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# Crystal structure of the *meso* compound (2*R*,6*S*)-4-(5-bromopyrimidin-2-yl)-2,6-dimethylmorpholine

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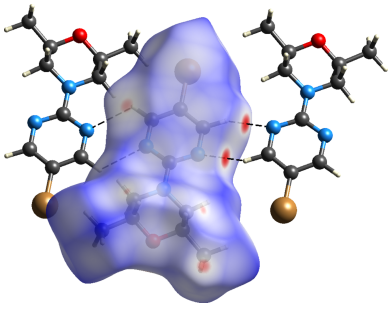
The title compound, $C_{10}H_{14}N_3OBr$, was prepared by a nucleophilic aromatic substitution reaction between 2,6-dimethylmorpholine and 2-chloropyrimidine, followed by bromination of the pyrimidine ring. The compound crystallizes in the monoclinic system (space group $P2_1/c$) with four molecules in the unit cell ($Z$ = 4). The molecule exhibits approximate $C_S$ point-group symmetry (r.m.s. deviation: 0.1072 Å). The arrangement of the molecules in the solid state is dominated by close packing. C—H···N contacts between pyrimidinyl rings in adjacent molecules with an $R_2^2(6)$ motif are encountered, whereas the bromine atom does not exhibit any short contacts that could be regarded as halogen bonds.

## 1. Chemical context

2,6-Dimethylmorpholine, usually the *cis* isomer, is a common building block in medicinal chemistry as it allows for modulating lipophilicity, basicity, metabolic stability and binding to the biological target. The antifungal agent amorolfin and the antineoplastic compound sonidegib are examples of approved and marketed drugs containing a *cis*-2,6-dimethylmorpholine group. In the context of our antimycobacterial drug discovery efforts, 4-arylmorpholine building blocks have attracted our interest (Palme *et al.*, 2025). We synthesized and crystallographically characterized (2*R*,6*S*)-4-(5-bromopyrimidin-2-yl)-2,6-dimethylmorpholine (**4**) in two steps from commercially available starting materials (Fig. 1), adapting an established route (Cheprakova *et al.*, 2014). The first step was a nucleophilic aromatic substitution ($S_NAr$) reaction between 2-chloropyrimidine (**1**) and 2,6-dimethylmorpholine hydrochloride (**2**) in the presence of a base to yield 2,6-dimethyl-4-(pyrimidin-2-yl)morpholine (**3**; Hanyu *et al.*, 2009). [The configuration of 2,6-dimethylmorpholine hydrochloride as purchased was unspecified, but $^1$H and $^{13}$C NMR spectroscopy (see supporting information) indicate that only one isomer was present]. It is worth noting that Wei *et al.* (2019) reported the synthesis of **3** by a transition-metal-free cross-coupling



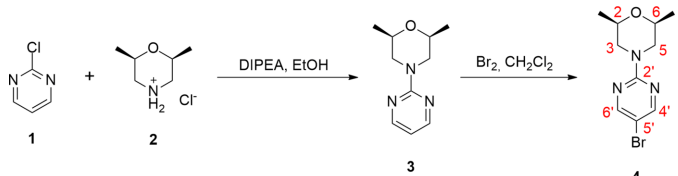
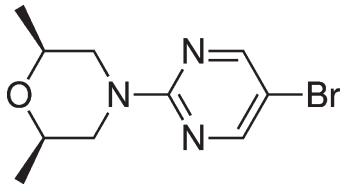



**Figure 1**
Two-step synthesis of **4**. DIPEA = *N*,*N*-Diisopropylethylamine (Hünig's base).

reaction of 2-cyanopyrimidine with 2,6-dimethylmorpholine, and that, recently, Hall *et al.* (2025) described a one-pot sequential desulfonylative fluorination of pyrimidine-2-sulfonyl fluoride followed by an $S_NAr$ reaction with 2,6-dimethylmorpholine. Finally, bromination of **3** in the second step gave compound **4** in good yield. Synthesis of **4** from 5-bromo-2-chloropyrimidine and 2,6-dimethylmorpholine under $S_NAr$ conditions has been described in the patent literature (Heng *et al.*, 2008; Yoshihara *et al.*, 2011; Wu *et al.*, 2015; You *et al.*, 2023; Shojaei *et al.*, 2023).




