Syntheses and crystal structures of a nitro–anthracene–isoxazole and its oxidation product

Chun Li, Matthew J. Weaver, Michael J. Campbell and Nicholas R. Natale

*Department of Chemistry, Ithaca College, 953 Danby Road, Ithaca, NY 14850, USA, and †Department of Biomedical and Pharmaceutical Sciences, University of Montana, Missoula, MT 59812, USA. *Correspondence e-mail: nicholas.natale@umontana.edu

The syntheses and structures of an unexpected by-product from an iodination reaction, namely, ethyl 5-methyl-3-(10-nitroanthracen-9-yl)isoxazole-4-carboxylate, C_{21}H_{16}N_{2}O_{5}, (I), and its oxidation product, ethyl 3-(9-hydroxy-10-oxo-9,10-dihydroanthracen-9-yl)-5-methylisoxazole-4-carboxylate, C_{21}H_{17}NO_{5} (V) are described. Compound (I) crystallizes with two molecules in the asymmetric unit in which the dihedral angles between the anthracene fused-ring systems and isoxazole ring mean planes are 88.67 (16) and 85.64 (16)°; both molecules feature a disordered nitro group. In (V), which crystallizes with one molecule in the asymmetric unit, the equivalent dihedral angle between the almost planar anthrone ring system (r.m.s. deviation = 0.029 Å) and the pendant isoxazole ring is 89.65 (5)°. In the crystal of (I), the molecules are linked by weak C—H· · ·O interactions into a three-dimensional network and in the extended structure of (V), inversion dimers linked by pairwise O—H· · ·O hydrogen bonds generate $R_{2}^{2}(14)$ loops.

1. Chemical context

In the course of our study of aryl-isoxazole amide (AIM) antitumor agents, we have a standard operating procedure to identify by-products of the synthesis (Weaver, Campbell et al., 2020), and have used the mechanistic insights gained in order to optimize and improve subsequent syntheses.

During recent structure–activity relationship studies, we encountered complications in constructing sterically hindered examples, which we desired for their calculated pharmacokinetic properties. After obtaining mediocre results with bromine as a leaving group in Suzuki couplings, we pursued a fairly routine alternative of moving to the next halogen down in the periodic table. We have encountered more complications in this study than in the previous twenty papers we have
published in this area (e.g. Weaver, Stump et al., 2020 and Weaver et al., 2015), and herein report the crystal structures of two compounds observed.

Using conditions usually reported for iodination, the main product observed for reaction of (II) was the nitro ester (I) rather than the expected iodo product (III), which was obtained in small amounts (Fig. 1). The nitro product so obtained exhibits most of the stereoelectronic properties of previously studied analogues that we have considered to be essential for their biological activity (Han et al., 2009). The nitro group is disordered and found in two distinct conformations in the unit cell. We attribute this to an extreme peri-effect, which substantially raises the energy of the co-planar conformer.

In order to improve on the accuracy of the crystal structure of (I) we attempted numerous recrystallizations; however, what was observed was the addition of oxygen to compound (I), which we attribute to cycloaddition of dioxygen to an endo-peroxide (IV) (Klaper et al., 2016), and ring opening with loss of a leaving group to the oxidation product anthraquinone (V). Usually, anthracenes are oxidized in vivo predominantly by cytochrome P450, leading to a potentially toxic arene oxide (Silverman et al., 2014). The rationale for the isoxazole series is that the C-5 isoxazole methyl group represents an opportunity for safer metabolism (Natale et al., 2010). The observation in this manuscript suggests that intramolecular dioxygenation, which would likely be mediated in vivo by mono amine oxidase (MAO), is another plausible route (Silverman, 2002). The observation of a possible endo-peroxide pathway in this study suggests that the metabolism of these 10-substituted anthracenyl isoxazole analogues could go through dioxygenation catalysed by COX (cyclooxygenase) and other prostaglandin synthases in vivo (Silverman, 2002).

2. Structural commentary

The first title compound (I), C_{21}H_{16}N_{2}O_{5}, crystallizes in the monoclinic Cc space group with two independent molecules in the asymmetric unit (Fig. 2). The dihedral angle between the anthracene ring mean plane and the isoxazole ring mean plane indicate near orthogonality: 88.67 (16) and 85.64 (16)\degree for molecules A (containing C1) and B (containing C22), respectively. Each independent anthryl ring contains a 10-nitro group with the O atoms disordered over two orientations. The isoxazole group and its attached ethyl ester moiety are virtually co-planar, with the twist angles found to be 3.1 (2)\degree between the C15–C17/O1/N1 and O2/C19/O3/C20 planes in molecule A, and 4.2 (2)\degree between the C36–C38/O6/N3 and O7/C40/O8/C41 planes in molecule B. The ester ethyl group is exo- with respect to the anthryl ring in the solid state but this conformation is not completely retained in solution as the proton NMR indicates significant anisotropy at the methyl group of the ethyl ester (δ = 0.41), which indicates at the very least a significant population of the endo-orientation. In addition, many of our other reported anthracenyl isoxazole esters have shown the ester ethyl group in an endo-orienta-
The partial packing of compound (I). For clarity, only hydrogen bonds C1—H1···O5 and C3—H3···O7 are shown as dashed lines, and H atoms not involved in these hydrogen bonds are removed.

Figure 6
(a) The Hirshfeld surface of (I) mapped over $d_{norm}$. Short and long contacts are indicated as red and blue spots, respectively. Contacts with distances approximately equal to the sum of the van der Waals radii are colored white. (b) Weak $\pi$···$\pi$ interactions are shown as green dashed lines on a surface mapped over curvedness. The $\pi$···$\pi$ stacking is indicated by the green flat regions surrounded by dark blue edges.

4. Hirshfeld surface analysis

Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was performed, and the associated two-dimensional fingerprint plots (McKinnon et al., 2007) were generated to quantify the intermolecular interactions using Crystal Explorer 21.5 (Spackman et al., 2021). The Hirshfeld surface of (I) is mapped over $d_{norm}$ in a fixed color scale of −0.31 (red) to 1.26 (blue) arbitrary units (Fig. 6). The delineated two-dimensional fingerprint plots shown in Fig. 7 indicate that two main contributions to the overall Hirshfeld surface area arise from H···H contacts (35.3%) and O···H/H···O contacts (29.0%) with C···H/H···C interactions contributing 17.5% of the Hirshfeld surface.

The Hirshfeld surface of compound V is mapped over $d_{norm}$ in a fixed color scale of −0.58 (red) to 1.31 (blue) arbitrary units (Fig. 8a), showing two short contacts from O···H hydrogen bonds in red spots. The delineated two-dimensional fingerprint plots (Fig. 9) indicate that H···H contacts contribute 47.7% of the Hirshfeld surface. Aromatic $\pi$···$\pi$ stacking is...
also identifiable from the Hirshfeld surface mapped over the shape-index property (Fig. 8b).

5. Database survey

A search for the 9-nitroanthracenyl moiety in the Cambridge Structural Database (CSD version 5.43, November 2021 update; Groom et al., 2016) resulted in 14 hits, of which two crystal structures of 9-nitroanthracene itself were reported, namely refcodes NTRANT (Trotter, 1959) and NTRANT01 (Glagovich et al., 2004). The reported angles between the NO$_2$ plane and the anthracene plane are 84.78 and 69.40°, respectively, which agree with our observation of the disordered NO$_2$ group in (I).

A search in the same database for the 10-hydroxy anthrone fragment resulted in 59 hits, of which 10 structures had an aromatic ring at the 10-position, namely refcodes COBWEY (Barker et al., 2019), DULVUB (Skrzat & Roszak, 1986), ELULII (Stepovik et al., 2015), EVETIL (Mao et al., 2021), JAYPAA (Roszak et al., 1990), MOTJIQ (Chen et al., 2015), MOTKEN (Chen et al., 2015), QAJPUQ (Forensi et al., 2020), SAMNEC (Hoffend et al., 2013) and WOKYIH (Pullella et al., 2019). The anthrone unit in these 10 structures are either essentially planar or in a shallow boat conformation. The aromatic rings at the 10-position in these compounds are all at a vertical orientation relative to the anthrone ring. It may be noted that an anthrone isoxazole ester we reported in 2014, refcode TIYZEI, also shares similar structural features (Duncan et al., 2014).

