Structural characterization and antimycobacterial evaluation of a benzimidazole analogue of the antituberculosis clinical drug candidate TBA-7371

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The crystal structure and in vitro antimycobacterial properties of N-(2-fluoroethyl)-1-[(6-methoxy-5-methylpyrimidin-4-yl)methyl]-1H-benzo[d]imidazole-4-carboxamide (C\textsubscript{17}H\textsubscript{18}FN\textsubscript{5}O\textsubscript{2}, I), a previously reported benzimidazole analogue of the 1,4-azaindole-based antituberculosis drug candidate TBA-7371, are reported. The structure determination was achieved using Hirshfeld atom refinement. Compound I crystallizes in the triclinic system (space group \textit{P}\textsubscript{1}) with two molecules in the asymmetric unit (\textit{Z} = 2). The two crystallographically distinct molecules exhibit a similar conformation with the amide groups in a \textit{Z} conformation, forming an intramolecular \textit{N}amide—\textit{H}—\textit{C}\textsubscript{1}/\textsubscript{C}\textsubscript{1}/\textsubscript{C}\textsubscript{1}/\textsubscript{N}benzimidazole hydrogen bond. The most significant supramolecular feature in the solid-state is a relatively short \textit{C}benzimidazole—\textit{H}—\textit{C}\textsubscript{1}/\textsubscript{C}\textsubscript{1}/\textsubscript{C}\textsubscript{1}/\textsubscript{N}pyrimidine hydrogen bond. Antimycobacterial testing confirmed in vitro activity against \textit{Mycobacterium smegmatis}, but no growth inhibition of \textit{Mycobacterium abscessus} was found.

1. Chemical context

TBA-7371 (Fig. 1) is a 1,4-azaindole-based drug candidate for the treatment of tuberculosis, which has advanced to a Phase 2a clinical study (ClinicalTrials.gov identifier: NCT04176250). The compound is a non-covalent inhibitor of the mycobacterial enzyme decaprenylphosphoryl-\textbeta-d-ribose-2'-epimerase (DprE1), which is essential for cell-wall synthesis in \textit{Mycobacterium tuberculosis}, the causative agent of tuberculosis (Shirude \textit{et al.}, 2013, 2014; Chikhale \textit{et al.}, 2018). As
shown in Fig. 1, scaffold morphing, a medicinal chemistry approach to the design of new ligands for the same target with a different core, led to the identification of \(N\)-(2-fluoroethyl)-1-[(6-methoxy-5-methylpyrimidin-4-yl)methyl]-1\(H\)-benzo[\(d\)]imidazole-4-carboxamide (I) (Manjunatha et al., 2019). In I, the 1,4-azaindole core has been replaced by a benzimidazole core, while the 6-methoxy-5-methylpyrimidine-4-yl group and the amide side chain were maintained. Compound I exhibits potent DprE1 inhibition and antimycobacterial activity (vide infra).

Late steps in the synthesis of I, following the previously published route (Manjunatha et al., 2019), are sketched in Fig. 2. Benzimidazole derivative A was reacted with 4-(chloromethyl)-6-methoxy-5-methylpyrimidine to give B. It is worth mentioning that N-alkylation in part occurred at position 3 of the benzimidazole scaffold, affording side product C. Regioisomers B and C were separated by flash chromatography, resulting in an approximate 3.75:1 ratio.

Compound C was identified by \(^1H\) and \(^{13}C\) NMR spectroscopy and APCI mass spectrometry (see Supporting Information). Hydrolysis of B followed by amide coupling with 2-fluoroethanamine gave the target compound I. X-ray crystallography unambiguously confirmed the structure.

2. Structural commentary

Compound I crystallizes in the triclinic space group \(P\bar{T}\) with two crystallographically distinct molecules (Fig. 3). In both molecules, the tilt of the 6-methoxy-5-methylpyrimidin-4-yl group of the plane out of the central benzimidazole moiety renders the conformers axially chiral. The \(C2—N1—C11—C12\) torsion angle is 101.9 (1)° in molecule 1 and 79.0 (1)° in molecule 2. The enantiomeric conformers in the chosen asymmetric unit thus exhibit the same handedness, but the corresponding oppositely handed conformers are present in the centrosymmetric crystal structure. The most marked structural difference between the two unique molecules is the orientation of the 2-fluoroethyl group about the \(C9—C10\) bond with \(N2—C9—C10—F1 = 68.1 (1)°\) for molecule 1 and \(61.8 (1)°\) for molecule 2.

The plane of the amide group and the mean plane of the benzimidazole moiety are nearly co-planar in molecules 1 and 2. The angle between the two planes is 8.8 (1)° in molecule 1 and 7.7 (1)° in molecule 2. The amide group adopts a Z conformation in both molecules and forms an intramolecular N—H···H hydrogen bond to atom N3 of the benzimidazole system (Table 1), resulting in a six-membered hydrogen-bonded ring with an \(S(6)\) motif (Bernstein et al., 1995). This is in line with Etter’s second hydrogen-bond rule for organic compounds, which states that intramolecular six-membered

![Figure 2](image_url)

**Figure 2**

Synthesis of I, following the published procedure (Manjunatha et al., 2019). Reagents and solvents: (a) 4-(chloromethyl)-6-methoxy-5-methylpyrimidine, Cs\(_2\)CO\(_3\), NaI, DMF; (b) LiOH, MeOH; (c) HATU, 2-fluoroethanamine, NMP.

![Figure 3](image_url)

**Figure 3**

Asymmetric unit of I. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by small spheres of arbitrary radius. Dashed lines represent hydrogen bonds. The number after the underscore indicates unique molecule 1 or 2.
hydrogen-bonded rings form in preference to intermolecular hydrogen bonds (Etter, 1990).

3. Supramolecular features

The most significant supramolecular feature of the title compound’s solid-state structure is a short C—H⋯N contact between the amidine C2—H2 group of the benzimidazole moiety in molecule 2 and N5 of the pyrimidine ring in molecule 1 (Fig. 3), which provides structural evidence for a C—H⋯N weak hydrogen bond (Table 1). The amidine C2—H2 group in molecule 1 forms a short C—H⋯O contact to the amide carbonyl group of molecule 2. The geometric parameters including a D—H⋯A angle $>140^\circ$ (Wood et al., 2009) are characteristic of a weak hydrogen bond (Thakuria et al., 2017). F⋯F interactions are not encountered in the crystal structure, but parallel arrangements between the pyrimidine ring of molecule 1 and the benzimidazole moiety of a neighbouring molecule 2 (Fig. 4) and between the benzimidazole moieties of two molecules 1 about a center of symmetry are notable features (Fig. 5). The latter and the stacking of these units with the pyrimidine rings of molecule 2 in the b* axis direction no doubt contribute to the 040 reflection having by far the strongest intensity in the diffraction data set. A packing index of 71.9% (Kitaigorodskii, 1973), as calculated with PLATON (Spek, 2020), suggests that the solid-state structure appears to be mainly governed by close packing.

