Crystal structures of 2,3,7,8,12,13,17,18-octabromo-5,10,15,20-tetrakis(pentafluorophenyl)porphyrin as the chloroform monosolvate and tetrahydrofuran monosolvate

Christopher J. Kingsbury, Keith J. Flanagan, Marc Kielmann, Brendan Twamley and Mathias O. Senge

Acta Cryst. (2020). E76, 214–220
Crystal structures of 2,3,7,8,12,13,17,18-octa-bromo-5,10,15,20-tetrakis(pentafluorophenyl)porphyrin as the chloroform monosolvate and tetrahydrofuran monosolvate

Christopher J. Kingsbury, Keith J. Flanagan, Marc Kielmann, Brendan Twamley and Mathias O. Senge*

School of Chemistry, Trinity Biomedical Science Institute, 152–160 Pearse Street, Trinity College Dublin, The University of Dublin, Dublin 2, Ireland. *Correspondence e-mail: sengem@tcd.ie

The crystal structures of the title compounds, two solvates (CHCl₃ and THF) of a symmetric and highly substituted porphyrin, C₄₄H₂Br₈F₂₀N₄ or OBrTPFPP, are described. These structures each feature a non-planar porphyrin ring, exhibiting a similar conformation of the strained ring independent of solvent identity. These distorted porphyrins are able to form hydrogen bonds and van der Waals halogen interactions with enclathrated solvent; supramolecular interactions of proximal macrocycles are additionally affected by solvent choice. The crystal studied for compound 1\(\cdot\)CHCl₃ was refined as an inversion twin. One pentafluorophenyl group was modelled as disordered over two sites [occupancy ratio = 0.462 (7):0.538 (7)]. The chloroform solvate was also modelled as disordered over two orientations [occupancy ratio = 0.882 (7):0.118 (7)].

1. Chemical context

Highly substituted porphyrins are a subclass of porphyrin compounds where the meso and \(\beta\) positions are substituted with non-H atoms. When large substituents are introduced to the periphery of the porphyrin ring, this tends to overcrowd the macrocycle and induce conformational distortion, increasing with the steric demand (Senge & Kalisch, 1997; Medforth et al., 1992). Among the most studied substitution patterns are those with variously functionalized aryl rings at the 5,10,15,20-positions and with halogen, alkane and aryl substituents at the 2,3,7,8,12,13,17,18-positions (Senge, 2000, 2006).

There are numerous approaches used to introduce conformational distortion to porphyrins, including coordination of specific metal centers, incorporation of a strapping motif, or decorating the ring with sterically demanding substituents (Schindler et al., 2018; Senge, 2006). Recent publications show uses for distorted porphyrins as free-base catalysts and sensors, and these compounds demonstrate unique and tuneable porphyrin inner core interactions (Aoki et al., 2019; Norvaisa et al., 2019; Kielmann et al., 2019; Kielmann & Senge, 2019). For example, non-planar metal-free porphyrins show promise as organocatalysts, acting as hydrogen-bond donors (Kielmann et al., 2019). Moreover, the porphyrin scaffold is customizable, and the potential for tuneable basicity and catalytic activity based on variable substitution patterns has been explored (Roucan et al., 2017). The distortion of the porphyrin ring, when compared to the planar parent...
compound, affects the photophysical and electronic properties of both free-base macrocycles and of derived metal complexes (Parusel et al., 2000; Gentemann et al., 1994; Röder et al., 2010). With this in mind, halogenated porphyrins specifically are of interest as ligands in catalytic metal complexes, owing to non-planar conformation, as well as the electron-deficient character of the porphyrin ring (Dolphin et al., 1997; Henling et al., 1993).

The title compound has been previously characterized as a dichlorobenzene solvate (Takeuchi et al., 1994). Structural differences between this literature compound and the structures presented herein arise from intermolecular interactions with chloroform and THF. Additionally, in the published structure, the solvent could not be adequately modelled. These three structures are compared below.