## 2. Structural commentary

Fig. 2 shows the molecular structure of **4** in the crystal. X-ray crystallography confirmed that the 2,6-dimethylmorpholine ring is *cis*-configured. As expected, it adopts a chair conformation with the two methyl groups in equatorial positions. The 2,6-dimethylmorpholine group and the pyrimidine ring are slightly inclined relative to one another about the C8—N4 bond, resulting in a r.m.s. deviation from exact molecular $C_S$ point group symmetry of 0.1072 Å. The geometry at N4 of the morpholine ring deviates marginally from planarity, as indicated by Σ(C—N—C) = 357.89 (9)°, which is barely smaller than 360° expected for an ideal planar coordination. The pyramidal height, *i.e.* the perpendicular distance of N4 to the plane specified by C3, C5 and C8, is small [0.1198 (6) Å]. This indicates that the lone pair of N4 is conjugated with the aromatic system of the pyrimidine ring.

The high-frequency shift of the $^1$H NMR signal assigned to the equatorial H atoms at C3 and C5 (4.45–4.33 ppm) can most likely be attributed to an anisotropic shielding effect exerted by the lone pairs of N1 and N3. Such a high-frequency shift of the morpholine H-3$_{eq}$/H-5$_{eq}$ signal was not observed for 2,6-dimethyl-4-phenylmorpholine (Yuan *et al.*, 2024) or other *cis*-2,6-dimethylmorpholine derivatives (Brügel, 1969). The C8—N4 bond is shorter than the corresponding C—N bond in 4-phenylmorpholine (WOXMOP; Jiang *et al.*, 2023) by 0.048 (2) Å.

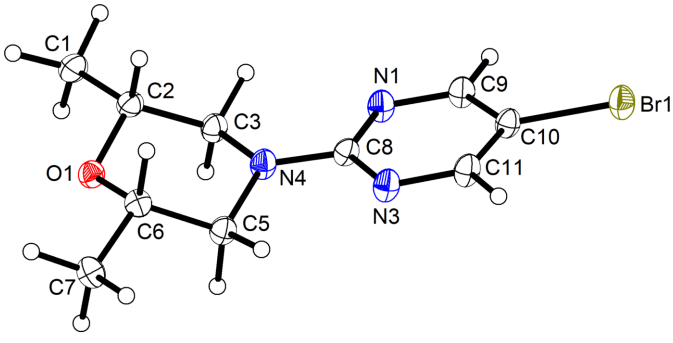



**Figure 2**
Molecular structure of **4** in the crystal. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7$A$···O1$^{i}$ | 1.078 (12) | 2.499 (12) | 3.4295 (9) | 143.9 (8) |
| C9—H9···N1$^{ii}$ | 1.065 (10) | 2.580 (10) | 3.2779 (9) | 122.5 (7) |
| C11—H11···N3$^{iii}$ | 1.095 (11) | 2.423 (11) | 3.3610 (9) | 142.8 (8) |

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+1$.

## 3. Supramolecular features

The most prominent supramolecular feature in the crystal structure of **4** is weak C—H···N hydrogen bonding between the pyrimidine rings of adjacent symmetry-related molecules (Fig. 3), resulting in centrosymmetric $R_2^2(6)$ motifs (Bernstein *et al.*, 1995). The geometric parameters listed in Table 1 suggest that the C11—H11···N3$^{iii}$ hydrogen bond is more favourable than C9—H9···N1$^{ii}$, as indicated by shorter H···$A$ and $D$···$A$ distances and a $D$—H···$A$ angle closer to linearity. In addition, a C$_{methyl}$—H···O$_{morpholine}$ intermolecular short contact (C7—H7$A$···O1$^{i}$) can be identified. Such contacts are ubiquitous in crystal structures of organic molecules (Desiraju, 1995). Short contacts to bromine that could be interpreted as halogen bonds are not encountered.

A Hirshfeld surface analysis was undertaken to investigate close intermolecular contacts and supramolecular assembly in the crystal structure in a more objective and quantitative manner (Spackman & Jayatilaka, 2009). Fig. 4 shows the Hirshfeld surface mapped with the normalized contact distance ($d_{norm}$), whereby red, white and blue regions respectively indicate intermolecular contacts shorter, approximately equal and longer than the sum of the van der

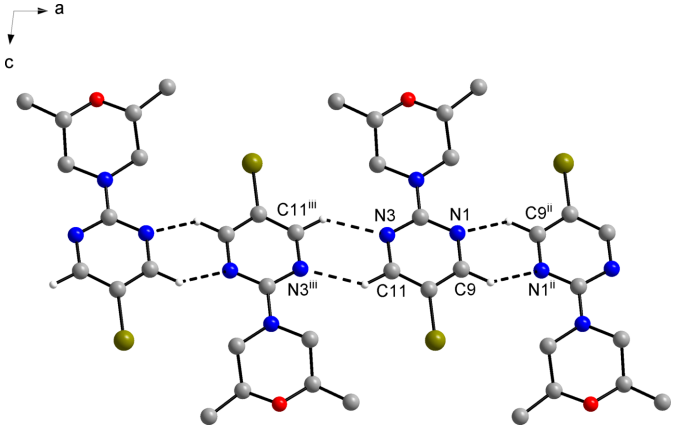



**Figure 3**
Part of the crystal structure of **4** viewed along the *b*-axis direction. H atoms not involved in C—H···N weak hydrogen bonds are omitted for clarity. Dashed lines represent weak hydrogen bonds. Colour scheme: C, grey; H, white; Br, dark yellow; N, blue; O, red. Symmetry codes: (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+1$.

