Crystal structures of two dioxomolybdenum complexes stabilized by salan ligands featuring phenyl and cyclohexyl backbones

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Two cis-dioxomolybdenum complexes based on salan ligands with different backbones are reported. The first complex, dioxido[2,2'-][1,2-phenylenebis(iminomethylene)]bis(phenolato)molybdenum(VI) dimethylformamide disolvate, [Mo(C20H18N2O2)O2]C12C3H7NO (PhLMoO2, 1b), features a phenyl backbone, while the second complex, (6,6'-[[cyclohexane-1,2-diyl]bis(azane-diyl)]bis(2,4-di-tert-butylphenolato))dioxidomolybdenum(VI) methanol disolvate, [Mo(C36H56N2O2)O2]C12CH3OH (CyLMoO2, 2b), is based on a cyclohexyl backbone. These complexes crystallized as solvated species, 1b·2DMF and 2b·2MeOH. The salan ligands PhLH2 (1a) and CyLH2 (2a) coordinate to the molybdenum center in these complexes 1b and 2b in a κ²Nκ²O fashion, forming a distorted octahedral geometry. The Mo—N and Mo—O distances are 2.3475 (16) and 1.9567 (16) Å, respectively, in 1b while the corresponding measurements are Mo—N = 2.3412 (12) Å, and Mo—O = 1.9428 (10) Å for 2b. A key geometrical feature is that the N—Mo—N angle of 72.40 (4)° in CyLMoO2 is slightly less than that of the PhLMoO2 angle of 75.18 (6)°, which is attributed to the flexibility of the cyclohexane ring between the nitrogen as compared to the rigid phenyl ring in the PhLMoO2.

1. Chemical context

Molybdenum centers are present in the active sites of various enzymes including nitrogenases, sulfite oxidase, xanthine oxidase, and DMSO reductase that catalyze two-electron redox processes (Hille et al., 2014; Enemark et al., 2004; Hille, 1996). This is attributed to the large number of stable oxidation states and coordination environments that can be achieved, as well as the solubility of molybdate salts in water. A majority of these enzymes are referred to as oxo-molybdenum enzymes due to the presence of at least one Mo=O moiety in the active site. The sulfite oxidase family of enzymes contains a cis-dioxo molybdenum(VI) (L₆MoO₂) center in its active site (Hille et al., 2014). Apart from being studied as models to understand biological systems, oxomolybdenum complexes have also found utility in processes such as olefin metathesis, olefin epoxidation, cytotoxic studies, and cyclic ester polymerizations (Hossain et al. 2020; Mayilmurugan et al. 2013; Yang et al. 2007). Mononuclear molybdenum complexes are generally distinguished by stretching frequencies [υ(O=Mo=O)] in the 910–950 cm⁻¹ and 890–925 cm⁻¹ regions, which are characteristic of a cis-MoO₂ fragment (Chakravarthy & Chand, 2011). A variety of ligand architectures have been successful in stabilizing the oxomolyb-
denum core in these complexes (Ziegler et al. 2009; Subramanian et al. 1984; Rajan et al. 1983). Dioxomolybdenum complexes stabilized by salan ligands have been used extensively for various applications (Roy et al., 2017; Whiteoak et al., 2009). The modular nature for the synthesis of salan ligands allows for incorporation of steric and electronic variations in the ligand framework to tune the reactivity of the molybdenum center. We are exploring the utility of dioxomolybdenum complexes in catalyzing the deoxydehydration (DODH) reaction with a focus on understanding ligand effects on catalytic activity. This work reports synthesis and crystal structures of two molybdenum complexes including a crystallographically uncharacterized complex, dioxido[2,2'-[1,2-phenylenebis(iminomethylene)bis(phenolato)]molybdenum(VI), PbLMoO2 (1b) (Rajan et al., 1983). The second is a known complex with a new unit cell, (Ziegler et al., 2009), 6,6'[[[(cyclohexane-1,2-diyli)bis(azanediyl)]bis(methylene)]bis(2,4-di-tert-butylphenolato)]dioxidomolybdenum(VI), CyLMoO2 (2b).

2. Structural commentary

The asymmetric unit of PbLMoO2 (1b) contains two molecules of PbLMoO2 and four molecules of dimethylformamide (DMF), as shown in Fig. 1. Fig. 2 shows one molecule of PbLMoO2 with hydrogen atoms and solvent removed for clarity. In this system, the salan ligand PbLH2 (1a) coordinates to the molybdenum center in a κ²N,κ²O fashion, forming a distorted octahedral geometry. The angles formed around the molybdenum core are 80.23 (6)° for O1—Mo01—N1, 157.78 (6)° for O1—Mo01—O2, 75.18 (6)° for N1—Mo01—N2, and 109.80 (7)° for O3—Mo01—O4. These angles are consistent with a system that is significantly distorted from octahedral geometry with bond angles resulting from the salan ligand ranging from 75.18 (6)° to 84.38 (7)°, while the angle between the ‘oxo’ oxygens of 109.80 (7)° is close to the ideal tetrahedral angle of 109.5°. Analogous bond angles in the second molecule in the unit cell are the same within 0.01 Å.

The bond distances between the molybdenum center and ligand atoms for Mo01—N1 and Mo01—O1 are 2.3475 (16) Å and 1.9567 (16) Å, respectively. The notable bond distances from the salan ligand ranging from 75.18 (6)° to 84.38 (7)°, while the angle between the ‘oxo’ oxygens of 109.80 (7)° is close to the ideal tetrahedral angle of 109.5°. Analogous bond angles in the second molecule in the unit cell are the same within 0.01 Å as distances for O1—C1 and N1—C8, respectively. The other bond distances have variations of 0.2–0.3 Å, with N3—C27 at 1.519 (3) Å, C26—C27 at 1.490 (3) Å, and C28—C33 at 1.392 (3) Å.

The asymmetric unit of CyLMoO2 (2b) contains one molecule of CyLMoO2 and two molecules of methanol (MeOH) (Fig. 3). The salan ligand CyLH2 (2a) binds in the same κ²N,κ²O fashion that complex 1b does. Fig. 4 shows CyLMoO2 with the hydrogen atoms removed for clarity. The complex also has a distorted octahedral geometry with angles of O3—Mo01—O1 at 96.36 (5)°, O1—Mo01—N1 at 76.73 (4)°, N1—Mo01—N2 at 72.40 (4)°, O2—Mo01—O3 at 72.40 (4)°, N2—Mo01—O2 at 78.91 (4)°, O2—Mo01—O4 at 100.19 (5)°, O2—Mo01—O3 at 94.58 (5)°. These...
angles are between 5 and 10° of the ideal 90° for octahedral geometry. The N1—Mo01—N2 angle at 72.40 (4)° is slightly less than that of the PhLMoO2 angle of 75.81 (6)°, which is attributed to the flexibility of the cyclohexane ring between the nitrogen atoms compared to the rigid phenyl ring in the PhLMoO2. Metal–ligand bond distances are found for Mo01—O1 at 1.9428 (10) Å, Mo01—O2 at 1.9484 (10) Å, Mo01—O3 at 1.7125 (10) Å, Mo01—O4 at 1.7226 (11) Å, Mo01—N1 at 2.3412 (12) Å, and Mo01—N2 at 2.3384 (12) Å. Other ligand distances and bond lengths within the phenyl rings are consistent with analogous distances in PhLMoO2 (1b). The cyclohexane bond distances are consistent with single C—C bonds. The bond lengths observed are not statistically different than those reported by Ziegler et al. (2009). There are a few statistically different angles, specifically around the molybdenum center where Table 1 shows the correlating bond angles. These bond-angle differences are most likely due to improved R1 of 2.78% as compared to the previously reported R1 of 5.5% and higher solvent disorder in the reported structure.

3. Supramolecular features

PhLMoO2 (1b): A single molecule of PhLMoO2 is hydrogen bonded to one disordered DMF molecule, as shown in Fig. 5, with a distance of 2.03 Å for O11⋯H008 (Table 2). A second hydrogen bond interaction is between O9—H00D with a distance of 2.16 (3) Å. Corresponding hydrogen bond distances in the second molecule in the unit cell are similar. There are three formula units within the contents of the unit cell. Perpendicular π-stacking between PhLMoO2 molecules is observed between C5 and the aryl ring centroid (C35–C39) with a distance of 4.597 Å.

CyLMoO2 (2b): There are four molecules of CyLMoO2 in the unit cell of this system and the complex is stabilized via hydrogen bonding to the solvent MeOH molecule (1.94 Å for O4⋯H5A and 2.00 Å for O5⋯H2; Table 3), as seen in Fig. 6. There is no indication that there are π-stacking interactions between the two molecules. In comparing the hydrogen bonding interactions in the two complexes, PhLMoO2 demonstrates a tighter packing arrangement due to the rigid phenyl ring compared to the flexible cyclohexane ring in CyLMoO2.
bonding with the previously reported structure, the main difference is the formation of hydrogen-bonded tetramers containing two molecules of 2b and two molecules of methanol in the current structure. The previously reported structure had one resolved molecule of methanol and one disordered oxygen atom, which form a hydrogen-bonded trimer with one molecule of \(^{31}\)LMoO\(_2\) (Ziegler et al., 2009).

4. Database survey

A database search of the Cambridge Structural Database (CSD; Groom et al., 2016) (webCSD accessed September 22, 2021) and SciFinder (SciFinder, 2021) did not yield any exact matches to the crystal structure for \(^{31}\)LMoO\(_2\) (1b). There was a similar crystal structure found with the imine form of the ligand (Salen)MoO\(_2\). A search for \(^{31}\)LMoO\(_2\) (2b) in the CSD (webCSD accessed September 22, 2021) shows that there is a known structure of the molecule with a different unit cell with accession code HUWGOW (Ziegler et al., 2009). The SciFinder search resulted in the same sources being found. The current structure for \(^{31}\)LMoO\(_2\) (2b) was solved in space group \(P\frac{2_1}{n}\) compared with \(P\frac{3_1}{1}\) for HUWGOW. The primary additional differences in the structures is an improved R1 of 2.78% and more clearly resolved methanol solvent, as compared to the previously reported R1 of 5.5% and more disordered methanol solvent (Ziegler et al., 2009).

5. Synthesis and crystallization

The salan ligands used for stabilizing [MoO\(_2\)]\(^{2+}\) in the complexes \(^{31}\)LMoO\(_2\) (1b) (Rajan et al., 1983) and \(^{31}\)LMoO\(_2\) (2b) (Ziegler et al., 2009) were synthesized by the reductive amination of the corresponding salicylaldehyde and diamine. The ligands \(^{31}\)LH\(_2\) (1a) and \(^{31}\)LH\(_2\) (2a) were synthesized as off-white solids in 86% and 58% yields, respectively. The reaction scheme is shown in Fig. 7. Both ligands were successfully characterized by NMR and IR spectroscopy. A salient feature in the \(^1\)H NMR spectra of both ligands as compared to the precursor salen compounds was the disappearance of the aldimine peak (~8.50 ppm) and the appearance of the benzonic resonances ~4.00 ppm. The molybdenum complexes \(^{31}\)LMoO\(_2\) (1b) and \(^{31}\)LMoO\(_2\) (2b) were synthesized in 86% and 42% yields, respectively, by the reaction of the corresponding ligands with MoO\(_2\)(acac)\(_2\) in methanol or acetonitrile as solvent. Complexes 1b and 2b were also characterized by NMR and IR spectroscopy. Both complexes exhibited stretches [[(Mo=O) = 916 and 876 cm\(^{-1}\) (1b); 903 and 875 cm\(^{-1}\) (2b)] characteristic of a cis-dioxo molybdenum core in the IR spectrum.

**Procedure for synthesis of ligands**

\(^{31}\)LH\(_2\) (1a): To a solution of 1,2-phenylenediamine (0.764 g, 7.20 mmol) in methanol (ca 7 ml) was added a solution of salicylaldehyde (1.76 ml, 14.9 mmol) in methanol (ca 8 ml). The mixture was stirred for 6 h at room temperature. The orange precipitate that formed during this period was filtered and washed with methanol, then dried under high vacuum to yield the salophen product as an orange solid (2.19 g, 98%).\(^1\)H NMR (CDCl\(_3\), 400 MHz, 300 K) \(\delta\) 13.0 (s, 2H), 8.63 (s, 2H), 7.38 (d, \(^3\)J\(_{HH}\) = 8 Hz, 2H), 7.35–7.33 (m, 2H), 7.26–7.22 (m, 2H), 7.05 (d, \(^3\)J\(_{HH}\) = 8 Hz, 2H), 6.92 (t, \(^3\)J\(_{HH}\) = 8 Hz, 2H).

