Bis(oxotremorine) fumarate bis(fumaric acid)

Marilyn Naeem, Andrew R. Chadeayne, James A. Golen and David R. Manke*

The title compound, bis(oxotremorine) fumarate bis(fumaric acid) [systematic name: 1-[4-(2-oxopyrrolidin-1-yl)but-2-ynyl]pyrrolidinium (2E)-but-2-enedioate bis[(2E)-but-2-enedioic acid]], C12H19N2O+/C4H2O4 2/C0 /C12H4O4, has a single oxotremorine monocation protonated at the pyrrolidine nitrogen, one fumaric acid molecule and half of a fumarate dianion in the asymmetric unit. The ions and fumaric acid molecules are held together by N—H/C1/C1/C1 O and O—H/C1/C1/C1 O hydrogen bonds in 40-membered rings with graph-set notation R6(40). The fumarate ions join these rings into infinite chains along [001].

Structure description
Oxotremorine is a selective agonist of the muscarinic acetylcholine receptor, which reproduces many of the symptoms observed in Parkinson’s disease. This property has made it an invaluable tool in studying potential pharmaceuticals for Parkinson’s (Ringdahl & Jenden, 1983). A salt of oxotremorine that is commonly used in biological studies is produced by treating oxotremorine free base with fumaric acid. The resulting salt is reported as the sesquifumarate, indicating that the compound possesses an empirical formula with a 1:1.5 ratio of cation to fumarate dianion. However, the structure reported here shows that in the solid-state, the compound consists of two monocationic, protonated oxotremorines, one doubly deprotonated dianionic fumarate, and two fully protonated fumaric acid molecules. One half of these ions and molecules are present in the asymmetric unit (Fig. 1).

The only compound found by searching on ‘sesquifumarate’ in the Cambridge Structural Database (CSD, version 5.43, update of March 2022; Groom et al., 2016) is that of the anti-arrhythmic agent tedisamil, which also exists as the bis(cation) bis(fumaric acid) fumarate and not the technical sesquifumarate (Jones et al., 2004: CSD refcode EYOYUM). There are seven other bis(cation) bis(fumaric acid) fumarate salts (Haynes et al., 2006: RESGEC, RESGUS; Provins et al., 2006: SEGSAZ; Li & Zheng, 2005:
The hydrogen-bonding network forms chains along [001], which consist of \( R_6^2(40) \) rings that are joined together by the fumarate dianions. The ring structure is shown above. Hydrogen atoms not involved in hydrogen bonds, and the second component of the disordered fumarate dianion are omitted for clarity.

In the structure of the title compound, the pyrrolidinium \( N-H \) of oxotremorine has bifurcated hydrogen bonds to two O atoms of a symmetry-generated fumarate dianion. One fumaric acid \( O-H \) hydrogen bonds to the carbonyl oxygen of the oxopyrrolidine of oxotremorine. The other fumaric acid \( O-H \) hydrogen bonds to the carbonyl oxygen of the oxopyrrolidine of oxotremorine. The N—H of oxotremorine has bifurcated hydrogen bonds to two oxoatoms (Table 1). These hydrogen bonds connect two oxotremorine cations, two fumaric acid molecules and two fumarate dianions into rings that have graph-set notation \( R_6^2(40) \) (Etter et al., 1990) (Fig. 2). The fumarate ions connect these rings together into infinite one-dimensional chains along [001]. The crystal packing of the title compound is shown in Fig. 3.

The fumaric acid and the fumarate dianion are near planar with r.m.s. deviations from planarity of 0.092 and 0.033 Å,