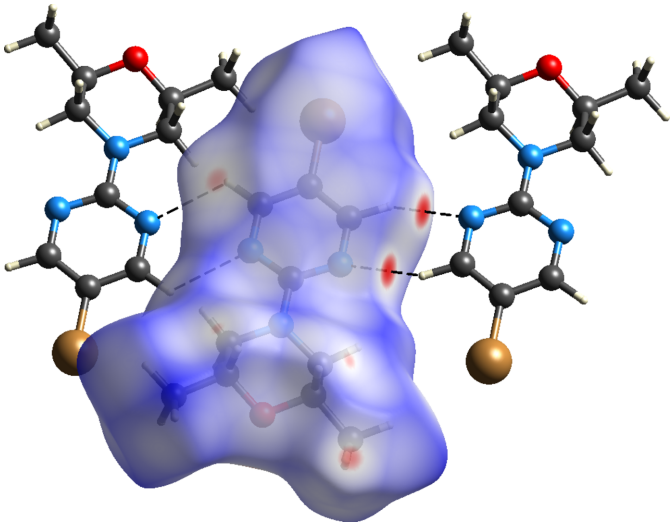


**Figure 4**
Hirshfeld surface mapped with $d_{norm}$ for **4**. Colour scheme for the atoms: C, dark grey; H, white; Br, bronze; N, blue; O, red.

Waals radii (Bondi, 1964). Here, the two major red concave areas result from the C11—H11···N3$^{iii}$ weak hydrogen bonds. Smaller red areas arise from the C9—H9···N1$^{ii}$ and C7—H7*A*···O1$^{i}$ short contacts as well as H···H intermolecular contacts involving the axial H atoms bonded to C2 and C6.

The corresponding two-dimensional fingerprint plot (Fig. 5) shows spikes from N···H/H···N (11.5% of the surface area included), O···H/H···O (5.2%) and Br···H/H···Br contacts (19.1%) as well as wings from C···H/H···C contacts (4.2%).

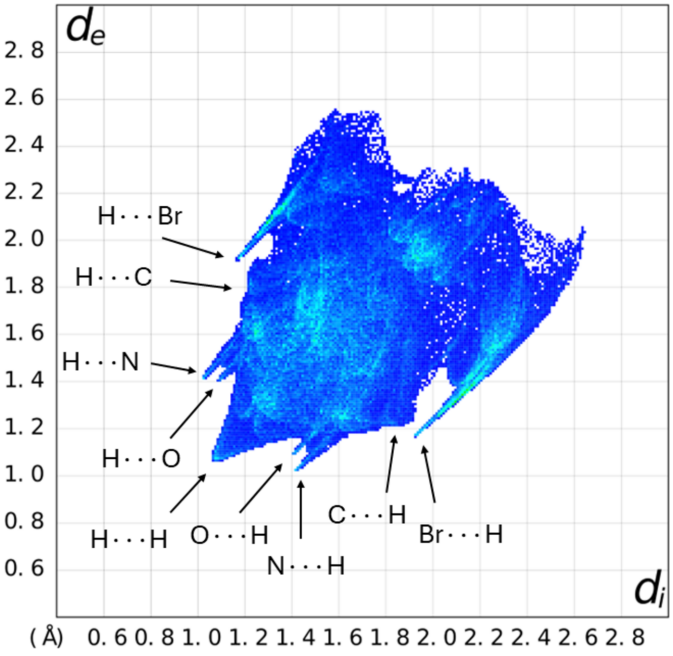



**Figure 5**
The two-dimensional fingerprint plot for **4**. $d_i$ and $d_e$ are the distances from the Hirshfeld surface to the nearest atoms inside and outside the surface, respectively. Dashed lines represent weak hydrogen bonds.

A triangular feature on the diagonal characteristic of face-to-face aromatic stacking is not observed, and C···N/N···C and C···C contacts together only contribute 4.3% to the surface area included. H···H contacts account for 52.2% of the surface area. The tip on the diagonal centred at $d_e + d_i < 2.4$ Å (*i.e.* less than twice the van der Waals radius of hydrogen) mirrors the small and weak red spots at the morpholine H atoms in the axial 2,6-positions in the $d_{norm}$ plot in Fig. 4. A slight asymmetry about the diagonal in the fingerprint plot is noticeable, in particular for the Br···H/H···Br spikes, which usually signals packing inefficiencies. Nonetheless, the packing index of 71% falls within the typical range observed for organic molecular crystals (Kitajgorodskij, 1973).