To a mixture of methanol (ca. 8 ml) and diethyl ether (ca 8 ml), was added salophen (1.52 g, 4.81 mmol) followed by NaBH\(_4\) (1.67 g, 44.4 mmol), and the reaction mixture was stirred at room temperature for 1 h. When the yellow color of the solution changed to colorless, it was transferred into a separatory funnel and DI H\(_2\)O (ca 15 ml) was added followed by ethyl acetate (2 \(\times\) ca 15 ml) for extraction. The organic solution was separated and combined, then washed with saturated NaCl solution (ca 20 ml). The organic layer was dried over anhydrous Na\(_2\)SO\(_4\) and filtered. The filtrate was concentrated under vacuum to give a light-yellow solid, which was dried under high vacuum. The color of the solid changed

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**Table 2**

| D—H ··· A | D—H | H···A | D···A | D—H ··· A |
|-----------|-----|------|------|-----------|
| N2—H008···O11 | 1.00 | 2.03 | 2.958 (2) | 154 |
| N4—H009···O10 | 1.00 | 1.99 | 2.924 (3) | 154 |
| N1—H00D···O12 | 0.85 (3) | 2.15 (3) | 2.949 (3) | 157 (2) |
| N3—H00E···O9 | 0.79 (3) | 2.16 (3) | 2.885 (3) | 154 (3) |

Symmetry code: (i) \(-x+1, -y+1, -z+1\).

**Table 3**

| D—H ··· A | D—H | H···A | D···A | D—H ··· A |
|-----------|-----|------|------|-----------|
| N2—H2···O5' | 1.00 | 2.00 | 2.9319 (16) | 153 |
| O5—H5A···O4 | 0.84 | 1.94 | 2.7837 (16) | 177 |

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**Figure 6**

View of four molecules of \(^{31}\)LMoO\(_2\) and six molecules of methanol in the unit cell with 50% probability ellipsoids, highlighting intermolecular distances. Distances between H atoms are listed without standard deviations because the H atoms were positionally fixed.
to light brown after 2 h under high vacuum to yield the product (1.32 g, 86%).\(^1\)H NMR (CDCl\(_3\), 400 MHz, 301 K) \(\delta\) 7.24–7.19 (m, 4H), 6.96–6.94 (m, 4H), 6.89 (t, \(J_{HH} = 8\) Hz, 2H), 6.86 (t, \(J_{HH} = 8\) Hz, 2H), 4.40 (s, 4H).

**\(\text{CyLH}_2\) (2a):** A 100 mL round-bottom flask was charged with trans-1,2-diaminocyclohexane (0.448 g, 4.38 mmol), methanol (ca. 16 mL), and 3,5-di-tert-butylsalicylaldehyde (2.05 g, 17.5 mmol). The solution was stirred for 24 h at room temperature. The solution resulted in a bright-yellow precipitate. The precipitate was dried under high vacuum to remove any residual solvent and yield the salen product (3.85 g, 81%).\(^1\)H NMR (CDCl\(_3\), 400 MHz, 301 K) \(\delta\) 17.5 mmol). NaBH\(_4\) (9 equivalents) was slowly added into the reaction mixture until the solution was colorless. The reaction mixture was then stirred for 10 min. The yellow precipitate that formed was filtered and then dried under vacuum to yield the complex as yellow solid (1.24 g, 86%).\(^1\)H NMR (DMSO-\(d_6\), 400 MHz, 301 K) \(\delta\) 7.55 (d, \(J_{HH} = 8\) Hz, 1H), 7.37–7.35 (m, 1H), 7.19–7.10 (m, 4H), 7.07–7.05 (m, 1H), 7.02–6.98 (m, 2H), 6.91 (d, \(J_{HH} = 8\) Hz, 1H), 6.85–6.83 (m, 1H), 6.80 (d, \(J_{HH} = 8\) Hz, 1H), 6.76–6.68 (m, 2H), 6.63 (d, \(J_{HH} = 8\) Hz, 1H), 6.59 (d, \(J_{HH} = 8\) Hz, 1H), 6.42 (d, \(J_{HH} = 12\) Hz, 1H), 5.24 (d, \(J_{HH} = 16\) Hz, 1H), 5.16 (d, \(J_{HH} = 16\) Hz, 1H), 4.94 (d, \(J_{HH} = 16\) Hz, 1H), 4.20 (d, \(J_{HH} = 12\) Hz, 1H). \(^{13}\)C{\(^1\)H} NMR (DMSO-\(d_6\), 100 MHz, 301 K) \(\delta\) 163.0, 160.2, 155.6, 148.0, 141.1, 130.5, 129.1, 129.0, 128.9, 128.0, 127.9, 125.9, 124.3, 122.9, 120.1, 119.2, 119.1, 118.9, 117.8, 115.3, 111.1, 53.7, 53.6. Selected IR (cm\(^{-1}\)): 3127 v(2′ N–H); 916, 876 v(Mo=O).

**Crystals of \(\text{PhL}_{\text{MoO}}\) (1b)** were grown by forming a supersaturated solution of the complex in DMF and layering with hexanes. The solution was placed in a refrigerator at 268 K for 1 month. Orange-yellow crystals were observed to grow for 1.5 months. Orange-yellow crystals were observed to grow and were collected for structural determination.

Procedure for synthesis of molybdenum complexes

Dioxido[2,2′-\{[(Cyclohexane-1,2-diyl)bis(azanediyl)]bis(methyl-\)ene\}]bis(2,4-di-tert-butylphenolato) dioxidomolybdenum(VI) (\(\text{C\text{yLMO}_2}\) 2b): A round-bottom flask equipped with a magnetic stirring bar was charged with MoO\(_2\)(acac\(_2\)) (0.165 g, 0.51 mmol) and methanol (ca. 10 mL). The solution was stirred, and 2a (0.27 g, 0.51 mmol) was added to the MoO\(_2\)(acac\(_2\)) dissolved in methanol. The solution was stirred overnight when it turned orange. The solution was filtered, and the solvent removed by evaporation under vacuum to obtain an orange precipitate. The precipitate was triturated with methanol, producing an orange solid, which was separated by gravity filtration and washed twice with cold methanol (0.108 g, 42%). \(^1\)H NMR (CDCl\(_3\), 400 MHz, 301 K) \(\delta\) 7.26 (s, 2H), 7.21 (d, \(J_{HH} = 12\) Hz, 2H), 7.18–7.15 (m, 4H), 7.07–7.05 (m, 1H), 6.91 (d, \(J_{HH} = 8\) Hz, 1H), 6.85–6.83 (m, 1H), 6.80 (d, \(J_{HH} = 8\) Hz, 1H), 6.76–6.68 (m, 2H), 6.63 (d, \(J_{HH} = 8\) Hz, 1H), 6.59 (d, \(J_{HH} = 8\) Hz, 1H), 6.42 (d, \(J_{HH} = 12\) Hz, 1H), 5.24 (d, \(J_{HH} = 16\) Hz, 1H), 5.16 (d, \(J_{HH} = 16\) Hz, 1H), 4.94 (d, \(J_{HH} = 16\) Hz, 1H), 4.20 (d, \(J_{HH} = 12\) Hz, 1H). 

**Figure 7**

Synthesis of the dioxomolybdenum complexes 1b and 2b.
Table 4
Experimental details.

|          | 1b                                                                 | 2b                                                                 |
|----------|----------------------------------------------------------------------|----------------------------------------------------------------------|
| Chemical data | [Mo(C₇₀H₆₀N₂O₂)O₂].2C₂H₇NO                                         | [Mo(C₇₀H₆₀N₂O₂)O₂].2CH₂O                                          |
| M₀       | 592.49                                                             | 740.84                                                             |
| Crystal system, space group | Triclinic, P T                                                    | Monoclinic, P2₁/n                                                  |
| Temperature (K) | 100                                                                | 105                                                                |
| a, b, c (Å) | 9.601, 12.860, 21.428                                              | 18.4889 (14), 10.9722 (8), 19.1517 (14)                           |
| α, β, γ (°) | 91.44, 91.49, 93.22                                               | 90, 94.035 (2), 90                                               |
| V (Å³)   | 2639.8                                                             | 3875.6 (5)                                                        |
| Z        | 4                                                                  | 4                                                                  |
| Radiation type | Mo Kα                                                            | Mo Kα                                                            |
| μ (mm⁻¹) | 0.54                                                              | 0.38                                                              |
| Crystal size (mm) | 0.34 × 0.29 × 0.29                                                | 0.2 × 0.18 × 0.1                                                  |
| Data collection | Brucker APEXII CCD                                                  | Brucker APEXII CCD                                                |
| Diffractometer | Bruker APEXII CCD                                                | Bruker APEXII CCD                                                |
| Absorption correction | Multi-scan (SADABS; Bruker, 2016)                     | Multi-scan (SADABS; Bruker, 2016)                                  |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 146655, 7625, 6364                                      | 29075, 9532, 8724                                                |
| Rₑₒₜ | 0.056                                                             | 0.026                                                             |
| (sin θλ)max (Å⁻¹) | 0.641                                                            | 0.667                                                             |
| Refinement |                                                     |                                                                  |
| R(F²) > 2σ(F²), W(R(F²)), S | 0.035, 0.065, 1.06                                           | 0.028, 0.070, 1.07                                               |
| No. of reflections | 7625                                                                 | 9532                                                              |
| No. of parameters | 683                                                                | 440                                                              |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H-atom parameters constrained                                    |
| ∆ρmax, ∆ρmin (e Å⁻³) | 0.35, −0.38                                                        | 0.52, −0.52                                                      |

Computer programs: APEX2 and SAINT (Bruker, 2016), SHELXT (Sheldrick, 2015), SHELXL (Sheldrick, 2008), and OLEX2 (Dolomanov et al., 2009).

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33.0, 31.6, 31.6, 31.5, 29.9, 29.9, 28.9, 24.5, 24.3, 24.1. Selected 1R (cm⁻¹): 903, 875 v(Mo=O).

Crystals of CH₃ using HFIX commands, and refined using a riding model (sin θλ)max > 2σ(I) reflections [Mo(C₇₀H₆₀N₂O₂)O₂] were grown by using a super-saturated solution of the complex dissolved in methanol and allowed to undergo slow evaporation over 2 d. A similar vial was also refrigerated where crystals were seen to form as well. The crystals from the slow evaporation set up were cropped and the orange–yellow crystals were used for structure determination.

6. Refinement
Crystal data, data collection, and refinement details are listed in Table 4. Hydrogen atoms were placed at ideal positions with C—H distances at 0.95 for CH and 0.99 Å for sp³ CH₂ and CH₃ using HFIX commands, and refined using a riding model with U(eq)(H) = 1.2U(eq)(C) for CH₃, CH₂, and CH₂. The structure for [Mo(C₇₀H₆₀N₂O₂)O₂] was initially refined in the trigonal crystal system P321; however, this resulted in the solvent DMF having a high level of disorder with many checkCIF errors.