| D—H···A D—H H···A D···A D—H···A |
|---|---|---|---|---|
| C14—H14···O5' | 0.93 | 2.65 | 3.498 (2) | 152 |
| C3—H3B···O1' | 0.97 | 2.43 | 3.381 (3) | 166 |
| C5—H5B···O2'' | 0.97 | 2.51 | 3.434 (2) | 159 |
| C8—H8A···O4'' | 0.97 | 2.54 | 3.191 (2) | 125 |
| C8—H8A···O6'' | 0.97 | 2.53 | 3.456 (3) | 161 |
| C8—H8B···O5'' | 0.97 | 2.52 | 3.480 (2) | 173 |
| C9—H9A···O6'' | 0.97 | 2.56 | 3.228 (3) | 126 |
| C10—H10A···O5'' | 0.97 | 2.66 | 3.624 (3) | 175 |
| C11—H11A···O4'' | 0.97 | 2.64 | 3.553 (3) | 157 |
| C12—H12B···O7'' | 0.97 | 2.68 | 3.399 (2) | 132 |
| C12—H12B···O7A'' | 0.97 | 2.37 | 3.139 (15) | 135 |
| O4—H4···O6 | 0.91 (1) | 1.58 (1) | 2.483 (2) | 167 (2) |
| O3—H3···O1 | 0.90 (1) | 1.69 (1) | 2.5739 (16) | 167 (2) |
| N2—H2···O6v | 0.908 (19) | 2.554 (18) | 3.172 (3) | 125.8 (17) |
| N2—H2···O7v | 0.908 (19) | 1.809 (19) | 2.705 (2) | 168.3 (17) |
| N2—H2···O6A'' | 0.908 (19) | 2.43 (3) | 3.131 (19) | 133.6 (15) |
| N2—H2···O7A'' | 0.908 (19) | 1.61 (2) | 2.489 (15) | 161.7 (17) |

Symmetry codes: (i) \( x-1, y, z \); (ii) \( -x, -y, -z+1 \); (iii) \( -x+1, -y+1, -z+1 \); (iv) \( -x+2, -y+1, -z+1 \); (v) \( -x+2, -y, -z+1 \); (vi) \( -x+1, -y, -z+1 \); (vii) \( x-1, y-1, z+1 \).
respectively. The C—O distances of the fumarate molecules are delocalized with values of 1.270 (3) and 1.243 (2) Å. The C—O distances in the fumaric acid molecules are localized, with the carbonyl distances being 1.209 (2) and 1.203 (2) Å and the carbon–hydroxyl distances being 1.310 (2) and 1.316 (18) Å. The C—O distances and the location of the hydrogen atoms from the difference-Fourier map make the assignment of fumarate and fumaric acid clear.

In the reported structure, the aminobut-2-ynlammonium unit has a near anti conformation, with a N2—C8—C5—N1 torsion angle of 163.17 (13)°. The other known structure of oxotremorine is reported as the sesquioxalate, but is similarly composed of the bis(oxotremorine) bis(oxalic acid) oxalate, oxotremorine is reported as the sesquioxalate, but is similarly composed of the bis(oxotremorine) bis(oxalic acid) oxalate, and shows a torsion angle of 38.35 (3)° for the equivalent nitrogen and carbon atoms (Clarke et al., 1975: OXTREO).

The other two similar structures reported, trimethyl-[4-(2-ethylaminoethyl)amino]-acetylenic imidazole (Moon et al., 1991: KOGCEO), and a related acetylenic imidazole (Clarke et al., 1991: KOGCEO) show equivalent torsion angles of 38.35 (3)° for the equivalent nitrogen and carbon atoms, respectively. The C—N distances of the imidazole molecules being 1.316 (18) Å. The C—O distances and the location of the hydrogen atoms from the difference-Fourier map make the assignment of fumarate and fumaric acid clear.

### Synthesis and crystallization

Single crystals suitable for X-ray diffraction studies were grown by dissolving 15 mg of oxotremorine sesquifumarate purchased from Sigma–Aldrich in 5 ml of water. Solvent was allowed to evaporate at ambient temperature and pressure and crystals formed after 12 h.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The fumarate dianion is disordered over two positions (C17, C18, O6, O7 and C17A, C18A, O6A, O7A), which were modeled using a SAME restraint, as well as 180°. The two components showed a 0.855 (4) to 0.145 (4) occupancy ratio.

