Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Potassium [1-(*tert*-butoxycarbonyl)-1*H*-indol-3-yl]trifluoroborate hemihydrate

**Guillaume Berionni, Peter Mayer* and Herbert Mayr**

Ludwig-Maximilians-Universität, Department of Chemistry, Butenandtstrasse 5–13, 81377 München, Germany
Correspondence e-mail: pemay@cup.uni-muenchen.de

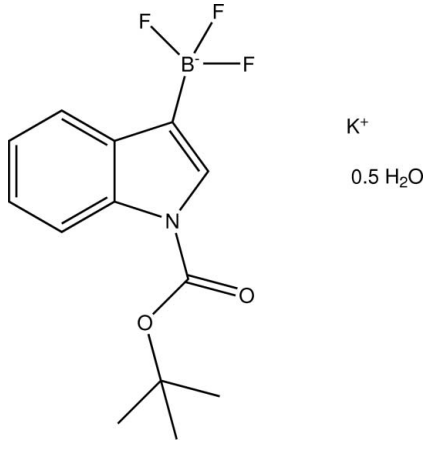
Received 13 March 2012; accepted 2 April 2012

Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.032; $wR$ factor = 0.075; data-to-parameter ratio = 17.5.

The asymmetric unit of the title salt, $K^+\cdot C_{13}H_{14}BF_3NO_2\cdot 0.5H_2O$, consists of two derivatized indolyltrifluoridoborate anions, two potassium cations and one water molecule. Within the indolyltrifluoroborate anions, the least-square planes consisting of the carboxyl group and the adjacent quarternary C atom of the *tert*-butyl groups deviate significantly from coplanarity with the indolyl planes [20.44 (11) and 21.02 (10)°]. The potassium ions are coordinated by six atoms (one $K^+$ ion by two O and four F atoms, and the second $K^+$ ion by one O and five F atoms), however, one of the potassium ions undergoes an additional weak potassium–$\pi$ interaction (K···centroid = 3.722 Å). The packing is stabilized by sequential O—H···O hydrogen bonds along [100] between water molecules and also by O—H···F hydrogen bonds.

## Related literature

For background to organotrifluoroborates and the synthesis, see: Mothes *et al.* (2008); Molander *et al.* (2009); Kassis *et al.* (2009); Reiter *et al.* (2010); Darses & Genet (2008). For related structures, see: Baran *et al.* (2005); Davies *et al.* (2005, 2007); Lu & Lin (2011).




## Experimental

### Crystal data

$K^+\cdot C_{13}H_{14}BF_3NO_2\cdot 0.5H_2O$
$M_r$ = 332.17
Orthorhombic, $P2_12_12_1$
$a$ = 5.8428 (1) Å
$b$ = 16.3177 (2) Å
$c$ = 32.1286 (5) Å
$V$ = 3063.17 (8) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.38 mm$^{-1}$
$T$ = 173 K
0.27 × 0.19 × 0.10 mm

### Data collection

Nonius KappaCCD diffractometer
24771 measured reflections
7001 independent reflections
6369 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.075
$S$ = 1.03
7001 reflections
400 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$
Absolute structure: Flack (1983), 2995 Friedel pairs
Flack parameter: 0.00 (3)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H51···F3$^{i}$ | 0.81 (1) | 2.14 (1) | 2.903 (2) | 156 (3) |
| O5—H51···F1$^{i}$ | 0.81 (1) | 2.62 (2) | 3.188 (2) | 128 (2) |
| O5—H52···O5$^{ii}$ | 0.81 (1) | 2.41 (1) | 3.1968 (16) | 164 (3) |

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

Data collection: *COLLECT* (Hooft, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

The authors thank Professor Peter Klüfers for generous allocation of diffractometer time.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HP2034).

## References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Baran, P. S., Guerrero, C. A., Ambhaikar, N. B. & Hafensteiner, B. D. (2005). *Angew. Chem. Intl. Ed.* **44**, 606–609.
Darses, S. & Genet, J.-P. (2008). *Chem. Rev.* **108**, 288–325.
Davies, S. G., Garner, A. C., Ouzman, J. V. A., Roberts, P. M., Smith, A. D., Snow, E. J., Thomson, J. E., Tamayo, J. A. & Vickers, R. J. (2007). *Org. Biomol. Chem.* **5**, 2138–2147.
Davies, J. R., Kane, P. D., Moody, C. J. & Slawin, A. M. Z. (2005). *J. Org. Chem.* **70**, 5840–5851.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Hooft, R. W. W. (2004). *COLLECT*. Bruker–Nonius BV, Delft, The Netherlands.

Kassis, P., Beneteau, V., Merour, J.-Y. & Routier, S. (2009). *Synthesis*, pp. 2447–2453.
Lu, T.-J. & Lin, C.-K. (2011). *J. Org. Chem.* **76**, 1621–1633.
Molander, G. A., Canturk, B. & Kennedy, L. E. (2009). *J. Org. Chem.* **74**, 973–980.
Mothes, C., Lavielle, S. & Karoyan, P. (2008). *J. Org. Chem.* **73**, 6706–6710.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Reiter, M., Torssell, S., Lee, S. & MacMillan, D. W. C. (2010). *Chem. Sci.* **1**, 37–42.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supplementary materials

*Acta Cryst.* (2012). E**68**, m551–m552 [doi:10.1107/S1600536812014225]

## Potassium [1-(*tert*-butoxycarbonyl)-1*H*-indol-3-yl]trifluoroborate hemihydrate

**Guillaume Berionni, Peter Mayer and Herbert Mayr**

### Comment

Organotrifluoroborates and, in particular, indolyltrifluoroborates [Molander *et al.* (2009), Kassis *et al.* (2009), Reiter *et al.* (2010)] are synthetically useful nucleophiles for Suzuki-Miyaura cross-coupling and other CC bond-forming reactions [Darses & Genet (2008)].

The asymmetric unit contains two formula units of the title compound (Fig. 1). The B–C bond distances in the two indolyltrifluoroborate anions are found to be 1.596 (3) Å and 1.600 (3) Å. These bond distances are close to the mean distance of 1.619 Å determined from 33 crystal structures of organotrifluoroborates (CSD version 5.33, Nov 2011). In the title compound, the 1-*tert*-butoxycarbonyl group is not coplanar with the indolyl ring, but deviates with plane-plane angles of 20.44 (11)° and 21.02 (10)°, in which the plane of a *tert*-butoxycarbonyl group is defined by its C and O atoms with the exception of the methyl groups. This structural feature is observed in several of the dozen of crystal structures of 3-substituted 1-(*tert*-Butoxycarbonyl)-1*H*-indolyl derivatives [Baran *et al.* (2005), Davies *et al.* (2005), Davies *et al.* (2007) and Lu & Lin (2011)].

The coordination sphere of K1 consists of two oxygen atoms and four fluorine atoms in bond distances ranging between 2.58 Å and 2.99 Å. Additionally the five-membered ring of an adjacent indolyl moiety is bound by a weak potassium-$\pi$ interaction (distance K1–*Cg*(N2, C14—C17) = 3.722 Å). The other potassium ion is coordinated by five fluorine atoms and one oxygen atom in bond distances ranging from 2.62 Å to 2.77 Å. The water molecule is coordinated solely to K1 and forms sequential hydrogen bonds of the type O–H···O along [100]. The other proton of the water molecule acts as donor in hydrogen bonds of the type O–H···F. Layers parallel to *ab* are formed by the combination of hydrogen bonds and coordination of the potassium ions (Fig. 2). Weak C–H···$\pi$ interactions between methyl-hydrogen atoms and the six-membered rings of the indolyl moieties are established with C···*Cg* distances of 3.836 (3) Å and 3.715 (2) Å. The packing of the title compound is shown in Fig. 3.

### Experimental

In a 100 ml Schlenck flask under argon, 1.00 g (2.92 mmol,1 eq.) of *tert*-butyl-3-iodo-1*H*-indole-1-carboxylate [Mothes *et al.* (2008)] was dissolved in 20 ml of freshly distilled THF and cooled to -78 °C. With a syringe, 1.37 ml of nBuLi (2.13 *M* in hexane, 2.92 mmol,1 eq.) was added dropwise, and the solution was stirred for 30 min at this temperature whilst turning orange pale. Neat triisopropyl borate (3.36 mmol, 0.55 ml, 1.15 eq.) was then slowly dropped to the mixture, and after removing the cooling bath the temperature reached 0 °C in 30 min. An aqueous solution of $KHF_2$ (17.5 mmol, 1.35 g, 6 eq. dissolved in 5 ml of $H_2O$) was added slowly to the limpid solution under vigorous stirring and after 15 minutes the solvents were removed *in vacuo*. The waxy solid was dissolved in 50 ml of hot acetone and filtrated. The filtrate was concentrated to 10 ml before adding 10 ml of diethylether. After one night in the fridge, colorless crystalline plates of the title compound were obtained (650 mg, 70%).