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Computing details

For both structures, data collection: APEX2 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015); program(s) used to refine structure: SHELXL (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

(6,6′-[[Cyclohexane-1,2-diyl]bis(azanediyl)]bis(methylene)]bis(2,4-di-tert-butylphenolato))dioxidomolybdenum(VI) methanol disolvate (2b)

Crystal data

\[
\begin{align*}
\text{[Mo(C_{36}H_{56}N_{2}O_{2})O_{2}]} & \cdot 2\text{CH}_4\text{O} \\
M_r & = 740.84 \\
\text{Monoclinic, } P2_1/n & \\
a & = 18.4889 (14) \text{ Å} \\
b & = 10.9722 (8) \text{ Å} \\
c & = 19.1517 (14) \text{ Å} \\
\beta & = 94.035 (2)^\circ \\
V & = 3875.6 (5) \text{ Å}^3 \\
Z & = 4 \\
\end{align*}
\]

\[
F(000) = 1584 \\
D_\text{x} = 1.270 \text{ Mg m}^{-3} \\
\text{Mo } K\alpha \text{ radiation, } \lambda = 0.71073 \text{ Å} \\
\text{Cell parameters from 9945 reflections} \\
\theta = 5.3–51.4^\circ \\
\mu = 0.38 \text{ mm}^{-1} \\
T = 105 \text{ K} \\
\text{Prism, clear yellow} \\
0.2 \times 0.18 \times 0.1 \text{ mm}
\]

Data collection

Bruker APEXII CCD

diffractometer

\(\varphi\) and \(\omega\) scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

\(T_{\text{min}} = 0.672, T_{\text{max}} = 0.750\)

29075 measured reflections

9532 independent reflections

8724 reflections with \(I > 2\sigma(I)\)

\(R_{\text{int}} = 0.026\)

\(\theta_{\text{max}} = 28.3^\circ, \theta_{\text{min}} = 5.3^\circ\)

\(h = -24 \rightarrow 24\)

\(k = -14 \rightarrow 14\)

\(l = -25 \rightarrow 25\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R(F^2 > 2\sigma(F^2)) = 0.028\)

\(wR(F^2) = 0.070\)

\(S = 1.07\)

9532 reflections

440 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

\(w = 1/[\sigma(F_c^2) + (0.0277P)^2 + 2.9594P]\)

where \(P = (F_c^2 + 2F_s^2)/3\)
(Δ/σ)_{max} = 0.002 
\Delta \rho_{max} = 0.52 \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 (Å²)**

|      | x         | y         | z         | Uiso*/Ueq |
|------|-----------|-----------|-----------|-----------|
| Mo01 | 0.59832 (2) | 0.45373 (2) | 0.68071 (2) | 0.00996 (4) |
| O1   | 0.67906 (5) | 0.43892 (9) | 0.62159 (5) | 0.01218 (19) |
| O3   | 0.64642 (6) | 0.52033 (10) | 0.75027 (6) | 0.0154 (2) |
| O2   | 0.51926 (5) | 0.39987 (10) | 0.73535 (5) | 0.01299 (19) |
| O4   | 0.55503 (6) | 0.56653 (10) | 0.63112 (6) | 0.0166 (2) |
| N2   | 0.54408 (6) | 0.30514 (11) | 0.60679 (6) | 0.0124 (2) |
| H2   | 0.562452 | 0.317877 | 0.559459 | 0.015* |
| N1   | 0.64967 (6) | 0.26557 (11) | 0.71382 (6) | 0.0108 (2) |
| H1   | 0.621681 | 0.233132 | 0.752592 | 0.013* |
| O5   | 0.44789 (6) | 0.68127 (12) | 0.54536 (6) | 0.0239 (3) |
| H5A  | 0.481126 | 0.646691 | 0.570127 | 0.036* |
| C22  | 0.46379 (8) | 0.32549 (14) | 0.59954 (7) | 0.0142 (3) |
| H22A | 0.442308 | 0.276831 | 0.559782 | 0.017* |
| H22B | 0.453789 | 0.412566 | 0.589333 | 0.017* |
| C3   | 0.84869 (7) | 0.31009 (13) | 0.70189 (7) | 0.0111 (3) |
| H3   | 0.864314 | 0.255829 | 0.738559 | 0.013* |
| C5   | 0.87410 (7) | 0.43712 (13) | 0.60581 (7) | 0.0121 (3) |
| H5   | 0.908405 | 0.472482 | 0.577181 | 0.015* |
| C24  | 0.46022 (7) | 0.32631 (13) | 0.73127 (7) | 0.0119 (3) |
| C1   | 0.75049 (7) | 0.41320 (13) | 0.63556 (7) | 0.0102 (2) |
| C25  | 0.43157 (7) | 0.28483 (13) | 0.79316 (7) | 0.0126 (3) |
| C2   | 0.77461 (7) | 0.33656 (13) | 0.69077 (7) | 0.0108 (2) |
| C33  | 0.46569 (8) | 0.32001 (14) | 0.86602 (7) | 0.0142 (3) |
| C4   | 0.89997 (7) | 0.36090 (13) | 0.66085 (7) | 0.0118 (3) |
| C26  | 0.36957 (8) | 0.21086 (14) | 0.78600 (8) | 0.0146 (3) |
| H26  | 0.348850 | 0.184092 | 0.827336 | 0.017* |
| C23  | 0.42907 (8) | 0.28965 (14) | 0.66554 (7) | 0.0142 (3) |
| C9   | 0.74008 (9) | 0.66304 (14) | 0.55600 (9) | 0.0201 (3) |
| H9A  | 0.697246 | 0.641207 | 0.580670 | 0.030* |
| H9B  | 0.725631 | 0.716410 | 0.516425 | 0.030* |
| H9C  | 0.775058 | 0.705636 | 0.588184 | 0.030* |
| C6   | 0.80076 (7) | 0.46405 (12) | 0.59066 (7) | 0.0111 (2) |
| C15  | 0.72567 (7) | 0.28252 (13) | 0.74267 (7) | 0.0110 (2) |
| H15A | 0.745494 | 0.202582 | 0.758523 | 0.013* |
| H15B | 0.725879 | 0.336533 | 0.784094 | 0.013* |
| C7   | 0.77512 (8) | 0.54657 (13) | 0.52888 (7) | 0.0128 (3) |
| C10  | 0.83846 (8) | 0.58516 (15) | 0.48592 (8) | 0.0170 (3) |
| Atom  | x     | y     | z     | U(eq)  |
|-------|-------|-------|-------|--------|
| C10   | 0.873925 | 0.631277 | 0.515788 | 0.025* |
| C16   | 0.64240 (8) | 0.17496 (13) | 0.65555 (7) | 0.0141 (3) |
| H16   | 0.672705 | 0.202504 | 0.617289 | 0.017* |
| C21   | 0.66673 (8) | 0.04744 (13) | 0.67866 (8) | 0.0160 (3) |
| H21A  | 0.642144 | 0.024197 | 0.720976 | 0.019* |
| H21B  | 0.719628 | 0.047899 | 0.691019 | 0.019* |
| C27   | 0.33673 (8) | 0.17452 (14) | 0.72175 (8) | 0.0157 (3) |
| C28   | 0.36837 (8) | 0.21471 (14) | 0.66180 (8) | 0.0162 (3) |
| H28   | 0.347797 | 0.190093 | 0.617174 | 0.019* |
| C31   | 0.98156 (7) | 0.33692 (13) | 0.67445 (7) | 0.0129 (3) |
| C36   | 0.54757 (8) | 0.29016 (15) | 0.87359 (8) | 0.0194 (3) |
| H36A  | 0.555113 | 0.205208 | 0.860075 | 0.029* |
| H36B  | 0.565991 | 0.302178 | 0.922347 | 0.029* |
| H36C  | 0.573525 | 0.344170 | 0.843165 | 0.029* |
| C19   | 0.56900 (10) | −0.04481 (15) | 0.59694 (9) | 0.0219 (3) |
| H19A  | 0.539944 | −0.068313 | 0.636294 | 0.026* |
| H19B  | 0.559450 | −0.104765 | 0.558777 | 0.026* |
| C20   | 0.64927 (9) | −0.04664 (15) | 0.62109 (9) | 0.0224 (3) |
| H20A  | 0.678364 | −0.029062 | 0.580807 | 0.027* |
| H20B  | 0.662603 | −0.128871 | 0.638897 | 0.027* |
| C14   | 1.02015 (9) | 0.45629 (15) | 0.69599 (9) | 0.0222 (3) |
| H14A  | 1.012181 | 0.516351 | 0.658365 | 0.033* |
| H14B  | 1.072247 | 0.441111 | 0.704617 | 0.033* |
| H14C  | 1.006671 | 0.487651 | 0.738759 | 0.033* |
| C34   | 0.43001 (10) | 0.25202 (16) | 0.92482 (8) | 0.0229 (3) |
| H34A  | 0.378309 | 0.272408 | 0.923167 | 0.034* |
| H34B  | 0.453306 | 0.276340 | 0.970230 | 0.034* |
| H34C  | 0.435662 | 0.163973 | 0.918539 | 0.034* |
| C12   | 0.99721 (8) | 0.24308 (14) | 0.73274 (8) | 0.0161 (3) |
| H12A  | 0.979753 | 0.274088 | 0.776503 | 0.024* |
| H12B  | 1.049566 | 0.228556 | 0.739032 | 0.024* |
| H12C  | 0.972342 | 0.166547 | 0.720004 | 0.024* |
| C8    | 0.72060 (9) | 0.47859 (16) | 0.47828 (8) | 0.0193 (3) |
| H8A   | 0.677274 | 0.458088 | 0.502541 | 0.029* |
| H8B   | 0.742917 | 0.403623 | 0.462137 | 0.029* |
| H8C   | 0.706920 | 0.530810 | 0.437985 | 0.029* |
| C35   | 0.45589 (9) | 0.45675 (14) | 0.87842 (8) | 0.0205 (3) |
| H35A  | 0.476047 | 0.502693 | 0.840440 | 0.031* |
| H35B  | 0.481207 | 0.479742 | 0.923160 | 0.031* |
| H35C  | 0.404140 | 0.475293 | 0.879611 | 0.031* |
| C17   | 0.56291 (8) | 0.17702 (14) | 0.62828 (8) | 0.0144 (3) |
| H17   | 0.533304 | 0.155719 | 0.668282 | 0.017* |
| C13   | 1.01268 (8) | 0.28886 (15) | 0.60759 (8) | 0.0186 (3) |
| H13A  | 0.987537 | 0.213576 | 0.592770 | 0.028* |
| H13B  | 1.064551 | 0.272195 | 0.616823 | 0.028* |
| H13C  | 1.005854 | 0.350083 | 0.570454 | 0.028* |
| Atom  | U\textsubscript{11}    | U\textsubscript{22}    | U\textsubscript{33}    | U\textsubscript{12}    | U\textsubscript{13}    | U\textsubscript{23}    |
|-------|----------------|----------------|----------------|----------------|----------------|----------------|
| Mo01  | 0.00740 (6) | 0.01008 (6) | 0.01251 (6) | 0.00059 (4) | 0.00143 (4) | 0.00055 (4) |
| O1    | 0.0078 (4) | 0.0146 (5) | 0.0143 (5) | 0.0017 (4) | 0.0018 (4) | 0.0034 (4) |
| O3    | 0.0134 (5) | 0.0137 (5) | 0.0190 (5) | −0.0010 (4) | 0.0010 (4) | −0.0023 (4) |
| O2    | 0.0102 (4) | 0.0154 (5) | 0.0135 (5) | −0.0020 (4) | 0.0023 (4) | −0.0009 (4) |
| O4    | 0.0135 (5) | 0.0168 (5) | 0.0197 (5) | 0.0044 (4) | 0.0030 (4) | 0.0037 (4) |
| N2    | 0.0099 (5) | 0.0152 (6) | 0.0119 (5) | 0.0022 (5) | 0.0000 (4) | 0.0012 (4) |
| N1    | 0.0079 (5) | 0.0117 (5) | 0.0127 (5) | −0.0015 (4) | 0.0006 (4) | 0.0002 (4) |
| O5    | 0.0216 (6) | 0.0340 (7) | 0.0158 (5) | 0.0087 (5) | −0.0007 (4) | 0.0025 (5) |
| C22   | 0.0102 (6) | 0.0187 (7) | 0.0133 (6) | −0.0004 (5) | −0.0014 (5) | 0.0016 (5) |
| C3    | 0.0104 (6) | 0.0096 (6) | 0.0131 (6) | 0.0002 (5) | −0.0002 (5) | 0.0003 (5) |
| C5    | 0.0101 (6) | 0.0122 (6) | 0.0144 (6) | −0.0018 (5) | 0.0029 (5) | 0.0005 (5) |
| C24   | 0.0072 (6) | 0.0128 (6) | 0.0156 (6) | 0.0009 (5) | 0.0004 (5) | 0.0007 (5) |
| C1    | 0.0077 (6) | 0.0100 (6) | 0.0131 (6) | −0.0003 (5) | 0.0013 (5) | −0.0009 (5) |
| C25   | 0.0104 (6) | 0.0125 (6) | 0.0148 (6) | 0.0023 (5) | 0.0006 (5) | 0.0009 (5) |
| C2    | 0.0094 (6) | 0.0099 (6) | 0.0131 (6) | −0.0020 (5) | 0.0014 (5) | −0.0002 (5) |
| C33   | 0.0157 (7) | 0.0140 (7) | 0.0130 (6) | 0.0010 (5) | 0.0022 (5) | 0.0004 (5) |
| C4    | 0.0090 (6) | 0.0114 (6) | 0.0150 (6) | −0.0002 (5) | 0.0010 (5) | −0.0013 (5) |
| C26   | 0.0113 (6) | 0.0144 (7) | 0.0185 (7) | 0.0013 (5) | 0.0042 (5) | 0.0029 (5) |
C23  0.0115 (6)  0.0178 (7)  0.0133 (6)  0.0002 (5)  −0.0001 (5)  0.0022 (5)
C9   0.0236 (8)  0.0144 (7)  0.0231 (7)  0.0052 (6)  0.0068 (6)  0.0051 (6)
C6   0.0108 (6)  0.0097 (6)  0.0129 (6)  −0.0004 (5)  0.0018 (5)  0.0004 (5)
C15  0.0083 (6)  0.0126 (6)  0.0120 (6)  −0.0017 (5)  −0.0004 (5)  0.0018 (5)
C7   0.0116 (6)  0.0133 (6)  0.0135 (6)  0.0002 (5)  0.0019 (5)  0.0028 (5)
C10  0.0149 (7)  0.0197 (7)  0.0166 (7)  −0.0022 (6)  0.0035 (5)  0.0055 (6)
C16  0.0140 (6)  0.0130 (7)  0.0150 (6)  0.0000 (5)  −0.0013 (5)  −0.0017 (5)
C21  0.0157 (7)  0.0118 (7)  0.0197 (7)  0.0019 (5)  −0.0041 (5)  −0.0005 (5)
C27  0.0093 (6)  0.0157 (7)  0.0219 (7)  −0.0005 (5)  0.0003 (5)  0.0025 (6)
C28  0.0213 (6)  0.0189 (7)  0.0169 (7)  −0.0008 (6)  −0.0028 (5)  0.0014 (6)
C11  0.0077 (6)  0.0140 (7)  0.0169 (6)  0.0000 (5)  0.0006 (5)  0.0010 (5)
C36  0.0177 (7)  0.0221 (8)  0.0177 (7)  0.0026 (6)  −0.0039 (6)  0.0001 (6)
C19  0.0274 (8)  0.0155 (7)  0.0218 (7)  −0.0022 (6)  −0.0057 (6)  −0.0017 (6)
C20  0.0258 (8)  0.0136 (7)  0.0269 (8)  0.0028 (6)  −0.0046 (6)  −0.0039 (6)
C14  0.0144 (7)  0.0178 (7)  0.0336 (9)  −0.0054 (6)  −0.0043 (6)  −0.0005 (6)
C34  0.0301 (9)  0.0251 (8)  0.0138 (7)  −0.0048 (7)  0.0042 (6)  0.0027 (6)
C12  0.0118 (6)  0.0191 (7)  0.0175 (7)  0.0027 (6)  0.0011 (5)  0.0035 (6)
C8   0.0168 (7)  0.0261 (8)  0.0145 (7)  −0.0049 (6)  −0.0012 (5)  0.0026 (6)
C35  0.0274 (8)  0.0161 (7)  0.0183 (7)  0.0028 (6)  0.0031 (6)  −0.0010 (6)
C17  0.0141 (6)  0.0133 (7)  0.0156 (7)  −0.0007 (5)  −0.0006 (5)  0.0005 (5)
C13  0.0134 (7)  0.0242 (8)  0.0185 (7)  0.0046 (6)  0.0033 (5)  0.0037 (6)
C18  0.0236 (8)  0.0161 (7)  0.0171 (7)  0.0005 (6)  −0.0068 (6)  −0.0025 (6)
C29  0.0103 (6)  0.0180 (7)  0.0270 (8)  −0.0030 (6)  0.0014 (6)  0.0012 (6)
C30  0.0221 (8)  0.0218 (8)  0.0345 (9)  −0.0053 (7)  0.0031 (7)  −0.0032 (7)
C37  0.0202 (8)  0.0358 (10)  0.0230 (8)  −0.0026 (7)  0.0027 (6)  −0.0067 (7)
C32  0.0226 (8)  0.0354 (10)  0.0324 (9)  −0.0141 (8)  0.0084 (7)  0.0008 (8)
C31  0.0135 (7)  0.0230 (9)  0.0563 (12) −0.0015 (7) −0.0078 (8)  0.0031 (8)
O0AA 0.0424 (9)  0.0457 (9)  0.0550 (10)  0.0012 (8)  0.0073 (7)  −0.0082 (8)
C38  0.0409 (12) 0.0465 (13)  0.0479 (13)  0.0002 (10)  0.0118 (10)  0.0003 (10)