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### Table 2

Experimental details.

| Parameter | Value |
|-----------|-------|
| Crystal data | C12H19N2O+·2C4H4O4− |
| Chemical formula | 2C12H19N2O+·2C4H4O4− |
| Mass | 760.78 |
| Crystal system, space group | Triclinic, P1 |
| Temperature (K) | 297 |
| α, β, γ (°) | 80.921 (3), 85.778 (5), 18.7260 (11) |
| V (Å³) | 94.922 (2), 90.428 (2), 98.945 (2) |
| Z | 1 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.10 |
| Crystal size (mm) | 0.30 × 0.20 × 0.04 |

Data collection

| Parameter | Value |
|-----------|-------|
| Monochromator | Mo Kα |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 27417, 3651, 2975 |
| R Donetsk, T | 0.035 |
| (∆2θ)max (Å⁻¹) | 0.612 |
| Refinement | |
| R[F² > 2σ(F²)], wR(F²), S | 0.042, 0.109, 1.03 |
| No. of reflections | 3651 |
| No. of parameters | 269 |
| No. of restraints | 8 |
| H-atoms treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.18, −0.15 |

Computer programs: APEX3 (Bruker, 2018), SAINT (Bruker, 2018), SHELXT2014 (Sheldrick, 2015a), SHELXL2018 (Sheldrick, 2015b), OLEX2 (Dolomanov et al., 2009), and pubCIF (Westrip, 2010).

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full crystallographic data

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1-[4-(2-Oxopyrrolidin-1-yl)but-2-ynyl]pyrrolidinium (2E)-but-2-enedioate bis[(2E)-but-2-enedioic acid]

Crystal data

\[2\text{C}_1\text{H}_4\text{N}_2\text{O}^+ \cdot \text{C}_4\text{H}_2\text{O}_4^- \cdot 2\text{C}_4\text{H}_6\text{O}_4 \]  
\( Z = 1 \)  

\( F(000) = 404 \)  

Triclinic, \( P\bar{1} \)  

\( a = 6.0921 \text{ (3) Å} \)  

\( b = 8.5778 \text{ (5) Å} \)  

\( c = 18.7260 \text{ (11) Å} \)  

\( \alpha = 94.922 \text{ (2)°} \)  

\( \beta = 90.428 \text{ (2)°} \)  

\( \gamma = 98.945 \text{ (2)°} \)  

\( V = 962.88 \text{ (9) Å}^3 \)  

Data collection

Bruker D8 Venture CMOS  

\( \phi \) and \( \omega \) scans  

Absorption correction: multi-scan  

(SADABS; Bruker, 2018)  

\( T_{\text{min}} = 0.717, T_{\text{max}} = 0.745 \)  

27417 measured reflections  

3651 independent reflections  

2975 reflections with \( I > 2\sigma(I) \)  

Refinement

Refinement on \( F^2 \)  

Least-squares matrix: full  

\( R[F^2 > 2\sigma(F^2)] = 0.042 \)  

\( wR(F^2) = 0.109 \)  

\( S = 1.03 \)  

3651 reflections  

269 parameters  

8 restraints  

Primary atom site location: structure-invariant direct methods  

Secondary atom site location: difference Fourier map  

Hydrogen site location: mixed  

\( w = 1/\left[\sigma^2(F_c^2) + (0.0473P)^2 + 0.2757P\right] \)  

where \( P = (F_c^2 + 2F_s^2)/3 \)  

\( (\Delta/\sigma)_{\text{max}} < 0.001 \)  

\( \Delta\rho_{\text{max}} = 0.18 \text{ e Å}^{-3} \)  

\( \Delta\rho_{\text{min}} = -0.15 \text{ e Å}^{-3} \)  