6. Synthesis and crystallization

Iodination of aromatic hydrocarbons with molecular iodine has been accomplished by several methods, typically using an oxidizing agent to generate the iodonium cation electrophile. Among the conditions we surveyed, fuming nitric acid (Bansal et al., 1987) with the anthracene isoxazole (II), appears to consistently produce the nitrated anthryl (I) rather than the desired iodo product (III). The anthryl isoxazole ester (II) was prepared as previously described (Mosher et al., 1996), and recrystallized before use. The ester

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**Figure 7**
The two-dimensional fingerprint plots for (I) delineated into (a) H···H contacts, (b) O···H/$\cdot$O contacts, (c) C···H/$\cdot$C/C1 contacts, and (d) N···H/$\cdot$N contacts. Other contact contributions less than 5% are omitted.

**Figure 8**
(a) The Hirshfeld surface of (V) mapped over $d_{norm}$. Short and long contacts are indicated as red and blue spots, respectively. Contacts with distances approximately equal to the sum of the van der Waals radii are colored white. Hydroxyl and carbonyl groups on the anthrone ring contributed major short contacts. (b) $\pi$--$\pi$ interactions (anthrone to anthrone and carbonyl to isoxazole ring) and $\sigma$--$\pi$ interaction (C--H bond to carbonyl) are shown as orange--red spots with green dashed lines in the shape-index map.

**Figure 9**
The two-dimensional fingerprint plots for (V) delineated into (a) H···H contacts, (b) O···H/$\cdot$O contacts, (c) C···H/$\cdot$C contacts, and (d) N···H/$\cdot$N contacts. Other contact contributions less than 5% are omitted.

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Table 3
Experimental details.

| (I)                     | (V)                     |
|-------------------------|-------------------------|
| Crystal data            |                         |
| Chemical formula        | C_{21}H_{16}N_{2}O_{5}  |
| M_0                     | 376.36                  |
| Crystal system, space group | Monoclinic, Cc           |
| Temperature (K)         | 100                     |
| a, b, c (Å)             | 16.4968 (10), 14.8697 (9), 16.1836 (9) |
| β (°)                   | 114.879 (3)             |
| V (Å³)                  | 3601.5 (4)              |
| Z                       | 8                       |
| Radiation type          | Mo Kα                   |
| μ (mm⁻¹)                | 0.10                    |
| Crystal size (mm)       | 0.29 × 0.24 × 0.22      |

Data collection

Diffractometer Bruker SMART Breeze CCD
Absorption correction –

No. of measured, independent and observed [I > 2σ(I)] reflections
Rint 0.054

(sin θ/λ)max (Å⁻¹) 0.633

Refinement

R[F² > 2σ(F²)], wR(F²), S
No. of reflections 7615
No. of parameters 546
No. of restraints 2
H-atom treatment H atoms treated by a mixture of independent and constrained refinement

Δρmax, Δρmin (e Å⁻³) 0.55, −0.19

Absolute structure

Flack x determined using 2257 quotients

Absolute structure parameter 0.5 (4)

Computer programs: APEX2 (Bruker, 2012), SAINT (Bruker, 2018), SHELXS (Sheldrick, 2008), SHELXL2018/1 (Sheldrick, 2015) and OLEX2 (Dolomanov et al., 2009).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. In compound (I), the nitro group is disordered in each of the two independent molecules in the asymmetric unit. The occupancies of each disordered part were refined, converging to 0.572 (13) and 0.428 (13) for molecule A and 0.64 (3) and 0.36 (3) for molecule B. EADP constraints were applied (Sheldrick, 2015) to each nitro group. The C-bound hydrogen atoms on both compounds were fixed geometrically and treated as riding with C–H = 0.95–0.98 Å and refined with Uiso(H) = 1.2Ueq(CH, CH2) or 1.5Ueq(CH3). The O-bound H atom in (V) was found in a difference-Fourier map and refined freely. Four reflections (110, 110, 111) in compound (I) and four reflections (100, 100, 110, 111) in compound (V) affected by the beam stop were omitted from the final cycles of refinement because of poor agreement between the observed and calculated intensities. The absolute structure of (I) was indeterminate in the present refinement.
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Computing details
For both structures, data collection: APEX2 (Bruker, 2012); cell refinement: SAINT (Bruker, 2018); data reduction: SAINT (Bruker, 2018); program(s) used to solve structure: SHELXS (Sheldrick, 2008); program(s) used to refine structure: SHELXL2018/1 (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Ethyl 5-methyl-3-(10-nitroanthracen-9-yl)isoxazole-4-carboxylate (I)

Crystal data

| C_{21}H_{16}N_{2}O_{5} | F(000) = 1568 |
|----------------------|--------------|
| Mr = 376.36          | D_{x} = 1.388 Mg m^{-3} |
| Monoclinic, Cc       | Mo Kα radiation, λ = 0.71073 Å |
| a = 16.4968 (10) Å   | Cell parameters from 7597 reflections |
| b = 14.8697 (9) Å    | θ = 2.7–21.0° |
| c = 16.1836 (9) Å    | µ = 0.10 mm^{-1} |
| β = 114.879 (3)°     | T = 100 K |
| V = 3601.5 (4) Å³    | Prism, yellow |
| Z = 8                | 0.29 × 0.24 × 0.22 mm |

Data collection

| Bruker SMART Breeze CCD diffractometer | 5596 reflections with I > 2σ(I) |
| Radiation source: 2 kW sealed X-ray tube | R_{int} = 0.054 |
| φ and ω scans | θ_{max} = 26.7°, θ_{min} = 2.7° |
| 45790 measured reflections | h = -20→20 |
| 7615 independent reflections | k = -18→18 |
| 7615 independent reflections | l = -20→20 |

Refinement

| Refinement on F^2 | H atoms treated by a mixture of independent and constrained refinement |
| Least-squares matrix: full | w = 1/[σ(F_c^2) + (0.0864P)^2 + 2.331P] |
| R(F^2 > 2σ(F^2)) = 0.059 | where P = (F_c^2 + 2F_s^2)/3 |
| wR(F^2) = 0.158 | (Δ/σ)_{max} < 0.001 |
| S = 1.02 | Δρ_{max} = 0.55 e Å^{-3} |
| 7615 reflections | Δρ_{min} = -0.19 e Å^{-3} |
| 546 parameters | Absolute structure: Flack x determined using |
| 2 restraints | 2257 quotients [(I+)-(I-)]/[I+] (Parsons et al., 2013) |
| Primary atom site location: structure-invariant direct methods | Absolute structure parameter: 0.5 (4) |
| Hydrogen site location: mixed |
**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