## 4. Database survey

The crystal structure most closely related to that of **4** in the Cambridge Structural Database (CSD; Groom *et al.*, 2016) is the structure of 4-(5-bromopyrimidin-2-yl)morpholine (ROTXOP; Cheprakova *et al.*, 2014). The corresponding 5-nitropyrimidine derivative has also been crystallographically characterized (YILPEQ; Gorbunov *et al.*, 2013). Therein, the coordination at the morpholine N atom is virtually planar [Σ(C—N—C) = 360.0 (2)°], which can be attributed to the electron-withdrawing effect of the nitro group. As of June 2026, there are no examples of crystal structures containing 2,6-dimethylmorpholine with N-bound unsubstituted aromatic groups in the CSD, but a relatively large number of crystal structures containing an unsubstituted 4-phenylmorpholine moiety. In most of these crystal structures, the coordination at the morpholine N atom is markedly pyramidal, as in 4-phenylmorpholine (WOXMOP; Jiang *et al.*, 2023).

## 5. Synthesis and crystallization

*General:* Starting materials were purchased and used as received. 2,6-Dimethylmorpholine hydrochloride was obtained from BLDpharm. Solvents were distilled before use. NMR spectra were recorded on an Agilent Technologies 600 MHz shielded VNMRS and an Agilent Technologies 400 MHz VNMRS spectrometer. Chemical shifts are reported relative to the residual solvent signal of chloroform-*d* ($\delta_H$ = 7.26 ppm, $\delta_C$ = 77.16 ppm) or DMSO-$d_6$ ($\delta_H$ = 2.50 ppm, $\delta_C$ = 39.51 ppm). Abbreviations: *s* = singlet, *d* = doublet, *t* = triplet, *dd* = doublet of doublets, *dqd* = doublet of quartet of doublets, *m* = multiplet. HRMS data were acquired on a Thermo Scientific Q Exactive GC Orbitrap GC-MS system.

*2,6-Dimethyl-4-(pyrimidin-2-yl)morpholine (**3**):* 2-Chloropyrimidine (**1**) (5.73 g, 50.0 mmol) and 2,6-dimethylmorpholine hydrochloride (**2**) (7.59 g, 50.0 mmol) were suspended in 50 mL of ethanol and 15 mL of DIPEA were added with stirring. The mixture was heated to reflux for 7 h. Subsequently, the solvent was removed under reduced pressure and the residue was taken up with ethyl acetate (50 mL). After washing successively with water (30 mL) and brine (30 mL), the organic layer was dried over magnesium sulfate and the solvent was evaporated under reduced pressure. The

crude product was purified by flash chromatography (Interchim puriFlash® 430) on silica gel using gradient elution with *n*-heptane/ethyl acetate to yield **3** as a colourless oil (8.41 g, 44.0 mmol, 88%). $^1$H NMR (600 MHz, chloroform-*d*) $\delta$ 8.31 (*d*, *J* = 4.8 Hz, 2H, H-4′/H-6′), 6.50 (*t*, *J* = 4.8 Hz, 1H, H-5′), 4.58–4.52 (*m*, 2H, H-3$_{eq}$/H-5$_{eq}$), 3.63 (*dqd*, *J* = 10.6, 6.3, 2.4 Hz, 2H, H-2$_{ax}$/H-6$_{ax}$), 2.60 (*dd*, *J* = 13.2, 10.6 Hz, 2H, H-3$_{ax}$/H-5$_{ax}$), 1.24 (*d*, *J* = 6.3 Hz, 6H, CH$_3$) ppm. $^{13}$C{$^1$H} NMR (151 MHz, chloroform-*d*): $\delta$ 161.1 (C-2′), 157.8 (C-4′/C-6′), 110.1 (C-5′), 71.9 (C-2/C-6), 49.5 (C-3/C-5), 19.0 (CH$_3$) ppm.