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.
**Refinement.** Hydrogen atoms H2, H3 and H4 were found from a difference-Fourier map and were refined isotropically, using *DFIX* restraints with O–H distances of 0.90 (1) Å. Isotropic displacement parameters were set to 1.2 $U_{eq}$ of the parent nitrogen atom and 1.5 $U_{eq}$ of the parent oxygen atom. All other hydrogen atoms were placed in calculated positions with C–H = 0.93 Å ($sp^2$) or 0.97 Å ($sp^3$). Isotropic displacement parameters were set to 1.2 $U_{eq}$ of the parent carbon atom.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| Atom | $x$       | $y$       | $z$       | $U_{iso}$ $^\star / U_{eq}$ | Occ. (<1) |
|------|-----------|-----------|-----------|------------------------------|-----------|
| O2   | 0.1880 (2) | 0.32007 (19) | 0.36008 (8) | 0.0785 (5)                  |           |
| O3   | 0.5181 (2) | 0.34987 (18) | 0.41423 (7) | 0.0648 (4)                  |           |
| O4   | 0.69301 (19) | 0.57836 (15) | 0.18323 (6) | 0.0575 (3)                  |           |
| O5   | 1.0269 (2) | 0.56449 (19) | 0.22771 (8) | 0.0732 (4)                  |           |
| C15  | 0.7126 (3) | 0.45921 (19) | 0.29059 (8) | 0.0457 (4)                  |           |
| H15  | 0.798508 | 0.415501 | 0.322444 | 0.055* |           |
| C13  | 0.3847 (3) | 0.36674 (19) | 0.36123 (9) | 0.0466 (4)                  |           |
| C14  | 0.4977 (3) | 0.44695 (18) | 0.30158 (8) | 0.0453 (4)                  |           |
| H14  | 0.411762 | 0.491313 | 0.269994 | 0.054* |           |
| C16  | 0.8265 (3) | 0.53832 (18) | 0.23060 (8) | 0.0445 (4)                  |           |
| O1   | 0.3504 (2) | 0.16999 (17) | 0.50962 (7) | 0.0673 (4)                  |           |
| N1   | 0.12900 (19) | 0.08762 (16) | 0.60010 (6) | 0.0421 (3)                  |           |
| N2   | 0.65707 (19) | 0.00872 (15) | 0.85784 (6) | 0.0380 (3)                  |           |
| C2   | 0.0229 (3) | 0.2911 (2) | 0.54231 (10) | 0.0578 (5)                  |           |
| H2A  | $-0.043039$ | 0.281057 | 0.494497 | 0.069* |           |
| H2B  | 0.094453 | 0.399740 | 0.553553 | 0.069* |           |
| C1   | 0.1861 (2) | 0.17907 (19) | 0.54731 (8) | 0.0445 (4)                  |           |
| C3   | $-0.1513 (3)$ | 0.2442 (3) | 0.59673 (12) | 0.0758 (6)                  |           |
| H3A  | $-0.168967$ | 0.334814 | 0.629641 | 0.091* |           |
| H3B  | $-0.293254$ | 0.203854 | 0.572933 | 0.091* |           |
| C4   | $-0.0716 (3)$ | 0.1172 (2) | 0.63662 (10) | 0.0576 (4)                  |           |
| H4A  | $-0.039682$ | 0.153881 | 0.686613 | 0.069* |           |
| H4B  | $-0.181912$ | 0.022144 | 0.633910 | 0.069* |           |
| C5   | 0.2587 (3) | $-0.02922 (19)$ | 0.62190 (8) | 0.0458 (4)                  |           |
| H5A  | 0.370750 | $-0.043844$ | 0.586468 | 0.055* |           |
| H5B  | 0.162152 | $-0.129865$ | 0.623917 | 0.055* |           |
| C6   | 0.3680 (2) | 0.01937 (19) | 0.69249 (8) | 0.0453 (4)                  |           |
| C7   | 0.4569 (3) | 0.0611 (2) | 0.74894 (9) | 0.0476 (4)                  |           |
| C8   | 0.5696 (3) | 0.1275 (2) | 0.81681 (9) | 0.0526 (4)                  |           |
| H8A  | 0.466071 | 0.177765 | 0.846459 | 0.063* |           |
| H8B  | 0.691969 | 0.208802 | 0.806785 | 0.063* |           |
| C9   | 0.8224 (3) | $-0.0784 (2)$ | 0.82116 (8) | 0.0463 (4)                  |           |
| H9A  | 0.963732 | $-0.010026$ | 0.817488 | 0.056* |           |
| H9B  | 0.769788 | $-0.122425$ | 0.773541 | 0.056* |           |
| C10  | 0.8423 (3) | $-0.2081 (3)$ | 0.86974 (11) | 0.0690 (5)                  |           |
| H10A | 0.867468 | $-0.304045$ | 0.841843 | 0.083* |           |
| H10B | 0.964896 | $-0.175271$ | 0.903809 | 0.083* |           |
| C11  | 0.6245 (3) | $-0.2359 (3)$ | 0.90830 (12) | 0.0729 (6)                  |           |
| H11A | 0.548597 | $-0.343053$ | 0.896019 | 0.087* |           |
|        |        |        |        |        |        |        |
|--------|--------|--------|--------|--------|--------|--------|
| H11B   | 0.649915 | -0.220532 | 0.959802 | 0.087*  |        |        |
| C12    | 0.4880 (3) | -0.1170 (2) | 0.88405 (9) | 0.0561 (4) |        |        |
| H12A   | 0.383755 | -0.164457 | 0.845940 | 0.067*  |        |        |
| H12B   | 0.406420 | -0.074741 | 0.923545 | 0.067*  |        |        |
| H4     | 0.773 (3) | 0.632 (3) | 0.1495 (10) | 0.089 (7)* |        |        |
| H3     | 0.440 (4) | 0.291 (3) | 0.4458 (11) | 0.098 (8)* |        |        |
| H2     | 0.724 (3) | 0.068 (2) | 0.8967 (10) | 0.053 (5)* |        |        |
| O6     | 0.8934 (4) | 0.7598 (3) | 0.10050 (12) | 0.0504 (5) | 0.855 (4) |        |
| O7     | 1.1822 (4) | 0.7858 (2) | 0.02939 (11) | 0.0519 (4) | 0.855 (4) |        |
| C17    | 1.0122 (5) | 0.7065 (2) | 0.05155 (12) | 0.0381 (5) | 0.855 (4) |        |
| C18    | 0.9417 (3) | 0.5389 (2) | 0.02174 (10) | 0.0442 (5) | 0.855 (4) |        |
| H4     | 0.773 (3) | 0.632 (3) | 0.1495 (10) | 0.089 (7)* |        |        |
| H3     | 0.440 (4) | 0.291 (3) | 0.4458 (11) | 0.098 (8)* |        |        |
| H2     | 0.724 (3) | 0.068 (2) | 0.8967 (10) | 0.053 (5)* |        |        |
| O6     | 0.8934 (4) | 0.7598 (3) | 0.10050 (12) | 0.0504 (5) | 0.855 (4) |        |
| O7     | 1.1822 (4) | 0.7858 (2) | 0.02939 (11) | 0.0519 (4) | 0.855 (4) |        |
| C17    | 1.0122 (5) | 0.7065 (2) | 0.05155 (12) | 0.0381 (5) | 0.855 (4) |        |
| C18    | 0.9417 (3) | 0.5389 (2) | 0.02174 (10) | 0.0442 (5) | 0.855 (4) |        |