**Geometric parameters (Å, °)**

| Bond                | Length (Å) | Angle (°) |
|---------------------|------------|-----------|
| Mo01—O1             | 1.9428 (10) |           |
| Mo01—O3             | 1.7125 (10) |           |
| Mo01—O2             | 1.9484 (10) |           |
| Mo01—O4             | 1.7226 (11) |           |
| Mo01—N2             | 2.3384 (12) |           |
| Mo01—N1             | 2.3412 (12) |           |
| O1—C1               | 1.3586 (16) |           |
| C2C2                | 1.3554 (17) |           |
| N2—H2               | 1.0000     |           |
| N2—C22              | 1.4979 (18) |           |
| N2—C17              | 1.4989 (19) |           |
| N1—H1               | 1.0000     |           |
| N1—C15              | 1.4850 (17) |           |
| N1—C16              | 1.4935 (18) |           |
| O5—H5A              | 0.8400     |           |
| O5—C37              | 1.421 (2)  |           |
| Bond | Length (Å) | Bond | Length (Å) |
|------|------------|------|------------|
| O3—Mo01—N2 | 1.610 (5) | C13—C11—C4 | 1.098 (3) |
| O3—Mo01—N1 | 1.892 (5) | C13—C11—C14 | 1.088 (3) |
| O2—Mo01—N2 | 1.789 (4) | C33—C36—H36A | 1.079 |
| O2—Mo01—N1 | 1.832 (4) | C33—C36—H36B | 1.095 |
| O4—Mo01—O1 | 1.948 (5) | C33—C36—H36C | 1.095 |
| O4—Mo01—O2 | 1.100 (5) | H36A—C36—H36B | 1.095 |
| O4—Mo01—N2 | 1.902 (5) | H36A—C36—H36C | 1.095 |
| O1—C11—N1 | 1.161 (5) | H36B—C36—H36C | 1.095 |
| N2—Mo01—N1 | 1.724 (4) | H19A—C19—H19B | 1.081 |
| C1—O1—Mo01 | 1.327 (5) | C20—C19—H19A | 1.096 |
| C24—O2—Mo01 | 1.141 (5) | C20—C19—H19B | 1.096 |
| Mo01—N2—H2 | 1.107 (9) | C20—C19—C18 | 1.110 (13) |
| C22—N2—Mo01 | 1.094 (9) | C18—C19—H19A | 1.096 |
| C22—N2—H2 | 1.107 (9) | C18—C19—H19B | 1.096 |
| C22—N2—C17 | 1.120 (11) | C18—C19—C18 | 111.2 (11) |
| C17—N2—Mo01 | 1.113 (8) | H20A—C20—H20B | 1.080 |
| C17—N2—H2 | 1.107 (9) | H20A—C20—H20B | 1.080 |
| C17—N2—C17 | 1.111 (9) | H20B—C20—H20B | 1.095 |
| C15—N1—Mo01 | 1.110 (8) | C7—C8—H8A | 1.095 |
| C15—N1—H1 | 1.071 (8) | C7—C8—H8B | 1.095 |
| C15—N1—C16 | 1.132 (11) | C7—C8—H8C | 1.095 |
| C16—N1—Mo01 | 1.111 (9) | H8A—C8—H8B | 1.095 |
| C16—N1—H1 | 1.071 (8) | H8A—C8—H8C | 1.095 |
| C37—O5—H5A | 1.095 | H14A—C14—H14B | 1.095 |
| N2—C22—H22A | 1.094 | H14A—C14—H14C | 1.095 |
| N2—C22—H22B | 1.094 | H14B—C14—H14B | 1.095 |
| N2—C22—C23 | 1.111 (11) | H14B—C14—H14C | 1.095 |
| H22A—C22—H22B | 1.080 | C33—C34—H34A | 1.095 |
| C23—C22—H22A | 1.094 | C33—C34—H34B | 1.095 |
| C23—C22—H22B | 1.094 | C33—C34—H34C | 1.095 |
| C2—C3—C3 | 1.119 (12) | C33—C34—H34A | 1.095 |
| C4—C3—H3 | 1.190 | C33—C34—H34B | 1.095 |
| C4—C3—C2 | 1.191 (12) | C33—C34—H34C | 1.095 |
| C4—C5—H5 | 1.198 | C33—C34—H34A | 1.095 |
| C6—C5—H5 | 1.198 | C33—C34—H34B | 1.095 |
| C6—C5—C4 | 1.238 (13) | C33—C34—H34C | 1.095 |
| O2—C24—C25 | 1.195 (12) | C7—C8—H8A | 1.095 |
| O2—C24—C23 | 1.199 (12) | C7—C8—H8B | 1.095 |
| C23—C24—C25 | 1.204 (13) | C7—C8—H8C | 1.095 |
| O1—C1—C2 | 1.121 (12) | C7—C8—H8A | 1.095 |
| O1—C1—C6 | 1.177 (12) | C7—C8—H8B | 1.095 |
| C2—C1—C6 | 1.203 (12) | C8—C9—H8B | 1.095 |
| C24—C25—C33 | 1.218 (13) | C8—C9—H8C | 1.095 |
| C24—C25—C24 | 1.172 (13) | C8—C9—H8C | 1.095 |
| C26—C25—C33 | 1.208 (13) | C33—C35—H35A | 1.095 |
| C26—C25—C24 | 1.172 (13) | C33—C35—H35B | 1.095 |
| C3—C2—C15 | 1.162 (12) | C33—C35—H35C | 1.095 |
| C1—C2—C3 | 1.197 (12) | H35A—C35—H35B | 1.095 |
| C1—C2—C15 | 1.239 (12) | H35A—C35—H35B | 1.095 |
C25—C33—C36 111.60 (12) H35A—C35—H35C 109.5
C34—C33—C25 111.87 (12) H35B—C35—H35C 109.5
C34—C33—C36 107.23 (13) N2—C17—C16 107.91 (12)
C35—C33—C25 109.93 (12) N2—C17—H17 107.5
C35—C33—C36 108.59 (13) N2—C17—C18 114.02 (12)
C35—C33—C34 107.46 (13) C16—C17—H17 107.5
C3—C4—C5 116.99 (12) C16—C17—C18 112.17 (13)
C3—C4—C11 122.44 (12) C18—C17—H17 107.5
C5—C4—C11 120.57 (12) C11—C13—H13A 109.5
C25—C26—H26 118.2 C11—C13—H13B 109.5
C27—C26—C25 123.69 (13) C11—C13—H13C 109.5
C27—C26—H26 118.2 H13A—C13—H13C 109.5
C24—C23—C22 120.24 (13) C13—C14—C15 116.99 (12)
C28—C23—C22 120.05 (13) H3—C14—C15 109.5
C28—C23—C24 119.62 (13) C14—C15—C16 113.51 (11)
C2—C15—C2 108.9 C14—C15—H15A 108.9
C2—C15—H15A 109.5 C14—C15—H15B 108.9
H15A—C15—H15B 107.7 C14—C15—H15C 108.9
H9A—C9—H9B 109.5 C15—C16—H16 108.7
C7—C9—H9A 109.5 C15—C16—H16 108.7
C7—C9—H9B 109.5 C15—C16—H16 108.7
C7—C9—H9C 109.5 H16—C15—C16 107.5
C9—C7—C8 109.99 (13) H16—C15—C16 107.5
C6—C7—C9 110.08 (12) H16—C15—C16 107.5
C6—C7—C8 110.57 (12) O5—C37—H37A 109.5
C6—C7—C8 110.57 (12) O5—C37—H37B 109.5
C10—C7—C9 107.85 (12) O5—C37—H37C 109.5
C10—C7—C6 111.63 (12) O5—C37—H37C 109.5
C10—C7—C8 107.54 (12) C37—O5—C38 107.54 (12)
C7—C10—H10A 109.5 H37A—C37—H37B 109.5
C7—C10—H10B 109.5 H37A—C37—H37C 109.5
C7—C10—H10C 109.5 H37B—C37—H37C 109.5
H10A—C10—H10B 109.5 H32A—C32—H32B 109.5
H10A—C10—H10C 109.5 H32A—C32—H32B 109.5
H10B—C10—H10C 109.5 H32A—C32—H32B 109.5
N1—C16—H16 108.7 H32A—C32—H32B 109.5
N1—C16—C21 112.61 (11) N2—C17—H17 107.5
N1—C16—C17 106.26 (11) N2—C17—H17 107.5
C21—C16—H16 108.7 H32A—C32—H32B 109.5
C17—C16—H16 108.7 C29—C31—H31B 109.5
C17—C16—C21 111.73 (12) H31A—C31—H31B 109.5
| Bond                  | Angle (°) | Bond                  | Angle (°) |
|----------------------|-----------|----------------------|-----------|
| C16—C21—H21A        | 109.3     | C16—C21—H21B        | 109.3     |
| C16—C21—C20         | 111.54 (12)| C38—O0AA—H0AA      | 109.5     |
| H21A—C21—H21B       | 108.0     | O0AA—C38—H38A       | 109.5     |
| C20—C21—H21A        | 109.3     | O0AA—C38—H38B       | 109.5     |
| C20—C21—H21B        | 109.3     | O0AA—C38—H38C       | 109.5     |
| C26—C27—C28         | 117.04 (13)| H38A—C38—H38B       | 109.5     |
| C26—C27—C29         | 122.83 (14)| H38A—C38—H38C       | 109.5     |
| C28—C27—C29         | 120.13 (13)| H38B—C38—H38C       | 109.5     |
| C23—C28—C27         | 121.89 (14)|                     |           |
| Mo01—O1—C1—C6       | 31.67 (19)| C1—C6—C7—C10       | −177.16 (13)|
| Mo01—O1—C1—C6       | −150.18 (10)| C1—C6—C7—C8      | −58.64 (17)|
| Mo01—O2—C24—C25     | 158.74 (11)| C25—C24—C23—C22   | −174.72 (13)|
| Mo01—O2—C24—C23     | −19.8 (2) | C25—C24—C23—C28     | 1.8 (2) |
| Mo01—N2—C22—C23     | −73.12 (13)| C25—C26—C27—C28   | 0.2 (2) |
| Mo01—N2—C17—C16     | −38.13 (13)| C25—C26—C27—C29   | −179.20 (14)|
| Mo01—N2—C17—C18     | −163.45 (10)| C2—C3—C4—C5      | −2.2 (2) |
| Mo01—N1—C15—C2      | −61.41 (13)| C2—C3—C4—C11      | 177.34 (13)|
| Mo01—N1—C16—C21     | −172.68 (9) | C2—C1—C6—C5       | −2.0 (2) |
| Mo01—N1—C16—C17     | −50.05 (12)| C2—C1—C6—C7       | 178.87 (13)|
| O1—C1—C2—C3         | 178.24 (12)| C33—C25—C26—C27   | −179.11 (14)|
| O1—C1—C2—C15        | −4.4 (2)  | C4—C3—C2—C1       | 2.1 (2) |
| O1—C1—C6—C5         | 179.79 (12)| C4—C3—C2—C15      | −175.52 (13)|
| O1—C1—C6—C7         | 0.69 (19)  | C4—C5—C6—C1       | 1.9 (2) |
| O2—C24—C25—C33      | −0.4 (2)  | C4—C5—C6—C7       | −178.97 (13)|
| O2—C24—C25—C26      | 178.78 (13)| C4—C5—C6—C3       | 127.48 (15)|
| O2—C24—C23—C22      | 3.8 (2)   | C4—C5—C6—C3       | 7.33 (19) |
| O2—C24—C23—C28      | −179.71 (13)| C26—C25—C33—C36   | −111.97 (16)|
| N2—C22—C23—C24      | 45.68 (19) | C26—C25—C33—C35   | −1.3 (2) |
| N2—C22—C23—C28      | −130.82 (14)| C26—C27—C29—C30   | −126.65 (16)|
| N2—C17—C18—C19      | 178.60 (13)| C26—C27—C29—C32   | −6.2 (2) |
| N1—C16—C21—C20      | 172.29 (13)| C26—C27—C29—C31   | 113.07 (17)|
| N1—C16—C17—N2       | 56.94 (14) | C23—C24—C25—C33   | 178.13 (13)|
| N1—C16—C17—C18      | −176.64 (12)| C23—C24—C25—C26   | −2.7 (2) |
| C22—N2—C17—C16      | −163.01 (11)| C6—C5—C4—C3      | 0.1 (2) |
| C22—N2—C17—C18      | 71.67 (16) | C6—C5—C4—C11      | −179.40 (13)|
| C22—C23—C28—C27     | 176.79 (14)| C6—C1—C2—C3      | 0.1 (2) |
| C3—C2—C15—N1        | −154.63 (12)| C6—C1—C2—C15     | 177.52 (13)|
| C3—C4—C11—C14       | −114.88 (15)| C15—N1—C16—C21   | 62.12 (15)|
| C3—C4—C11—C12       | 5.36 (19)  | C15—N1—C16—C17   | −175.25 (11)|
| C3—C4—C11—C13       | 125.68 (14)| C16—N1—C15—C2     | 64.63 (15)|
| C5—C4—C11—C14       | 64.61 (17) | C16—C21—C20—C19   | −55.27 (18)|
| C5—C4—C11—C12       | −175.14 (13)| C16—C17—C18—C19   | 55.56 (18)|
| C5—C4—C11—C13       | −54.82 (17)| C21—C16—C17—N2   | −179.87 (11)|
| C5—C6—C7—C9         | −115.97 (15)| C21—C16—C17—C18  | −53.46 (17)|
| C5—C6—C7—C10        | 3.78 (19)  | C28—C27—C29—C30   | 53.95 (19)|
| C5—C6—C7—C8         | 122.30 (14)| C28—C27—C29—C32   | 174.37 (15)|
C24—C25—C33—C36 −53.41 (18)  C28—C27—C29—C31 −66.33 (19)
C24—C25—C33—C34 −173.56 (13)  C20—C19—C18—C17 −57.38 (18)
C24—C25—C33—C35 67.14 (17)  C17—N2—C22—C23 54.25 (15)
C24—C25—C26—C27 1.7 (2)  C17—C16—C21—C20 52.79 (17)
C24—C23—C28—C27 0.3 (2)  C18—C19—C20—C21 57.58 (18)
C1—C2—C15—N1 27.90 (19)  C17—C16—C21—C20 52.79 (17)
C1—C6—C7—C9 63.09 (17)