2. Structural commentary

The crystal structure of the title compound (2,3,7,8,12,13,17,18-octabromo-5,10,15,20-tetrakis(pentafluorophenyl)porphyrin monochloroform solvate, (1-CHCl₃) shows a single H₂OBrTPFPP molecule and one chloroform solvate in the asymmetric unit. This highly substituted porphyrin ring exhibits peri interactions from the appended bromine atoms crowding with pentafluorophenyl rings, forcing the bromine atoms substantially out of the mean plane of the porphyrin ring at a mean deviation of 2.14 (14) Å. One of these pentafluorophenyl rings is disordered over two positions, related by a co-planar shift; the co-crystallized CHCl₃ is also disordered over two orientations. A view of the molecular structure of H₂OBrTPFPP is shown in Fig. 1.

A second crystal structure of (2,3,7,8,12,13,17,18-octabromo-5,10,15,20-tetrakis(pentafluorophenyl)porphyrin monotetrahydrofuran solvate (1-THF) displays essentially the same conformation of the macrocycle; differences in the packing of these compounds are discussed below. The D24 values, a summation of atomic deviations from mean plane of the macrocycle are similar in 1-CHCl₃ and 1-THF; a view of the skeletal deviations from the mean-plane in the crystal structure of these two compounds is shown in Fig. 4b.

Normal Structure Decomposition (NSD, Jentzen et al., 1995; Schindler et al., 2018) analysis is the standard method of comparing the mode and extent of distortion between porphyrin structures. NSD is the decomposition of the atomic coordinates of a porphyrin into defined in-plane and out-of-plane distortion modes, based on a least-squares fit of the atomic coordinates to the calculated lowest frequency vibrational modes. The porphyrin rings of the title compounds are shown to exhibit significant out-of-plane saddle-type [B2u (min)] distortion in both crystal structures reported here. This saddling distortion is a direct result of large substituents appended to the porphyrin ring – the saddle distortion alleviates steric demand by removing the restraint of co-planarity from the Br and aryl groups. Slight isotropic contraction, or bre mode distortion, of this porphyrin ring when projected into the xy-plane [A1g (min)] is an effect of the large skewing, or pyrrole tilt – the reported Cl₄TPFPP and F₈TPFPP porphyrins do not show this A₁g contraction with similar bond distances reported, as shown in Table 3 and Fig. 5.

3. Supramolecular features

In 1-CHCl₃, solvent chloroform molecules are nestled between two adjacent porphyrin rings and disordered over two similar
orientations. In the dominant orientation, CHCl₃ molecules show weak C—H $\cdots$ Cl/C/N interactions ($\sim$2.7 Å H $\cdots$ N) to imine pyrrole rings of one porphyrin, and Cl $\cdots$ F contact ($\sim$3.0 Å) to an adjacent porphyrin (Table 1). These interactions are shown in Fig. 2.

This solvent-mediated supramolecular motif serves to arrange the porphyrin rings directly above and below one another, in an approximately face-to-face arrangement. As a result of this arrangement, the porphyrin molecules form stacks which extend along the b-axis direction. The adjacent stacks of porphyrin units in the ac direction interdigitate with one another, as shown in Fig. 3.

In 1-THF, the central core of the porphyrin displays traditional hydrogen bonding (Table 2) from one pyrrole group to the THF oxygen atom [N $\cdots$ O 2.849 (6) Å], with a longer distance to the other available pyrrole N—H group (N $\cdots$ O = 3.8 Å). The THF solvate is not observed to form similar bimodal intramolecular interactions as the chloroform solvate, and porphyrin molecules do not form the infinite stacking arrangements seen in 1-CHCl₃. The porphyrin molecules display multiple halogen–halogen interactions from the bromine and fluorine atoms in both structures.