*(2R,6S)-4-(5-Bromopyrimidin-2-yl)-2,6-dimethylmorpholine (**4**):* Compound **3** (5.00 g, 26.1 mmol) was dissolved in 50 mL of dichloromethane and 0.80 mL (31.2 mmol) of bromine were added dropwise with stirring. After stirring for 12 h at room temperature, the progress of the reaction was checked by TLC. An additional 0.20 mL (7.8 mmol) of bromine was added and stirring was continued for 1 h. Subsequently, approx. 30 mL of a saturated aqueous sodium thiosulfate solution were added, whereupon the mixture became colourless. The organic layer was separated and the aqueous phase was extracted with dichloromethane (2 × 30 mL) followed by ethyl acetate (1 × 30 mL). After drying over magnesium sulfate, the combined organic layers were evaporated to dryness to obtain compound **4** as a white solid (6.58 g, 24.2 mmol, 93%). $^1$H NMR (402 MHz, DMSO-$d_6$) $\delta$ 8.45 (*s*, 2H, H-4′/H-6′), 4.45–4.33 (*m*, 2H, H-3$_{eq}$/H-5$_{eq}$), 3.53 (*dqd*, *J* = 10.7, 6.2, 2.5 Hz, 2H, H-2$_{ax}$/H-6$_{ax}$), 2.52 (*dd*, *J* = 13.2, 10.7 Hz, 2H, H-3$_{ax}$/H-5$_{ax}$), 1.13 (*d*, *J* = 6.2 Hz, 6H, CH$_3$) ppm. $^{13}$C{$^1$H} APT NMR (101 MHz, DMSO-$d_6$): $\delta$ 159.2 (C-2′), 157.9 (C-4′/C-6′), 105.5 (C-5′), 70.8 (C-2/C-6), 48.9 (C-3/C-5), 18.6 (CH$_3$) ppm. HRMS(EI): *m*/*z* calculated for $C_{10}H_{14}N_3OBr^+$ 271.031486 $[M]^+$, found 271.031420. Crystals suitable for single-crystal X-ray diffraction analysis were grown from a solution in dichloromethane by slow evaporation of the solvent at ambient conditions.

## 6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal structure was initially refined with *SHELXL* (Sheldrick, 2015*b*). Subsequently, Hirshfeld atom refinement was performed with *NoSpherA2* (Kleemiss *et al.*, 2021) in *OLEX2* (Dolomanov *et al.*, 2009). *ORCA* 6.1 (Neese, 2025) was used to calculate the electron density at the B3LYP/def2-TZVPP level of theory (Becke, 1993; Lee *et al.*, 1988; Weigend & Ahlrichs, 2005), which was partitioned into Hirshfeld atoms and converted *via* Fourier transform into atomic form factors (Midgley *et al.*, 2021). Least-squares refinements against the non-spherical atomic form factors thus obtained were carried out with *olex2.refine* (Bourhis *et al.*, 2015). Anisotropic atomic displacement parameters (ADPs) were introduced for all non-H atoms. Positions and isotropic ADPs of H atoms were refined freely.

The deviation from molecular point-group symmetry was calculated with *MOLSYM* in *PLATON* (Spek, 2009) using the atomic weighting mode. Hirshfeld surface analysis was conducted with *CrystalExplorer 21* (Spackman *et al.*, 2021), which by default applies neutron-derived values for *X*—H bond lengths (Allen & Bruno, 2010).

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{10}H_{14}BrN_3O$ |
| $M_r$ | 272.15 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 100 |
| *a*, *b*, *c* (Å) | 10.3934 (6), 4.2323 (2), 25.8356 (14) |
| $\beta$ (°) | 97.076 (3) |
| $V$ (Å$^3$) | 1127.8 (1) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 3.63 |
| Crystal size (mm) | 0.18 × 0.10 × 0.07 |
| | |
| Data collection | |
| Diffractometer | Bruker AXS D8 VENTURE |
| Absorption correction | Gaussian (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.679, 0.860 |
| No. of measured, independent and observed [$I \geq 2u(I)$] reflections | 97547, 3622, 3383 |
| $R_{int}$ | 0.042 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.727 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.013, 0.030, 1.06 |
| No. of reflections | 3622 |
| No. of parameters | 192 |
| H-atom treatment | All H-atom parameters refined |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.33, −0.24 |

Computer programs: *APEX6* (Bruker, 2024), *SAINT* (Bruker, 2019), *SHELXT* (Sheldrick, 2015*a*), *OLEX2.refine* (Bourhis *et al.*, 2015), *DIAMOND* (Brandenburg, 2018) and *publCIF* (Westrip, 2010).