| Atom | x     | y     | z     | U(eq)  |
|------|-------|-------|-------|--------|
| O1   | 0.3319(3) | 0.2201(3) | 0.5466(2) | 0.0487(9) |
| O2   | 0.3808(2)  | 0.0465(3)  | 0.3397(2)  | 0.0459(9)  |
| O3   | 0.4880(2)  | 0.0385(3)  | 0.4815(2)  | 0.0471(9)  |
| N1   | 0.2636(3)  | 0.2332(3)  | 0.4569(3)  | 0.0440(10) |
| H1   | 0.363669    | 0.290494    | 0.346904    | 0.047*     |
| C2   | 0.3469(4)   | 0.3427(4)   | 0.2259(4)   | 0.0519(14) |
| H2   | 0.397851    | 0.380760    | 0.250073    | 0.062*     |
| C3   | 0.2934(4)   | 0.3424(4)   | 0.1320(4)   | 0.0548(15) |
| H3   | 0.308016    | 0.380624    | 0.93210     | 0.066*     |
| C4   | 0.2203(4)   | 0.2876(4)   | 0.0955(4)   | 0.0471(13) |
| H4   | 0.184644    | 0.288156    | 0.031651    | 0.056*     |
| C5   | 0.0334(3)   | 0.0476(3)   | 0.1397(3)   | 0.0375(11) |
| H5   | 0.001(4)    | 0.040(3)    | 0.083(4)    | 0.045(15)* |
| C6   | 0.0166(4)   | −0.0086(4)  | 0.1956(4)   | 0.0447(13) |
| H6   | −0.030(4)   | −0.042(4)   | 0.171(4)    | 0.042(15)* |
| C7   | 0.0677(3)   | −0.0057(4)  | 0.2910(3)   | 0.0433(12) |
| H7   | 0.054540    | −0.045527   | 0.329570    | 0.052*     |
| C8   | 0.1361(3)   | 0.0548(3)   | 0.3273(3)   | 0.0363(11) |
| H8   | 0.170089    | 0.056581    | 0.391448    | 0.044*     |
| C9   | 0.2294(3)   | 0.1746(3)   | 0.3054(3)   | 0.0335(10) |
| C10  | 0.1272(3)   | 0.1697(3)   | 0.1202(3)   | 0.0359(11) |
| C11  | 0.1972(3)   | 0.2301(3)   | 0.1519(3)   | 0.0354(11) |
| C12  | 0.2511(3)   | 0.2315(3)   | 0.2482(3)   | 0.0326(10) |
| C13  | 0.1573(3)   | 0.1148(3)   | 0.2714(3)   | 0.0295(10) |
| C14  | 0.1042(3)   | 0.1105(3)   | 0.1749(3)   | 0.0325(10) |
| C15  | 0.2857(3)   | 0.1785(3)   | 0.4056(3)   | 0.0330(10) |
| C16  | 0.3647(3)   | 0.1298(3)   | 0.4562(3)   | 0.0353(10) |
| C17  | 0.3904(3)   | 0.1590(4)   | 0.5441(3)   | 0.0404(12) |
| C18  | 0.4659(4)   | 0.1363(4)   | 0.6323(3)   | 0.0537(15) |
| H18A | 0.516186    | 0.176866    | 0.642774    | 0.081*     |
| H18B | 0.484587    | 0.074058    | 0.630503    | 0.081*     |
| H18C | 0.446894    | 0.143120    | 0.681695    | 0.081*     |
| C19  | 0.4107(3)   | 0.0671(4)   | 0.4186(3)   | 0.0402(12) |
| C20  | 0.5375(4)   | −0.0214(4)  | 0.4483(4)   | 0.0517(14) |
| H20A | 0.555653    | 0.011073    | 0.406087    | 0.062*     |
| H20B | 0.499576    | −0.072871   | 0.415356    | 0.062*     |
| C21  | 0.6175(4)   | −0.0541(4)  | 0.5297(4)   | 0.0585(15) |
| H21A | 0.651142    | −0.096711   | 0.509947    | 0.088*     |
|        | x     | y     | z     | Ueq  |
|--------|-------|-------|-------|------|
| H21B   | 0.597864 | -0.083994 | 0.572036 | 0.088* |
| H21C   | 0.655804 | -0.002855 | 0.560146 | 0.088* |
| O4     | 0.1027 (18) | 0.111 (3) | -0.022 (3) | 0.072 (6) | 0.572 (13) |
| O5     | -0.0003 (7) | 0.1959 (10) | -0.0102 (7) | 0.068 (3) | 0.572 (13) |
| N2     | 0.076 (3) | 0.156 (2) | 0.025 (3) | 0.039 (4) | 0.572 (13) |
| O4A    | 0.073 (3) | 0.105 (4) | -0.022 (4) | 0.072 (6) | 0.428 (13) |
| O5A    | 0.0259 (10) | 0.2395 (14) | -0.0113 (10) | 0.068 (3) | 0.428 (13) |
| N2A    | 0.066 (4) | 0.178 (3) | 0.017 (4) | 0.039 (4) | 0.428 (13) |
| O6     | 0.4350 (3) | 0.7239 (3) | 0.2788 (2) | 0.0553 (11) |
| O7     | 0.3756 (2) | 0.5736 (2) | 0.4917 (2) | 0.0424 (8) |
| O8     | 0.2920 (2) | 0.5316 (2) | 0.3474 (2) | 0.0422 (8) |
| N3     | 0.4850 (3) | 0.7577 (3) | 0.3678 (3) | 0.0561 (13) |
| N4     | 0.6061 (3) | 0.7796 (3) | 0.8043 (3) | 0.0495 (12) |
| C22    | 0.3713 (4) | 0.8310 (4) | 0.4975 (4) | 0.0506 (14) |
| H22    | 0.347573 | 0.821907 | 0.433552 | 0.061* |
| C23    | 0.3270 (4) | 0.8841 (4) | 0.5326 (4) | 0.0600 (16) |
| H23    | 0.272392 | 0.911450 | 0.492941 | 0.072* |
| C24    | 0.3606 (4) | 0.8994 (4) | 0.6269 (4) | 0.0568 (15) |
| H24    | 0.328476 | 0.936589 | 0.650609 | 0.068* |
| C25    | 0.4393 (4) | 0.8609 (3) | 0.6845 (4) | 0.0506 (14) |
| H25    | 0.461760 | 0.872032 | 0.748095 | 0.061* |
| C26    | 0.6969 (3) | 0.6606 (4) | 0.7304 (3) | 0.0471 (13) |
| H26    | 0.721891 | 0.669551 | 0.794480 | 0.057* |
| C27    | 0.7377 (4) | 0.6055 (5) | 0.6938 (5) | 0.0650 (17) |
| H27    | 0.792063 | 0.577059 | 0.732452 | 0.078* |
| C28    | 0.7018 (4) | 0.5891 (5) | 0.6000 (4) | 0.0635 (17) |
| H28    | 0.731967 | 0.549990 | 0.575834 | 0.076* |
| C29    | 0.6250 (4) | 0.6284 (4) | 0.5438 (4) | 0.0481 (13) |
| H29    | 0.600529 | 0.615429 | 0.480499 | 0.058* |
| C30    | 0.4988 (4) | 0.7306 (4) | 0.5209 (3) | 0.0405 (12) |
| C31    | 0.5696 (3) | 0.7629 (3) | 0.7055 (3) | 0.0364 (11) |
| C32    | 0.4875 (3) | 0.8051 (3) | 0.6514 (3) | 0.0381 (11) |
| C33    | 0.4527 (3) | 0.7886 (3) | 0.5551 (3) | 0.0400 (12) |
| C34    | 0.5802 (3) | 0.6883 (3) | 0.5776 (3) | 0.0349 (11) |
| C35    | 0.6165 (3) | 0.7057 (3) | 0.6735 (3) | 0.0369 (11) |
| C36    | 0.4597 (4) | 0.7112 (3) | 0.4208 (3) | 0.0413 (12) |
| C37    | 0.3956 (3) | 0.6454 (3) | 0.3713 (3) | 0.0370 (11) |
| C38    | 0.3817 (3) | 0.6569 (4) | 0.2828 (3) | 0.0429 (12) |
| C39    | 0.3211 (4) | 0.6178 (4) | 0.1946 (3) | 0.0478 (13) |
| H39A   | 0.316485 | 0.552762 | 0.201431 | 0.072* |
| H39B   | 0.344798 | 0.629473 | 0.149346 | 0.072* |
| H39C   | 0.261828 | 0.645184 | 0.174453 | 0.072* |
| C40    | 0.3545 (3) | 0.5804 (3) | 0.4101 (3) | 0.0365 (11) |
| C41    | 0.2435 (4) | 0.4683 (4) | 0.3801 (4) | 0.0487 (13) |
| H41A   | 0.226101 | 0.498471 | 0.424769 | 0.058* |
| H41B   | 0.282252 | 0.416477 | 0.410479 | 0.058* |
| C42    | 0.1625 (4) | 0.4370 (4) | 0.3006 (4) | 0.0563 (15) |
| H42A   | 0.180119 | 0.410741 | 0.255014 | 0.084* |
### Atomic displacement parameters (Å²)