4. Database survey

A search of the Cambridge Structural Database (CSD; Groom et al., 2016) for acyclic 1-alkyl benzimidazole-4-carboxamides via WebCSD (accessed on 21 October 2022; CCDC, 2017) yielded the structure of 1-(2,6-difluorobenzyl)-2-(2,6-dimethoxy-5-methylpyrimidin-4-yl group and 6-methoxy-5-methylpyrimidin moieties in an AABB fashion in the b* axis direction. Dashed lines represent hydrogen bonds. H atoms have been omitted for clarity, except for amide and amidine H atoms.

5. Antimycobacterial evaluation

Manjunatha et al. (2019) reported an in vitro minimal inhibitory concentration (MIC) of 1.56–3.12 μM for 1 against M. tuberculosis H37Rv and MIC 0.78–1.56 μM against Mycobacterium smegmatis. Potent inhibition of the M. tuberculosis DprE1 and molecular docking suggested a mode of action similar to TBA-7371. We re-evaluated the in vitro activity of 1 against M. smegmatis mc2 155, using broth microdilution assays (for the assay protocols, see supporting information and Richter et al., 2018). We determined a MIC90 of 12.5 μM in Middlebrook 7H9 medium supplemented with 10% ADS (albumin-dextrose-saline) and 0.05% polysorbate 80, and...
indole TBA-7371 (Sarathy et al., 2022) showed moderate activity against several M. abscessus strains and clinical isolates for the 3,4-dihydrocarbostyril-based non-covalent DprE1 inhibitor and Phase 2b/c clinical antituberculosis drug candidate OPC-167832.

6.25 μM in Mueller Hinton II Broth with 0.05% polysorbate 80.

The non-tuberculous Mycobacterium abscessus is an opportunistic pathogen, which can cause difficult-to-treat skin, soft tissue and pulmonary infections, in particular in patients with structural lung diseases such as cystic fibrosis (Boudehen et al., 2021). Screening of antitubercular agents for activity against M. abscessus has been proposed (Ganapathy & Dick, 2022). Mechanism-based covalent DprE1 inhibitors with potent activity against M. tuberculosis and other mycobacteria like M. smegmatis form covalent adducts with the thiol group of Cys387 on the FAD substrate binding domain (Shetye et al., 2020). These compounds are usually inactive against M. abscessus, since the M. abscessus DprE1 has an alanine residue in the corresponding amino-acid position, which prevents covalent linkage. Testing of non-covalent DprE1 inhibitors against M. abscessus, however, could be a promising approach to identifying potential lead structures. Therefore, we also tested 1 against M. abscessus ATCC19977 in vitro. In both Middlebrook 7H9 medium supplemented with 10% ADS and 0.05% polysorbate 80 and Mueller Hinton II Broth with 0.05% polysorbate 80, however, no growth inhibition could be detected (MIC₉₀ > 100 μM). While this work was in progress, the same observation was reported for the parent 1,4-azaindole TBA-7371 (Sarathy et al., 2022). It is worth noting, however, that Sarathy et al. (2022) found moderate in vitro activity against several M. abscessus strains and clinical isolates for the 3,4-dihydrocarbostyril-based non-covalent DprE1 inhibitor and Phase 2b/c clinical antituberculosis drug candidate OPC-167832.

6. Synthesis and crystallization

Compound 1 was synthesized as described by Manjunatha et al. (2019). Analytical data for A, B, C and 1 can be found in the supporting information. Crystals of 1 suitable for X-ray diffraction were grown from a solution in ethyl acetate/n-heptane (1:1) by slow evaporation of the solvents at room temperature.

7. Refinement

Initially, the structure was refined to convergence using independent atom model refinement with SHELXL2018/3 (Sheldrick, 2015b). The final structure refinement was carried out by Hirshfeld atom refinement with aspherical scattering factors using NospherA2 (Kleemiss et al., 2021; Midgley et al., 2021) partitioning in OLEX2 (Dolomanov et al., 2009) based on electron density from iterative single-determinant SCF single-point DFT calculations using ORCA (Neese et al., 2020) with a B3LYP functional (Becke, 1993; Lee et al., 1988) and a def2-TZVPP basis set. Crystal data, data collection and structure refinement details are summarized in Table 2.

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**Table 2**

| Crystal data                              | C₁₇H₁₈FN₅O₂ |
|-------------------------------------------|-------------|
| Chemical formula                          |             |
| Mₘ                                         | 343.36      |
| Crystal system, space group               | Triclinic, P� |
| Temperature (K)                           | 100         |
| a, b, c (Å)                               | 7.6940 (19), 15.013 (4), 15.281 (4) |
| α, β, γ (°)                               | 71.040 (4), 77.874 (5), 87.780 (4) |
| V (Å³)                                    | 1631.3 (7)  |
| Z                                          | 4           |
| Radiation type                            | Mo Kα       |
| μ (mm⁻¹)                                  | 0.10        |
| Crystal size (mm)                         | 0.10 × 0.05 × 0.02 |

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**Figure 6**

Structure overlay of the benzene rings of the two unique molecules of 1 (molecule 1: green; molecule 2: orange) and the benzene ring of CT319 in the crystal structure of its non-covalent complex with M. tuberculosis DprE1 (pink; PDB code: 4FDQ; resolution: 2.4 Å), showing the similar conformations of the benzamide moieties.
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Computing details
Data collection: APEX3 (Bruker, 2017); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT (Bruker, 2004); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: olex2.refine (Bourhis et al., 2015); molecular graphics: DIAMOND (Brandenburg, 2018) and Mercury (Macrae et al., 2020); software used to prepare material for publication: enCIFer (Allen et al., 2004) and pubICIF (Westrip, 2010).