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# supporting information

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## Crystal structure of the *meso* compound (2*R*,6*S*)-4-(5-bromopyrimidin-2-yl)-2,6-dimethylmorpholine

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### Computing details

**(2*R*,6*S*)-4-(5-Bromopyrimidin-2-yl)-2,6-dimethylmorpholine**

*Crystal data*

$C_{10}H_{14}BrN_3O$
$M_r = 272.15$
Monoclinic, $P2_1/c$
$a = 10.3934$ (6) Å
$b = 4.2323$ (2) Å
$c = 25.8356$ (14) Å
$\beta = 97.076$ (3)°
$V = 1127.8$ (1) Å$^3$
$Z = 4$
$F(000) = 551.505$
$D_x = 1.603$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9300 reflections
$\theta = 3.2$–$31.0°$
$\mu = 3.63$ mm$^{-1}$
$T = 100$ K
Prism, colourless
0.18 × 0.10 × 0.07 mm

*Data collection*

Bruker AXS D8 VENTURE diffractometer
Radiation source: I$\mu$S Diamond
Incoatec Helios mirrors monochromator
Detector resolution: 7.391 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: gaussian (SADABS; Krause *et al.*, 2015)
$T_{min} = 0.679$, $T_{max} = 0.860$
97547 measured reflections
3622 independent reflections
3383 reflections with $I \geq 2u(I)$
$R_{int} = 0.042$
$\theta_{max} = 31.1°$, $\theta_{min} = 2.0°$
$h = -15 \rightarrow 15$
$k = -6 \rightarrow 6$
$l = -37 \rightarrow 37$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.013$
$wR(F^2) = 0.030$
$S = 1.06$
3622 reflections
192 parameters
0 restraints
0 constraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0129P)^2 + 0.1241P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.0001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$

*Special details*

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

**Refinement**. Refinement using NoSpherA2, an implementation of NOn-SPHERical Atom-form-factors in Olex2. Please cite: F. Kleemiss *et al.* Chem. Sci. DOI 10.1039/D0SC05526C - 2021 NoSpherA2 implementation of HAR makes use of tailor-made aspherical atomic form factors calculated on-the-fly from a Hirshfeld-partitioned electron density (ED) - not from spherical-atom form factors.
The ED is calculated from a gaussian basis set single determinant SCF wavefunction - either Hartree-Fock or DFT using selected funtionals - for a fragment of the crystal. This fragment can be embedded in an electrostatic crystal field by employing cluster charges or modelled using implicit solvation models, depending on the software used.
The following options were used: SOFTWARE: ORCA 6.1 PARTITIONING: NoSpherA2 INT ACCURACY: Normal METHOD: B3LYP BASIS SET: def2-TZVPP CHARGE: 0 MULTIPLICITY: 1 DATE: 2026-04-01_20-42-29
The minimum and maximum estimated transmissions from the multi-scan scaling are 0.6613 and 0.8743 (SADABS).