### 4. Database survey

Previous structures of H₂X₈F₂₀TPP-solvant have been described for X = H (Birnbaum et al., 1995; Dogutan et al., 2010), F (Leroy et al., 1999), Cl (Birnbaum et al., 1995), and Br (Birnbaum et al., 1995) (Fig. 4a). The increasing distortion of macrocycles with increasingly larger halogens can be observed in the plot of skeletal deviations shown in Fig. 4c.
The macrocycle structures of 1-CHCl$_3$ and 1-THF can be directly compared to the previous structure 1-C$_6$H$_4$Cl$_2$; these three structures all exhibit approximately the same macrocycle bond distances and angles, shown in Table 3. The supramolecular interactions of 1-C$_6$H$_4$Cl$_2$ could not be reliably determined given that the solvent was only partially modelled in the reported structure. The face-to-face stacking centroid-to-centroid distance of porphyrin macrocycles in 1-C$_6$H$_4$Cl$_2$ was 6.93 Å, whereas for 1-CHCl$_3$, the separation was 6.83 Å. It is additionally possible that the solvent in the former case was in fact dichloromethane, which was present in the crystalization solution and displays a similar Cl/Cl/Cl/Cl separation.

The NSD analysis parameters of similar literature structures are summarized in Fig. 5, as a comparator to the structures in this work. The NSD parameter, in Å, is equal to one quarter of the sum of the displacements of all 24 atoms of the simplified distortion model, which can be attributed to this distortion mode; the error value shown is the sum error ($\sigma_{oop}$) of the least-squares fit of all six lowest frequency modes. As expected, an increasing saddle-type distortion is found for increasing size of the halogen atom, with little deviation from planarity apparent where \(X=\text{H or F}\) \(sad=0.001 (9) \text{ Å (H)}\) and 0.000 (0) Å \(\text{ (F)}\). Significant saddling distortion was apparent for \(X=\text{Cl}\) \(sad=1.91 (2) \text{ Å}\), and greater for \(X=\text{Br}\) \(1\text{-C}_6\text{H}_4\text{Cl}_2\) \(sad=2.72 (5) \text{ Å}\), \(1\text{-CHCl}_3\) \(sad=3.45 (7) \text{ Å}\) and \(1\text{-THF}\) \(sad=3.16 (7) \text{ Å}\), showing the dependence of distortion on steric bulk, which outweighs solvent contributions.

Analogous studies of porphyrins with increasing steric demand have observed increasing distortion for the porphyrin structures (ethyl)$_x$5,10,15,20-tetraphenylporphyrin \(X=2,4,6,8\) \(x=2\text{ (TATPOT01)}, 4\text{ (TATPUZ01)}, 6\text{ (TATQEK01)}, 8\text{ (SATQOU)}\).
Table 3
Calculated mean distances, angles and structural parameters (Å, °) for compounds 1–6.