N-(2-Fluoroethyl)-1-[(6-methoxy-5-methylpyrimidin-4-yl)methyl]-1,3-benzodiazole-4-carboxamide

Crystal data
C_{17}H_{18}FN_{5}O_{2}  
Mr = 343.36
Triclinic, P\textoverline{1}  
a = 7.6940 (19) Å  
b = 15.013 (4) Å  
c = 15.281 (4) Å  
α = 71.040 (4)°  
β = 77.874 (5)°  
γ = 87.780 (4)°  
V = 1631.3 (7) Å³

Data collection
Bruker AXS Kappa Mach3 APEX II diffractometer  
Radiation source: Incoatec I\textalpha S  
Incoatec Helios mirrors monochromator  
Detector resolution: 66.67 pixels mm⁻¹
φ- and ω-scans  
Absorption correction: gaussian
(SADABS; Krause et al., 2015)  
T_{\text{min}} = 0.994, T_{\text{max}} = 0.999

Refinement
Refinement on F^2  
Least-squares matrix: full  
R[F^2 > 2\sigma(F^2)] = 0.031
wR(F^2) = 0.072
S = 1.07
8147 reflections  
595 parameters  
0 restraints  
0 constraints  
Primary atom site location: dual  
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined

\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0302P)^2 + 0.1357P} \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

\( \Delta/\sigma \) max = −0.001
\( \Delta \rho_{\text{max}} = 0.34 \text{ e Å}^{-3} \)
\( \Delta \rho_{\text{min}} = −0.32 \text{ e Å}^{-3} \)

Special details

Experimental. Crystal mounted on a MiTeGen loop using Perfluoropolyether PFO-XR75.

Refinement. Refinement of \( F^2 \) against ALL reflections. The weighted R-factor \( wR \) and goodness of fit \( S \) are based on \( F^2 \), conventional R-factors \( R \) are based on \( F \), with \( F \) set to zero for negative \( F^2 \). The threshold expression of \( F^2 > 2\sigma(F^2) \) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on \( F^2 \) are statistically about twice as large as those based on \( F \), and R- factors based on ALL data will be even larger.