**Atomic displacement parameters (Å²)**

|        |        |        |        |        |        |        |
|--------|--------|--------|--------|--------|--------|--------|
| U₁₁    | 0.0462 (7) | 0.1052 (12) | 0.0848 (10) | -0.0063 (7) | -0.0006 (6) | 0.0461 (9) |
| U₂₂    | 0.0503 (7) | 0.0919 (10) | 0.0556 (7) | 0.0070 (6) | 0.0046 (6) | 0.0340 (7) |
| U₃₃    | 0.0508 (7) | 0.0692 (8) | 0.0536 (7) | 0.0034 (6) | 0.0024 (5) | 0.0224 (6) |
| U₁₂    | 0.0460 (7) | 0.0988 (11) | 0.0808 (9) | 0.0140 (7) | 0.0119 (6) | 0.0357 (8) |
| U₁₃    | 0.0450 (9) | 0.0463 (9) | 0.0464 (9) | 0.0088 (7) | 0.0000 (7) | 0.0097 (7) |
| U₂₃    | 0.0485 (9) | 0.0434 (8) | 0.0455 (8) | 0.0073 (7) | 0.0010 (7) | 0.0118 (7) |
| C2     | 0.0646 (10) | 0.0583 (11) | 0.0530 (10) | 0.0192 (9) | -0.0103 (8) | 0.0070 (8) |
| C1     | 0.0542 (9) | 0.0542 (9) | 0.0372 (8) | 0.0050 (7) | -0.0054 (6) | 0.0096 (7) |
| C3     | 0.0560 (11) | 0.0972 (16) | 0.0831 (14) | 0.0325 (11) | 0.0053 (10) | 0.0203 (12) |
| C4     | 0.0504 (9) | 0.0678 (11) | 0.0563 (10) | 0.0127 (8) | 0.0130 (8) | 0.0077 (8) |
| C5     | 0.0472 (8) | 0.0510 (9) | 0.0409 (8) | 0.0088 (7) | -0.0014 (7) | 0.0115 (7) |
| C6     | 0.0423 (8) | 0.0526 (9) | 0.0438 (9) | 0.0090 (7) | 0.0019 (7) | 0.0178 (7) |
| C7     | 0.0446 (8) | 0.0573 (10) | 0.0439 (9) | 0.0126 (7) | -0.0001 (7) | 0.0138 (7) |
| C8     | 0.0626 (10) | 0.0514 (10) | 0.0463 (9) | 0.0166 (8) | -0.0070 (8) | 0.0060 (7) |
| C9     | 0.0411 (8) | 0.0559 (10) | 0.0412 (8) | 0.0090 (7) | 0.0030 (6) | -0.0020 (7) |
| C10    | 0.0741 (13) | 0.0688 (13) | 0.0702 (12) | 0.0277 (10) | -0.0078 (10) | 0.0112 (10) |
| C11    | 0.0741 (13) | 0.0640 (12) | 0.0794 (14) | -0.0056 (10) | -0.0103 (11) | 0.0305 (11) |
| C12    | 0.0410 (8) | 0.0748 (12) | 0.0499 (9) | -0.0049 (8) | 0.0030 (7) | 0.0164 (8) |
| O6     | 0.0583 (12) | 0.0436 (10) | 0.0486 (13) | 0.0053 (7) | 0.0167 (7) | 0.0044 (8) |
| O7     | 0.0575 (14) | 0.0492 (13) | 0.0408 (7) | -0.0149 (9) | 0.0115 (9) | -0.0009 (9) |
| C17    | 0.0421 (14) | 0.0411 (9) | 0.0297 (10) | 0.0013 (10) | -0.0020 (11) | 0.0048 (7) |
| C18    | 0.0432 (10) | 0.0447 (11) | 0.0402 (10) | -0.0065 (8) | 0.0062 (8) | 0.0030 (8) |
### Geometric parameters (Å, °)