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$   | $U^{13}$   | $U^{23}$   |
|-----|------------|------------|------------|------------|------------|------------|
| O1  | 0.052 (2)  | 0.056 (2)  | 0.0267 (19)| 0.0025 (19)| 0.0058 (17)| −0.0110 (15)|
| O2  | 0.045 (2)  | 0.058 (2)  | 0.0280 (19)| 0.0043 (17)| 0.0083 (16)| −0.0063 (15)|
| O3  | 0.037 (2)  | 0.066 (2)  | 0.0312 (19)| 0.0086 (18)| 0.0079 (16)| −0.0030 (17)|
| N1  | 0.044 (2)  | 0.049 (3)  | 0.029 (2)  | 0.006 (2)  | 0.0054 (19)| 0.0002 (19)|
| C1  | 0.031 (3)  | 0.041 (3)  | 0.038 (3)  | −0.006 (2) | 0.008 (2)  | 0.003 (2)  |
| C2  | 0.044 (3)  | 0.057 (3)  | 0.055 (4)  | −0.015 (3) | 0.021 (3)  | 0.001 (3)  |
| C3  | 0.050 (3)  | 0.069 (4)  | 0.048 (3)  | −0.011 (3) | 0.023 (3)  | 0.018 (3)  |
| C4  | 0.048 (3)  | 0.060 (3)  | 0.031 (3)  | −0.003 (3) | 0.015 (2)  | 0.006 (2)  |
| C5  | 0.031 (3)  | 0.050 (3)  | 0.026 (2)  | −0.009 (2) | 0.007 (2)  | −0.008 (2) |
| C6  | 0.035 (3)  | 0.053 (3)  | 0.044 (3)  | −0.019 (3) | 0.015 (2)  | −0.009 (2) |
| C7  | 0.044 (3)  | 0.050 (3)  | 0.039 (3)  | −0.012 (2) | 0.020 (2)  | 0.001 (2)  |
| C8  | 0.037 (3)  | 0.046 (3)  | 0.027 (2)  | −0.004 (2) | 0.014 (2)  | −0.003 (2) |
| C9  | 0.029 (2)  | 0.040 (3)  | 0.029 (2)  | −0.001 (2) | 0.0097 (19)| −0.002 (2) |
| C10 | 0.032 (2)  | 0.049 (3)  | 0.022 (2)  | 0.000 (2)  | 0.0062 (19)| −0.001 (2) |
| C11 | 0.031 (2)  | 0.042 (3)  | 0.032 (2)  | −0.002 (2) | 0.012 (2)  | 0.006 (2)  |
| C12 | 0.028 (2)  | 0.038 (2)  | 0.030 (2)  | −0.0012 (19)| 0.0099 (19)| 0.0026 (19)|
| C13 | 0.029 (2)  | 0.033 (2)  | 0.025 (2)  | 0.0011 (18)| 0.0104 (19)| −0.0022 (18)|
| C14 | 0.026 (2)  | 0.043 (3)  | 0.029 (2)  | −0.001 (2) | 0.0112 (19)| −0.005 (2) |
| C15 | 0.037 (3)  | 0.035 (3)  | 0.024 (2)  | −0.007 (2) | 0.011 (2)  | −0.0030 (19)|
| C16 | 0.033 (2)  | 0.043 (3)  | 0.023 (2)  | −0.006 (2) | 0.0047 (19)| −0.004 (2) |
| C17 | 0.038 (3)  | 0.047 (3)  | 0.026 (2)  | −0.005 (2) | 0.004 (2)  | −0.004 (2) |
| C18 | 0.053 (3)  | 0.073 (4)  | 0.019 (2)  | 0.000 (3)  | −0.001 (2) | −0.007 (2) |
| C19 | 0.041 (3)  | 0.048 (3)  | 0.028 (3)  | −0.008 (2) | 0.011 (2)  | −0.003 (2) |
| C20 | 0.051 (3)  | 0.060 (4)  | 0.045 (3)  | 0.010 (3)  | 0.021 (3)  | 0.002 (3)  |
| C21 | 0.049 (3)  | 0.069 (4)  | 0.050 (4)  | 0.009 (3)  | 0.014 (3)  | 0.009 (3)  |
| O4  | 0.100 (19)| 0.068 (6)  | 0.035 (2)  | 0.004 (14)| 0.015 (12)| −0.010 (3) |
| O5  | 0.031 (6)  | 0.118 (10)| 0.044 (3)  | 0.013 (5)  | 0.007 (4)  | 0.017 (5)  |
| N2  | 0.037 (10)| 0.044 (15)| 0.025 (7)  | 0.008 (10)| 0.003 (7)  | 0.018 (9)  |
| O4A | 0.100 (19)| 0.068 (6)  | 0.035 (2)  | 0.004 (14)| 0.015 (12)| −0.010 (3) |
| O5A | 0.031 (6)  | 0.118 (10)| 0.044 (3)  | 0.013 (5)  | 0.007 (4)  | 0.017 (5)  |
| N2A | 0.037 (10)| 0.044 (15)| 0.025 (7)  | 0.008 (10)| 0.003 (7)  | 0.018 (9)  |
| O6  | 0.065 (3)  | 0.058 (2)  | 0.031 (2)  | −0.017 (2) | 0.0094 (19)| 0.0037 (16)|
| O7  | 0.047 (2)  | 0.052 (2)  | 0.0246 (17)| 0.0098 (17)| 0.0108 (15)| 0.0032 (14)|
| O8  | 0.050 (2)  | 0.0442 (19)| 0.0293 (18)| −0.0018 (17)| 0.0132 (16)| −0.0044 (15)|
| N3  | 0.063 (3)  | 0.058 (3)  | 0.034 (2)  | −0.016 (2) | 0.008 (2)  | −0.003 (2) |
| N4  | 0.052 (3)  | 0.050 (3)  | 0.032 (2)  | −0.015 (2) | 0.003 (2)  | −0.006 (2) |
| C22 | 0.045 (3)  | 0.056 (3)  | 0.038 (3)  | 0.005 (3)  | 0.005 (2)  | 0.002 (3)  |
| C23 | 0.055 (4)  | 0.053 (3)  | 0.057 (4)  | 0.017 (3)  | 0.010 (3)  | 0.003 (3)  |
C24  0.064 (4)  0.045 (3)  0.060 (4)  0.014 (3)  0.024 (3)  −0.002 (3)
C25  0.061 (4)  0.039 (3)  0.045 (3)  −0.005 (3)  0.015 (3)  −0.008 (2)
C26  0.041 (3)  0.059 (3)  0.030 (3)  0.002 (3)  0.004 (2)  0.014 (2)
C27  0.047 (3)  0.085 (5)  0.058 (4)  0.024 (3)  0.016 (3)  0.016 (3)
C28  0.058 (4)  0.085 (5)  0.047 (3)  0.026 (3)  0.021 (3)  0.008 (3)
C29  0.044 (3)  0.067 (4)  0.032 (3)  0.007 (3)  0.014 (2)  0.007 (3)
C30  0.045 (3)  0.041 (3)  0.029 (3)  −0.004 (2)  0.010 (2)  0.000 (2)
C31  0.042 (3)  0.037 (2)  0.023 (2)  −0.008 (2)  0.007 (2)  −0.0019 (19)
C32  0.042 (3)  0.033 (2)  0.034 (3)  −0.005 (2)  0.012 (2)  −0.001 (2)
C33  0.041 (3)  0.038 (3)  0.030 (3)  −0.003 (2)  0.004 (2)  −0.003 (2)
C34  0.035 (3)  0.037 (2)  0.027 (2)  −0.006 (2)  0.007 (2)  0.0038 (19)
C35  0.035 (3)  0.040 (3)  0.028 (2)  −0.009 (2)  0.005 (2)  0.005 (2)
C36  0.048 (3)  0.041 (3)  0.025 (2)  0.004 (2)  0.006 (2)  0.006 (2)
C37  0.039 (3)  0.039 (3)  0.023 (2)  0.005 (2)  0.003 (2)  −0.0026 (19)
C38  0.043 (3)  0.043 (3)  0.032 (3)  −0.002 (2)  0.006 (2)  0.002 (2)
C39  0.052 (3)  0.056 (3)  0.028 (3)  −0.007 (3)  0.009 (2)  −0.002 (2)
C40  0.038 (3)  0.035 (3)  0.034 (3)  0.010 (2)  0.012 (2)  −0.002 (2)
C41  0.051 (3)  0.057 (3)  0.042 (3)  −0.004 (3)  0.024 (3)  −0.001 (2)
C42  0.050 (3)  0.066 (4)  0.054 (4)  −0.002 (3)  0.023 (3)  −0.010 (3)
O9   0.093 (9)  0.100 (9)  0.038 (4)  −0.031 (8)  0.032 (5)  −0.006 (4)
O10  0.065 (7)  0.104 (8)  0.044 (4)  −0.036 (7)  0.008 (5)  −0.009 (5)
O9A  0.100 (16) 0.045 (7)  0.032 (6)  0.009 (7)  0.023 (7)  0.007 (5)
O10A 0.048 (10) 0.047 (8)  0.048 (7)  −0.023 (7)  0.003 (7)  −0.014 (5)