### Refinement

C-bound H atoms were positioned geometrically (C—H = 0.98 Å for aliphatic, 0.95 Å for aromatic H) and treated as riding on their parent atoms [$U_{iso}$(H) = 1.2$U_{eq}$(C, aromatic), $U_{iso}$(H) = 1.5$U_{eq}$(C, aliphatic)]. The methyl groups were allowed to rotate along the C–O bonds to best fit the experimental electron density. The hydrogen atoms of the water molecule were fixed to O–H distances of 0.82 (1) Å [$U_{iso}$(H) = 1.2$U_{eq}$(O)].

### Computing details

Data collection: *COLLECT* (Hooft, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

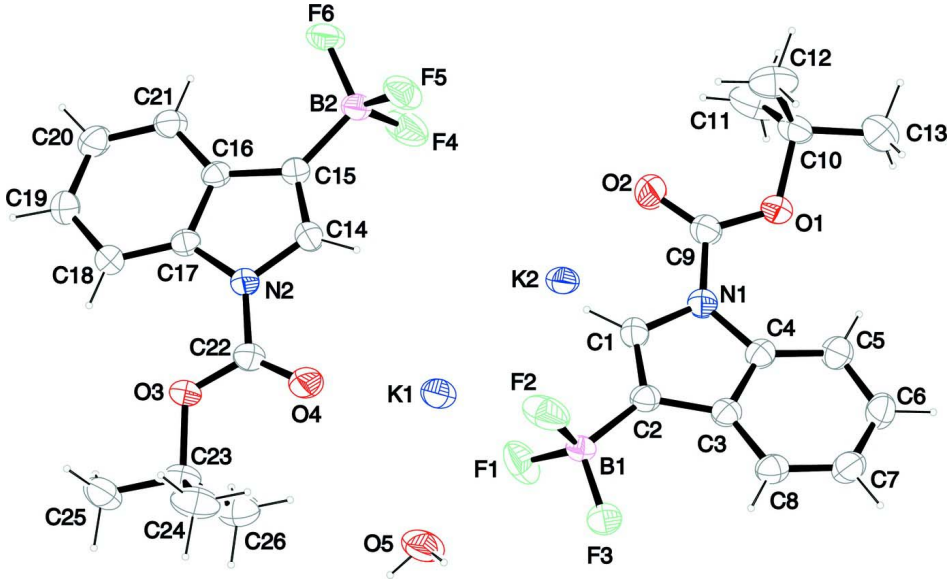



**Figure 1**

The molecular structures of the asymmetric unit (contains two formula units of the title compound), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

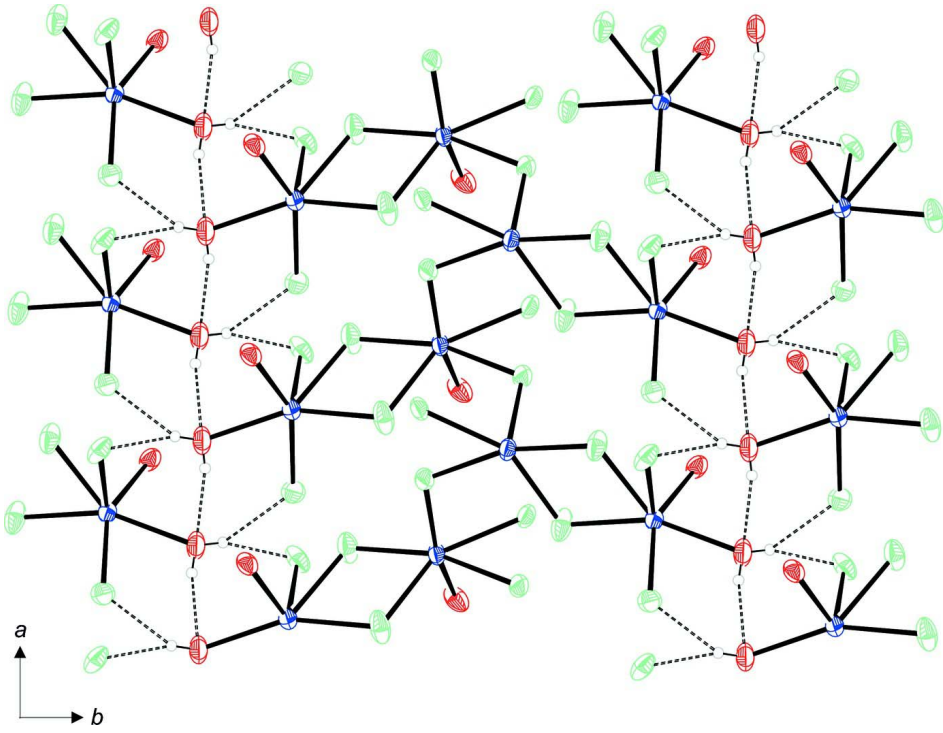



**Figure 2**

View on a layer formed by potassium ions and their coordinating O and F atoms. All other atoms have been omitted for clarity. Dashed lines indicate hydrogen bonds, fat solid lines K–O and K–F bonds.

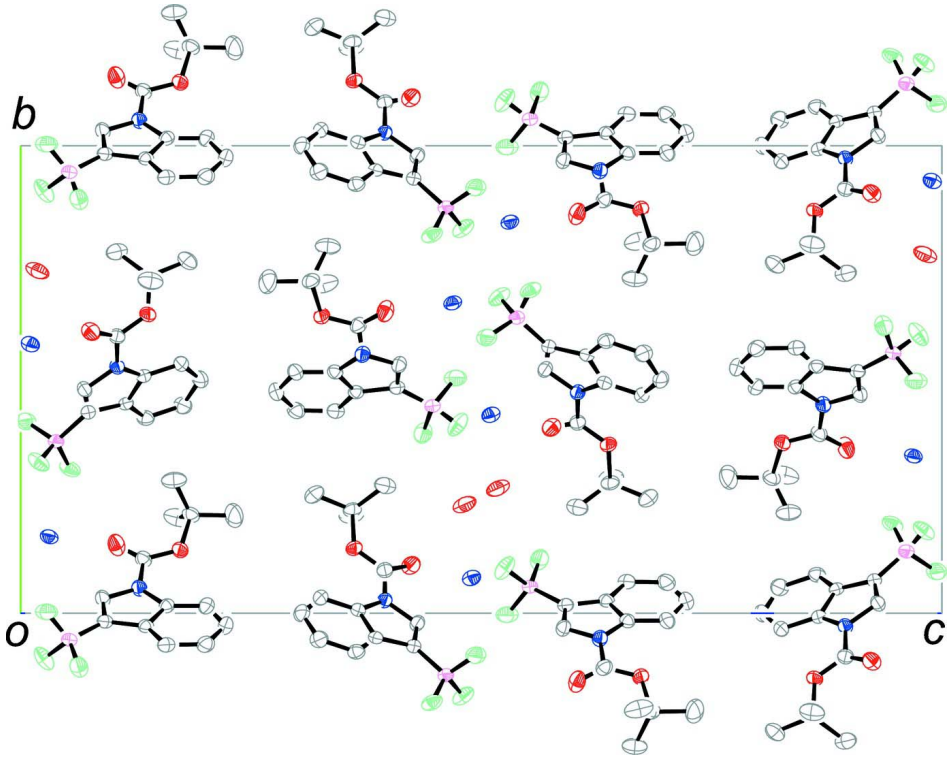



**Figure 3**

The packing of the title compound viewed along [100]. Hydrogen atoms have been omitted for clarity. Fragments located outside the unit cell have been completed.