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

|     | x     | y     | z     | Uiso/\(U_{eq}\) |
|-----|-------|-------|-------|-----------------|
| C2_1| 0.24678 (14) | 0.11875 (7) | 0.50249 (7) | 0.0171 (2) |
| H2_1| 0.1399 (16) | 0.1169 (8) | 0.4698 (8) | 0.033 (3)* |
| C3A_1| 0.40214 (12) | 0.12795 (6) | 0.60043 (7) | 0.01224 (19) |
| C4_1| 0.46666 (13) | 0.12885 (6) | 0.67969 (7) | 0.0138 (2) |
| C5_1| 0.64993 (14) | 0.12841 (7) | 0.67153 (8) | 0.0188 (2) |
| H5_1| 0.7017 (16) | 0.1288 (9) | 0.7320 (9) | 0.037 (3)* |
| C6_1| 0.76757 (14) | 0.12644 (8) | 0.58895 (8) | 0.0223 (2) |
| H6_1| 0.9064 (17) | 0.1261 (8) | 0.5876 (9) | 0.040 (3)* |
| C7_1| 0.70606 (13) | 0.12396 (7) | 0.51032 (8) | 0.0187 (2) |
| H7_1| 0.7946 (16) | 0.1207 (8) | 0.4507 (9) | 0.038 (3)* |
| C7A_1| 0.52267 (12) | 0.12461 (6) | 0.51864 (7) | 0.0131 (2) |
| C8_1| 0.35297 (14) | 0.12221 (7) | 0.77376 (7) | 0.0175 (2) |
| C9_1| 0.05871 (17) | 0.09121 (8) | 0.87354 (8) | 0.0265 (3) |
| H9a_1| 0.1274 (19) | 0.0422 (11) | 0.9242 (10) | 0.057 (4)* |
| H9b_1| −0.0673 (19) | 0.0628 (10) | 0.8694 (10) | 0.050 (4)* |
| C10_1| 0.01296 (17) | 0.17191 (8) | 0.91114 (8) | 0.0258 (3) |
| H10a_1| −0.0661 (19) | 0.1461 (10) | 0.9831 (11) | 0.057 (4)* |
| H10b_1| 0.1311 (18) | 0.2145 (9) | 0.9030 (9) | 0.047 (4)* |
| C11_1| 0.48068 (16) | 0.10678 (7) | 0.36547 (7) | 0.0188 (2) |
| H11a_1| 0.3719 (18) | 0.0773 (9) | 0.3485 (9) | 0.044 (4)* |
| H11b_1| 0.5883 (18) | 0.0590 (9) | 0.3682 (9) | 0.048 (4)* |
| C12_1| 0.54232 (13) | 0.19816 (6) | 0.28726 (7) | 0.01278 (19) |
| C13_1| 0.66938 (12) | 0.19801 (6) | 0.20828 (7) | 0.01218 (19) |
| C14_1| 0.70948 (12) | 0.28746 (6) | 0.13868 (6) | 0.01223 (19) |
| C15_1| 0.51185 (13) | 0.35548 (7) | 0.22720 (7) | 0.0151 (2) |
| H15_1| 0.4475 (15) | 0.4202 (8) | 0.2342 (8) | 0.031 (3)* |
| C16_1| 0.75853 (15) | 0.11175 (7) | 0.19506 (8) | 0.0172 (2) |
| H16a_1| 0.8798 (19) | 0.1290 (10) | 0.1421 (10) | 0.054 (4)* |
| H16b_1| 0.8024 (18) | 0.0689 (10) | 0.2556 (10) | 0.052 (4)* |
| H16c_1| 0.677 (2) | 0.0719 (11) | 0.1756 (10) | 0.061 (4)* |
| C17_1| 0.88043 (14) | 0.38333 (7) | −0.00710 (7) | 0.0181 (2) |
| H17a_1| 0.9829 (17) | 0.3724 (8) | −0.0611 (9) | 0.040 (3)* |
|  |  |  |  |  |
|---|---|---|---|---|
| H17b_1 | 0.7668 (16) | 0.4161 (8) | −0.0362 (8) | 0.033 (3)* |
| H17c_1 | 0.9341 (16) | 0.4290 (9) | 0.0248 (9) | 0.037 (3)* |
| N1_1 | 0.41820 (11) | 0.11800 (5) | 0.45741 (5) | 0.01466 (17) |
| N2_1 | 0.17564 (12) | 0.11793 (6) | 0.78099 (6) | 0.0202 (2) |
| N2a_1 | 0.1293 (17) | 0.1211 (9) | 0.7235 (10) | 0.036 (4)* |
| N3_1 | 0.22925 (10) | 0.12443 (6) | 0.58814 (6) | 0.01580 (18) |
| N4_1 | 0.46198 (11) | 0.27632 (6) | 0.29767 (6) | 0.01515 (18) |
| N5_1 | 0.63354 (10) | 0.36553 (5) | 0.14786 (6) | 0.01407 (17) |
| O1_1 | 0.41907 (11) | 0.11614 (5) | 0.84196 (5) | 0.02732 (19) |
| O2_1 | 0.83091 (9) | 0.29152 (5) | 0.06084 (5) | 0.01625 (15) |
| F1_1 | −0.09275 (9) | 0.23342 (5) | 0.85713 (5) | 0.0407 (2) |
| C2_2 | 0.63497 (13) | 0.58977 (7) | 0.02129 (7) | 0.0161 (2) |
| H2_2 | 0.6202 (16) | 0.5177 (9) | 0.0635 (9) | 0.038 (3)* |
| C3A_2 | 0.71862 (12) | 0.72002 (6) | −0.08890 (7) | 0.01256 (19) |
| C4_2 | 0.79584 (12) | 0.79156 (7) | −0.17228 (7) | 0.0130 (2) |
| C5_2 | 0.75312 (13) | 0.88396 (7) | −0.17785 (7) | 0.0161 (2) |
| H5_2 | 0.8156 (16) | 0.90545 (7) | −0.10381 (7) | 0.0188 (2) |
| C6_2 | 0.6057 (16) | 0.9770 (9) | −0.1100 (8) | 0.036 (3)* |
| C7_2 | 0.55929 (14) | 0.83519 (7) | −0.02112 (7) | 0.0171 (2) |
| H7_2 | 0.4735 (16) | 0.8528 (8) | 0.0344 (8) | 0.034 (3)* |
| C7A_2 | 0.60227 (12) | 0.74268 (7) | −0.01578 (7) | 0.0140 (2) |
| C8_2 | 0.91827 (12) | 0.77332 (7) | −0.25397 (7) | 0.0138 (2) |
| C9_2 | 1.08197 (14) | 0.65409 (8) | −0.31197 (8) | 0.0188 (2) |
| C10_2 | 1.1513 (16) | 0.5933 (9) | −0.2806 (9) | 0.039 (3)* |
| H9a_2 | 1.1762 (16) | 0.7109 (9) | −0.3576 (9) | 0.035 (3)* |
| C10b_2 | 0.97648 (16) | 0.62865 (9) | −0.37364 (8) | 0.0269 (3) |
| C11_2 | 0.8958 (17) | 0.6864 (9) | −0.4029 (9) | 0.044 (3)* |
| C11b_2 | 1.0606 (18) | 0.6044 (9) | −0.4256 (10) | 0.054 (4)* |
| C11a_2 | 0.43832 (14) | 0.64282 (8) | 0.14634 (7) | 0.0192 (2) |
| C11bb_2 | 0.3521 (16) | 0.5797 (9) | 0.1648 (9) | 0.039 (3)* |
| C12_2 | 0.3542 (16) | 0.7039 (9) | 0.1409 (9) | 0.038 (3)* |
| C12a_2 | 0.54170 (13) | 0.63169 (7) | 0.22336 (7) | 0.0154 (2) |
| C13_2 | 0.45344 (13) | 0.63352 (7) | 0.31175 (7) | 0.0161 (2) |
| C13_2a | 0.56317 (15) | 0.62089 (7) | 0.37788 (7) | 0.0201 (2) |
| C15_2 | 0.80399 (15) | 0.60741 (8) | 0.27110 (8) | 0.0257 (3) |
| H15_2 | 0.9430 (17) | 0.5958 (9) | 0.2557 (9) | 0.043 (3)* |
| C16_2 | 0.25762 (15) | 0.64610 (8) | 0.33854 (8) | 0.0215 (2) |
| C16a_2 | 0.2278 (19) | 0.6794 (10) | 0.3907 (11) | 0.061 (4)* |
| C16b_2 | 0.2055 (19) | 0.6939 (11) | 0.2826 (11) | 0.063 (4)* |
| C16c_2 | 0.187 (2) | 0.5814 (12) | 0.3640 (11) | 0.075 (5)* |
| C17_2 | 0.5973 (2) | 0.61725 (10) | 0.52889 (10) | 0.0379 (3) |
| H17a_2 | 0.513 (2) | 0.6183 (12) | 0.5923 (13) | 0.078 (5)* |
| H17b_2 | 0.6689 (18) | 0.5546 (10) | 0.5403 (10) | 0.053 (4)* |
| H17c_2 | 0.691 (2) | 0.6769 (12) | 0.5007 (11) | 0.063 (4)* |
| N1_2 | 0.54919 (11) | 0.65749 (6) | 0.05356 (6) | 0.01600 (18) |
| N2_2 | 0.97000 (11) | 0.68391 (6) | −0.23867 (6) | 0.01570 (18) |
| H2a_2 | 0.9130 (17) | 0.6344 (9) | −0.1783 (10) | 0.034 (3)* |
N3_2  0.73649 (10)  0.62313 (5)  -0.06356 (6)  0.01434 (17)
N4_2  0.71695 (11)  0.61835 (6)  0.20201 (6)  0.0216 (2)
N5_2  0.73631 (12)  0.60823 (6)  0.35843 (6)  0.0252 (2)
O1_2  0.96957 (9)  0.83717 (5)  -0.32943 (5)  0.01908 (16)
O2_2  0.48417 (11)  0.62231 (5)  0.46393 (5)  0.02792 (19)
F1_2   0.85963 (9)  0.55284 (5)  -0.31786 (5)  0.03320 (17)

Atomic displacement parameters (Å²)