Geometric parameters (Å, º)

O1—N1   1.428 (5)  O6—N3   1.418 (6)
O1—C17  1.338 (7)  O6—C38  1.349 (7)
O2—C19  1.199 (6)  O7—C40  1.219 (6)
O3—C19  1.324 (6)  O8—C40  1.319 (6)
O3—C20  1.455 (7)  O8—C41  1.470 (6)
N1—C15  1.318 (6)  N3—C36  1.301 (7)
C1—H1   0.9500  N4—C31  1.474 (6)
C1—C2   1.365 (7)  N4—O9   1.237 (9)
C1—C12  1.423 (7)  N4—O10  1.203 (11)
C2—H2   0.9500  N4—O9A  1.281 (13)
C2—C3   1.401 (8)  N4—O10A 1.206 (14)
C3—H3   0.9500  C22—H22 0.9500
C3—C4   1.366 (8)  C22—C23 1.352 (8)
C4—H4   0.9500  C22—C33 1.420 (8)
C4—C11  1.416 (7)  C23—H23 0.9500
C5—H5   0.86 (6)  C23—C24 1.406 (9)
C5—C6   1.343 (7)  C24—H24 0.9500
C5—C14  1.416 (7)  C24—C25 1.366 (9)
C6—H6   0.85 (6)  C25—H25 0.9500
C6—C7   1.415 (7)  C25—C32 1.403 (8)
C7—H7   0.9500  C26—H26 0.9500
C7—C8   1.367 (7)  C26—C27 1.346 (9)