**Potassium [1-(*tert*-butoxycarbonyl)-1*H*-indol-3-yl]trifluoroborate hemihydrate**

*Crystal data*

$K^+ \cdot C_{13}H_{14}BF_3NO_2 \cdot 0.5H_2O$
$M_r = 332.17$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.8428$ (1) Å
$b = 16.3177$ (2) Å
$c = 32.1286$ (5) Å
$V = 3063.17$ (8) Å$^3$
$Z = 8$
$F(000) = 1368$
$D_x = 1.441$ (1) Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 12366 reflections
$\theta = 3.1$–$27.5°$
$\mu = 0.38$ mm$^{-1}$
$T = 173$ K
Block, colourless
0.27 × 0.19 × 0.10 mm

*Data collection*

Nonius KappaCCD diffractometer
Radiation source: rotating anode
MONTEL, graded multilayered X-ray optics monochromator
CCD; rotation images scans
24771 measured reflections
7001 independent reflections
6369 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -7 \rightarrow 7$
$k = -21 \rightarrow 21$
$l = -41 \rightarrow 41$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.075$
$S = 1.03$
7001 reflections
400 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 1.0137P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$
Absolute structure: Flack (1983), 2995 Friedel pairs
Flack parameter: 0.00 (3)

*Special details*

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

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| K1 | 0.23271 (7) | −0.07549 (2) | −0.010496 (14) | 0.03663 (10) |
| K2 | −0.44437 (8) | 0.16361 (2) | 0.030974 (13) | 0.03596 (10) |
| F1 | −0.1583 (2) | −0.09666 (9) | 0.02545 (4) | 0.0531 (3) |
| F2 | −0.4208 (3) | 0.00302 (7) | 0.02809 (4) | 0.0579 (4) |
| F3 | −0.4763 (2) | −0.11459 (8) | 0.06318 (4) | 0.0515 (3) |
| F4 | 1.2008 (3) | 0.08962 (7) | −0.00590 (4) | 0.0599 (4) |
| F5 | 0.9079 (2) | 0.17683 (7) | −0.01961 (4) | 0.0475 (3) |
| F6 | 1.2432 (2) | 0.19073 (7) | −0.05353 (4) | 0.0441 (3) |
| O1 | 0.2775 (2) | 0.13553 (7) | 0.17310 (4) | 0.0355 (3) |
| O2 | 0.3463 (3) | 0.14600 (10) | 0.10405 (4) | 0.0501 (4) |
| O3 | 0.6469 (2) | −0.13910 (7) | −0.13800 (4) | 0.0331 (3) |
| O4 | 0.4899 (3) | −0.10246 (8) | −0.07628 (4) | 0.0427 (3) |
| O5 | 0.0801 (3) | −0.23252 (10) | −0.01815 (6) | 0.0562 (4) |
| H51 | 0.097 (5) | −0.2697 (12) | −0.0347 (7) | 0.067* |
| H52 | −0.052 (2) | −0.2317 (18) | −0.0103 (9) | 0.067* |
| N1 | 0.0878 (3) | 0.05375 (9) | 0.12880 (5) | 0.0323 (3) |
| N2 | 0.7777 (3) | −0.02748 (9) | −0.10475 (4) | 0.0294 (3) |
| C1 | 0.0068 (3) | 0.03130 (11) | 0.08919 (6) | 0.0332 (4) |
| H1 | 0.0749 | 0.0484 | 0.0638 | 0.040* |
| C2 | −0.1791 (3) | −0.01743 (10) | 0.09157 (6) | 0.0289 (4) |
| C3 | −0.2240 (3) | −0.02728 (10) | 0.13595 (5) | 0.0285 (4) |
| C4 | −0.0545 (3) | 0.01594 (10) | 0.15856 (5) | 0.0290 (4) |
| C5 | −0.0473 (4) | 0.01580 (11) | 0.20163 (6) | 0.0362 (4) |
| H5 | 0.0708 | 0.0433 | 0.2164 | 0.043* |