| Bond                  | Distance (Å) | Angle (°) | Bond                  | Distance (Å) | Angle (°) |
|-----------------------|--------------|-----------|-----------------------|--------------|-----------|
| O2—C13                | 1.2026 (19)  |           | C5—H5A                |              |           |
| O3—C13                | 1.310 (2)    |           | C5—H5B                |              |           |
| O3—H3                 | 0.902 (10)   |           | C5—C6                 | 1.473 (2)    |           |
| O4—C16                | 1.3016 (19)  |           | C6—C7                 | 1.185 (2)    |           |
| O4—H4                 | 0.914 (10)   |           | C7—C8                 | 1.467 (2)    |           |
| O5—C16                | 1.2092 (19)  |           | C8—H8A                | 0.9700       |           |
| C15—H15               | 0.9300       |           | C8—H8B                | 0.9700       |           |
| C15—C14               | 1.316 (2)    |           | C9—H9A                | 0.9700       |           |
| C15—C16               | 1.483 (2)    |           | C9—H9B                | 0.9700       |           |
| C13—C14               | 1.480 (2)    |           | C9—C10                | 1.515 (3)    |           |
| C14—H14               | 0.9300       |           | C10—H10A              | 0.9700       |           |
| O1—C1                 | 1.2372 (19)  |           | C10—H10B              | 0.9700       |           |
| N1—C1                 | 1.3293 (19)  |           | C10—C11               | 1.511 (3)    |           |
| N1—C4                 | 1.451 (2)    |           | C11—H11A              | 0.9700       |           |
| N1—C5                 | 1.452 (2)    |           | C11—H11B              | 0.9700       |           |
| N2—C8                 | 1.486 (2)    |           | C11—C12               | 1.509 (3)    |           |
| N2—C9                 | 1.4833 (19)  |           | C12—H12A              | 0.9700       |           |
| N2—C12                | 1.490 (2)    |           | C12—H12B              | 0.9700       |           |
| N2—H2                 | 0.908 (19)   |           | O6—C17                | 1.270 (3)    |           |
| C2—H2A                | 0.9700       |           | O7—C17                | 1.243 (2)    |           |
| C2—H2B                | 0.9700       |           | C17—C18               | 1.492 (3)    |           |
| C2—C1                 | 1.495 (2)    |           | C18—C18               | 1.295 (4)    |           |
| C2—C3                 | 1.510 (3)    |           | C18—H18               | 0.9300       |           |
| C3—H3A                | 0.9700       |           | O6A—C17A              | 1.273 (14)   |           |
| C3—H3B                | 0.9700       |           | O7A—C17A              | 1.222 (14)   |           |
| C3—C4                 | 1.509 (3)    |           | C17A—C18A             | 1.502 (14)   |           |
| C4—H4A                | 0.9700       |           | C18A—C18A             | 1.25 (2)     |           |
| C4—H4B                | 0.9700       |           | C18A—H18A             | 0.9300       |           |
| C13—O3—H3             | 108.7 (16)   |           | H5A—C5—H5B            | 107.9        |           |