|   | U¹¹ | U²² | U³³ | U¹² | U¹³ | U²³ |
|---|-----|-----|-----|-----|-----|-----|
|C2_1 | 0.0170 (5) | 0.0220 (5) | 0.0124 (5) | 0.0007 (4) | -0.0054 (4) | -0.0044 (4) |
|C3A_1 | 0.0134 (5) | 0.0129 (4) | 0.0095 (5) | 0.0004 (4) | -0.0016 (4) | -0.0029 (4) |
|C4_1 | 0.0163 (5) | 0.0139 (5) | 0.0113 (5) | 0.0005 (4) | -0.0031 (4) | -0.0041 (4) |
|C5_1 | 0.0171 (5) | 0.0217 (5) | 0.0184 (6) | -0.0004 (4) | -0.0067 (4) | -0.0057 (4) |
|C6_1 | 0.0120 (5) | 0.0290 (6) | 0.0242 (6) | -0.0018 (4) | -0.0022 (4) | -0.0071 (5) |
|C7_1 | 0.0138 (5) | 0.0211 (5) | 0.0174 (5) | -0.0009 (4) | 0.0025 (4) | -0.0045 (4) |
|C7A_1 | 0.0140 (5) | 0.0130 (4) | 0.0100 (5) | -0.0002 (4) | 0.0005 (4) | -0.0024 (4) |
|C8_1 | 0.0251 (6) | 0.0171 (5) | 0.0112 (5) | 0.0044 (4) | -0.0043 (4) | -0.0059 (4) |
|C9_1 | 0.0330 (7) | 0.0225 (6) | 0.0184 (6) | 0.0017 (5) | 0.0070 (5) | -0.0068 (5) |
|C10_1 | 0.0307 (7) | 0.0269 (6) | 0.0178 (6) | 0.0104 (5) | 0.0001 (5) | -0.0088 (5) |
|C11_1 | 0.0304 (6) | 0.0133 (5) | 0.0103 (5) | -0.0028 (4) | 0.0001 (4) | -0.0030 (4) |
|C12_1 | 0.0182 (5) | 0.0107 (4) | 0.0090 (5) | -0.0004 (4) | -0.0029 (4) | -0.0026 (4) |
|C13_1 | 0.0141 (5) | 0.0120 (5) | 0.0104 (5) | -0.0004 (4) | -0.0017 (4) | -0.0039 (4) |
|C14_1 | 0.0141 (4) | 0.0127 (5) | 0.0097 (5) | -0.0003 (4) | -0.0026 (4) | -0.0032 (4) |
|C15_1 | 0.0187 (5) | 0.0126 (5) | 0.0122 (5) | 0.0021 (4) | -0.0008 (4) | -0.0031 (4) |
|C16_1 | 0.0198 (5) | 0.0142 (5) | 0.0180 (6) | 0.0025 (4) | -0.0038 (5) | -0.0061 (4) |
|C17_1 | 0.0174 (5) | 0.0197 (5) | 0.0137 (5) | -0.0031 (4) | 0.0004 (4) | -0.0025 (4) |
|N1_1 | 0.0194 (4) | 0.0146 (4) | 0.0086 (4) | -0.0007 (3) | -0.0010 (3) | -0.0029 (3) |
|N2_1 | 0.0228 (5) | 0.0221 (5) | 0.0142 (5) | 0.0016 (4) | 0.0011 (4) | -0.0071 (4) |
|N3_1 | 0.0198 (4) | 0.0130 (4) | 0.0116 (4) | 0.0009 (3) | -0.0005 (3) | -0.0039 (3) |
|N4_1 | 0.0195 (4) | 0.0127 (5) | 0.0132 (5) | -0.0111 (3) | 0.0013 (3) | -0.0044 (3) |
|C2_2 | 0.0188 (5) | 0.0155 (5) | 0.0119 (5) | 0.0025 (4) | -0.0030 (4) | -0.0017 (4) |
|C3A_2 | 0.0144 (5) | 0.0129 (5) | 0.0102 (5) | 0.0018 (4) | -0.0036 (4) | -0.0031 (4) |
|C4_2 | 0.0147 (5) | 0.0132 (5) | 0.0113 (5) | 0.0015 (4) | -0.0034 (4) | -0.0038 (4) |
|C5_2 | 0.0201 (5) | 0.0125 (5) | 0.0166 (5) | 0.0007 (4) | -0.0058 (4) | -0.0048 (4) |
|C6_2 | 0.0226 (5) | 0.0157 (5) | 0.0211 (6) | 0.0041 (4) | -0.0074 (4) | -0.0089 (4) |
|C7_2 | 0.0196 (5) | 0.0204 (5) | 0.0156 (5) | 0.0057 (4) | -0.0063 (4) | -0.0105 (4) |
|C7A_2 | 0.0161 (5) | 0.0168 (5) | 0.0106 (5) | 0.0041 (4) | -0.0046 (4) | -0.0059 (4) |
|C8_2 | 0.0150 (5) | 0.0150 (5) | 0.0111 (5) | -0.0010 (4) | -0.0027 (4) | -0.0035 (4) |
|C9_2 | 0.0164 (5) | 0.0215 (6) | 0.0190 (5) | 0.0007 (4) | -0.0011 (4) | -0.0089 (5) |
|C10_2 | 0.0316 (6) | 0.0330 (7) | 0.0236 (6) | 0.0063 (5) | -0.0092 (5) | -0.0176 (5) |
|C11_2 | 0.0171 (5) | 0.0295 (6) | 0.0107 (5) | 0.0041 (5) | -0.0047 (4) | -0.0053 (4) |
|C12_2 | 0.0172 (5) | 0.0181 (5) | 0.0111 (5) | 0.0019 (4) | -0.0051 (4) | -0.0035 (4) |
|C13_2 | 0.0224 (5) | 0.0151 (5) | 0.0116 (5) | -0.0004 (4) | -0.0055 (4) | -0.0041 (4) |
### Geometric parameters (Å, °)