| | | | | |
|---|---|---|---|---|
| C6 | −0.2203 (4) | −0.02636 (12) | 0.22244 (6) | 0.0404 (5) |
| H6 | −0.2196 | −0.0279 | 0.2520 | 0.049* |
| C7 | −0.3937 (4) | −0.06622 (12) | 0.20090 (6) | 0.0412 (5) |
| H7 | −0.5118 | −0.0930 | 0.2160 | 0.049* |
| C8 | −0.3975 (3) | −0.06758 (11) | 0.15802 (6) | 0.0339 (4) |
| H8 | −0.5162 | −0.0955 | 0.1436 | 0.041* |
| C9 | 0.2502 (4) | 0.11582 (11) | 0.13339 (6) | 0.0343 (4) |
| C10 | 0.4090 (4) | 0.21055 (11) | 0.18483 (6) | 0.0359 (4) |
| C11 | 0.6548 (4) | 0.20440 (15) | 0.17096 (10) | 0.0607 (7) |
| H11A | 0.7180 | 0.1515 | 0.1796 | 0.091* |
| H11B | 0.7438 | 0.2488 | 0.1836 | 0.091* |
| H11C | 0.6625 | 0.2090 | 0.1406 | 0.091* |
| C12 | 0.2916 (4) | 0.28447 (12) | 0.16649 (8) | 0.0499 (6) |
| H12A | 0.3055 | 0.2831 | 0.1361 | 0.075* |
| H12B | 0.3636 | 0.3344 | 0.1772 | 0.075* |
| H12C | 0.1294 | 0.2839 | 0.1742 | 0.075* |
| C13 | 0.3855 (7) | 0.20916 (19) | 0.23156 (8) | 0.0838 (11) |
| H13A | 0.2229 | 0.2093 | 0.2391 | 0.126* |
| H13B | 0.4599 | 0.2577 | 0.2434 | 0.126* |
| H13C | 0.4583 | 0.1596 | 0.2426 | 0.126* |
| C14 | 0.8113 (3) | 0.02142 (11) | −0.06941 (6) | 0.0318 (4) |
| H14 | 0.7153 | 0.0204 | −0.0456 | 0.038* |
| C15 | 0.9966 (3) | 0.07020 (10) | −0.07336 (5) | 0.0275 (4) |
| C16 | 1.0897 (3) | 0.05186 (9) | −0.11437 (5) | 0.0258 (3) |
| C17 | 0.9503 (3) | −0.00726 (10) | −0.13362 (5) | 0.0265 (3) |
| C18 | 0.9858 (3) | −0.03336 (11) | −0.17426 (6) | 0.0341 (4) |
| H18 | 0.8871 | −0.0719 | −0.1873 | 0.041* |
| C19 | 1.1730 (4) | −0.00034 (13) | −0.19486 (6) | 0.0416 (5) |
| H19 | 1.2024 | −0.0166 | −0.2227 | 0.050* |
| C20 | 1.3184 (4) | 0.05562 (12) | −0.17607 (7) | 0.0412 (5) |
| H20 | 1.4465 | 0.0760 | −0.1911 | 0.049* |
| C21 | 1.2797 (3) | 0.08230 (11) | −0.13564 (6) | 0.0334 (4) |
| H21 | 1.3802 | 0.1204 | −0.1228 | 0.040* |
| C22 | 0.6229 (3) | −0.09235 (11) | −0.10457 (6) | 0.0318 (4) |
| C23 | 0.5320 (3) | −0.22094 (11) | −0.14000 (6) | 0.0335 (4) |
| C24 | 0.2743 (4) | −0.21102 (13) | −0.14087 (9) | 0.0508 (6) |
| H24A | 0.2026 | −0.2642 | −0.1466 | 0.076* |
| H24B | 0.2320 | −0.1720 | −0.1627 | 0.076* |
| H24C | 0.2214 | −0.1905 | −0.1139 | 0.076* |
| C25 | 0.6211 (4) | −0.25395 (13) | −0.18113 (7) | 0.0471 (5) |
| H25A | 0.7885 | −0.2570 | −0.1802 | 0.071* |
| H25B | 0.5741 | −0.2173 | −0.2038 | 0.071* |
| H25C | 0.5581 | −0.3088 | −0.1860 | 0.071* |
| C26 | 0.6157 (4) | −0.27308 (13) | −0.10437 (7) | 0.0478 (5) |
| H26A | 0.7833 | −0.2720 | −0.1036 | 0.072* |
| H26B | 0.5631 | −0.3296 | −0.1082 | 0.072* |
| H26C | 0.5550 | −0.2515 | −0.0781 | 0.072* |
| B1 | −0.3097 (3) | −0.05677 (11) | 0.05280 (6) | 0.0262 (4) |
| B2 | 1.0867 (4) | 0.13229 (12) | −0.03848 (6) | 0.0298 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| K1 | 0.0355 (2) | 0.03093 (19) | 0.0434 (2) | −0.00376 (18) | 0.00348 (19) | −0.00575 (17) |
| K2 | 0.0414 (2) | 0.02571 (18) | 0.0408 (2) | −0.00664 (17) | 0.00910 (19) | −0.00547 (16) |
| F1 | 0.0414 (7) | 0.0679 (8) | 0.0500 (7) | −0.0022 (6) | 0.0071 (6) | −0.0286 (6) |
| F2 | 0.0777 (9) | 0.0317 (6) | 0.0642 (8) | 0.0021 (7) | −0.0338 (8) | −0.0013 (5) |
| F3 | 0.0583 (8) | 0.0541 (7) | 0.0420 (7) | −0.0296 (7) | 0.0088 (6) | −0.0065 (5) |
| F4 | 0.0919 (11) | 0.0359 (6) | 0.0519 (7) | −0.0013 (7) | −0.0370 (8) | −0.0022 (5) |
| F5 | 0.0419 (7) | 0.0433 (6) | 0.0574 (8) | −0.0076 (6) | 0.0157 (6) | −0.0225 (5) |
| F6 | 0.0435 (7) | 0.0332 (6) | 0.0558 (7) | −0.0152 (6) | 0.0148 (6) | −0.0137 (5) |
| O1 | 0.0416 (8) | 0.0321 (6) | 0.0327 (7) | −0.0097 (6) | −0.0022 (6) | −0.0023 (5) |
| O2 | 0.0538 (10) | 0.0598 (10) | 0.0367 (8) | −0.0258 (8) | 0.0127 (7) | −0.0080 (7) |
| O3 | 0.0378 (7) | 0.0276 (6) | 0.0340 (7) | −0.0098 (5) | −0.0004 (6) | −0.0035 (5) |
| O4 | 0.0422 (8) | 0.0414 (8) | 0.0446 (8) | −0.0145 (7) | 0.0127 (7) | −0.0065 (6) |
| O5 | 0.0654 (11) | 0.0346 (8) | 0.0688 (12) | −0.0079 (8) | 0.0163 (10) | −0.0192 (7) |
| N1 | 0.0332 (9) | 0.0338 (8) | 0.0298 (8) | −0.0077 (7) | 0.0014 (7) | −0.0021 (6) |
| N2 | 0.0310 (8) | 0.0291 (7) | 0.0280 (7) | −0.0059 (6) | 0.0025 (6) | −0.0019 (6) |
| C1 | 0.0350 (10) | 0.0363 (9) | 0.0283 (9) | −0.0063 (8) | 0.0040 (7) | −0.0039 (7) |
| C2 | 0.0301 (9) | 0.0234 (8) | 0.0333 (9) | −0.0002 (7) | 0.0028 (7) | −0.0010 (7) |
| C3 | 0.0303 (9) | 0.0227 (8) | 0.0325 (9) | 0.0042 (7) | 0.0031 (7) | 0.0001 (6) |
| C4 | 0.0293 (9) | 0.0264 (8) | 0.0314 (9) | 0.0032 (8) | 0.0017 (8) | 0.0022 (7) |
| C5 | 0.0416 (11) | 0.0345 (9) | 0.0324 (9) | 0.0009 (9) | −0.0057 (9) | 0.0041 (7) |
| C6 | 0.0509 (13) | 0.0414 (10) | 0.0290 (9) | 0.0011 (10) | 0.0012 (9) | 0.0050 (8) |
| C7 | 0.0468 (12) | 0.0350 (10) | 0.0418 (11) | −0.0037 (9) | 0.0098 (10) | 0.0040 (8) |
| C8 | 0.0343 (10) | 0.0276 (9) | 0.0399 (10) | 0.0001 (8) | 0.0030 (8) | 0.0008 (7) |
| C9 | 0.0343 (10) | 0.0336 (9) | 0.0350 (9) | −0.0044 (9) | 0.0017 (9) | −0.0044 (7) |
| C10 | 0.0375 (11) | 0.0302 (9) | 0.0400 (10) | −0.0056 (8) | −0.0069 (9) | −0.0050 (8) |
| C11 | 0.0311 (11) | 0.0428 (12) | 0.108 (2) | 0.0024 (10) | −0.0107 (13) | −0.0169 (13) |
| C12 | 0.0343 (12) | 0.0331 (10) | 0.0822 (17) | 0.0015 (9) | −0.0019 (11) | 0.0036 (10) |
| C13 | 0.135 (3) | 0.0742 (19) | 0.0425 (14) | −0.054 (2) | −0.0084 (17) | −0.0072 (13) |
| C14 | 0.0348 (10) | 0.0327 (9) | 0.0279 (9) | −0.0042 (8) | 0.0026 (7) | −0.0050 (7) |
| C15 | 0.0310 (9) | 0.0241 (8) | 0.0274 (8) | −0.0007 (7) | 0.0005 (7) | 0.0006 (6) |
| C16 | 0.0274 (9) | 0.0205 (7) | 0.0296 (9) | 0.0013 (7) | 0.0011 (7) | 0.0028 (6) |
| C17 | 0.0276 (8) | 0.0230 (8) | 0.0290 (8) | 0.0011 (7) | −0.0007 (7) | 0.0024 (6) |
| C18 | 0.0435 (11) | 0.0300 (9) | 0.0290 (9) | −0.0046 (8) | 0.0016 (8) | −0.0018 (7) |
| C19 | 0.0533 (13) | 0.0384 (10) | 0.0331 (10) | −0.0045 (10) | 0.0127 (9) | −0.0047 (8) |
| C20 | 0.0435 (11) | 0.0372 (10) | 0.0429 (11) | −0.0076 (9) | 0.0174 (9) | −0.0015 (8) |
| C21 | 0.0319 (10) | 0.0286 (8) | 0.0396 (10) | −0.0046 (8) | 0.0031 (8) | −0.0010 (7) |
| C22 | 0.0317 (10) | 0.0298 (9) | 0.0338 (9) | −0.0054 (7) | −0.0013 (8) | −0.0011 (7) |
| C23 | 0.0301 (10) | 0.0268 (8) | 0.0435 (10) | −0.0073 (8) | −0.0057 (8) | −0.0037 (7) |
| C24 | 0.0318 (11) | 0.0388 (11) | 0.0818 (17) | −0.0052 (9) | −0.0098 (11) | −0.0083 (11) |
| C25 | 0.0520 (14) | 0.0404 (11) | 0.0488 (13) | −0.0093 (10) | −0.0064 (11) | −0.0128 (9) |
| C26 | 0.0493 (14) | 0.0346 (10) | 0.0595 (14) | −0.0018 (10) | −0.0088 (11) | 0.0058 (10) |
| B1 | 0.0258 (10) | 0.0216 (9) | 0.0313 (10) | 0.0000 (7) | 0.0003 (8) | −0.0012 (7) |
| B2 | 0.0339 (11) | 0.0230 (9) | 0.0325 (10) | −0.0025 (8) | −0.0013 (9) | −0.0011 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| K1—F1 | 2.5832 (13) | C3—C4 | 1.416 (3) |
| K1—O4 | 2.6303 (14) | C4—C5 | 1.384 (2) |
| K1—F2$^{i}$ | 2.6977 (13) | C5—C6 | 1.394 (3) |
| K1—F4$^{ii}$ | 2.7048 (12) | C5—H5 | 0.9500 |
| K1—O5 | 2.7243 (16) | C6—C7 | 1.389 (3) |
| K1—F3$^{i}$ | 2.9833 (14) | C6—H6 | 0.9500 |
| K1—B1$^{i}$ | 3.373 (2) | C7—C8 | 1.378 (3) |
| K1—C15$^{ii}$ | 3.4111 (17) | C7—H7 | 0.9500 |
| K1—C14$^{ii}$ | 3.485 (2) | C8—H8 | 0.9500 |
| K1—H52 | 3.05 (3) | C10—C11 | 1.507 (3) |
| K2—F2 | 2.6256 (12) | C10—C12 | 1.508 (3) |
| K2—F5$^{ii}$ | 2.6315 (12) | C10—C13 | 1.508 (3) |
| K2—O2$^{ii}$ | 2.6627 (14) | C11—H11A | 0.9800 |
| K2—F4$^{iii}$ | 2.6756 (14) | C11—H11B | 0.9800 |
| K2—F6$^{iv}$ | 2.7158 (12) | C11—H11C | 0.9800 |
| K2—F5$^{iv}$ | 2.7671 (12) | C12—H12A | 0.9800 |
| K2—F6$^{iii}$ | 3.3016 (14) | C12—H12B | 0.9800 |
| K2—B2$^{iv}$ | 3.344 (2) | C12—H12C | 0.9800 |
| F1—B1 | 1.406 (2) | C13—H13A | 0.9800 |
| F2—B1 | 1.415 (2) | C13—H13B | 0.9800 |
| F2—K1$^{ii}$ | 2.6977 (13) | C13—H13C | 0.9800 |
| F3—B1 | 1.396 (2) | C14—C15 | 1.350 (2) |
| F3—K1$^{ii}$ | 2.9832 (14) | C14—K1$^{i}$ | 3.485 (2) |
| F4—B2 | 1.423 (2) | C14—H14 | 0.9500 |
| F4—K2$^{v}$ | 2.6756 (14) | C15—C16 | 1.457 (2) |
| F4—K1$^{i}$ | 2.7047 (12) | C15—B2 | 1.600 (3) |
| F5—B2 | 1.409 (2) | C15—K1$^{i}$ | 3.4111 (17) |
| F5—K2$^{i}$ | 2.6316 (12) | C16—C21 | 1.395 (2) |
| F5—K2$^{vi}$ | 2.7671 (12) | C16—C17 | 1.406 (2) |
| F6—B2 | 1.407 (2) | C17—C18 | 1.389 (2) |
| F6—K2$^{vi}$ | 2.7157 (12) | C18—C19 | 1.387 (3) |
| F6—K2$^{v}$ | 3.3017 (14) | C18—H18 | 0.9500 |
| O1—C9 | 1.325 (2) | C19—C20 | 1.386 (3) |
| O1—C10 | 1.494 (2) | C19—H19 | 0.9500 |
| O2—C9 | 1.203 (2) | C20—C21 | 1.388 (3) |
| O2—K2$^{i}$ | 2.6627 (14) | C20—H20 | 0.9500 |
| O3—C22 | 1.325 (2) | C21—H21 | 0.9500 |
| O3—C23 | 1.496 (2) | C23—C26 | 1.508 (3) |
| O4—C22 | 1.207 (2) | C23—C24 | 1.515 (3) |
| O5—H51 | 0.812 (10) | C23—C25 | 1.519 (3) |
| O5—H52 | 0.813 (10) | C24—H24A | 0.9800 |
| N1—C9 | 1.396 (2) | C24—H24B | 0.9800 |
| N1—C1 | 1.406 (2) | C24—H24C | 0.9800 |
| N1—C4 | 1.409 (2) | C25—H25A | 0.9800 |
| N2—C22 | 1.393 (2) | C25—H25B | 0.9800 |
| N2—C14 | 1.402 (2) | C25—H25C | 0.9800 |
| N2—C17 | 1.409 (2) | C26—H26A | 0.9800 |
| C1—C2 | 1.348 (3) | C26—H26B | 0.9800 |