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| Bond                | Length (Å) | Bond                | Length (Å) |
|---------------------|------------|---------------------|------------|
| C8—H8               | 0.9500     | C26—C35             | 1.425 (7)  |
| C8—C13              | 1.415 (6)  | C27—H27             | 0.9500     |
| C9—C12              | 1.408 (7)  | C27—C28             | 1.399 (9)  |
| C9—C13              | 1.399 (6)  | C28—H28             | 0.9500     |
| C9—C15              | 1.493 (6)  | C28—C29             | 1.343 (8)  |
| C10—C11             | 1.380 (7)  | C29—H29             | 0.9500     |
| C10—C14             | 1.407 (7)  | C29—C34             | 1.406 (7)  |
| C10—N2              | 1.43 (5)   | C30—C33             | 1.408 (8)  |
| C10—N2A             | 1.55 (6)   | C30—C34             | 1.416 (7)  |
| C11—C12             | 1.433 (6)  | C30—C36             | 1.498 (7)  |
| C13—C14             | 1.436 (6)  | C31—C32             | 1.413 (7)  |
| C15—C16             | 1.413 (7)  | C31—C35             | 1.388 (7)  |
| C16—C17             | 1.373 (7)  | C32—C33             | 1.438 (7)  |
| C16—C19             | 1.485 (7)  | C34—C35             | 1.433 (7)  |
| C17—C18             | 1.485 (7)  | C36—C37             | 1.415 (7)  |
| C18—H18A            | 0.9800     | C37—C38             | 1.362 (7)  |
| C18—H18B            | 0.9800     | C37—C40             | 1.465 (7)  |
| C18—H18C            | 0.9800     | C38—C39             | 1.474 (7)  |
| C20—H20A            | 0.9900     | C39—H39A            | 0.9800     |
| C20—H20B            | 0.9900     | C39—H39B            | 0.9800     |
| C20—C21             | 1.500 (8)  | C39—H39C            | 0.9800     |
| C21—H21A            | 0.9800     | C41—H41A            | 0.9900     |
| C21—H21B            | 0.9800     | C41—H41B            | 0.9900     |
| C21—H21C            | 0.9800     | C41—C42             | 1.488 (8)  |
| O4—N2               | 1.22 (6)   | C42—H42A            | 0.9800     |
| O5—N2               | 1.29 (4)   | C42—H42B            | 0.9800     |
| O4A—N2A             | 1.27 (9)   | C42—H42C            | 0.9800     |
| O5A—N2A             | 1.11 (4)   |                      |            |
| C17—O1—N1           | 109.5 (3)  | O5A—N2A—O4A        | 131 (6)    |
| C19—O3—C20          | 114.9 (4)  | C38—O6—N3          | 109.0 (4)  |
| C15—N1—O1           | 104.3 (4)  | C40—O8—C41        | 116.3 (4)  |
| C2—C1—H1            | 119.9      | C36—N3—O6          | 105.7 (4)  |
| C2—C1—C12           | 120.2 (5)  | O9—N4—C31         | 116.7 (5)  |
| C12—C1—H1           | 119.9      | O10—N4—C31        | 118.0 (6)  |
| C1—C2—H2            | 119.5      | O10—N4—O9         | 125.2 (7)  |
| C1—C2—C3            | 120.9 (5)  | O9A—N4—C31       | 118.6 (6)  |
| C3—C2—H2            | 119.5      | O10A—N4—C31      | 121.6 (8)  |
| C2—C3—H3            | 119.7      | O10A—N4—O9A     | 119.7 (9)  |
| C4—C3—C2            | 120.6 (5)  | C23—C22—H22      | 119.6      |
| C4—C3—H3            | 119.7      | C23—C22—C33     | 120.9 (5)  |
| C3—C4—H4            | 119.7      | C33—C22—H22     | 119.6      |
| C3—C4—C11           | 120.7 (5)  | C22—C23—H23     | 119.5      |
| C11—C4—H4           | 119.7      | C22—C23—C24    | 121.0 (5)  |
| C6—C5—H5            | 115 (4)    | C24—C23—H23   | 119.5      |
| C6—C5—C14           | 120.7 (5)  | C23—C24—H24    | 120.0      |
| C14—C5—H5           | 124 (4)    | C25—C24—C23  | 120.0 (6)  |
| C5—C6—H6            | 116 (4)    | C25—C24—H24  | 120.0      |
C5—C6—C7 121.2 (5) C24—C25—C32 121.1 (5)
C7—C6—H6 122 (4) C24—C25—H25 119.4
C6—C7—H7 120.2 C25—C26—C35 120.2 (5)
C8—C7—C6 119.6 (5) C25—C26—H26 119.9
C8—C7—H7 120.2 C27—C26—H26 119.9
C7—C8—C13 121.4 (4) C24—C25—H25 119.3
C13—C8—H8 119.3 C26—C27—C28 121.5 (5)
C12—C9—C15 118.5 (4) C28—C27—H27 119.3
C13—C9—C12 122.2 (4) C27—C28—H28 119.8
C13—C9—C15 119.3 (4) C29—C28—C27 120.4 (6)
C11—C10—C14 125.3 (4) C29—C28—H28 119.8
C11—C10—N2 121.3 (18) C28—C29—H29 119.6
C11—C10—N2A 115 (2) C28—C29—C34 120.9 (5)
C14—C10—N2 124.3 (4) C34—C29—H29 119.9
C14—C10—N2A 117.0 (4) C34—C29—C35 118.9 (5)
C10—C11—C12 117.0 (4) C35—C30—C33 125.5 (4)
C10—C11—C4 124.3 (4) C35—C30—C36 122.2 (5)
C10—C11—C12 117.8 (4) C30—C33—C32 118.9 (5)
C9—C12—C1 119.5 (4) C30—C33—C34 118.9 (5)
C9—C12—C11 121.6 (4) C30—C33—C35 118.9 (5)
C9—C12—C11 119.5 (4) C30—C33—C36 118.9 (5)
C8—C13—C14 123.2 (4) C31—C32—C33 119.5 (5)
C9—C13—C8 119.0 (4) C31—C32—C34 122.7 (4)
C9—C13—C14 119.3 (4) C31—C32—C35 122.7 (4)
C5—C14—C13 123.7 (4) C31—C32—C36 122.7 (4)
N1—C15—C9 119.8 (4) C31—C32—C37 122.7 (4)
N1—C15—C16 112.6 (4) C31—C32—C38 122.7 (4)
C16—C15—C9 127.6 (4) C31—C32—C39 122.7 (4)
C16—C15—C19 126.2 (4) C31—C32—C40 122.7 (4)
C17—C16—C15 104.2 (4) C31—C32—C41 122.7 (4)
C17—C16—C19 129.4 (4) C31—C32—C42 122.7 (4)
O1—C17—C16 109.5 (4) C31—C32—C43 122.7 (4)
O1—C17—C18 116.7 (4) C31—C32—C44 122.7 (4)
C16—C17—C18 133.8 (5) C31—C32—C45 122.7 (4)
C17—C18—H18A 109.5 C38—C37—C36 105.3 (5)
C17—C18—H18B 109.5 C38—C37—C37 129.0 (5)
C17—C18—H18C 109.5 O6—C38—C37 108.6 (4)
H18A—C18—H18B 109.5 O6—C38—C39 115.7 (4)
H18A—C18—H18C 109.5 C37—C38—C39 135.6 (5)
H18B—C18—H18C 109.5 C38—C39—H39A 109.5
O2—C19—O3 124.6 (5) C38—C39—H39B 109.5
O2—C19—C16 123.0 (5) C38—C39—H39C 109.5
O3—C19—C16 112.3 (4) H39A—C39—H39B 109.5
O3—C20—H20A 110.3 H39A—C39—H39C 109.5
O3—C20—H20B 110.3 H39B—C39—H39C 109.5
| Bond/Angle                  | Value   | Bond/Angle                  | Value   |
|---------------------------|---------|---------------------------|---------|
| O3—C20—C21               | 107.3 (5)| O7—C40—O8                | 124.2 (5)|
| H20A—C20—H20B            | 108.5   | O7—C40—C37               | 123.0 (5)|
| C21—C20—H20A             | 110.3   | O8—C40—C37               | 112.7 (4)|
| C21—C20—H20B             | 110.3   | O8—C41—H41A              | 110.0   |
| C20—C21—H21A             | 109.5   | O8—C41—H41B              | 110.0   |
| C20—C21—H21B             | 109.5   | O8—C41—C42               | 108.4 (5)|
| C20—C21—H21C             | 109.5   | H41A—C41—H41B            | 108.4   |
| H21A—C21—H21B            | 109.5   | H42A—C42—H42B            | 109.5   |
| H21A—C21—H21C            | 109.5   | H42A—C42—H42C            | 109.5   |
| O4—N2—C10                | 123 (3) | C41—C42—H42B             | 109.5   |
| O4—N2—O5                 | 122 (4) | C41—C42—H42C             | 109.5   |
| O5—N2—C10                | 116 (3) | C41—C42—C42              | 109.5   |
| O4A—N2A—C10              | 108 (3) | C42—C41—C42              | 109.5   |
| O5A—N2A—C10              | 121 (5) | C42—C41—H41A             | 110.0   |

| Bond/Angle                  | Value   | Bond/Angle                  | Value   |
|---------------------------|---------|---------------------------|---------|
| O4—N2—C15—C9             | −179.2 (4)| N2A—C10—C14—C5           | 9 (3)   |
| N1—O1—C15—C16            | 0.1 (5) | N2A—C10—C14—C13          | −172 (2)|
| N1—O1—C17—C16            | −0.1 (6) | O6—N3—C36—C30            | −179.1 (5)|
| N1—O1—C17—C18            | −179.4 (5)| O6—N3—C36—C37            | 1.3 (6) |
| N1—C15—C16—C17           | −0.1 (6) | N3—O6—C38—C37            | −0.5 (6)|
| N1—C15—C16—C19           | −176.0 (5)| N3—O6—C38—C39            | 176.6 (5) |
| C1—C2—C3—C4              | −0.7 (9) | N3—C36—C37—C38           | −1.6 (6)|
| C2—C1—C12—C9             | 179.1 (5) | N3—C36—C37—C40           | 178.6 (5) |
| C2—C1—C12—C11            | 0.6 (7) | N4—C31—C32—C25           | 0.1 (7) |
| C2—C3—C4—C11             | 0.0 (9) | N4—C31—C32—C33           | 179.7 (4) |
| C3—C4—C11—C10            | −176.7 (5)| N4—C31—C35—C26           | 1.3 (7) |
| C3—C4—C11—C12            | 1.0 (8) | N4—C31—C35—C34           | 179.0 (4) |
| C4—C11—C12—C1             | −1.2 (7) | N4—C31—C35—C34           | 179.0 (4) |
| C4—C11—C12—C9             | −179.8 (5)| N4—C31—C35—C34           | 179.0 (4) |
| C5—C6—C7—C8              | −0.6 (8) | C22—C23—C24—C25          | 0.4 (10)|
| C6—C5—C14—C10            | 178.2 (5)| C22—C23—C33—C30          | 177.8 (6) |
| C6—C5—C14—C13            | −1.0 (7) | C23—C22—C33—C32          | −1.1 (8)|
| C6—C7—C8—C13             | −0.2 (8) | C23—C22—C33—C32          | −0.6 (9)|
| C7—C8—C13—C9             | −176.8 (5)| C24—C25—C32—C31          | 179.5 (5) |
| C7—C8—C13—C14            | 0.3 (7) | C25—C25—C32—C33          | −0.1 (8)|
| C8—C13—C14—C5            | 0.2 (6) | C25—C32—C33—C22          | 0.9 (7) |
| C8—C13—C14—C10           | −179.0 (4)| C25—C32—C33—C30          | −178.0 (5)|
| C9—C13—C14—C5            | 177.5 (4) | C26—C27—C28—C29          | −0.1 (11)|
| C9—C13—C14—C10           | −1.7 (6) | C27—C26—C35—C31          | 179.3 (5) |
| C9—C15—C16—C17           | 179.1 (5)| C27—C26—C35—C34          | 1.6 (8) |
| C9—C15—C16—C19           | 3.2 (8) | C27—C26—C35—C34          | 1.6 (10)|
| C10—C11—C12—C1            | 176.6 (5)| C28—C29—C34—C30          | −179.5 (5)|
| C10—C11—C12—C9            | −2.0 (7) | C28—C29—C34—C30          | −1.5 (8)|
| C11—C10—C14—C5           | −178.2 (5)| C30—C34—C35—C26          | −0.1 (7)|
| C11—C10—C14—C13          | 1.0 (7) | C30—C34—C35—C31          | −177.9 (4)|
| C11—C10—N2—O4            | 77 (4)  | C30—C34—C35—C31          | 178.0 (4) |
| C11—C10—N2—O5            | −101 (3)| C30—C36—C37—C38          | 178.9 (5)|