Hydrogen-bond geometry (Å, °)

\[ \begin{array}{cccccc}
D—H···A & D—H & H···A & D···A & D—H···A \\
N2—H2···O5^i & 1.00 & 2.00 & 2.9319 (16) & 153 \\
O5—H5A···O4 & 0.84 & 1.94 & 2.7837 (16) & 177 \\
\end{array} \]

Symmetry code: (i) \(-x+1, -y+1, -z+1\).

Dioxido(2,2’-[1,2-phenylenebis(iminomethylene)]bis(phenolato))molybdenum(VI) dimethylformamide disolvate (1b)

Crystal data

\[ \text{[Mo(C}_{20}\text{H}_{18}\text{N}_{2}\text{O}_{2})\text{O}_2]·2\text{C}_{3}\text{H}_7\text{NO}} \]

\[ \begin{array}{cccc}
Z &=& 4 \\
F(000) &=& 1224 \\
D_\text{x} &=& 1.491 \text{ Mg m}^{-3} \\
\text{Mo } K\alpha \text{ radiation, } \lambda = 0.71073 \text{ Å} \\
\end{array} \]

Cell parameters from 9515 reflections

\[ \begin{array}{cccc}
a &=& 9.601 \text{ Å} \\
b &=& 12.860 \text{ Å} \\
c &=& 21.428 \text{ Å} \\
\alpha &=& 91.44^\circ \\
\beta &=& 91.49^\circ \\
\gamma &=& 93.22^\circ \\
V &=& 2639.8 \text{ Å}^3 \\
\end{array} \]

Data collection

Bruker APEXII CCD

diffractometer

\[ \phi \text{ and } \omega \text{ scans} \]

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

\[ T_{\text{min}} = 0.664, T_{\text{max}} = 0.737 \]

146655 measured reflections

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\[ R[F^2 > 2\sigma(F^2)] = 0.035 \]

\[ wR(F^2) = 0.065 \]

\[ S = 1.06 \]

7625 reflections

683 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

\[ \Delta \sigma/\sigma \text{max} = 0.002 \]

\[ \Delta \rho_{\text{max}} = 0.35 \text{ e Å}^{-3} \]

\[ \Delta \rho_{\text{min}} = -0.38 \text{ e Å}^{-3} \]
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 (Å²)