| | | | |
|---|---|---|---|
| C1—H1 | 0.9500 | C26—H26C | 0.9800 |
| C2—C3 | 1.459 (2) | B1—K1$^{ii}$ | 3.373 (2) |
| C2—B1 | 1.596 (3) | B2—K2$^{vi}$ | 3.344 (2) |
| C3—C8 | 1.401 (3) | | |

| | | | |
|---|---|---|---|
| F1—K1—O4 | 147.37 (4) | K1—O5—H52 | 106 (2) |
| F1—K1—F2$^{i}$ | 121.45 (5) | H51—O5—H52 | 109 (3) |
| O4—K1—F2$^{i}$ | 91.15 (5) | C9—N1—C1 | 120.89 (15) |
| F1—K1—F4$^{ii}$ | 92.74 (5) | C9—N1—C4 | 130.33 (15) |
| O4—K1—F4$^{ii}$ | 104.47 (5) | C1—N1—C4 | 107.54 (15) |
| F2$^{i}$—K1—F4$^{ii}$ | 63.48 (4) | C22—N2—C14 | 121.35 (15) |
| F1—K1—O5 | 67.98 (5) | C22—N2—C17 | 130.21 (15) |
| O4—K1—O5 | 87.55 (5) | C14—N2—C17 | 107.45 (14) |
| F2$^{i}$—K1—O5 | 137.21 (5) | C2—C1—N1 | 111.93 (16) |
| F4$^{ii}$—K1—O5 | 156.82 (6) | C2—C1—H1 | 124.0 |
| F1—K1—F3$^{i}$ | 96.93 (4) | N1—C1—H1 | 124.0 |
| O4—K1—F3$^{i}$ | 106.04 (4) | C1—C2—C3 | 105.39 (16) |
| F2$^{i}$—K1—F3$^{i}$ | 46.31 (3) | C1—C2—B1 | 125.33 (16) |
| F4$^{ii}$—K1—F3$^{i}$ | 102.06 (4) | C3—C2—B1 | 129.24 (16) |
| O5—K1—F3$^{i}$ | 93.27 (5) | C8—C3—C4 | 118.68 (16) |
| F1—K1—B1$^{i}$ | 116.33 (5) | C8—C3—C2 | 132.59 (17) |
| O4—K1—B1$^{i}$ | 92.69 (5) | C4—C3—C2 | 108.70 (15) |
| F2$^{i}$—K1—B1$^{i}$ | 23.80 (4) | C5—C4—N1 | 131.36 (18) |
| F4$^{ii}$—K1—B1$^{i}$ | 86.07 (4) | C5—C4—C3 | 122.22 (17) |
| O5—K1—B1$^{i}$ | 113.52 (6) | N1—C4—C3 | 106.41 (15) |
| F3$^{i}$—K1—B1$^{i}$ | 24.40 (4) | C4—C5—C6 | 117.27 (19) |
| F1—K1—C15$^{ii}$ | 90.01 (5) | C4—C5—H5 | 121.4 |
| O4—K1—C15$^{ii}$ | 82.64 (5) | C6—C5—H5 | 121.4 |
| F2$^{i}$—K1—C15$^{ii}$ | 104.16 (4) | C7—C6—C5 | 121.43 (18) |
| F4$^{ii}$—K1—C15$^{ii}$ | 46.38 (4) | C7—C6—H6 | 119.3 |
| O5—K1—C15$^{ii}$ | 118.01 (5) | C5—C6—H6 | 119.3 |
| F3$^{i}$—K1—C15$^{ii}$ | 148.14 (4) | C8—C7—C6 | 121.14 (19) |
| B1$^{i}$—K1—C15$^{ii}$ | 127.92 (4) | C8—C7—H7 | 119.4 |
| F1—K1—C14$^{ii}$ | 71.25 (5) | C6—C7—H7 | 119.4 |
| O4—K1—C14$^{ii}$ | 92.47 (5) | C7—C8—C3 | 119.15 (19) |
| F2$^{i}$—K1—C14$^{ii}$ | 124.35 (4) | C7—C8—H8 | 120.4 |
| F4$^{ii}$—K1—C14$^{ii}$ | 61.90 (4) | C3—C8—H8 | 120.4 |
| O5—K1—C14$^{ii}$ | 98.42 (5) | O2—C9—O1 | 126.80 (18) |
| F3$^{i}$—K1—C14$^{ii}$ | 158.52 (4) | O2—C9—N1 | 122.11 (17) |
| B1$^{i}$—K1—C14$^{ii}$ | 147.81 (4) | O1—C9—N1 | 111.08 (16) |
| C15$^{ii}$—K1—C14$^{ii}$ | 22.54 (4) | O1—C10—C11 | 111.15 (16) |
| F1—K1—K2$^{i}$ | 110.59 (3) | O1—C10—C12 | 108.85 (16) |
| O4—K1—K2$^{i}$ | 98.19 (3) | C11—C10—C12 | 111.8 (2) |
| F2$^{i}$—K1—K2$^{i}$ | 31.13 (3) | O1—C10—C13 | 101.06 (17) |
| F4$^{ii}$—K1—K2$^{i}$ | 32.38 (3) | C11—C10—C13 | 112.4 (2) |
| O5—K1—K2$^{i}$ | 166.44 (5) | C12—C10—C13 | 111.1 (2) |
| F3$^{i}$—K1—K2$^{i}$ | 73.36 (2) | C10—C11—H11A | 109.5 |
| B1$^{i}$—K1—K2$^{i}$ | 54.20 (3) | C10—C11—H11B | 109.5 |
| C15$^{ii}$—K1—K2$^{i}$ | 75.08 (3) | H11A—C11—H11B | 109.5 |