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C11—C10—N2A—O4A 115 (4) C31—C32—C33—C22 −178.7 (5)
C11—C10—N2A—O5A −64 (6) C31—C32—C33—C30 2.3 (7)
C12—C1—C2—C3 0.4 (9) C32—C31—C35—C22 −176.7 (5)
C12—C9—C13—C8 177.7 (4) C32—C31—C35—C34 0.9 (7)
C12—C9—C13—C14 0.7 (7) C33—C22—C23—C24 0.5 (10)
C12—C9—C15—N1 91.6 (6) C33—C30—C34—C29 178.1 (5)
C12—C9—C15—C16 −87.6 (6) C33—C30—C34—C35 0.1 (7)
C13—C9—C12—C1 −177.3 (5) C33—C30—C36—N3 95.1 (7)
C13—C9—C12—C11 1.3 (7) C33—C30—C36—C37 −85.4 (7)
C13—C9—C15—N1 −88.6 (6) C34—C30—C33—C25 178.2 (5)
C13—C9—C15—C16 92.3 (6) C34—C30—C33—C32 179.7 (5)
C14—C5—C6—C7 1.2 (8) C34—C30—C33—C32 −1.4 (8)
C14—C10—C11—C4 178.5 (5) C34—C30—C33—C22 177.5 (4)
C14—C10—C11—C12 0.8 (7) C34—C30—C33—C22 −177.5 (4)
C14—C10—N2—O4 −97 (4) C34—C30—C33—C22 176.3 (5)
C14—C10—N2—O5 85 (3) C34—C30—C36—N3 −85.9 (6)
C14—C10—N2A—O4A −71 (5) C34—C30—C36—C37 93.6 (7)
C14—C10—N2A—O5A 110 (5)
C15—C9—C12—C1 2.6 (7) C35—C26—C27—C28 −1.5 (10)
C15—C9—C12—C11 −178.9 (4) C35—C30—C33—C22 −1.4 (8)
C15—C9—C13—C8 −2.1 (7) C35—C30—C33—C32 179.7 (5)
C15—C9—C13—C14 −179.2 (4) C35—C30—C33—C32 −178.8 (4)
C15—C9—C15—N1 −88.6 (6) C35—C30—C34—C29 −0.8 (7)
C15—C9—C15—C16 92.3 (6) C35—C30—C34—C35 −178.8 (4)
C15—C16—C17—O1 0.1 (5) C36—C37—C38—O6 1.2 (6)
C15—C16—C17—C18 179.3 (6) C36—C37—C38—C39 −175.0 (6)
C15—C16—C17—C19 2.6 (7) C36—C37—C40—O7 −4.6 (7)
C16—C17—C19—O2 −1.2 (7) C36—C37—C40—O8 176.0 (5)
C16—C17—C19—O3 179.8 (5) C36—C37—C40—O8 −179.0 (5)
C17—O1—N1—C15 0.0 (5) C36—C37—C40—O7 176.3 (5)
C17—O1—N1—C15 −175.8 (5) C37—C38—C39—C40 2.6 (7)
C19—O3—C20—C21 −174.3 (5) C38—O6—N3—C36 −0.5 (6)
C19—O3—C20—C21 −179.4 (5) C38—O6—N3—C36 175.1 (5)
C20—O3—C19—O2 0.5 (7) C38—O6—N3—C36 −179.0 (5)
C20—O3—C19—O2 −175.8 (5) C38—O6—N3—C36 176.3 (5)
C3—H3···O7a 0.95 2.44 3.339 (6) 158

Hydrogen-bond geometry (Å, º)

| D—H···A | D—H  | H···A  | D···A     | D—H···A |
|---------|------|-------|-----------|---------|
| C1—H1···O5i | 0.95 | 2.46  | 3.366 (12) | 159     |
| C3—H3···O7a  | 0.95 | 2.44  | 3.339 (6)  | 158     |
C7—H7···O4\textsuperscript{iii} 0.95 2.40 3.24 (4) 147
C7—H7···O4\textsuperscript{A}iii 0.95 2.46 3.34 (6) 154

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

Ethyl 3-(9-hydroxy-10-oxo-9,10-dihydroanthracen-9-yl)-5-methylisoxazole-4-carboxylate (V)

Crystal data

C\textsubscript{21}H\textsubscript{17}NO\textsubscript{5}  
Mr = 363.36  
Monoclinic, \textit{P}2\textsubscript{1}/\textit{c}  
a = 8.2862 (4) Å  
b = 23.5895 (11) Å  
c = 8.6219 (4) Å  
β = 97.728 (2)°  
V = 1669.99 (14) Å\textsuperscript{3}  
Z = 4  
F(000) = 760  
D\textsubscript{x} = 1.445 Mg m\textsuperscript{−3}  
Mo \textit{Kα} radiation, λ = 0.71073 Å  
Cell parameters from 9893 reflections  
θ = 2.5–28.3°  
µ = 0.10 mm\textsuperscript{−1}  
T = 100 K  
Prism, yellow  
0.28 × 0.20 × 0.19 mm

Data collection

Bruker SMART Breeze CCD diffractometer  
Radiation source: 2 kW sealed X-ray tube  
φ and ω scans  
Absorption correction: numerical  
(SADABS; Krause \textit{et al.}, 2015)  
T\textsubscript{min} = 0.945, T\textsubscript{max} = 1.000  
44252 measured reflections  
4112 independent reflections  
3252 reflections with I > 2σ(I)  
R\textsubscript{int} = 0.051  
θ\textsubscript{max} = 28.3°, θ\textsubscript{min} = 1.7°  
h = −10→11  
k = −31→31  
l = −11→11

Refinement

Refinement on F\textsuperscript{2}  
Least-squares matrix: full  
R[F\textsuperscript{2} > 2σ(F\textsuperscript{2})] = 0.051  
wR(F\textsuperscript{2}) = 0.114  
S = 1.13  
4112 reflections  
250 parameters  
0 restraints

Primary atom site location: structure-invariant direct methods  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
\(w = 1/[σ(\textit{F}_\text{o}^2) + (0.0241P)^2 + 1.7289P]\)  
where P = (\textit{F}_\text{c}^2 + 2\textit{F}_\text{c}^\text{2})/3  
\((Δρ)_{\text{max}} < 0.001  
Δρ_{\text{max}} = 0.37 \text{ e Å}^{-3}  
Δρ_{\text{min}} = −0.21 \text{ e Å}^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å\textsuperscript{2})

\begin{tabular}{cccccc}
O1 & 0.65863 (16) & 0.55415 (6) & 0.82793 (16) & 0.0252 (3) 
O2 & 0.18140 (16) & 0.55228 (6) & 1.16490 (15) & 0.0203 (3) 
O3 & 0.44673 (16) & 0.68686 (5) & 0.90343 (16) & 0.0217 (3) 
O4 & 0.37570 (18) & 0.77723 (6) & 0.93929 (18) & 0.0306 (3) 
O5 & 0.03664 (16) & 0.70944 (5) & 1.19895 (16) & 0.0226 (3) 
\end{tabular}
Atomic displacement parameters (Å²)