|     | x          | y          | z          | Uiso/ULEQ |
|-----|------------|------------|------------|-----------|
| Mo01| 0.90908 (2)| 0.88143 (2)| 0.23791 (2)| 0.01047 (6) |
| Mo02| 0.40906 (2)| 0.38140 (2)| 0.26211 (2)| 0.01045 (6) |
| O5  | 0.36926 (14)| 0.42698 (12)| 0.17689 (7) | 0.0135 (3)  |
| O1  | 0.86930 (14)| 0.92696 (12)| 0.32311 (7) | 0.0134 (3)  |
| O3  | 1.00613 (14)| 0.98497 (13)| 0.21447 (8) | 0.0165 (3)  |
| O8  | 0.51765 (15)| 0.28410 (13)| 0.23806 (8) | 0.0152 (3)  |
| O7  | 0.50605 (15)| 0.48499 (13)| 0.28555 (8) | 0.0169 (3)  |
| N2  | 0.72468 (16)| 0.77486 (14)| 0.26078 (8) | 0.0098 (3)  |
| H008| 0.736567| 0.755680| 0.30560| 0.012* |
| N4  | 0.22457 (16)| 0.27483 (14)| 0.23925 (9) | 0.0103 (4)  |
| H009| 0.231199| 0.252423| 0.194450| 0.012* |
| O4  | 1.01781 (15)| 0.78420 (13)| 0.26191 (8) | 0.0152 (3)  |
| O2  | 0.87093 (14)| 0.83127 (13)| 0.15364 (7) | 0.0141 (3)  |
| O6  | 0.37087 (14)| 0.33128 (13)| 0.34638 (7) | 0.0141 (3)  |
| N1  | 0.71375 (17)| 0.97852 (14)| 0.21773 (9) | 0.0101 (3)  |
| N3  | 0.21399 (17)| 0.47869 (14)| 0.28219 (9) | 0.0101 (3)  |
| O9  | 0.2105 (3)| 0.59956 (16)| 0.39734 (9) | 0.0395 (5)  |
| O10 | 0.29034 (18)| 0.15986 (15)| 0.12529 (8) | 0.0235 (4)  |
| O11 | 0.79013 (18)| 0.65963 (14)| 0.37463 (8) | 0.0234 (4)  |
| O12 | 0.7110 (3)| 1.09959 (16)| 0.10270 (9) | 0.0388 (5)  |
| C40 | 0.2694 (2)| 0.27959 (17)| 0.37595 (10) | 0.0133 (4)  |
| C33 | 0.09199 (19)| 0.32526 (16)| 0.24527 (10) | 0.0097 (4)  |
| N6  | 0.2294 (2)| 0.00745 (18)| 0.07856 (10) | 0.0229 (4)  |
| C13 | 0.59214 (19)| 0.82531 (17)| 0.25477 (10) | 0.0100 (4)  |
| N7  | 0.7296 (2)| 0.50764 (18)| 0.42150 (10) | 0.0231 (5)  |
| C2  | 0.6928 (2)| 1.05716 (17)| 0.32295 (11) | 0.0132 (4)  |
| C44 | 0.2062 (2)| 0.0933 (2)| 0.11184 (11) | 0.0201 (5)  |
| H00P| 0.114108| 0.101089| 0.125731| 0.024* |
| C20 | 0.7691 (2)| 0.77942 (17)| 0.12402 (10) | 0.0138 (4)  |
| C26 | 0.1928 (2)| 0.55726 (17)| 0.17703 (11) | 0.0133 (4)  |
| C35 | 0.1970 (2)| 0.20156 (17)| 0.34516 (11) | 0.0138 (4)  |
| C32 | −0.0270 (2)| 0.27625 (17)| 0.22922 (10) | 0.0124 (4)  |
| H00T| −0.028382| 0.209159| 0.209538| 0.015* |
| C21 | 0.2697 (2)| 0.48024 (18)| 0.14646 (11) | 0.0140 (4)  |
| N5  | 0.2261 (2)| 0.77472 (17)| 0.40850 (10) | 0.0223 (4)  |
| C47 | 0.7060 (2)| 0.5934 (2)| 0.38822 (11) | 0.0196 (5)  |
| H00W| 0.612568| 0.601350| 0.374316| 0.024* |
| N8  | 0.7263 (2)| 1.27474 (17)| 0.09144 (10) | 0.0223 (4)  |
| C12 | 0.4730 (2)| 0.77623 (17)| 0.27093 (10) | 0.0125 (4)  |
| Atom | X       | Y       | Z       | U11     | U22     | U33     | U12     | U13     | U23     |
|------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| H00Y | 0.473625| 0.710632| 0.290319| 0.015   |
| C39  | 0.2388  | 0.3050  | 0.43809 (11) | 0.0201 (5) |
| H00Z | 0.291477| 0.368086| 0.458964 | 0.024   |
| C8   | 0.58707 (19) | 0.92488 (16) | 0.22796 (10) | 0.0091 (4) |
| C29  | -0.0390 (2) | 0.47127 (17) | 0.28501 (10) | 0.0123 (4) |
| H011 | -0.037498| 0.538591| 0.304393 | 0.015   |
| C19  | 0.7389  (2) | 0.8050 (2) | 0.06185 (12) | 0.0203 (5) |
| H012 | 0.791590| 0.859381| 0.042563 | 0.024   |
| C15  | 0.6965 (2) | 0.70132 (17) | 0.15474 (11) | 0.0142 (4) |
| C28  | 0.08690 (19) | 0.42482 (17) | 0.27184 (10) | 0.0091 (4) |
| C41  | 0.1618 (3) | 0.6874 (2) | 0.39361 (12) | 0.0283 (6) |
| H015 | 0.068263| 0.689541| 0.378304 | 0.034   |
| C9   | 0.4611 (2) | 0.97132 (17) | 0.21493 (10) | 0.0125 (4) |
| H016 | 0.460785| 1.037336| 0.196026 | 0.015   |
| C1   | 0.7697  (2) | 0.98025 (18) | 0.35362 (11) | 0.0138 (4) |
| C5   | 0.6467 (3) | 1.0132 (2) | 0.44922 (12) | 0.0256 (6) |
| H018 | 0.630612| 0.998654| 0.491758 | 0.031   |
| C34  | 0.2286 (2) | 0.17687 (17) | 0.27776 (11) | 0.0134 (4) |
| H01G | 0.158999| 0.123370| 0.260434 | 0.016   |
| H01H | 0.322041| 0.148400| 0.275457 | 0.016   |
| C23  | 0.1469 (3) | 0.5132 (2) | 0.05077 (12) | 0.0258 (6) |
| H01I | 0.130229| 0.498464| 0.007471 | 0.031   |
| C6   | 0.7459 (2) | 0.9580 (2) | 0.41654 (12) | 0.0204 (5) |
| H01B | 0.796107| 0.906242| 0.436774 | 0.024   |
| C50  | 0.6613 (3) | 1.1871 (2) | 0.10638 (12) | 0.0282 (6) |
| H3AA | 0.569131| 1.190364| 0.121161 | 0.034   |
| C25  | 0.0929 (2) | 0.61129 (19) | 0.14338 (11) | 0.0189 (5) |
| H01J | 0.041127| 0.662013| 0.164100 | 0.023   |
| C52  | 0.6615 (3) | 1.3763 (2) | 0.09717 (13) | 0.0289 (6) |
| H4AA | 0.655244| 1.407393| 0.055983 | 0.043   |
| H    | 0.718693| 1.422943| 0.125606 | 0.043   |
| HA   | 0.567704| 1.365643| 0.113700 | 0.043   |
| C3   | 0.5933 (2) | 1.1153 (19) | 0.35649 (12) | 0.0194 (5) |
| H01F | 0.541726| 1.162479| 0.336215 | 0.023   |
| C36  | 0.0928 (2) | 0.1508 (2) | 0.37725 (12) | 0.0227 (5) |
| H01K | 0.038307| 0.096025| 0.356264 | 0.027   |
| C43  | 0.1617 (3) | 0.8762 (2) | 0.40289 (13) | 0.0286 (6) |
| H01V | 0.066608| 0.864836| 0.385335 | 0.043   |
| H01X | 0.217185| 0.920818| 0.375361 | 0.043   |
| H01  | 0.158583| 0.910086| 0.444276 | 0.043   |
| C14  | 0.7286 (2) | 0.67698 (17) | 0.22211 (11) | 0.0132 (4) |
| H01A | 0.658777| 0.624167| 0.236808 | 0.016   |
| H01C | 0.822034| 0.648551| 0.225945 | 0.016   |
| C7   | 0.7171 (2) | 1.07799 (17) | 0.25468 (11) | 0.0132 (4) |
| H01D | 0.643980| 1.122212| 0.238363 | 0.016   |
| H01E | 0.808727| 1.116005| 0.250437 | 0.016   |
| C27  | 0.2171 (2) | 0.57802 (17) | 0.24528 (11) | 0.0134 (4) |
| H01L | 0.144556| 0.623135| 0.260818 | 0.016   |
| Atom  | x     | y     | z     | Ueq  |
|-------|-------|-------|-------|------|
| H01M  | 0.308883 | 0.616118 | 0.252287 | 0.016* |
| C22   | 0.2461 (2) | 0.4580 (2) | 0.08324 (11) | 0.0201 (5) |
| H01Q  | 0.296160 | 0.406416 | 0.062409 | 0.024* |
| C38   | 0.1364 (3) | 0.2525 (2) | 0.46952 (12) | 0.0268 (6) |
| H01S  | 0.118153 | 0.270242 | 0.511713 | 0.032* |
| C18   | 0.6362 (3) | 0.7523 (2) | 0.03063 (12) | 0.0273 (6) |
| H01N  | 0.614082 | 0.767239 | 0.011561 | 0.033* |
| C16   | 0.5928 (2) | 0.6508 (2) | 0.12279 (12) | 0.0221 (5) |
| H01O  | 0.538427 | 0.597338 | 0.142117 | 0.027* |
| C17   | 0.5624 (3) | 0.6753 (2) | 0.06102 (13) | 0.0294 (6) |
| H01P  | 0.487821 | 0.637301 | 0.039190 | 0.035* |
| C37   | 0.0627 (3) | 0.1754 (2) | 0.43895 (13) | 0.0293 (6) |
| H01T  | -0.010064 | 0.137342 | 0.459158 | 0.035* |
| C4    | 0.5703 (3) | 1.0908 (2) | 0.41929 (12) | 0.0278 (6) |
| H01R  | 0.504234 | 1.127999 | 0.441810 | 0.033* |
| C46   | 0.3651 (3) | -0.0174 (2) | 0.05437 (13) | 0.0305 (6) |
| H1AA  | 0.357558 | -0.026473 | 0.008834 | 0.046* |
| HB    | 0.394488 | -0.082007 | 0.072697 | 0.046* |
| HC    | 0.434179 | 0.039525 | 0.065371 | 0.046* |
| C24   | 0.0704 (3) | 0.5906 (2) | 0.08077 (12) | 0.0271 (6) |
| H01U  | 0.004413 | 0.627654 | 0.057686 | 0.032* |
| C42   | 0.3670 (3) | 0.7719 (2) | 0.43037 (14) | 0.0314 (6) |
| H0AA  | 0.372846 | 0.789365 | 0.475180 | 0.047* |
| HD    | 0.425537 | 0.822557 | 0.407903 | 0.047* |
| HE    | 0.399668 | 0.701912 | 0.423092 | 0.047* |
| C51   | 0.8669 (3) | 1.2720 (2) | 0.06964 (14) | 0.0310 (6) |
| H5AA  | 0.902116 | 1.203125 | 0.076874 | 0.046* |
| HF    | 0.926724 | 1.325264 | 0.092367 | 0.046* |
| HG    | 0.867536 | 1.285745 | 0.024869 | 0.046* |
| C11   | 0.3464 (2) | 0.82277 (18) | 0.25892 (11) | 0.0153 (4) |
| H01W  | 0.261648 | 0.787886 | 0.270639 | 0.018* |
| C48   | 0.6238 (4) | 0.4321 (3) | 0.43236 (16) | 0.0491 (9) |
| H2AA  | 0.613110 | 0.425094 | 0.477441 | 0.074* |
| HH    | 0.647881 | 0.365190 | 0.413854 | 0.074* |
| HI    | 0.535937 | 0.452815 | 0.413397 | 0.074* |
| C30   | -0.1581 (2) | 0.42067 (18) | 0.27025 (11) | 0.0157 (5) |
| H01Y  | -0.244489 | 0.449657 | 0.279109 | 0.019* |
| C31   | -0.1535 (2) | 0.32303 (18) | 0.24115 (11) | 0.0156 (5) |
| H01Z  | -0.238872 | 0.286402 | 0.228821 | 0.019* |
| C49   | 0.8649 (3) | 0.4826 (2) | 0.44569 (13) | 0.0300 (6) |
| H02D  | 0.933605 | 0.539377 | 0.437160 | 0.045* |
| H02E  | 0.892550 | 0.417988 | 0.425468 | 0.045* |
| H02F  | 0.860619 | 0.473340 | 0.490867 | 0.045* |
| C45   | 0.1239 (4) | -0.0683 (3) | 0.06749 (17) | 0.0505 (9) |
| H02A  | 0.149070 | -0.133015 | 0.087006 | 0.076* |
| H02B  | 0.110078 | -0.080595 | 0.022377 | 0.076* |
| H02C  | 0.037358 | -0.045270 | 0.085225 | 0.076* |
| C10   | 0.3417 (2) | 0.92073 (18) | 0.22967 (11) | 0.0158 (5) |
Atomic displacement parameters (Å²)