| C16—O4—H4             | 110.0 (15)   |           | C6—C5—H5A             | 109.3        |           |
| C14—C15—H15           | 117.9        |           | C6—C5—H5B             | 109.3        |           |
| C14—C15—C16           | 124.13 (15)  |           | C7—C6—C5              | 178.83 (17)  |           |
| C16—C15—H15           | 117.9        |           | C6—C7—C8              | 174.81 (17)  |           |
| O2—C13—O3             | 123.57 (15)  |           | N2—C8—H8A             | 108.7        |           |
| O2—C13—C14            | 122.35 (15)  |           | N2—C8—H8B             | 108.7        |           |
| O3—C13—C14            | 114.08 (14)  |           | C7—C8—N2              | 114.07 (14)  |           |
| C15—C14—C13           | 123.90 (15)  |           | C7—C8—H8A             | 108.7        |           |
| C15—C14—H14           | 118.1        |           | C7—C8—H8B             | 108.7        |           |
| C13—C14—H14           | 118.1        |           | H8A—C8—H8B            | 107.6        |           |
| O4—C16—C15            | 114.39 (13)  |           | N2—C9—H9A             | 111.2        |           |
O5—C16—O4 123.90 (15) N2—C9—H9B 111.2
O5—C16—C15 121.70 (15) N2—C9—C10 102.94 (13)
C1—N1—C4 114.58 (13) H9A—C9—H9B 109.1
C1—N1—C5 123.48 (13) C10—C9—H9B 111.2
C4—N1—C5 121.90 (13) C10—C9—H10A 110.5
C8—N2—C12 116.01 (13) C9—C10—H10B 110.5
C8—N2—H2 103.0 (11) C9—C10—H10B 110.5
C9—N2—C8 116.32 (12) H10A—C10—H10B 108.7
C9—N2—C12 104.70 (13) C11—C10—C9 106.06 (15)
C9—N2—H2 108.6 (11) C11—C10—H10A 110.5
C12—N2—H2 107.8 (11) C11—C10—H10B 110.5
H2A—C2—H2B 108.8 C10—C11—C12 110.5
C1—C2—H2A 110.7 C10—C11—H11A 110.5
C1—C2—H2B 110.7 C10—C11—H11B 110.5
C1—C2—H2A 110.7 H11A—C11—H11B 108.7
C1—C2—C3 105.11 (15) C12—C11—C10 106.27 (15)
C3—C2—H2A 110.7 C12—C11—H11A 110.5
C3—C2—H2B 110.7 C12—C11—H11B 110.5
O1—C1—N1 123.90 (15) N2—C12—C11 103.58 (13)
O1—C1—C2 126.98 (15) N2—C12—H12A 111.0
N1—C1—C2 109.11 (14) N2—C12—H12B 111.0
C2—C3—H3A 110.4 C11—C12—H12A 111.0
C2—C3—H3B 110.4 C11—C12—H12B 111.0
H3A—C3—H3B 108.6 H12A—C12—H12B 109.0
C4—C3—C2 106.81 (15) O6—C17—C18 116.8 (2)
C4—C3—H3A 110.4 O7—C17—O6 123.1 (2)
C4—C3—H3B 110.4 O7—C17—C18 120.1 (2)
N1—C4—C3 104.08 (14) C17—C18—H18 117.8
N1—C4—H4A 110.9 C18—C18—C17 124.4 (2)
N1—C4—H4B 110.9 C18—C18—H18 117.8
C3—C4—H4A 110.9 O6A—C17A—C18A 116.1 (16)
C3—C4—H4B 110.9 O7A—C17A—O6A 123.5 (16)
H4A—C4—H4B 109.0 O7A—C17A—C18A 119.4 (16)
N1—C5—H5A 109.3 C17A—C18A—H18A 114.6
N1—C5—H5B 109.3 C18A—C18A—C17A 130.7 (16)
N1—C5—C6 111.67 (13) C18A—C18A—H18A 114.6
O2—C13—C14—C15 −159.84 (18) C5—N1—C1—O1 2.9 (2)
O3—C13—C14—C15 19.3 (2) C5—N1—C1—C2 −176.77 (14)
C14—C15—C16—O4 −8.4 (2) C5—N1—C4—C3 −179.65 (16)
C14—C15—C16—O5 170.45 (18) C8—N2—C9—C10 −169.44 (14)
C16—C15—C14—C13 179.50 (15) C8—N2—C9—C10 168.12 (15)
N2—C9—C10—C11 25.88 (19) C9—N2—C8—C7 59.62 (19)
C2—C3—C4—N1 −4.8 (2) C9—N2—C12—C11 38.48 (17)
C1—N1—C4—C3 2.4 (2) C9—C10—C11—C12 −2.6 (2)
C1—N1—C5—C6 109.44 (16) C10—C11—C12—N2 −21.6 (2)
C1—C2—C3—C4 5.5 (2) C12—N2—C8—C7 −64.18 (19)
C3—C2—C1—O1 176.15 (18) C12—N2—C9—C10 −39.98 (16)
C3—C2—C1—N1 −4.2 (2) O6—C17—C18—C18A −171.2 (3)
IUCrData (2022). 7, x220364