| Compound | 1-CHCl3 | 1-THF | 2 (HALZUP; Dogutan et al., 2010) | 3 (GODYON; Leroy et al., 1999) | 4 (ZALHUP; Birnbaum et al., 1995) | 1-C6H4Cl2 (ZALJEB; Birnbaum et al., 1995) | 6 (RONROB; Spyroulias et al., 1997) |
|----------|---------|-------|----------------------------------|---------------------------------|---------------------------------|--------------------------------------|---------------------------------|
| Bond lengths | | | | | | | |
| N–C1 | 1.368 (7) | 1.366 (6) | 1.358 | 1.370 | 1.372 | 1.367 | 1.362 |
| C1–Cm | 1.440 (20) | 1.450 (2) | 1.450 | 1.438 | 1.448 | 1.457 | 1.435 |
| C1–C1 | 1.406 (10) | 1.408 (4) | 1.395 | 1.399 | 1.402 | 1.406 | 1.415 |
| C1–C6 | 1.361 (10) | 1.357 (12) | 1.345 | 1.332 | 1.347 | 1.349 | 1.348 |
| Bond angles | | | | | | | |
| N–C1–Cm | 129.0 (2) | 128.3 (14) | 125.0 | 125.9 | 127.2 | 127.1 | 129.3 |
| C1–Cm–Ca | 107.4 (12) | 107.5 (9) | 107.1 | 108.1 | 108.0 | 107.4 | 107.7 |
| Ca–Cb–Cb | 123.0 (9) | 123.8 (7) | 125.8 | 125.4 | 125.7 | 123.4 | 120.9 |
| Ca–N–Ca | 109.0 (4) | 109.0 (3) | 108.1 | 108.7 | 109.5 | 108.7 | 109.4 |
| N–Ca–Cb | 110.0 (6) | 110.0 (6) | 108.8 | 107.5 | 107.2 | 108.1 | 107.5 |
| N–Ca–Cm | 123.1 (7) | 123.7 (12) | 125.0 | 125.7 | 127.2 | 127.1 | 129.3 |
| Pyrrole mean-plane inclination angles | | | | | | | |
| <pyrN221 | 27.8 | 25.6 | 0.7 | 3.1 | 14.6 | 25.6 | 39.1 |
| <pyrN222 | 26.9 | 29.5 | 0.4 | 3.4 | 15.7 | 20.4 | 39.1 |
| <pyrN223 | 36.9 | 36.5 | 0.7 | 3.1 | 14.9 | 25.6 | 39.1 |
| Mean(<pyr) | 32.1 | 22.5 | 0.4 | 3.4 | 14.0 | 20.4 | 39.1 |
| Structural parameters | | | | | | | |
| Δlip | 0.59 (11) | 0.42 (10) | 0.182 (12) | 0.23 (2) | 0.07 (4) | 0.31 (7) | 0.8 (2) |
| Δoop | 3.46 (7) | 3.17 (7) | 0.01 (9) | 0.246 (8) | 1.91 (2) | 2.73 (5) | 3.790 (120) |
| ΔN22–N21 | 2.931 (3) | 2.32 (5) | 2.895 | 2.942 | 2.939 | 2.86 | 2.958 |
| ΔN22–N23 | 2.895 (3) | 2.61 (6) | 2.927 | 2.942 | 2.942 | 2.923 | 2.958 |
| ΔN24–N21 | 2.928 (3) | 2.60 (6) | 2.895 | 2.942 | 2.925 | 2.86 | 2.958 |
| ΔCm | 0.543 | 0.497 | 0.008 | 0.046 | 0.308 | 0.442 | 0.616 |
| ΔCm | 0.075 | 0.090 | 0.018 | 0.046 | 0.088 | 0.077 | 0.034 |
| ΔCm | 0.036 | 0.017 | 0.009 | 0.061 | 0.009 | 0.091 | 0.317 |
| ΔCm | 0.418 | 0.379 | 0.007 | 0.029 | 0.250 | 0.339 | 0.409 |
| ΔCm | 1.156 | 1.060 | 0.005 | 0.055 | 0.626 | 0.903 | 1.264 |

(a) Simulated total in-plane distortion; (b) simulated total out-of-plane distortion; (c) average deviation from the least-squares plane of the 24-macrocycle atoms; (d) simulated displacement of the four internal nitrogen atoms from the 24-atom mean plane; (e) average deviation of the meso-carbon atoms from the 24-atom mean plane; (f) Average deviation of the o-carbon atoms from the 24-atom mean plane; (g) Average deviation of the p-carbon atoms from the 24-atom mean plane.

imply that the inductive or steric contribution of the fluoro substituents is negligible in causing increased distortion of the macrocycle.

5. Synthesis and crystallization

This compound was synthesized by a previously reported procedure (Mandon et al., 1992). Crystallization was performed by slow evaporation of a partially covered homogeneous solution at room temperature; of chloroform for 1-CHCl3 and THF for 1-THF.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4.