|        | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|--------|-----------|-----------|-----------|-----------|-----------|-----------|
| Mo01   | 0.00439 (8) | 0.00933 (10) | 0.01750 (10) | −0.00061 (6) | 0.00009 (6) | −0.00087 (7) |
| Mo02   | 0.00428 (8) | 0.00942 (10) | 0.01748 (10) | −0.00056 (6) | 0.00010 (6) | 0.00020 (6) |
| O5     | 0.0091 (6) | 0.0138 (8)  | 0.0178 (8)  | 0.00111 (6)  | 0.00001 (6)  | 0.00000 (6)  |
| O1     | 0.0087 (6) | 0.0138 (8)  | 0.0175 (8)  | 0.00133 (6)  | −0.00164 (6) | −0.0029 (6)  |
| O3     | 0.0082 (6) | 0.0153 (8)  | 0.0254 (9)  | −0.0046 (6)  | 0.0021 (6)   | −0.0019 (7)  |
| O8     | 0.0081 (6) | 0.0142 (8)  | 0.0235 (8)  | 0.00085 (6)  | 0.00000 (6)  | 0.0017 (7)   |
| O7     | 0.0084 (6) | 0.0169 (8)  | 0.0248 (9)  | −0.00329 (6) | −0.0035 (6)  | 0.0011 (7)   |
| N2     | 0.0056 (7) | 0.0088 (9)  | 0.0148 (9)  | −0.00111 (7) | −0.0022 (6)  | −0.0003 (7)  |
| N4     | 0.0060 (7) | 0.0090 (9)  | 0.0158 (9)  | 0.00011 (7)  | 0.00117 (6)  | −0.0022 (7)  |
| O4     | 0.0076 (6) | 0.0140 (8)  | 0.0237 (8)  | 0.00066 (6)  | −0.0014 (6)  | −0.0030 (7)  |
| O2     | 0.0093 (6) | 0.0152 (8)  | 0.0175 (8)  | −0.00411 (6) | 0.0021 (6)   | −0.0007 (6)  |
| O6     | 0.0085 (6) | 0.0155 (8)  | 0.0175 (8)  | −0.0043 (6)  | −0.0038 (6)  | 0.0003 (6)   |
| N1     | 0.0086 (7) | 0.0100 (9)  | 0.0115 (9)  | −0.00087 (7) | 0.0018 (6)   | 0.0010 (7)   |
| N3     | 0.0084 (7) | 0.0092 (9)  | 0.0123 (9)  | −0.0013 (7)  | −0.0025 (7)  | −0.0028 (7)  |
| O9     | 0.0747 (15)| 0.0213 (10) | 0.0206 (10) | −0.0087 (11) | −0.0054 (10) | −0.0046 (8)  |
| O10    | 0.0253 (8) | 0.0227 (9)  | 0.0220 (9)  | −0.0024 (8)  | 0.0047 (7)   | −0.0052 (7)  |
| O11    | 0.0260 (8) | 0.0214 (9)  | 0.0221 (9)  | −0.0029 (7)  | −0.0064 (7)  | 0.0034 (7)   |
| O12    | 0.0738 (15)| 0.0221 (10) | 0.0194 (10) | −0.0091 (10) | 0.0019 (10)  | 0.0024 (8)   |
| C40    | 0.0110 (8) | 0.0126 (10) | 0.0165 (11) | 0.0010 (8)   | −0.0008 (8)  | 0.0035 (8)   |
| C33    | 0.0077 (8) | 0.0099 (10) | 0.0117 (10) | 0.0008 (8)   | 0.0009 (7)   | 0.0004 (8)   |
| N6     | 0.0258 (10)| 0.0234 (11) | 0.0184 (11) | −0.0088 (9)  | 0.0065 (8)   | −0.0060 (9)  |
| C13    | 0.0089 (8) | 0.0097 (10) | 0.0114 (10) | 0.0014 (8)   | −0.0011 (7)  | −0.0011 (8)  |
| N7     | 0.0264 (10)| 0.0230 (11) | 0.0185 (11) | −0.0091 (9)  | −0.0071 (8)  | 0.0041 (9)   |
| C2     | 0.0099 (8) | 0.0101 (10) | 0.0189 (11) | −0.0033 (8)  | −0.0023 (8)  | −0.0040 (8)  |
| C44    | 0.0218 (10)| 0.0233 (13) | 0.0155 (11) | 0.0028 (10)  | 0.0039 (9)   | 0.0011 (10)  |
| C20    | 0.0105 (9) | 0.0123 (10) | 0.0184 (11) | 0.0015 (8)   | 0.0010 (8)   | −0.0046 (8)  |
| C26    | 0.0108 (8) | 0.0094 (10) | 0.0193 (11) | −0.0032 (8)  | 0.0011 (8)   | 0.0030 (8)   |
| C35    | 0.0109 (8) | 0.0099 (10) | 0.0206 (11) | 0.0000 (8)   | −0.0007 (8)  | 0.0034 (8)   |
| C32    | 0.0093 (8) | 0.0088 (10) | 0.0185 (11) | −0.0033 (8)  | −0.0011 (8)  | −0.0006 (8)  |
| C21    | 0.0075 (8) | 0.0148 (11) | 0.0198 (11) | −0.0011 (8)  | 0.0017 (8)   | 0.0048 (9)   |
| N5     | 0.0251 (10)| 0.0215 (11) | 0.0201 (11) | 0.0007 (9)   | −0.0033 (8)  | −0.0015 (9)  |
| C47    | 0.0215 (10)| 0.0227 (13) | 0.0142 (11) | −0.0004 (10) | −0.0039 (9)  | −0.0015 (10)|
|       |   x    |   y    |   z    |   x    |   y    |   z    |
|-------|--------|--------|--------|--------|--------|--------|
| C9    | 0.0096 | 0.0105 | 0.0174 | 0.0012 | -0.0016| 0.0002 |
| C1    | 0.0077 | 0.0137 | 0.0193 | -0.0017| -0.0018 | -0.0044|
| C5    | 0.0227 | 0.0393 | 0.0147 | 0.0034 | -0.0009| -0.0032|
| C34   | 0.0107 | 0.0086 | 0.0206 | -0.0002| -0.0011| 0.0003 |
| C23   | 0.0234 | 0.0392 | 0.0148 | 0.0030 | -0.0003| 0.0014 |
| C6    | 0.0162 | 0.0263 | 0.0185 | 0.0020 | -0.0047| -0.0004|
| C50   | 0.0402 | 0.0292 | 0.0138 | -0.0104| 0.0003 | 0.0013 |
| C25   | 0.0140 | 0.0187 | 0.0248 | 0.0034 | 0.0043 | 0.0065 |
| C52   | 0.0312 | 0.0275 | 0.0292 | 0.0091 | 0.0037 | 0.0028 |
| C3    | 0.0146 | 0.0185 | 0.0247 | 0.0035 | -0.0047| -0.0071|
| C36   | 0.0204 | 0.0192 | 0.0281 | -0.0040| 0.0011 | 0.0060 |
| C43   | 0.0304 | 0.0271 | 0.0286 | 0.0083 | -0.0035| -0.0032|
| C14   | 0.0109 | 0.0079 | 0.0208 | 0.0000 | 0.0007 | -0.0002|
| C7    | 0.0102 | 0.0072 | 0.0219 | -0.0015| -0.0005| -0.0007|
| C27   | 0.0105 | 0.0076 | 0.0218 | -0.0011| 0.0003 | 0.0005 |
| C22   | 0.0171 | 0.0254 | 0.0178 | 0.0025 | 0.0028 | -0.0017|
| C38   | 0.0296 | 0.0329 | 0.0179 | 0.0003 | 0.0042 | 0.0028 |
| C18   | 0.0309 | 0.0313 | 0.0188 | -0.0002| -0.0055| -0.0051|
| C16   | 0.0185 | 0.0188 | 0.0281 | -0.0041| 0.0002 | -0.0065|
| C17   | 0.0278 | 0.0337 | 0.0249 | -0.0030| -0.0078| -0.0119|
| C37   | 0.0288 | 0.0344 | 0.0253 | -0.0025| 0.0078 | 0.0125 |
| C4    | 0.0217 | 0.0379 | 0.0242 | 0.0118 | -0.0006| -0.0115|
| C46   | 0.0319 | 0.0318 | 0.0278 | 0.0052 | 0.0051 | -0.0110|
| C24   | 0.0222 | 0.0377 | 0.0230 | 0.0120 | 0.0008 | 0.0114 |
| C42   | 0.0288 | 0.0324 | 0.0326 | 0.0062 | -0.0088| -0.0059|
| C51   | 0.0274 | 0.0325 | 0.0341 | 0.0052 | 0.0079 | 0.0056 |
| C11   | 0.0069 | 0.0103 | 0.0280 | -0.0050| 0.0018 | -0.0036|
| C48   | 0.0536 | 0.0512 | 0.0385 | -0.0346| -0.0143| 0.0156 |
| C30   | 0.0081 | 0.0162 | 0.0231 | 0.0023 | 0.0027 | 0.0029 |
| C31   | 0.0074 | 0.0113 | 0.0275 | -0.0050| -0.0029| 0.0022 |
| C49   | 0.0310 | 0.0319 | 0.0276 | 0.0049 | -0.0063| 0.0108 |
| C45   | 0.0540 | 0.0502 | 0.0432 | -0.0335| 0.0155 | -0.0181|
| C10   | 0.0081 | 0.0165 | 0.0224 | 0.0014 | -0.0033| -0.0035|

