 (2) |
| N1—C16—C17—C18 | 179.53 (15) |
| O1—C17—C18—C19 | -179.66 (15) |
| C16—C17—C18—C19 | -2.0 (2) |
| C17—C18—C19—C20 | -1.2 (3) |
| C18—C19—C20—C21 | 2.4 (2) |
| C18—C19—C20—C22 | -176.54 (16) |
| C19—C20—C21—C16 | -0.6 (2) |
### Hydrogen-bond geometry (Å, °)

|          | D—H  | H···A | D···A  | D—H···A  |
|----------|------|------|-------|----------|
| O1—H10  | 0.82 (2) | 2.27 (2) | 2.754 (2) | 118.0 (17) |
| O2—H20  | 0.86 (2) | 2.11 (2) | 2.8602 (18) | 144.9 (18) |
| O2—H20  | 0.86 (2) | 2.17 (2) | 2.695 (2) | 119.1 (17) |