Compound 1-CHCl3 was refined as an inversion twin, with a Flack parameter of 0.010 (4), indicating a small inversion impurity in the single crystal. The pentafluorophenyl group bound to C5 was modelled as disordered over two equivalent [0.462 (7):0.538 (7)] coplanar positions, displaced by ≥0.3 Å at the centroid, which were constrained to have equal Uij parameters for atoms sharing sites, similar Uij parameters for bonded atoms, and idealized ring geometry. The most distant fluorine atoms had to be held to additional isotropic Uij restraints. The chloroform solvate was also modelled as disordered over two orientations, sharing approximate carbon and hydrogen positions. This second orientation was related by a partial rotation around the threefold axis and modelled such that these two orientations had a sum occupancy of one molecule. The dominant orientation was refined to 0.882 (7) occupancy, and C–Cl distances in the minor component had to be restrained to idealized bond distances. C atoms were held to equal Uij restraints and Cl atoms were restrained to similar Uij parameters.

In compound 1-THF pentafluorophenyl rings were modelled as disordered over two orientations with dominant orientations of 0.748 (18) and 0.694 (17) occupancy. Porphyrin-to-phenyl distances and carbon atom displacement parameters (Uij) were restrained. Idealized geometric constraints were imposed on the least occupied phenyl ring C10A–C10F. The ipso phenyl carbon atoms were constrained...
Table 4
Experimental details.

| 1-CHCl₃ | 1-THF |
|---------|-------|
| Crystal data | Crystal data |
| Chemical formula | C₄₄H₂Br₈F₂₀N₄CHCl₃ | C₄₄H₂Br₈F₂₀N₄C₆H₄O |
| Mᵣ | 1725.14 | 1677.88 |
| Crystal system, space group | Monoclinic, P2₁ | Monoclinic, P2₁/n |
| Temperature (K) | 100 | 100 |
| Temperature (°C) | 77 | 21.524 (9), 9.545 (4), 26.020 (11) |
| β (°) | 104.683 (1) | 107.426 (7) |
| V (Å³) | 2517.6 (2) | 5101 (4) |
| Z | 2 | 4 |
| Radiation type | Mo Kα | Mo Kα |
| μ (mm⁻¹) | 6.65 | 6.41 |
| Crystal size (mm) | 0.31 x 0.30 x 0.11 | 0.42 x 0.11 x 0.09 |

Data collection

Diffractometer | Bruker APEXII CCD | Bruker APEXII CCD |
|----------------|----------------|----------------|
| Absorption correction | Multi-scan (SADABS; Bruker, 2016) | Multi-scan (SADABS; Bruker, 2016) |
| No. of measured, independent and observed [I > 2σ(I)] reflections | ∞ | 18291, 12689, 10079 |
| Rint | 0.028 | 0.082 |
| (sinθ/λ)max (Å⁻¹) | 0.758 | 0.668 |

Refinement

R[F² > 2σ(F²)], wR(F²), S | 0.020, 0.043, 1.03 | 0.054, 0.095, 1.21 |
|----------------|----------------|----------------|
| No. of reflections | 18291 | 12689 |
| No. of parameters | 763 | 948 |
| No. of restraints | 244 | 718 |
| H-atom treatment | H-atoms parameters constrained | H-atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.52, −0.46 | 1.63, −0.98 |
| Absolute structure | Refined as an inversion twin. | – |
| Absolute structure parameter | 0.010 (4) | – |

Computer programs: APEX3 (Bruker, 2017), SAINT (Bruker, 2015), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), SHELXLE (Hubbschle et al., 2011) and OLEX2 (Dolomanov et al., 2009).

### References

Aoki, E., Suzuki, W., Kotani, H., Ishizuka, T., Sakai, H., Hasobe, T. & Kojima, T. (2019). Chem. Commun. 55, 4925–4928.

Birbaum, E. R., Hodge, J. A., Grinstaff, M. W., Schaefer, W. P., Henling, L., Labinger, J. A., Bercaw, J. E. & Gray, H. B. (1995). Inorg. Chem. 34, 3625–3632.

Bruker (2015). SAINT. Bruker AXS Inc., Madison, WI, USA.

Bruker (2016). SAINT/SAVES Bruker AXS Inc., Madison, WI, USA.

Bruker (2017). APEX3. Bruker AXS Inc., Madison, WI, USA.