| | | | |
|---|---|---|---|
| C14$^{ii}$—K1—K2$^{i}$ | 93.62 (3) | C10—C11—H11C | 109.5 |
| F1—K1—H52 | 53.4 (3) | H11A—C11—H11C | 109.5 |
| O4—K1—H52 | 100.0 (4) | H11B—C11—H11C | 109.5 |
| F2$^{i}$—K1—H52 | 143.8 (5) | C10—C12—H12A | 109.5 |
| F4$^{ii}$—K1—H52 | 142.8 (3) | C10—C12—H12B | 109.5 |
| O5—K1—H52 | 14.9 (3) | H12A—C12—H12B | 109.5 |
| F3$^{i}$—K1—H52 | 97.5 (5) | C10—C12—H12C | 109.5 |
| B1$^{i}$—K1—H52 | 120.5 (5) | H12A—C12—H12C | 109.5 |
| C15$^{ii}$—K1—H52 | 111.3 (5) | H12B—C12—H12C | 109.5 |
| C14$^{ii}$—K1—H52 | 89.7 (5) | C10—C13—H13A | 109.5 |
| K2$^{i}$—K1—H52 | 161.3 (5) | C10—C13—H13B | 109.5 |
| F2—K2—F5$^{ii}$ | 91.09 (5) | H13A—C13—H13B | 109.5 |
| F2—K2—O2$^{ii}$ | 86.99 (5) | C10—C13—H13C | 109.5 |
| F5$^{ii}$—K2—O2$^{ii}$ | 155.88 (5) | H13A—C13—H13C | 109.5 |
| F2—K2—F4$^{iii}$ | 64.83 (4) | H13B—C13—H13C | 109.5 |
| F5$^{ii}$—K2—F4$^{iii}$ | 111.69 (5) | C15—C14—N2 | 111.82 (16) |
| O2$^{ii}$—K2—F4$^{iii}$ | 89.19 (5) | C15—C14—K1$^{i}$ | 75.65 (11) |
| F2—K2—F6$^{iv}$ | 149.50 (4) | N2—C14—K1$^{i}$ | 106.29 (11) |
| F5$^{ii}$—K2—F6$^{iv}$ | 77.14 (4) | C15—C14—H14 | 124.1 |
| O2$^{ii}$—K2—F6$^{iv}$ | 92.55 (4) | N2—C14—H14 | 124.1 |
| F4$^{iii}$—K2—F6$^{iv}$ | 145.66 (4) | K1$^{i}$—C14—H14 | 88.3 |
| F2—K2—F5$^{iv}$ | 161.94 (4) | C14—C15—C16 | 105.29 (15) |
| F5$^{ii}$—K2—F5$^{iv}$ | 94.90 (3) | C14—C15—B2 | 124.85 (16) |
| O2$^{ii}$—K2—F5$^{iv}$ | 94.27 (5) | C16—C15—B2 | 129.85 (15) |
| F4$^{iii}$—K2—F5$^{iv}$ | 97.15 (4) | C14—C15—K1$^{i}$ | 81.81 (11) |
| F6$^{iv}$—K2—F5$^{iv}$ | 48.52 (3) | C16—C15—K1$^{i}$ | 103.96 (10) |
| F2—K2—F6$^{iii}$ | 97.69 (3) | B2—C15—K1$^{i}$ | 83.88 (10) |
| F5$^{ii}$—K2—F6$^{iii}$ | 85.04 (4) | C21—C16—C17 | 119.33 (16) |
| O2$^{ii}$—K2—F6$^{iii}$ | 119.05 (4) | C21—C16—C15 | 131.86 (16) |
| F4$^{iii}$—K2—F6$^{iii}$ | 42.94 (3) | C17—C16—C15 | 108.80 (15) |
| F6$^{iv}$—K2—F6$^{iii}$ | 108.98 (2) | C18—C17—C16 | 122.48 (16) |
| F5$^{iv}$—K2—F6$^{iii}$ | 65.98 (3) | C18—C17—N2 | 130.85 (16) |
| F2—K2—B2$^{iv}$ | 173.53 (5) | C16—C17—N2 | 106.60 (14) |
| F5$^{ii}$—K2—B2$^{iv}$ | 85.44 (4) | C19—C18—C17 | 116.58 (18) |
| O2$^{ii}$—K2—B2$^{iv}$ | 93.94 (5) | C19—C18—H18 | 121.7 |
| F4$^{iii}$—K2—B2$^{iv}$ | 121.56 (5) | C17—C18—H18 | 121.7 |
| F6$^{iv}$—K2—B2$^{iv}$ | 24.11 (4) | C20—C19—C18 | 122.10 (18) |
| F5$^{iv}$—K2—B2$^{iv}$ | 24.41 (4) | C20—C19—H19 | 119.0 |
| F6$^{iii}$—K2—B2$^{iv}$ | 87.47 (4) | C18—C19—H19 | 118.9 |
| F2—K2—B2$^{iii}$ | 82.84 (4) | C19—C20—C21 | 120.94 (18) |
| F5$^{ii}$—K2—B2$^{iii}$ | 103.06 (5) | C19—C20—H20 | 119.5 |
| O2$^{ii}$—K2—B2$^{iii}$ | 100.55 (5) | C21—C20—H20 | 119.5 |
| F4$^{iii}$—K2—B2$^{iii}$ | 20.62 (4) | C20—C21—C16 | 118.46 (17) |
| F6$^{iv}$—K2—B2$^{iii}$ | 127.00 (4) | C20—C21—H21 | 120.8 |
| F5$^{iv}$—K2—B2$^{iii}$ | 79.22 (4) | C16—C21—H21 | 120.8 |
| F6$^{iii}$—K2—B2$^{iii}$ | 23.21 (4) | O4—C22—O3 | 126.87 (17) |
| B2$^{iv}$—K2—B2$^{iii}$ | 103.25 (5) | O4—C22—N2 | 121.69 (17) |
| F2—K2—K2$^{vii}$ | 129.84 (3) | O3—C22—N2 | 111.43 (15) |
| F5$^{ii}$—K2—K2$^{vii}$ | 100.51 (3) | O3—C23—C26 | 109.01 (15) |