|   | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|----------|----------|----------|----------|----------|----------|
| O1| 0.0224 (7)| 0.0278 (7)| 0.0271 (7)| 0.0023 (6)| 0.0097 (6)| 0.0023 (6) |
| O2| 0.0205 (7)| 0.0183 (6)| 0.0235 (7)| −0.0023 (5)| 0.0081 (5)| 0.0017 (5) |
| O3| 0.0199 (7)| 0.0209 (6)| 0.0256 (7)| −0.0025 (5)| 0.0070 (5)| 0.0004 (5) |
| O4| 0.0339 (8)| 0.0194 (7)| 0.0397 (9)| −0.0005 (6)| 0.0090 (7)| 0.0073 (6) |
| O5| 0.0204 (7)| 0.0214 (7)| 0.0264 (7)| 0.0033 (5)| 0.0049 (5)| −0.0018 (5) |
### Geometric parameters (Å, °)

|     |    |    |    |    |    |
|-----|----|----|----|----|----|
| O1—C10 | 1.231 (2) | C7—C8 | 1.383 (3) |
| O2—C9  | 1.436 (2) | C8—H8 | 0.9500 |
| O2—H2  | 0.91 (3)  | C9—C12 | 1.392 (3) |
| O3—C19 | 1.336 (2) | C9—C13 | 1.520 (2) |
| O3—C20 | 1.458 (2) | C9—C15 | 1.528 (2) |
| O4—C19 | 1.208 (2) | C10—C11 | 1.474 (3) |
| O5—N1  | 1.418 (2) | C10—C14 | 1.483 (3) |
| O5—C17 | 1.336 (2) | C10—C14 | 1.483 (3) |
| N1—C15 | 1.304 (2) | C11—C12 | 1.397 (3) |
| C1—H1  | 0.9500  | C13—C14 | 1.393 (3) |
| C1—C2  | 1.384 (3) | C15—C16 | 1.439 (2) |
| C1—C13 | 1.392 (3) | C16—C17 | 1.365 (3) |
| C2—H2A | 0.9500  | C16—C19 | 1.466 (3) |
| C2—C3  | 1.391 (3) | C17—C18 | 1.483 (3) |
| C3—H3  | 0.9500  | C18—H18A | 0.9800 |
| C3—C4  | 1.380 (3) | C18—H18B | 0.9800 |
| C4—H4  | 0.9500  | C18—H18C | 0.9800 |
| C4—C14 | 1.397 (3) | C20—H20A | 0.9900 |
| C5—H5  | 0.9500  | C20—H20B | 0.9900 |
| C5—C6  | 1.377 (3) | C20—C21 | 1.501 (3) |
| C5—C11 | 1.401 (3) | C21—H21A | 0.9800 |
| C6—H6  | 0.9500  | C21—H21B | 0.9800 |
| C6—C7  | 1.387 (3) | C21—H21C | 0.9800 |
C7—H7  0.9500

C9—O2—H2  106.1 (16)  C8—C12—C9  118.00 (16)
C19—O3—C20  115.78 (15)  C8—C12—C11  119.62 (17)
C17—O5—N1  109.24 (14)  C11—C12—C9  122.37 (16)
C15—N1—O5  105.61 (14)  C1—C13—C9  118.24 (16)
C2—C1—H1  119.7  C4—C14—C10  119.11 (17)
C2—C1—C13  120.57 (18)  C4—C14—C11  122.62 (16)
C13—C1—H1  119.7  C1—C13—C14  119.05 (17)
C1—C2—H2A  119.8  C1—C13—C9  119.83 (17)
C1—C2—C3  120.38 (18)  C13—C14—C10  121.11 (16)
C3—C2—H2A  119.8  C1—C13—C14  117.35 (15)
C1—C2—C3  120.4  C1—C13—C16  111.38 (16)
C2—C3—H3  120.4  C16—C15—C9  131.27 (16)
C2—C3—C4  119.6  C16—C15—C16  113.44 (15)
C3—C4—H4  120.4  C19—C16—C17  116.12 (17)
C3—C4—C14  120.74 (18)  C19—C16—C19  119.86 (16)
C14—C4—H4  119.6  C17—C16—C17  116.12 (17)
C6—C5—H5  119.8  C17—C16—C18  119.86 (16)
C6—C5—C11  120.49 (18)  C17—C16—C18  116.12 (17)
C11—C5—H5  119.8  C17—C16—C19  119.86 (16)
C5—C6—H6  120.1  C17—C16—C19  119.86 (16)
C5—C6—C7  119.80 (18)  C17—C16—C19  119.86 (16)
C7—C6—H6  120.1  C17—C18—C19  113.44 (15)
C6—C7—C8  120.49 (18)  C17—C18—C20  123.74 (18)
C7—C6—H6  120.1  C17—C18—C21  123.74 (18)
C8—C7—C6  119.8  C17—C18—C20  123.74 (18)
C8—C7—H7  119.8  C17—C18—C21  123.74 (18)
C7—C8—H8  119.9  C17—C18—C20  123.74 (18)
C7—C8—C12  120.15 (18)  C17—C18—C21  123.74 (18)
C12—C8—H8  119.9  O4—C19—C16  113.44 (15)
O2—C9—C12  109.31 (14)  C9—C15—C16  −179.04 (17)
O2—C9—C13  109.03 (14)  C9—C15—C17  −3.3 (3)
O2—C9—C15  104.91 (14)  C10—C11—C12  −177.95 (16)
C12—C9—C15  108.81 (14)  C10—C11—C5  2.8 (3)
C13—C9—C12  114.27 (15)  C10—C11—C14  −177.95 (16)
C13—C9—C15  110.09 (14)  C10—C11—C10  2.8 (3)
O1—C10—C11  121.06 (17)  C9—C15—C16  −179.04 (17)
O1—C10—C14  120.73 (17)  C9—C15—C17  −3.3 (3)
C11—C10—C14  118.21 (16)  C10—C11—C12  −177.95 (16)
C5—C11—C10  119.19 (17)  C10—C11—C12  −177.95 (16)
C12—C11—C5  119.44 (17)  C10—C11—C9  2.8 (3)
C12—C11—C10  121.35 (16)  C11—C5—C6—C7  0.5 (3)
O2—C9—C12—C11 121.43 (17)
O2—C9—C13—C1 54.2 (2)
O2—C9—C13—C14 −123.89 (17)
O2—C9—C15—N1 1.7 (2)
O2—C9—C15—C16 −179.40 (17)
O5—N1—C15—C9 179.43 (14)
O5—N1—C15—C16 0.30 (19)
N1—O5—C17—C16 0.41 (19)
N1—O5—C17—C18 −179.49 (15)
N1—C15—C16—C17 −0.1 (2)
N1—C15—C16—C19 175.66 (19)
C1—C2—C3—C4 2.3 (3)
C1—C13—C14—C4 2.6 (3)
C1—C13—C14—C10 −176.28 (16)
C2—C1—C13—C9 179.77 (16)
C2—C1—C13—C14 −2.1 (3)
C2—C3—C4—C14 −1.8 (3)
C3—C4—C14—C10 178.25 (17)
C3—C4—C14—C13 −0.6 (3)
C5—C6—C7—C8 0.2 (3)
C5—C11—C12—C8 0.1 (3)
C5—C11—C12—C9 −179.15 (16)
C6—C5—C11—C12 −0.3 (3)
C6—C5—C11—C10 177.76 (17)
C6—C5—C11—C12 −0.3 (3)
C6—C7—C8—C12 0.2 (3)
C7—C8—C12—C9 179.26 (17)
C7—C8—C12—C11 0.0 (3)
C9—C13—C14—C4 −179.41 (16)
C9—C13—C14—C10 1.7 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O2—H2···O1i | 0.91 (3) | 1.93 (3) | 2.8359 (19) | 176 (2) |

Symmetry code: (i) −x+1, −y+1, −z+2.