Dugtan, D. K., Bediako, D. K., Teets, T. S., Schwalbe, M. & Nocera, D. G. (2010). Org. Lett. 12, 1036–1039.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). J. Appl. Cryst. 42, 339–341.

Dolphin, D., Traylor, T. G. & Xie, L. Y. (1997). Acc. Chem. Res. 30, 251–259.

Gentemann, S., Medforth, C. J., Forsyth, T. P., Nurco, D. J., Smith, K. M., Fajer, J. & Holten, D. (1994). J. Am. Chem. Soc. 116, 7363–7368.

Henling, L. M., Schaefer, W. P., Hodge, J. A., Hughes, M. E., Gray, H. B., Lyons, J. E. & Ellis, P. E. (1993). Acta Cryst. C49, 1743–1747.

Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). J. Appl. Crystallogr. 44, 1281–1284.

Jentzen, W., Simpson, M. C., Hobbs, J. D., Song, X., Ema, T., Nelson, N. Y., Medforth, C. J., Smith, K. M., Veyrat, M., Mazzanti, M., Ramasseul, R., Marchon, T., Takeuchi, T., Goddard, W. A. III & Shelnutt, J. A. (1995). J. Am. Chem. Soc. 117, 11085–11097.

Kielmann, M., Grover, N., Kalisch, W. W. & Senge, M. O. (2019). Eur. J. Org. Chem. pp. 2448–2452. Kielmann, M. & Senge, M. O. (2019). Angew. Chem. Int. Ed. 58, 418–441.

Kojima, T. (2019). Acc. Chem. Res. 52, 1161–1165.

König, D., Borchard, T. & Fajer, J. (2015). Angew. Chem. Int. Ed. 54, 9859–9866.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.

König, D., Borchard, T., Fajer, J. & Holten, D. (2014). J. Am. Chem. Soc. 136, 11085–11097.
Schindler, J., Kupfer, S., Ryan, A. A., Flanagan, K. J., Senge, M. O. & Dietzek, B. (2018). Coord. Chem. Rev. 360, 1–16.

Senge, M. O. (2000). The Porphyrin Handbook, edited by K. M. Kadish, K. M. Smith and S. R. Guillard; Vol. 1, pp. 239–347; Academic Press, San Diego.

Senge, M. O. (2006). Chem. Commun. pp. 243–256.

Senge, M. O. & Kalisch, W. W. (1997). Inorg. Chem. 36, 6103–6116.

Sheldrick, G. M. (2015a). Acta Cryst. A71, 3–8.

Sheldrick, G. M. (2015b). Acta Cryst. C71, 3–8.

Spyroulias, G. A., Despotopoulos, A., Raptopoulou, C. P., Terzis, A. & Coutsolelos, A. G. (1997). Chem. Commun. pp. 783–784.

Takeuchi, T., Gray, H. B. & Goddard, W. A. (1994). J. Am. Chem. Soc. 116, 9730–9732.
Crystal structures of 2,3,7,8,12,13,17,18-octabromo-5,10,15,20-tetrakis(penta-fluorophenyl)porphyrin as the chloroform monosolvate and tetrahydrofuran monosolvate

Christopher J. Kingsbury, Keith J. Flanagan, Marc Kielmann, Brendan Twamley and Mathias O. Senge

Computing details

For both structures, data collection: APEX3 (Bruker, 2017); cell refinement: SAINT (Bruker, 2015); data reduction: SAINT (Bruker, 2015); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: Shelxle (Hübschle et al., 2011); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

2,3,7,8,12,13,17,18-Octabromo-5,10,15,20-tetrakis(pentafluorophenyl)porphyrin chloroform monosolvate (1.CHCl3)

Crystal data

C44H2Br8F20N4·CHCl3

F(000) = 1624

Mr = 1725.14

Monoclinic, P21

a = 15.5162 (7) Å

b = 6.8288 (3) Å

c = 24.5631 (12) Å

β = 104.683 (1)°

V = 2517.6 (2) Å³

Z = 2

Dx = 2.276 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9974 reflections

θ = 2.6–32.5°

µ = 6.65 mm⁻¹

T = 100 K

Tmin = 0.458, Tmax = 0.746

107677 measured reflections

18291 independent reflections

17164 reflections with I > 2σ(I)