| Bond/Distance | Value (Å) | Error | Value (Å) | Error |
|---------------|----------|-------|----------|-------|
| C2_1—H2_1    | 1.054    | 0.001 | C2_2—H2_2 | 1.060 |
| C2_1—N1_1    | 1.355 | 0.002 | C2_2—N1_2 | 1.359 |
| C2_1—N3_1    | 1.317 | 0.001 | C2_2—N3_2 | 1.313 |
| C3A_1—C4_1   | 1.406 | 0.002 | C3A_2—C4_2 | 1.404 |
| C3A_1—C7A_1  | 1.405 | 0.002 | C3A_2—C7A_2 | 1.404 |
| C3A_1—N3_1   | 1.387 | 0.001 | C3A_2—N3_2 | 1.387 |
| C4_1—C5_1    | 1.389 | 0.002 | C4_2—C5_2 | 1.392 |
| C4_1—C8_1    | 1.492 | 0.002 | C4_2—C8_2 | 1.492 |
| C5_1—H5_1    | 1.082 | 0.001 | C5_2—H5_2 | 1.101 |
| C5_1—C6_1    | 1.398 | 0.002 | C5_2—C6_2 | 1.402 |
| C6_1—H6_1    | 1.064 | 0.001 | C6_2—H6_2 | 1.070 |
| C6_1—C7_1    | 1.392 | 0.002 | C6_2—C7_2 | 1.391 |
| C7_1—H7_1    | 1.031 | 0.001 | C7_2—H7_2 | 1.059 |
| C7_1—C7A_1   | 1.389 | 0.002 | C7_2—C7A_2 | 1.393 |
| C7A_1—N1_1   | 1.383 | 0.001 | C7A_2—N1_2 | 1.381 |
| C8_1—N2_1    | 1.347 | 0.002 | C8_2—N2_2 | 1.344 |
| C8_1—O1_1    | 1.229 | 0.001 | C8_2—O1_2 | 1.234 |
| C9_1—H9a_1   | 1.092 | 0.001 | C9_2—H9a_2 | 1.064 |
| C9_1—H9b_1   | 1.096 | 0.001 | C9_2—H9b_2 | 1.090 |
| C9_1—C10_1   | 1.502 | 0.002 | C9_2—C10_2 | 1.507 |
| C9_1—N2_1    | 1.447 | 0.002 | C9_2—N2_2 | 1.445 |
| C10_1—H10a_1 | 1.091 | 0.001 | C10_2—H10a_2 | 1.078 |
| C10_1—H10b_1 | 1.094 | 0.001 | C10_2—H10b_2 | 1.066 |
| C10_1—F1_1   | 1.387 | 0.001 | C10_2—F1_2 | 1.389 |
| C11_1—H11a_1 | 1.076 | 0.001 | C11_2—H11a_2 | 1.102 |
| C11_1—H11b_1 | 1.073 | 0.001 | C11_2—H11b_2 | 1.092 |
| C11_1—C12_1  | 1.510 | 0.002 | C11_2—C12_2 | 1.516 |
| C11_1—N1_1   | 1.448 | 0.001 | C11_2—N1_2 | 1.445 |
| C12_1—C13_1  | 1.384 | 0.001 | C12_2—C13_2 | 1.386 |
| C12_1—N4_1   | 1.344 | 0.002 | C12_2—N4_2 | 1.342 |
| C13_1—C14_1  | 1.412 | 0.001 | C13_2—C14_2 | 1.411 |
| C13_1—C16_1  | 1.493 | 0.002 | C13_2—C16_2 | 1.497 |
| C14_1—N5_1   | 1.324 | 0.002 | C14_2—N5_2 | 1.322 |
| Bond                  | Dist(Å) (E) | Bond                  | Dist(Å) (E) |
|----------------------|-------------|----------------------|-------------|
| C14_1—O2_1           | 1.3349(11)  | C14_2—O2_2           | 1.3344(13)  |
| C15_1—H15_1          | 1.100(11)   | C15_2—H15_2          | 1.066(13)   |
| C15_1—N4_1           | 1.3227(13)  | C15_2—N4_2           | 1.3289(14)  |
| C15_1—N5_1           | 1.3358(13)  | C15_2—N5_2           | 1.3305(15)  |
| C16_1—H16a_1         | 1.076(14)   | C16_2—H16a_2         | 1.055(16)   |
| C16_1—H16b_1         | 1.055(14)   | C16_2—H16b_2         | 1.063(16)   |
| C16_1—H16c_1         | 1.032(16)   | C16_2—H16c_2         | 1.047(17)   |
| C17_1—H17a_1         | 1.065(13)   | C17_2—H17a_2         | 1.051(17)   |
| C17_1—H17b_1         | 1.092(12)   | C17_2—H17b_2         | 1.055(14)   |
| C17_1—H17c_1         | 1.096(13)   | C17_2—H17c_2         | 1.083(16)   |
| C17_1—O2_1           | 1.4378(12)  | C17_2—O2_2           | 1.4341(15)  |
| N2_1—H2a_1           | 1.002(14)   | N2_2—H2a_2           | 1.002(14)   |
| N1_1—C2_1—H2_1       | 121.8(6)    | N1_2—C2_2—H2_2       | 121.1(7)    |
| N3_1—C2_1—H2_1       | 124.5(6)    | N3_2—C2_2—H2_2       | 125.3(7)    |
| N3_1—C2_1—N1_1       | 113.70(9)   | N3_2—C2_2—N1_2       | 113.60(9)   |
| C7A_1—C3A_1—C4_1     | 119.54(8)   | C7A_2—C3A_2—C4_2     | 120.12(8)   |
| N3_1—C3A_1—C4_1      | 130.61(9)   | N3_2—C3A_2—C4_2      | 130.02(9)   |
| N3_1—C3A_1—C7A_1     | 109.75(8)   | N3_2—C3A_2—C7A_2     | 109.86(8)   |
| C5_1—C4_1—C3A_1      | 117.17(9)   | C5_2—C4_2—C3A_2      | 117.23(9)   |
| C8_1—C4_1—C3A_1      | 124.80(9)   | C8_2—C4_2—C3A_2      | 123.55(8)   |
| C8_1—C4_1—C5_1       | 117.82(9)   | C8_2—C4_2—C5_2       | 119.22(9)   |
| H5_1—C5_1—C4_1       | 118.1(6)    | H5_2—C5_2—C4_2       | 117.9(6)    |
| C6_1—C5_1—C4_1       | 122.32(10)  | C6_2—C5_2—C4_2       | 121.84(9)   |
| C6_1—C5_1—H5_1       | 119.6(6)    | C6_2—C5_2—H5_2       | 120.2(6)    |
| H6_1—C6_1—C5_1       | 118.4(7)    | H6_2—C6_2—C5_2       | 120.5(6)    |
| C7_1—C6_1—C5_1       | 121.32(10)  | C7_2—C6_2—C5_2       | 121.47(9)   |
| C7_1—C6_1—H6_1       | 120.3(7)    | C7_2—C6_2—H6_2       | 118.0(6)    |
| H7_1—C7_1—C6_1       | 120.2(7)    | H7_2—C7_2—C6_2       | 120.4(6)    |
| C7A_1—C7_1—C6_1      | 116.21(10)  | C7A_2—C7_2—C6_2      | 116.54(9)   |
| C7A_1—C7_1—H7_1      | 123.5(7)    | C7A_2—C7_2—H7_2      | 123.1(6)    |
| C7_1—C7A_1—C3A_1     | 123.43(9)   | C7_2—C7A_2—C3A_2     | 122.79(9)   |
| N1_1—C7A_1—C3A_1     | 105.24(8)   | N1_2—C7A_2—C3A_2     | 105.16(8)   |
| N1_1—C7A_1—C7_1      | 131.26(9)   | N1_2—C7A_2—C7_2      | 132.05(9)   |
| N2_1—C8_1—C4_1       | 116.58(9)   | N2_2—C8_2—C4_2       | 115.46(8)   |
| O1_1—C8_1—C4_1       | 121.17(9)   | O1_2—C8_2—C4_2       | 121.45(8)   |
| O1_1—C8_1—N2_1       | 122.15(10)  | O1_2—C8_2—N2_2       | 123.08(9)   |
| H9b_1—C9_1—C9a_1     | 114.0(11)   | H9b_2—C9_2—C9a_2     | 110.2(9)    |
| C10_1—C9_1—C9a_1     | 105.0(8)    | C10_2—C9_2—C9a_2     | 107.5(7)    |
| C10_1—C9_1—H9b_1     | 106.5(7)    | C10_2—C9_2—H9b_2     | 107.5(6)    |
| N2_1—C9_1—C9a_1      | 108.6(7)    | N2_2—C9_2—C9a_2      | 109.5(7)    |
| N2_1—C9_1—H9b_1      | 109.1(7)    | N2_2—C9_2—H9b_2      | 109.8(6)    |
| N2_1—C9_1—C10_1      | 113.74(10)  | N2_2—C9_2—C10_2      | 112.27(9)   |
| H10a_1—C10_1—C9_1    | 109.9(7)    | H10a_2—C10_2—C9_2    | 110.4(7)    |
| H10b_1—C10_1—C9_1    | 111.7(7)    | H10b_2—C10_2—C9_2    | 111.2(7)    |