**Geometric parameters (Å, º)**

|       |       |       |       |       |       |       |
|-------|-------|-------|-------|-------|-------|-------|
| Mo01—O1 | 1.9567 (16) | C29—C30 | 1.311 (3) |
| Mo01—O3 | 1.6769 (16) | C19—H012 | 0.9500 |
| Mo01—O4 | 2.2493 (17) | C19—C18 | 1.322 (4) |
| Mo01—O2 | 1.7518 (14) | C15—C14 | 1.512 (3) |
| Mo01—O1 | 1.9213 (16) | C15—C16 | 1.324 (3) |
| Mo01—O2 | 2.3475 (16) | C41—H015 | 0.9500 |
| Mo01—N1 | 1.9665 (15) | C9—H016 | 0.9500 |
| Mo01—O8 | 1.7493 (15) | C9—C10 | 1.335 (3) |
| Mo01—O7 | 1.6423 (17) | C1—C6 | 1.407 (3) |
| Mo01—N4 | 2.2145 (18) | C5—H018 | 0.9500 |
| Mo01—O6 | 1.9692 (15) | C5—C6 | 1.410 (3) |
| Mo01—N3 | 2.3529 (16) | C5—C4 | 1.426 (4) |
| Bond | Distance (Å) | Bond | Distance (Å) | Distance (Å) |
|------|--------------|------|--------------|--------------|
| O5—C21 | 1.368 (2) | C34—H01G | 0.9900 |
| O1—C1 | 1.377 (2) | C34—H01H | 0.9900 |
| N2—H008 | 1.0000 | C23—H01I | 0.9500 |
| N2—C13 | 1.465 (2) | C23—C22 | 1.401 (3) |
| N2—C14 | 1.492 (3) | C23—C24 | 1.420 (4) |
| N4—H009 | 1.0000 | C6—H01B | 0.9500 |
| N4—C33 | 1.468 (2) | C50—H3AA | 0.9500 |
| N4—C34 | 1.525 (3) | C25—H01J | 0.9500 |
| O2—C20 | 1.295 (3) | C25—C24 | 1.370 (4) |
| O6—C40 | 1.332 (3) | C52—H4AA | 0.9800 |
| N1—C8 | 1.389 (3) | C52—H | 0.9800 |
| N1—C7 | 1.486 (3) | C52—HA | 0.9800 |
| N1—H00D | 0.85 (3) | C3—H01F | 0.9500 |
| N3—C28 | 1.379 (3) | C3—C4 | 1.399 (4) |
| N3—C27 | 1.519 (3) | C36—H01K | 0.9500 |
| N3—H00E | 0.79 (3) | C36—C37 | 1.393 (4) |
| O9—C41 | 1.250 (4) | C43—H01V | 0.9800 |
| O10—C44 | 1.169 (3) | C43—H01X | 0.9800 |
| O11—C47 | 1.187 (3) | C43—H01 | 0.9800 |
| O12—C50 | 1.249 (4) | C14—H01A | 0.9900 |
| C40—C35 | 1.338 (3) | C14—H01C | 0.9900 |
| C40—C39 | 1.405 (3) | C7—H01D | 0.9900 |
| C33—C32 | 1.307 (3) | C7—H01E | 0.9900 |
| C33—C28 | 1.392 (3) | C27—H01L | 0.9900 |
| N6—C44 | 1.332 (3) | C27—H01M | 0.9900 |
| N6—C46 | 1.464 (3) | C22—H01Q | 0.9500 |
| N6—C45 | 1.377 (4) | C38—H01S | 0.9500 |
| C13—C12 | 1.333 (3) | C38—C37 | 1.333 (4) |
| C13—C8 | 1.419 (3) | C18—H01N | 0.9500 |
| N7—C47 | 1.355 (3) | C18—C17 | 1.372 (4) |
| N7—C48 | 1.394 (4) | C16—H01O | 0.9500 |
| N7—C49 | 1.443 (3) | C16—C17 | 1.396 (4) |
| C2—C1 | 1.430 (3) | C17—H01P | 0.9500 |
| C2—C3 | 1.415 (3) | C37—H01T | 0.9500 |
| C2—C7 | 1.515 (3) | C4—H01R | 0.9500 |
| C44—H00P | 0.9500 | C46—H1AA | 0.9800 |
| C20—C19 | 1.407 (3) | C46—HB | 0.9800 |
| C20—C15 | 1.380 (3) | C46—HC | 0.9800 |
| C26—C21 | 1.425 (3) | C24—H01U | 0.9500 |
| C26—C25 | 1.410 (3) | C42—H0AA | 0.9800 |
| C26—C27 | 1.490 (3) | C42—HD | 0.9800 |
| C35—C34 | 1.513 (3) | C42—HE | 0.9800 |
| C35—C36 | 1.373 (3) | C51—H5AA | 0.9800 |
| C32—H00T | 0.9500 | C51—HF | 0.9800 |
| C32—C31 | 1.412 (3) | C51—HG | 0.9800 |
| C21—C22 | 1.388 (3) | C11—H01W | 0.9500 |
| N5—C41 | 1.280 (4) | C11—C10 | 1.424 (3) |
| N5—C43 | 1.482 (3) | C48—H2AA | 0.9800 |
| Bond          | Distance (Å) | Bond          | Distance (Å) |
|--------------|--------------|--------------|--------------|
| N5—C42       | 1.423 (3)    | C48—HH       | 0.9800       |
| C47—H00W     | 0.9500       | C48—HI       | 0.9800       |
| N8—C50       | 1.308 (3)    | C30—H01Y     | 0.9500       |
| N8—C52       | 1.481 (3)    | C30—C31      | 1.391 (3)    |
| N8—C51       | 1.442 (3)    | C31—H01Z     | 0.9500       |
| C12—H00Y     | 0.9500       | C49—H02D     | 0.9800       |
| C12—C11      | 1.405 (3)    | C49—H02E     | 0.9800       |
| C39—H00Z     | 0.9500       | C49—H02F     | 0.9800       |
| C39—C38      | 1.364 (4)    | C45—H02A     | 0.9800       |
| C8—C9        | 1.403 (2)    | C45—H02B     | 0.9800       |
| C29—H011     | 0.9500       | C45—H02C     | 0.9800       |
| C29—C28      | 1.410 (2)    | C10—H022     | 0.9500       |
| O1—Mo01—N2   | 77.65 (7)    | O1—C1—C6     | 118.21 (19)  |
| O1—Mo01—N1   | 80.23 (6)    | C6—C1—C2     | 120.4 (2)    |
| O3—Mo01—O1  | 100.51 (8)   | C6—C5—H018   | 119.6        |
| O3—Mo01—N2  | 161.90 (6)   | C6—C5—C4     | 120.9 (2)    |
| O3—Mo01—O4  | 109.80 (7)   | C4—C5—H018   | 119.6        |
| O3—Mo01—O2  | 92.31 (8)    | N4—C34—H01G  | 109.6        |
| O3—Mo01—N1  | 86.75 (6)    | N4—C34—H01H  | 109.6        |
| N2—Mo01—N1  | 75.18 (6)    | C35—C34—N4   | 110.35 (17)  |
| O4—Mo01—O1  | 94.14 (7)    | C35—C34—H01G | 109.6        |
| O4—Mo01—N2  | 88.30 (6)    | C35—C34—H01H | 109.6        |
| O4—Mo01—O2  | 98.32 (7)    | H01G—C34—H01H | 108.1 |
| O4—Mo01—N1  | 163.31 (7)   | C22—C23—H01I | 119.1        |
| O2—Mo01—O1  | 157.78 (6)   | C22—C23—C24  | 121.9 (2)    |
| O2—Mo01—N2  | 84.38 (7)    | C24—C23—H01I | 119.1        |
| O2—Mo01—N1  | 82.63 (6)    | C1—C6—C5     | 119.3 (2)    |
| O5—Mo02—N4  | 81.39 (7)    | C1—C6—H01B   | 120.4        |
| O5—Mo02—O6  | 158.10 (6)   | C5—C6—H01B   | 120.4        |
| O5—Mo02—N3  | 81.07 (6)    | O12—C50—N8   | 125.0 (3)    |
| O8—Mo02—O5  | 94.30 (7)    | O12—C50—H3AA | 117.5        |
| O8—Mo02—N4  | 89.52 (7)    | N8—C50—H3AA  | 117.5        |
| O8—Mo02—O6  | 98.33 (7)    | C26—C25—H01J | 120.0        |
| O8—Mo02—N3  | 163.76 (7)   | C24—C25—C26  | 120.0 (2)    |
| O7—Mo02—O5  | 96.96 (8)    | C24—C25—H01J | 120.0        |
| O7—Mo02—O8  | 109.00 (7)   | N8—C52—H4AA  | 109.5        |
| O7—Mo02—N4  | 161.48 (7)   | N8—C52—H     | 109.5        |
| O7—Mo02—O6  | 95.73 (8)    | N8—C52—HA    | 109.5        |
| O7—Mo02—N3  | 87.08 (7)    | H4AA—C52—H   | 109.5        |
| N4—Mo02—N3  | 74.43 (6)    | H4AA—C52—HA  | 109.5        |
| O6—Mo02—N4  | 80.91 (7)    | H—C52—HA     | 109.5        |
| O6—Mo02—N3  | 81.84 (6)    | C2—C3—H01F   | 119.8        |
| C21—O5—Mo02 | 137.80 (14)  | C4—C3—C2     | 120.5 (2)    |
| C1—O1—Mo01  | 138.48 (14)  | C4—C3—H01F   | 119.8        |
| Mo01—N2—H008| 107.3        | C35—C36—H01K | 118.3        |
| C13—N2—Mo01 | 112.59 (12)  | C35—C36—C37  | 123.5 (3)    |
| C13—N2—H008 | 107.3        | C37—C36—H01K | 118.3        |
| Bond/Angle | Distance (Å) | Torsion (°) |
|-----------|-------------|-------------|
| C13—N2—C14 | 113.36 (16) | N5—C43—H01V | 109.5 |
| C14—N2—Mo01 | 108.75 (12) | N5—C43—H01X | 109.5 |
| C14—N2—H008 | 107.3 | N5—C43—H01 | 109.5 |
| Mo02—N4—H009 | 107.0 | H01V—C43—H01X | 109.5 |
| C33—N4—Mo02 | 113.03 (13) | H01V—C43—H01 | 109.5 |
| C33—N4—H009 | 107.0 | H01X—C43—H01 | 109.5 |
| C33—N4—C34 | 111.85 (15) | N2—C14—C15 | 108.70 (16) |
| C34—N4—Mo02 | 110.51 (12) | N2—C14—H01A | 109.9 |
| C34—N4—H009 | 107.0 | N2—C14—H01C | 109.9 |
| C20—O2—Mo01 | 136.10 (14) | C15—C14—H01A | 109.9 |
| C40—O6—Mo02 | 138.81 (13) | C15—C14—H01C | 109.9 |
| Mo01—N1—H00D | 110.7 (19) | H01A—C14—H01C | 108.3 |
| C8—N1—Mo01 | 113.88 (13) | N1—C7—C2 | 110.47 (17) |
| C8—N1—C7 | 107.69 (16) | N1—C7—H01D | 109.6 |
| C8—N1—H00D | 106 (2) | N1—C7—H01E | 109.6 |
| C7—N1—Mo01 | 112.87 (12) | C2—C7—H01D | 109.6 |
| C7—N1—H00D | 105 (2) | C2—C7—H01E | 109.6 |
| Mo02—N3—H00E | 113.0 (19) | H01D—C7—H01E | 108.1 |
| C28—N3—Mo02 | 114.65 (13) | N3—C27—H01L | 109.1 |
| C28—N3—C27 | 109.21 (16) | N3—C27—H01M | 109.1 |
| C28—N3—H00E | 99 (2) | C26—C27—N3 | 112.45 (18) |
| C27—N3—Mo02 | 111.45 (12) | C26—C27—H01L | 109.1 |
| C27—N3—H00E | 109 (2) | C26—C27—H01M | 109.1 |
| O6—C40—C35 | 118.3 (2) | H01L—C27—H01M | 107.8 |
| O6—C40—C39 | 121.4 (2) | C21—C22—C23 | 118.6 (2) |
| C35—C40—C39 | 120.3 (2) | C21—C22—H01Q | 120.7 |
| C32—C33—N4 | 121.34 (19) | C23—C22—H01Q | 120.7 |
| C32—C33—C28 | 117.14 (18) | C39—C38—H01S | 121.2 |
| C28—C33—N4 | 121.41 (18) | C37—C38—C39 | 117.7 (2) |
| C44—N6—C46 | 124.5 (2) | C37—C38—H01S | 121.2 |
| C44—N6—C45 | 120.4 (2) | C19—C18—H01N | 121.0 |
| C45—N6—C46 | 115.0 (3) | C19—C18—C17 | 118.0 (2) |
| C12—C13—N2 | 120.75 (18) | C17—C18—H01N | 121.0 |
| C12—C13—C8 | 118.66 (17) | C15—C16—H01O | 119.6 |
| C8—C13—N2 | 120.47 (17) | C15—C16—C17 | 120.7 (3) |
| C47—N7—C48 | 121.9 (2) | C17—C16—H01O | 119.6 |
| C47—N7—C49 | 124.1 (2) | C18—C17—C16 | 122.5 (3) |
| C48—N7—C49 | 113.9 (2) | C18—C17—H01P | 118.8 |
| C1—C2—C7 | 119.57 (18) | C16—C17—H01P | 118.8 |
| C3—C2—C1 | 119.5 (2) | C36—C37—H01T | 120.1 |
| C3—C2—C7 | 120.89 (19) | C38—C37—C36 | 119.7 (2) |
| O10—C44—N6 | 125.1 (2) | C38—C37—H01T | 120.1 |
| O10—C44—H00P | 117.5 | C5—C4—H01R | 120.3 |
| N6—C44—H00P | 117.5 | C3—C4—C5 | 119.5 (2) |
| O2—C20—C19 | 118.2 (2) | C3—C4—H01R | 120.3 |
| O2—C20—C15 | 118.5 (2) | N6—C46—H1AA | 109.5 |
| C15—C20—C19 | 123.3 (2) | N6—C46—HB | 109.5 |
| C21—C26—C27 | 119.07 (18) | N6—C46—HC | 109.5 |
sup-19

Acta Cryst. (2022). E78, 244-250
| Bond                  | Angle (°)       |
|----------------------|-----------------|
| Mo01—O1—C1—C2       | −28.3 (3)       |
| Mo01—O1—C1—C6       | 152.37 (19)     |
| Mo01—N2—C13—C12     | −175.03 (17)    |
| Mo01—N2—C13—C8      | 8.9 (2)         |
| Mo01—N2—C14—C15     | −69.85 (16)     |
| Mo01—O2—C20—C19     | 144.35 (18)     |
| Mo01—O2—C20—C15     | −36.3 (3)       |
| Mo01—N1—C8—C13      | 6.3 (2)         |
| Mo01—N1—C8—C9       | −176.91 (16)    |
| Mo01—N1—C7—C2       | −70.89 (17)     |
| Mo02—O5—C21—C26     | 28.4 (3)        |
| Mo02—O5—C21—C22     | −152.48 (18)    |
| Mo02—N4—C33—C32     | 174.91 (17)     |
| Mo02—N4—C33—C28     | −9.1 (2)        |
| Mo02—N4—C34—C35     | 72.40 (16)      |
| Mo02—O6—C40—C35     | 37.3 (3)        |
| Mo02—O6—C40—C39     | −143.20 (19)    |
| Mo02—N3—C28—C33     | −6.2 (2)        |
| Mo02—N3—C28—C29     | 176.63 (15)     |
| O5—C21—C22—C23      | −178.6 (2)      |
| O1—C1—C6—C5         | 178.5 (2)       |
| N2—C13—C12—C11      | −173.9 (2)      |
| N2—C13—C8—N1        | −10.4 (3)       |
| N2—C13—C8—C9        | 172.8 (2)       |
| N4—C33—C32—C31      | 173.9 (2)       |
| N4—C33—C28—N3       | 10.4 (3)        |
| N4—C33—C28—C29      | −172.52 (19)    |
| O2—C20—C19—C18      | 179.4 (2)       |
| O2—C20—C15—C14      | 2.2 (3)         |
| O2—C20—C15—C16      | 179.62 (19)     |
| O6—C40—C35—C34      | −2.1 (3)        |
| O6—C40—C35—C36      | −179.60 (18)    |
| O6—C40—C39—C38      | −179.3 (2)      |
| N1—C8—C9—C10        | −175.1 (2)      |
| C40—C35—C34—N4      | −52.4 (2)       |
| C40—C35—C36—C37     | −1.3 (3)        |
| C40—C39—C38—C37     | −0.9 (4)        |
| C33—N4—C34—C35      | −54.5 (2)       |
| C33—C32—C31—C30     | −0.5 (3)        |
| C13—N2—C14—C15      | 56.2 (2)        |
| C13—C12—C11—C10     | 0.3 (3)         |
| C13—C8—C9—C10       | 1.6 (3)         |
| C2—C1—C6—C5         | −0.8 (4)        |
| C2—C3—C4—C5         | −0.8 (4)        |
| C20—C19—C18—C17     | 0.8 (4)         |
| C20—C15—C14—N2      | 53.3 (2)        |

Supporting information.
C20—C15—C16—C17  1.3 (3)  
C26—C21—C22—C23  0.5 (4)  
C26—C25—C24—C23  1.1 (4)  
C35—C40—C39—C38  0.2 (3)  
C35—C36—C37—C38  0.6 (4)  
C32—C33—C28—N3  −173.44 (19)  
C32—C33—C28—C29  3.6 (3)  
C21—C26—C25—C24  −0.7 (4)  
C21—C26—C27—N3  −48.4 (3)  
C12—C13—C8—N1  173.4 (2)  
C4—C5—C6—C1  0.3 (4)  
C46—N6—C44—O10  0.9 (4)  
C46—C6—C5—C2  1.1 (4)  
C26—C21—C22—C23  0.5 (4)  
C26—C25—C24—C23  1.1 (4)  
C35—C40—C39—C38  0.2 (3)  
C35—C36—C37—C38  0.6 (4)  
C32—C33—C28—N3  −173.44 (19)  
C32—C33—C28—C29  3.6 (3)  
C21—C26—C25—C24  −0.7 (4)  
C21—C26—C27—N3  −48.4 (3)  
C12—C13—C8—N1  173.4 (2)  
C4—C5—C6—C1  0.3 (4)  
C46—N6—C44—O10  0.9 (4)  
C46—C6—C5—C2  1.1 (4)  
C26—C21—C22—C23  0.5 (4)  
C26—C25—C24—C23  1.1 (4)  
C35—C40—C39—C38  0.2 (3)  
C35—C36—C37—C38  0.6 (4)  
C32—C33—C28—N3  −173.44 (19)  
C32—C33—C28—C29  3.6 (3)  
C21—C26—C25—C24  −0.7 (4)  
C21—C26—C27—N3  −48.4 (3)  
C12—C13—C8—N1  173.4 (2)  
C45—N6—C44—O10  177.3 (3)  

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H   | H···A | D···A  | D—H···A |
|---------|-------|-------|--------|--------|
| N2—H008···O11 | 1.00  | 2.03  | 2.958 (2) | 154 |
| N4—H009···O10  | 1.00  | 1.99  | 2.924 (3) | 154 |
| N1—H00D···O12  | 0.85 (3) | 2.15 (3) | 2.949 (3) | 157 (2) |
| N3—H00E···O9   | 0.79 (3) | 2.16 (3) | 2.885 (3) | 154 (3) |