Rint = 0.028

θmax = 32.6°, θmin = 1.4°

h = −23→23

k = −10→10

l = −37→36

Refinement

Refinement on F²

Least-squares matrix: full

R[F²] = 0.020

wR(F²) = 0.043

S = 1.03

18291 reflections

763 parameters

244 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/\left[\sigma^2(F_o^2) + (0.0025P)^2 + 1.005P\right]$ where $P = (F_o^2 + 2F_c^2)/3$

$\Delta/\sigma_{\text{max}} = 0.002$
$\Delta \rho_{\text{max}} = 0.52 \text{ e Å}^{-3}$
$\Delta \rho_{\text{min}} = -0.46 \text{ e Å}^{-3}$

Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.010 (4)

**Special details**

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

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

|    | x    | y    | z    | $U_{eq}$ | Occ. (<1) |
|----|------|------|------|----------|-----------|
| C1 | 0.79032 (16) | 0.5406 (4) | 0.87273 (10) | 0.0134 (4) |
| Br1 | 0.87104 (2) | 0.43518 (5) | 0.99180 (2) | 0.02349 (6) |
| C2 | 0.86821 (16) | 0.5046 (4) | 0.91809 (10) | 0.0152 (4) |
| Br2 | 1.05536 (2) | 0.43897 (5) | 0.93835 (2) | 0.02288 (6) |
| C3 | 0.94138 (16) | 0.5053 (4) | 0.89661 (10) | 0.0153 (4) |
| Br3 | 1.06413 (2) | 0.81653 (4) | 0.70329 (2) | 0.02160 (6) |
| C4 | 0.91257 (15) | 0.5427 (4) | 0.83707 (10) | 0.0132 (4) |
| Br4 | 0.92250 (2) | 0.68017 (4) | 0.57534 (2) | 0.01783 (5) |
| C5 | 0.96138 (16) | 0.5753 (4) | 0.79756 (10) | 0.0145 (4) |
| Br5 | 0.67971 (2) | -0.08433 (4) | 0.56768 (2) | 0.02307 (5) |
| C6 | 0.92364 (15) | 0.5776 (4) | 0.73877 (10) | 0.0134 (4) |
| F6 | 0.94084 (10) | 0.1746 (3) | 0.61796 (6) | 0.0201 (3) |
| Br6 | 0.49352 (2) | -0.08753 (4) | 0.62177 (2) | 0.02119 (5) |
| C7 | 0.96548 (16) | 0.6523 (4) | 0.69563 (10) | 0.0154 (4) |
| F7 | 0.98329 (10) | 0.0661 (3) | 0.52270 (7) | 0.0227 (3) |
| Br7 | 0.37497 (2) | 0.69017 (4) | 0.73639 (2) | 0.01604 (5) |
| C8 | 0.91110 (16) | 0.5994 (4) | 0.64558 (10) | 0.0139 (4) |
| F8 | 0.86376 (11) | 0.1210 (3) | 0.42128 (7) | 0.0246 (4) |
| Br8 | 0.51364 (2) | 0.86107 (4) | 0.86350 (2) | 0.01969 (5) |
| C9 | 0.83861 (15) | 0.4866 (3) | 0.65798 (10) | 0.0133 (4) |
| F9 | 0.70704 (11) | 0.3005 (3) | 0.41561 (6) | 0.0247 (3) |
| C10 | 0.77912 (15) | 0.3636 (4) | 0.62036 (10) | 0.0139 (4) |
| F10 | 0.66599 (10) | 0.4240 (3) | 0.51083 (6) | 0.0229 (3) |
| C11 | 0.70159 (15) | 0.2861 (4) | 0.63184 (10) | 0.0137 (4) |