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.86770 (7) | 0.35247 (18) | 0.28616 (3) | 0.02637 (14) |
| H1A | 0.8346 (11) | 0.407 (2) | 0.2447 (5) | 0.049 (3)* |
| H1B | 0.8798 (11) | 0.100 (2) | 0.2894 (4) | 0.048 (3)* |
| H1C | 0.9610 (11) | 0.460 (3) | 0.2975 (4) | 0.051 (3)* |
| C2 | 0.76932 (6) | 0.47733 (16) | 0.31971 (3) | 0.02041 (12) |
| H2 | 0.7597 (9) | 0.736 (2) | 0.3144 (4) | 0.034 (2)* |
| C3 | 0.80623 (6) | 0.40775 (17) | 0.37755 (3) | 0.02130 (12) |
| H3A | 0.8193 (10) | 0.150 (2) | 0.3831 (4) | 0.040 (3)* |
| H3B | 0.8993 (9) | 0.525 (2) | 0.3919 (4) | 0.034 (2)* |
| C5 | 0.57364 (6) | 0.42407 (17) | 0.38639 (3) | 0.02103 (12) |
| H5A | 0.5027 (10) | 0.546 (2) | 0.4076 (4) | 0.039 (2)* |
| H5B | 0.5608 (10) | 0.170 (2) | 0.3940 (4) | 0.039 (3)* |
| C6 | 0.54734 (6) | 0.48648 (16) | 0.32811 (3) | 0.01974 (12) |
| H6 | 0.5531 (8) | 0.746 (2) | 0.3220 (3) | 0.032 (2)* |
| C7 | 0.41680 (7) | 0.36042 (17) | 0.30472 (3) | 0.02331 (13) |
| H7A | 0.3993 (11) | 0.410 (3) | 0.2635 (5) | 0.056 (3)* |
| H7B | 0.3402 (11) | 0.467 (3) | 0.3244 (5) | 0.053 (3)* |
| H7C | 0.4119 (11) | 0.106 (2) | 0.3097 (4) | 0.048 (3)* |
| C8 | 0.73225 (6) | 0.65197 (15) | 0.45507 (2) | 0.01870 (11) |
| C9 | 0.88456 (6) | 0.79021 (17) | 0.52262 (3) | 0.02176 (12) |
| H9 | 0.9834 (10) | 0.791 (3) | 0.5395 (4) | 0.043 (2)* |
| C10 | 0.78849 (6) | 0.91794 (15) | 0.54884 (2) | 0.01989 (12) |
| C11 | 0.66181 (6) | 0.90051 (17) | 0.52476 (3) | 0.02281 (13) |
| H11 | 0.5802 (10) | 0.999 (3) | 0.5425 (4) | 0.047 (3)* |
| Br1 | 0.829245 (7) | 1.105440 (18) | 0.615057 (3) | 0.02623 (3) |
| N1 | 0.85771 (5) | 0.65779 (14) | 0.47578 (2) | 0.02223 (11) |
| N3 | 0.63261 (5) | 0.76677 (15) | 0.47811 (2) | 0.02287 (11) |
| N4 | 0.70440 (5) | 0.52684 (15) | 0.40639 (2) | 0.02233 (11) |
| O1 | 0.64614 (4) | 0.34162 (11) | 0.302198 (18) | 0.02004 (9) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0264 (3) | 0.0294 (4) | 0.0253 (3) | −0.0036 (3) | 0.0111 (3) | −0.0039 (3) |
| C2 | 0.0226 (3) | 0.0190 (3) | 0.0205 (3) | −0.0011 (2) | 0.0062 (2) | −0.0007 (2) |
| C3 | 0.0178 (3) | 0.0270 (3) | 0.0197 (3) | 0.0014 (2) | 0.0045 (2) | −0.0018 (2) |
| C5 | 0.0168 (3) | 0.0271 (3) | 0.0194 (3) | 0.0011 (2) | 0.0027 (2) | 0.0002 (2) |
| C6 | 0.0206 (3) | 0.0179 (3) | 0.0203 (3) | 0.0019 (2) | 0.0011 (2) | 0.0004 (2) |
| C7 | 0.0212 (3) | 0.0234 (3) | 0.0244 (3) | 0.0028 (2) | −0.0009 (2) | −0.0021 (2) |
| C8 | 0.0149 (3) | 0.0239 (3) | 0.0175 (3) | 0.0030 (2) | 0.0029 (2) | 0.0010 (2) |
| C9 | 0.0153 (3) | 0.0303 (3) | 0.0196 (3) | 0.0036 (2) | 0.0019 (2) | −0.0023 (2) |
| C10 | 0.0182 (3) | 0.0245 (3) | 0.0173 (3) | 0.0035 (2) | 0.0033 (2) | −0.0004 (2) |
| C11 | 0.0169 (3) | 0.0325 (3) | 0.0193 (3) | 0.0064 (2) | 0.0036 (2) | −0.0016 (3) |
| Br1 | 0.02647 (4) | 0.03261 (4) | 0.01976 (4) | 0.00209 (3) | 0.00346 (2) | −0.00558 (3) |
| N1 | 0.0148 (2) | 0.0317 (3) | 0.0204 (3) | 0.0039 (2) | 0.00277 (19) | −0.0036 (2) |
| N3 | 0.0151 (2) | 0.0347 (3) | 0.0190 (2) | 0.0052 (2) | 0.00271 (19) | −0.0018 (2) |
| N4 | 0.0155 (2) | 0.0329 (3) | 0.0189 (3) | 0.0012 (2) | 0.00342 (19) | −0.0032 (2) |
| O1 | 0.0220 (2) | 0.0190 (2) | 0.0194 (2) | 0.00032 (17) | 0.00371 (17) | −0.00088 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—H1A | 1.108 (11) | C6—C7 | 1.5128 (9) |
| C1—H1B | 1.079 (10) | C6—O1 | 1.4310 (8) |
| C1—H1C | 1.078 (11) | C7—H7A | 1.078 (12) |
| C1—C2 | 1.5147 (9) | C7—H7B | 1.094 (11) |
| C2—H2 | 1.105 (10) | C7—H7C | 1.085 (10) |
| C2—C3 | 1.5250 (10) | C8—N1 | 1.3473 (8) |
| C2—O1 | 1.4252 (8) | C8—N3 | 1.3471 (8) |
| C3—H3A | 1.107 (10) | C8—N4 | 1.3626 (8) |
| C3—H3B | 1.109 (10) | C9—H9 | 1.065 (10) |
| C3—N4 | 1.4572 (8) | C9—C10 | 1.3838 (9) |
| C5—H5A | 1.101 (10) | C9—N1 | 1.3315 (9) |
| C5—H5B | 1.104 (10) | C10—C11 | 1.3872 (9) |
| C5—C6 | 1.5201 (9) | C10—Br1 | 1.8860 (7) |
| C5—N4 | 1.4595 (8) | C11—H11 | 1.095 (11) |
| C6—H6 | 1.111 (10) | C11—N3 | 1.3321 (9) |
| | | | |
| H1B—C1—H1A | 107.5 (8) | O1—C6—C5 | 109.70 (5) |
| H1C—C1—H1A | 109.7 (8) | O1—C6—H6 | 107.6 (4) |
| H1C—C1—H1B | 107.6 (8) | O1—C6—C7 | 108.79 (5) |
| C2—C1—H1A | 109.2 (6) | H7A—C7—C6 | 111.1 (6) |
| C2—C1—H1B | 112.4 (6) | H7B—C7—C6 | 109.7 (6) |
| C2—C1—H1C | 110.3 (6) | H7B—C7—H7A | 109.6 (9) |
| H2—C2—C1 | 109.3 (5) | H7C—C7—C6 | 110.8 (6) |
| C3—C2—C1 | 112.79 (6) | H7C—C7—H7A | 107.7 (8) |
| C3—C2—H2 | 108.7 (5) | H7C—C7—H7B | 107.8 (8) |
| O1—C2—C1 | 108.73 (5) | N3—C8—N1 | 125.24 (6) |
| O1—C2—H2 | 107.2 (5) | N4—C8—N1 | 117.32 (5) |