| H10b_1—C10_1—H10a_1  | 114.6(10)   | H10b_2—C10_2—H10a_2  | 113.6(10)   |
| F1_1—C10_1—C9_1      | 109.35(10)  | F1_2—C10_2—C9_2      | 108.97(9)   |
| F1_1—C10_1—H10a_1    | 106.5(7)    | F1_2—C10_2—H10a_2    | 106.7(7)    |
| Bond  | Distance (Å) | Bond  | Distance (Å) |
|-------|--------------|-------|--------------|
| F1_1—C10_1—H10b_1 | 104.4 (7) | F1_2—C10_2—H10b_2 | 105.6 (7) |
| H11b_1—C11_1—H11a_1 | 109.0 (10) | H11b_2—C11_2—H11a_2 | 108.4 (9) |
| C12_1—C11_1—H11a_1 | 108.2 (7) | C12_2—C11_2—H11a_2 | 109.4 (6) |
| C12_1—C11_1—H11b_1 | 108.6 (7) | C12_2—C11_2—H11b_2 | 109.5 (7) |
| N1_1—C11_1—H11a_1 | 107.6 (7) | N1_2—C11_2—H11a_2 | 108.5 (6) |
| N1_1—C11_1—H11b_1 | 109.8 (7) | N1_2—C11_2—H11b_2 | 106.9 (6) |
| N1_1—C11_1—C12_1 | 113.63 (8) | N1_2—C11_2—C12_2 | 113.96 (8) |
| C13_1—C12_1—C11_1 | 120.19 (8) | C13_2—C12_2—C11_2 | 119.79 (9) |
| N4_1—C12_1—C11_1 | 116.34 (8) | N4_2—C12_2—C11_2 | 117.08 (9) |
| N4_1—C12_1—C13_1 | 123.40 (8) | N4_2—C12_2—C13_2 | 123.11 (9) |
| C14_1—C13_1—C12_1 | 114.47 (8) | C14_2—C13_2—C12_2 | 114.56 (9) |
| C16_1—C13_1—C12_1 | 124.13 (9) | C16_2—C13_2—C12_2 | 124.46 (9) |
| C16_1—C13_1—C14_1 | 121.40 (9) | C16_2—C13_2—C14_2 | 120.97 (9) |
| N5_1—C14_1—C13_1 | 123.18 (9) | N5_2—C14_2—C13_2 | 123.50 (10) |
| O2_1—C14_1—C13_1 | 117.08 (8) | O2_2—C14_2—C13_2 | 117.25 (10) |
| O2_1—C14_1—N5_1 | 119.74 (8) | O2_2—C14_2—N5_2 | 119.77 (9) |
| N4_1—C15_1—H15_1 | 117.2 (6) | N4_2—C15_2—H15_2 | 117.0 (7) |
| N5_1—C15_1—H15_1 | 116.0 (6) | N5_2—C15_2—H15_2 | 115.9 (7) |
| N5_1—C15_1—N4_1 | 126.82 (9) | N5_2—C15_2—N4_2 | 127.12 (10) |
| H16a_1—C16_1—C13_1 | 111.8 (7) | H16a_2—C16_2—C13_2 | 111.5 (8) |
| H16b_1—C16_1—C13_1 | 113.0 (7) | H16b_2—C16_2—C13_2 | 113.1 (8) |
| H16b_1—C16_1—H16a_1 | 102.7 (10) | H16b_2—C16_2—H16a_2 | 101.5 (11) |
| H16c_1—C16_1—C13_1 | 111.6 (8) | H16c_2—C16_2—C13_2 | 111.4 (9) |
| H16e_1—C16_1—H16a_1 | 108.3 (11) | H16e_2—C16_2—H16a_2 | 108.5 (12) |
| H16c_1—C16_1—H16b_1 | 109.1 (11) | H16c_2—C16_2—H16b_2 | 110.3 (12) |
| H17b_1—C17_1—H17a_1 | 110.8 (9) | H17b_2—C17_2—H17a_2 | 110.4 (12) |
| H17c_1—C17_1—H17a_1 | 108.6 (9) | H17c_2—C17_2—H17a_2 | 111.0 (12) |
| H17c_1—C17_1—H17b_1 | 109.7 (9) | H17c_2—C17_2—H17b_2 | 108.9 (11) |
| O2_1—C17_1—H17a_1 | 105.9 (6) | O2_2—C17_2—H17a_2 | 106.2 (9) |
| O2_1—C17_1—H17b_1 | 110.8 (6) | O2_2—C17_2—H17b_2 | 110.5 (7) |
| O2_1—C17_1—H17c_1 | 111.0 (6) | O2_2—C17_2—H17c_2 | 109.8 (8) |
| C7A_1—N1_1—C2_1 | 106.63 (8) | C7A_2—N1_2—C2_2 | 106.66 (8) |
| C11_1—N1_1—C2_1 | 126.92 (9) | C11_2—N1_2—C2_2 | 126.10 (9) |
| C11_1—N1_1—C7A_1 | 126.35 (8) | C11_2—N1_2—C7A_2 | 126.98 (9) |
| C9_1—N2_1—C8_1 | 119.57 (10) | C9_2—N2_2—C8_2 | 122.13 (9) |
| H2a_1—N2_1—C8_1 | 118.7 (8) | H2a_2—N2_2—C8_2 | 118.6 (7) |
| H2a_1—N2_1—C9_1 | 120.7 (8) | H2a_2—N2_2—C9_2 | 118.4 (7) |
| C3A_1—N3_1—C2_1 | 104.67 (8) | C3A_2—N3_2—C2_2 | 104.70 (8) |
| C15_1—N4_1—C12_1 | 115.96 (8) | C15_2—N4_2—C12_2 | 115.91 (9) |
| C15_1—N5_1—C14_1 | 116.15 (8) | C15_2—N5_2—C14_2 | 115.79 (9) |
| C17_1—O2_1—C14_1 | 117.07 (8) | C17_2—O2_2—C14_2 | 116.88 (10) |
| C2_1—N1_1—C7A_1—C3A_1 | 0.69 (8) | C2_2—N1_2—C7A_2—C3A_2 | 1.07 (8) |
| C2_1—N1_1—C7A_1—C7_1 | 177.71 (8) | C2_2—N1_2—C7A_2—C7_2 | -179.36 (8) |
| C2_1—N1_1—C11_1—C12_1 | 101.85 (10) | C2_2—N1_2—C11_2—C12_2 | 78.97 (10) |
| C2_1—N3_1—C3A_1—C4_1 | -175.83 (7) | C2_2—N3_2—C3A_2—C4_2 | -179.62 (7) |
| C2_1—N3_1—C3A_1—C7A_1 | 0.29 (8) | C2_2—N3_2—C3A_2—C7A_2 | -0.08 (8) |
| C3A_1—C4_1—C5_1—C6_1 | -0.47 (11) | C3A_2—C4_2—C5_2—C6_2 | -0.20 (11) |
\[ \text{C3A}_1—\text{C4}_1—\text{C8}_1—\text{N2}_1 \quad -2.07 \text{ (10)} \quad \text{C3A}_2—\text{C4}_2—\text{C8}_2—\text{N2}_2 \quad -8.31 \text{ (10)} \]
\[ \text{C3A}_1—\text{C7A}_1—\text{C7}_1—\text{C6}_1 \quad 0.27 \text{ (11)} \quad \text{C3A}_2—\text{C7A}_2—\text{C7}_2—\text{C6}_2 \quad 0.26 \text{ (11)} \]
\[ \text{C3A}_1—\text{C7A}_1—\text{N1}_1—\text{C11}_1 \quad -175.96 \text{ (7)} \quad \text{C3A}_2—\text{C7A}_2—\text{N1}_2—\text{C11}_2 \quad 175.46 \text{ (7)} \]
\[ \text{C4}_1—\text{C5}_1—\text{C6}_1—\text{C7}_1 \quad -0.58 \text{ (12)} \quad \text{C4}_2—\text{C5}_2—\text{C6}_2—\text{C7}_2 \quad -0.19 \text{ (12)} \]
\[ \text{C4}_1—\text{C8}_1—\text{N2}_1—\text{C9}_1 \quad 166.74 \text{ (9)} \quad \text{C4}_2—\text{C8}_2—\text{N2}_2—\text{C9}_2 \quad 177.21 \text{ (8)} \]
\[ \text{C5}_1—\text{C7}_1—\text{C7A}_1—\text{N1}_1 \quad 0.27 \text{ (11)} \quad \text{C5}_2—\text{C7}_2—\text{C7A}_2—\text{N1}_2 \quad 0.16 \text{ (11)} \]
\[ \text{C4}_1—\text{C8}_1—\text{N2}_1—\text{C9}_1 \quad 0.26 \text{ (11)} \quad \text{C4}_2—\text{C8}_2—\text{N2}_2—\text{C9}_2 \quad 0.16 \text{ (11)} \]
\[ \text{C3A}_1—\text{C7A}_1—\text{N1}_1—\text{C11}_1 \quad 0.27 \text{ (11)} \quad \text{C3A}_2—\text{C7A}_2—\text{N1}_2—\text{C11}_2 \quad 0.16 \text{ (11)} \]
\[ \text{C11}_1—\text{C12}_1—\text{C13}_1—\text{C14}_1 \quad 177.37 \text{ (9)} \quad \text{C11}_2—\text{C12}_2—\text{C13}_2—\text{C14}_2 \quad 178.99 \text{ (9)} \]
\[ \text{C11}_1—\text{C12}_1—\text{C13}_1—\text{C16}_1 \quad -2.24 \text{ (12)} \quad \text{C11}_2—\text{C12}_2—\text{C13}_2—\text{C16}_2 \quad -0.06 \text{ (12)} \]
\[ \text{C11}_1—\text{C12}_1—\text{C13}_1—\text{C16}_1 \quad -0.06 \text{ (12)} \quad \text{C11}_2—\text{C12}_2—\text{C13}_2—\text{C16}_2 \quad -0.37 \text{ (12)} \]
\[ \text{C13}_1—\text{C14}_1—\text{N5}_1—\text{C15}_1 \quad -175.94 \text{ (9)} \quad \text{C13}_2—\text{C14}_2—\text{N5}_2—\text{C15}_2 \quad -175.04 \text{ (10)} \]