Symmetry code: (i) $-x+2, -y+1, -z$.

 Hydrogen-bond geometry (Å, °)

| D—H···A  | D—H | H···A | D···A  | D—H···A |
|---------|-----|------|-------|---------|
| C14—H14···O5$^\text{ii}$ | 0.93 | 2.65 | 3.498 (2) | 152 |
| C3—H3B···O1$^\text{ii}$ | 0.97 | 2.43 | 3.381 (3) | 166 |
| C5—H5B···O2$^\text{iii}$ | 0.97 | 2.51 | 3.434 (2) | 159 |
| C8—H8A···O4$^\text{iv}$ | 0.97 | 2.54 | 3.191 (2) | 125 |
| C8—H8A···O6$^\text{iv}$ | 0.97 | 2.53 | 3.456 (3) | 161 |
| C9—H9A···O6$^\text{v}$ | 0.97 | 2.52 | 3.480 (2) | 173 |
| C10—H10A···O5$^\text{vi}$ | 0.97 | 2.66 | 3.624 (3) | 175 |
| C11—H11A···O4$^\text{vii}$ | 0.97 | 2.64 | 3.553 (3) | 157 |
| C12—H12A···O7$^\text{viii}$ | 0.97 | 2.68 | 3.399 (2) | 132 |
| C12—H12A···O7$^\text{A}$ $^\text{viii}$ | 0.97 | 2.37 | 3.139 (15) | 135 |
| O4—H4···O6 | 0.91 (1) | 1.58 (1) | 2.483 (2) | 167 (2) |
| O3—H3···O1 | 0.90 (1) | 1.69 (1) | 2.5739 (16) | 167 (2) |
| N2—H2···O6$^\text{v}$ | 0.908 (19) | 2.554 (18) | 3.172 (3) | 125.8 (14) |
| N2—H2···O7$^\text{v}$ | 0.908 (19) | 1.809 (19) | 2.705 (2) | 168.3 (17) |
| N2—H2···O6$^\text{v}$ | 0.908 (19) | 2.43 (3) | 3.131 (19) | 133.6 (15) |
| N2—H2···O7$^\text{A}$ $^\text{v}$ | 0.908 (19) | 1.61 (2) | 2.489 (15) | 161.7 (17) |

Symmetry codes: (ii) $x-1, y, z$; (iii) $-x, -y, -z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+2, -y, -z+1$; (vi) $-x+2, -y, -z+1$; (vii) $-x+1, -y, -z+1$; (viii) $x-1, y-1, z+1$. 

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data reports

C4—N1—C1—O1 $-179.16$ (16) O7—C17—C18—C18$^\text{i}$ $7.4$ (4) 
C4—N1—C1—C2 $1.17$ (19) O6A—C17A—C18A—C18A$^\text{i}$ $-1$ (3) 
C4—N1—C5—C6 $-68.36$ (19) O7A—C17A—C18A—C18A$^\text{i}$ $-169$ (2)