| | | | |
|---|---|---|---|
| O1—C2—C3 | 109.93 (5) | N4—C8—N3 | 117.42 (6) |
| H3A—C3—C2 | 109.2 (6) | C10—C9—H9 | 121.0 (5) |
| H3B—C3—C2 | 110.2 (5) | N1—C9—H9 | 117.2 (5) |
| H3B—C3—H3A | 108.0 (7) | N1—C9—C10 | 121.75 (6) |
| N4—C3—C2 | 108.86 (5) | C11—C10—C9 | 117.51 (6) |
| N4—C3—H3A | 111.1 (5) | Br1—C10—C9 | 120.90 (5) |
| N4—C3—H3B | 109.5 (5) | Br1—C10—C11 | 121.60 (5) |
| H5B—C5—H5A | 105.4 (7) | H11—C11—C10 | 122.2 (6) |
| C6—C5—H5A | 111.2 (5) | N3—C11—C10 | 121.75 (6) |
| C6—C5—H5B | 109.6 (5) | N3—C11—H11 | 116.0 (6) |
| N4—C5—H5A | 109.6 (5) | C9—N1—C8 | 116.93 (6) |
| N4—C5—H5B | 110.8 (5) | C11—N3—C8 | 116.82 (6) |
| N4—C5—C6 | 110.17 (5) | C5—N4—C3 | 114.79 (5) |
| H6—C6—C5 | 107.9 (4) | C8—N4—C3 | 121.42 (5) |
| C7—C6—C5 | 112.17 (6) | C8—N4—C5 | 121.68 (5) |
| C7—C6—H6 | 110.6 (4) | C6—O1—C2 | 110.32 (5) |
| | | | |
| C1—C2—C3—N4 | −178.16 (6) | C7—C6—C5—N4 | 175.48 (6) |
| C1—C2—O1—C6 | −172.02 (5) | C8—N1—C9—C10 | −0.37 (7) |
| C2—C3—N4—C5 | 51.43 (6) | C8—N3—C11—C10 | −0.71 (7) |
| C2—C3—N4—C8 | −144.83 (5) | C9—C10—C11—N3 | −0.09 (8) |
| C2—O1—C6—C5 | −62.59 (5) | C9—N1—C8—N3 | −0.53 (8) |
| C2—O1—C6—C7 | 174.37 (5) | C9—N1—C8—N4 | 177.77 (6) |
| C3—C2—O1—C6 | 64.06 (6) | C11—C10—C9—N1 | 0.66 (8) |
| C3—N4—C5—C6 | −50.89 (6) | C11—N3—C8—N1 | 1.07 (8) |
| C3—N4—C8—N1 | −0.77 (7) | C11—N3—C8—N4 | −177.23 (6) |
| C3—N4—C8—N3 | 177.67 (6) | Br1—C10—C9—N1 | −179.78 (5) |
| C5—N4—C8—N1 | 161.85 (6) | Br1—C10—C11—N3 | −179.64 (5) |
| C5—N4—C8—N3 | −19.71 (7) | N4—C3—C2—O1 | −56.64 (6) |
| C6—C5—N4—C8 | 145.42 (5) | N4—C5—C6—O1 | 54.47 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7*A*···O1$^{i}$ | 1.078 (12) | 2.499 (12) | 3.4295 (9) | 143.9 (8) |
| C9—H9···N1$^{ii}$ | 1.065 (10) | 2.580 (10) | 3.2779 (9) | 122.5 (7) |
| C11—H11···N3$^{iii}$ | 1.095 (11) | 2.423 (11) | 3.3610 (9) | 142.8 (8) |

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