**Hydrogen-bond geometry (Å, †)**

| D—H···A    | D—H   | H···A   | D···A   | D—H···A |
|------------|-------|---------|---------|---------|
| C2_1—H2_1···O1_2i | 1.054 (12) | 2.353 (12) | 3.2907 (14) | 147.5 (9) |
| C7_1—H7_1···O1_2ii | 1.031 (12) | 2.245 (13) | 3.2211 (14) | 157.4 (10) |
| N2_1—H2a_1···N3_1 | 1.002 (14) | 2.035 (14) | 2.8581 (14) | 137.9 (10) |
| C2_2—H2_2···N5_1 | 1.060 (13) | 2.240 (13) | 3.2922 (15) | 171.4 (9) |
| C7_2—H7_2···O1_1iii | 1.059 (12) | 2.394 (12) | 3.0915 (14) | 122.3 (8) |
| C11_2—H11b_2···F1_1iv | 1.092 (12) | 2.186 (12) | 3.1839 (13) | 150.7 (10) |
| C16_2—H16c_2···F1_2v | 1.047 (17) | 2.403 (17) | 3.2827 (15) | 140.9 (12) |
| N2_2—H2a_2···N3_2 | 1.002 (14) | 1.942 (14) | 2.7823 (13) | 139.6 (10) |

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