| | | | |
|---|---|---|---|
| O2$^{ii}$—K2—K2$^{vii}$ | 99.12 (4) | O3—C23—C24 | 110.57 (16) |
| F4$^{iii}$—K2—K2$^{vii}$ | 65.53 (3) | C26—C23—C24 | 113.36 (19) |
| F6$^{iv}$—K2—K2$^{vii}$ | 80.36 (3) | O3—C23—C25 | 101.55 (15) |
| F5$^{iv}$—K2—K2$^{vii}$ | 32.19 (2) | C26—C23—C25 | 110.46 (18) |
| F6$^{iii}$—K2—K2$^{vii}$ | 36.60 (2) | C24—C23—C25 | 111.24 (19) |
| B2$^{iv}$—K2—K2$^{vii}$ | 56.35 (4) | C23—C24—H24A | 109.5 |
| B2$^{iii}$—K2—K2$^{vii}$ | 47.03 (3) | C23—C24—H24B | 109.5 |
| F2—K2—K2$^{viii}$ | 124.95 (4) | H24A—C24—H24B | 109.5 |
| F5$^{ii}$—K2—K2$^{viii}$ | 34.07 (2) | C23—C24—H24C | 109.5 |
| O2$^{ii}$—K2—K2$^{viii}$ | 138.78 (3) | H24A—C24—H24C | 109.5 |
| F4$^{iii}$—K2—K2$^{viii}$ | 125.95 (3) | H24B—C24—H24C | 109.5 |
| F6$^{iv}$—K2—K2$^{viii}$ | 46.46 (3) | C23—C25—H25A | 109.5 |
| F5$^{iv}$—K2—K2$^{viii}$ | 63.69 (3) | C23—C25—H25B | 109.5 |
| F6$^{iii}$—K2—K2$^{viii}$ | 84.93 (2) | H25A—C25—H25B | 109.5 |
| B2$^{iv}$—K2—K2$^{viii}$ | 51.37 (4) | C23—C25—H25C | 109.5 |
| B2$^{iii}$—K2—K2$^{viii}$ | 108.06 (4) | H25A—C25—H25C | 109.5 |
| K2$^{vii}$—K2—K2$^{viii}$ | 80.496 (13) | H25B—C25—H25C | 109.5 |
| F2—K2—K1$^{ii}$ | 32.08 (3) | C23—C26—H26A | 109.5 |
| F5$^{ii}$—K2—K1$^{ii}$ | 102.39 (3) | C23—C26—H26B | 109.5 |
| O2$^{ii}$—K2—K1$^{ii}$ | 88.57 (3) | H26A—C26—H26B | 109.5 |
| F4$^{iii}$—K2—K1$^{ii}$ | 32.77 (3) | C23—C26—H26C | 109.5 |
| F6$^{iv}$—K2—K1$^{ii}$ | 178.10 (3) | H26A—C26—H26C | 109.5 |
| F5$^{iv}$—K2—K1$^{ii}$ | 129.88 (3) | H26B—C26—H26C | 109.5 |
| F6$^{iii}$—K2—K1$^{ii}$ | 69.12 (2) | F3—B1—F1 | 105.97 (14) |
| B2$^{iv}$—K2—K1$^{ii}$ | 154.26 (4) | F3—B1—F2 | 106.27 (15) |
| B2$^{iii}$—K2—K1$^{ii}$ | 51.23 (3) | F1—B1—F2 | 104.90 (16) |
| K2$^{vii}$—K2—K1$^{ii}$ | 97.952 (11) | F3—B1—C2 | 114.77 (15) |
| K2$^{viii}$—K2—K1$^{ii}$ | 132.537 (15) | F1—B1—C2 | 111.89 (15) |
| B1—F1—K1 | 140.70 (11) | F2—B1—C2 | 112.31 (14) |
| B1—F2—K2 | 133.82 (11) | F3—B1—K1$^{ii}$ | 61.98 (9) |
| B1—F2—K1$^{ii}$ | 105.92 (10) | F1—B1—K1$^{ii}$ | 94.60 (10) |
| K2—F2—K1$^{ii}$ | 116.79 (5) | F2—B1—K1$^{ii}$ | 50.28 (8) |
| B1—F3—K1$^{ii}$ | 93.63 (10) | C2—B1—K1$^{ii}$ | 152.40 (12) |
| B2—F4—K2$^{v}$ | 117.91 (11) | F6—B2—F5 | 106.25 (14) |
| B2—F4—K1$^{i}$ | 118.65 (10) | F6—B2—F4 | 106.25 (16) |
| K2$^{v}$—F4—K1$^{i}$ | 114.86 (5) | F5—B2—F4 | 106.46 (16) |
| B2—F5—K2$^{i}$ | 143.41 (11) | F6—B2—C15 | 113.72 (15) |
| B2—F5—K2$^{vi}$ | 101.39 (10) | F5—B2—C15 | 112.58 (16) |
| K2$^{i}$—F5—K2$^{vi}$ | 113.74 (4) | F4—B2—C15 | 111.08 (14) |
| B2—F6—K2$^{vi}$ | 103.84 (10) | F6—B2—K2$^{vi}$ | 52.05 (8) |
| B2—F6—K2$^{v}$ | 89.19 (10) | F5—B2—K2$^{vi}$ | 54.21 (8) |
| K2$^{vi}$—F6—K2$^{v}$ | 96.95 (3) | F4—B2—K2$^{vi}$ | 117.36 (11) |
| C9—O1—C10 | 120.25 (14) | C15—B2—K2$^{vi}$ | 131.55 (12) |
| C9—O2—K2$^{i}$ | 161.75 (14) | F6—B2—K2$^{v}$ | 67.61 (10) |
| C22—O3—C23 | 120.08 (14) | F5—B2—K2$^{v}$ | 103.06 (11) |
| C22—O4—K1 | 161.78 (13) | C15—B2—K2$^{v}$ | 141.36 (12) |
| K1—O5—H51 | 136 (2) | K2$^{vi}$—B2—K2$^{v}$ | 81.60 (4) |
| | | | |
| O4—K1—F1—B1 | −147.17 (17) | C21—C16—C17—N2 | 178.67 (15) |

| | | | |
|---|---|---|---|
| F2$^{i}$—K1—F1—B1 | 35.8 (2) | C15—C16—C17—N2 | −1.82 (18) |
| F4$^{ii}$—K1—F1—B1 | −24.58 (19) | C22—N2—C17—C18 | 16.4 (3) |
| O5—K1—F1—B1 | 168.7 (2) | C14—N2—C17—C18 | −175.10 (19) |
| F3$^{i}$—K1—F1—B1 | 77.93 (19) | C22—N2—C17—C16 | −166.55 (17) |
| B1$^{i}$—K1—F1—B1 | 62.39 (19) | C14—N2—C17—C16 | 1.93 (18) |
| C15$^{ii}$—K1—F1—B1 | −70.90 (19) | C16—C17—C18—C19 | 2.4 (3) |
| C14$^{ii}$—K1—F1—B1 | −83.63 (19) | N2—C17—C18—C19 | 178.99 (18) |
| K2$^{i}$—K1—F1—B1 | 3.15 (19) | C17—C18—C19—C20 | 0.2 (3) |
| F5$^{ii}$—K2—F2—B1 | 92.47 (17) | C18—C19—C20—C21 | −1.2 (3) |
| O2$^{ii}$—K2—F2—B1 | −63.47 (17) | C19—C20—C21—C16 | −0.4 (3) |
| F4$^{iii}$—K2—F2—B1 | −153.99 (18) | C17—C16—C21—C20 | 2.9 (3) |
| F6$^{iv}$—K2—F2—B1 | 26.4 (2) | C15—C16—C21—C20 | −176.48 (19) |
| F5$^{iv}$—K2—F2—B1 | −158.03 (15) | K1—O4—C22—O3 | −162.5 (3) |
| F6$^{iii}$—K2—F2—B1 | 177.62 (17) | K1—O4—C22—N2 | 18.9 (6) |
| B2$^{iii}$—K2—F2—B1 | −164.50 (18) | C23—O3—C22—O4 | −11.1 (3) |
| K2$^{vii}$—K2—F2—B1 | −162.85 (15) | C23—O3—C22—N2 | 167.62 (15) |
| K2$^{viii}$—K2—F2—B1 | 88.44 (17) | C14—N2—C22—O4 | 10.1 (3) |
| K1$^{ii}$—K2—F2—B1 | −155.6 (2) | C17—N2—C22—O4 | 177.22 (18) |
| F5$^{ii}$—K2—F2—K1$^{ii}$ | −111.94 (6) | C14—N2—C22—O3 | −168.73 (16) |
| O2$^{ii}$—K2—F2—K1$^{ii}$ | 92.12 (7) | C17—N2—C22—O3 | −1.6 (3) |
| F4$^{iii}$—K2—F2—K1$^{ii}$ | 1.60 (6) | C22—O3—C23—C26 | −59.2 (2) |
| F6$^{iv}$—K2—F2—K1$^{ii}$ | −177.97 (6) | C22—O3—C23—C24 | 66.1 (2) |
| F5$^{iv}$—K2—F2—K1$^{ii}$ | −2.4 (2) | C22—O3—C23—C25 | −175.75 (16) |
| F6$^{iii}$—K2—F2—K1$^{ii}$ | −26.80 (7) | K1$^{ii}$—F3—B1—F1 | −86.68 (13) |
| B2$^{iii}$—K2—F2—K1$^{ii}$ | −8.92 (6) | K1$^{ii}$—F3—B1—F2 | 24.56 (13) |
| K2$^{vii}$—K2—F2—K1$^{ii}$ | −7.26 (9) | K1$^{ii}$—F3—B1—C2 | 149.32 (13) |
| K2$^{viii}$—K2—F2—K1$^{ii}$ | −115.98 (5) | K1—F1—B1—F3 | −170.68 (12) |
| F1—K1—O4—C22 | 137.5 (4) | K1—F1—B1—F2 | 77.1 (2) |
| F2$^{i}$—K1—O4—C22 | −45.0 (4) | K1—F1—B1—C2 | −44.9 (2) |
| F4$^{ii}$—K1—O4—C22 | 17.9 (4) | K1—F1—B1—K1$^{ii}$ | 127.17 (15) |
| O5—K1—O4—C22 | 177.8 (4) | K2—F2—B1—F3 | 128.95 (14) |
| F3$^{i}$—K1—O4—C22 | −89.5 (4) | K1$^{ii}$—F2—B1—F3 | −28.49 (15) |
| B1$^{i}$—K1—O4—C22 | −68.7 (4) | K2—F2—B1—F1 | −119.07 (15) |
| C15$^{ii}$—K1—O4—C22 | 59.1 (4) | K1$^{ii}$—F2—B1—F1 | 83.50 (13) |
| C14$^{ii}$—K1—O4—C22 | 79.5 (4) | K2—F2—B1—C2 | 2.7 (2) |
| K2$^{i}$—K1—O4—C22 | −14.5 (4) | K1$^{ii}$—F2—B1—C2 | −154.75 (12) |
| C9—N1—C1—C2 | −167.77 (17) | K2—F2—B1—K1$^{ii}$ | 157.44 (19) |
| C4—N1—C1—C2 | 0.8 (2) | C1—C2—B1—F3 | 170.96 (17) |
| N1—C1—C2—C3 | 0.3 (2) | C3—C2—B1—F3 | −6.6 (3) |
| N1—C1—C2—B1 | −177.75 (16) | C1—C2—B1—F1 | 50.2 (2) |
| C1—C2—C3—C8 | 176.85 (19) | C3—C2—B1—F1 | −127.44 (19) |
| B1—C2—C3—C8 | −5.2 (3) | C1—C2—B1—F2 | −67.5 (2) |
| C1—C2—C3—C4 | −1.3 (2) | C3—C2—B1—F2 | 114.9 (2) |
| B1—C2—C3—C4 | 176.68 (16) | C1—C2—B1—K1$^{ii}$ | −112.6 (3) |
| C9—N1—C4—C5 | −14.9 (3) | C3—C2—B1—K1$^{ii}$ | 69.8 (3) |
| C1—N1—C4—C5 | 178.0 (2) | K2$^{vi}$—F6—B2—F5 | 0.78 (16) |
| C9—N1—C4—C3 | 165.54 (18) | K2$^{v}$—F6—B2—F5 | 97.73 (13) |
| C1—N1—C4—C3 | −1.53 (19) | K2$^{vi}$—F6—B2—F4 | −112.32 (13) |
| C8—C3—C4—C5 | 3.7 (3) | K2$^{v}$—F6—B2—F4 | −15.37 (13) |

| | | | |
|---|---|---|---|
| C2—C3—C4—C5 | −177.83 (17) | K2$^{vi}$—F6—B2—C15 | 125.18 (13) |
| C8—C3—C4—N1 | −176.70 (15) | K2$^{v}$—F6—B2—C15 | −137.87 (14) |
| C2—C3—C4—N1 | 1.74 (19) | K2$^{v}$—F6—B2—K2$^{vi}$ | 96.95 (5) |
| N1—C4—C5—C6 | 178.10 (18) | K2$^{vi}$—F6—B2—K2$^{v}$ | −96.95 (5) |
| C3—C4—C5—C6 | −2.5 (3) | K2$^{i}$—F5—B2—F6 | −164.53 (12) |
| C4—C5—C6—C7 | −0.4 (3) | K2$^{vi}$—F5—B2—F6 | −0.76 (15) |
| C5—C6—C7—C8 | 1.9 (3) | K2$^{i}$—F5—B2—F4 | −51.6 (2) |
| C6—C7—C8—C3 | −0.6 (3) | K2$^{vi}$—F5—B2—F4 | 112.20 (12) |
| C4—C3—C8—C7 | −2.1 (3) | K2$^{i}$—F5—B2—C15 | 70.4 (2) |
| C2—C3—C8—C7 | 179.89 (19) | K2$^{vi}$—F5—B2—C15 | −125.86 (12) |
| K2$^{i}$—O2—C9—O1 | 142.3 (4) | K2$^{i}$—F5—B2—K2$^{vi}$ | −163.8 (2) |
| K2$^{i}$—O2—C9—N1 | −38.6 (6) | K2$^{i}$—F5—B2—K2$^{v}$ | −94.39 (17) |
| C10—O1—C9—O2 | 10.7 (3) | K2$^{vi}$—F5—B2—K2$^{v}$ | 69.38 (8) |
| C10—O1—C9—N1 | −168.51 (15) | K2$^{v}$—F4—B2—F6 | 21.72 (18) |
| C1—N1—C9—O2 | −8.6 (3) | K1$^{i}$—F4—B2—F6 | −124.53 (12) |
| C4—N1—C9—O2 | −174.2 (2) | K2$^{v}$—F4—B2—F5 | −91.24 (15) |
| C1—N1—C9—O1 | 170.70 (17) | K1$^{i}$—F4—B2—F5 | 122.51 (12) |
| C4—N1—C9—O1 | 5.1 (3) | K2$^{v}$—F4—B2—C15 | 145.88 (12) |
| C9—O1—C10—C11 | −62.8 (2) | K1$^{i}$—F4—B2—C15 | −0.4 (2) |
| C9—O1—C10—C12 | 60.7 (2) | K2$^{v}$—F4—B2—K2$^{vi}$ | −33.50 (17) |
| C9—O1—C10—C13 | 177.7 (2) | K1$^{i}$—F4—B2—K2$^{vi}$ | −179.74 (6) |
| C22—N2—C14—C15 | 168.35 (16) | K1$^{i}$—F4—B2—K2$^{v}$ | −146.25 (17) |
| C17—N2—C14—C15 | −1.4 (2) | C14—C15—B2—F6 | −164.37 (17) |
| C22—N2—C14—K1$^{i}$ | 87.62 (16) | C16—C15—B2—F6 | 17.0 (3) |
| C17—N2—C14—K1$^{i}$ | −82.10 (13) | K1$^{i}$—C15—B2—F6 | 120.06 (14) |
| N2—C14—C15—C16 | 0.2 (2) | C14—C15—B2—F5 | −43.5 (2) |
| K1$^{i}$—C14—C15—C16 | 102.32 (12) | C16—C15—B2—F5 | 137.88 (18) |
| N2—C14—C15—B2 | −178.71 (16) | K1$^{i}$—C15—B2—F5 | −119.03 (14) |
| K1$^{i}$—C14—C15—B2 | −76.62 (16) | C14—C15—B2—F4 | 75.8 (2) |
| N2—C14—C15—K1$^{i}$ | −102.09 (14) | C16—C15—B2—F4 | −102.8 (2) |
| C14—C15—C16—C21 | −179.56 (18) | K1$^{i}$—C15—B2—F4 | 0.26 (15) |
| B2—C15—C16—C21 | −0.7 (3) | C14—C15—B2—K2$^{vi}$ | −104.92 (19) |
| K1$^{i}$—C15—C16—C21 | −94.39 (19) | C16—C15—B2—K2$^{vi}$ | 76.4 (2) |
| C14—C15—C16—C17 | 1.01 (19) | K1$^{i}$—C15—B2—K2$^{vi}$ | 179.51 (13) |
| B2—C15—C16—C17 | 179.87 (17) | C14—C15—B2—K2$^{v}$ | 112.3 (2) |
| K1$^{i}$—C15—C16—C17 | 86.18 (13) | C16—C15—B2—K2$^{v}$ | −66.3 (3) |
| C21—C16—C17—C18 | −4.0 (3) | K1$^{i}$—C15—B2—K2$^{v}$ | 36.76 (17) |
| C15—C16—C17—C18 | 175.51 (16) | | |

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*−1, *y*, *z*; (iii) *x*−2, *y*, *z*; (iv) *x*−3/2, −*y*+1/2, −*z*; (v) *x*+2, *y*, *z*; (vi) *x*+3/2, −*y*+1/2, −*z*; (vii) *x*−1/2, −*y*+1/2, −*z*; (viii) *x*+1/2, −*y*+1/2, −*z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H51···F3$^{ix}$ | 0.81 (1) | 2.14 (1) | 2.903 (2) | 156 (3) |
| O5—H51···F1$^{ix}$ | 0.81 (1) | 2.62 (2) | 3.188 (2) | 128 (2) |
| O5—H52···O5$^{x}$ | 0.81 (1) | 2.41 (1) | 3.1968 (16) | 164 (3) |

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