| F11 | 0.41703 (10) | 0.4175 (3) | 0.58292 (6) | 0.0224 (3) |
| C12 | 0.64841 (16) | 0.1194 (4) | 0.60940 (10) | 0.0150 (4) |
| F12 | 0.24742 (11) | 0.2960 (3) | 0.54385 (7) | 0.0268 (4) |
| C13 | 0.57470 (16) | 0.1181 (4) | 0.63059 (10) | 0.0149 (4) |
| F13 | 0.16081 (11) | 0.1268 (3) | 0.61447 (9) | 0.0321 (4) |
| C14 | 0.57932 (15) | 0.2834 (4) | 0.66693 (10) | 0.0130 (4) |
| F14 | 0.24506 (11) | 0.0721 (3) | 0.72457 (8) | 0.0270 (4) |
| C15 | 0.51835 (15) | 0.3577 (3) | 0.69576 (10) | 0.0127 (4) |
| F15 | 0.41634 (10) | 0.1838 (2) | 0.76383 (6) | 0.0196 (3) |
C16  0.54601 (15)  0.4764 (3)  0.74374 (10)  0.0126 (4)  
F16  0.59460 (13)  0.3120 (3)  0.91627 (7)  0.0328 (4)  
C17  0.49068 (15)  0.6028 (3)  0.76885 (10)  0.0138 (4)  
F17  0.54619 (14)  0.3700 (4)  1.01268 (8)  0.0471 (6)  
C18  0.54346 (15)  0.6649 (4)  0.81857 (10)  0.0141 (4)  
F18  0.60364 (13)  0.6916 (4)  1.07618 (7)  0.0433 (5)  
C19  0.63021 (15)  0.5740 (4)  0.82428 (10)  0.0133 (4)  
F19  0.71156 (14)  0.3700 (4)  1.04248 (8)  0.0435 (5)  
C20  0.70187 (16)  0.6028 (3)  0.87352 (10)  0.0140 (4)  
F20  0.76215 (12)  0.8962 (4)  0.94747 (8)  0.0315 (4)  
N21  0.88463 (13)  0.4908 (3)  0.71443 (8)  0.0129 (4)  
H21  0.82117 (13)  0.5520 (3)  0.82522 (8)  0.0136 (4)  
N22  0.8289 (3)    1.0578 (6)  0.84825 (15)  0.0405 (6)  
F5    1.0834 (12)  0.304 (4)    0.7761 (9)    0.0289 (15)  
F4    1.2643 (6)    0.3383 (11)  0.8118 (4)    0.0363 (11)  
F3    1.3335 (5)    0.6633 (11)  0.8705 (8)    0.0379 (10)  
F2    1.2238 (5)    0.9445 (11)  0.8947 (3)    0.0382 (11)  
F1    1.0460 (9)    0.603 (3)    0.8183 (10)   0.0210 (7)    
C1S   0.7222 (3)    1.0817 (7)  0.7756 (2)    0.0287 (9)    
H     0.762829      1.220837  0.76873      0.034*       
Cl2S  0.68818 (11)  0.9313 (2)  0.75235 (8)  0.0489 (5)    0.882 (7)    
Cl3S  0.86152 (16)  1.0294 (2)  0.73764 (8)  0.0461 (4)    0.882 (7)    
N24   0.63172 (13)  0.4721 (3)  0.77706 (8)  0.0132 (4)    
C51   1.0608 (6)    0.603 (3)   0.8183 (10)  0.0210 (7)    0.462 (7)    
C52   1.0984 (7)    0.767 (2)   0.8488 (8)   0.0255 (12)   0.462 (7)    
C53   1.1905 (7)    0.7875 (14)  0.8658 (6)   0.0287 (14)   0.462 (7)    
C54   1.2450 (6)    0.6439 (12)  0.8522 (5)   0.0292 (14)   0.462 (7)    
C55   1.2074 (9)    0.4800 (15)  0.8217 (6)   0.0280 (13)   0.462 (7)    
C56   1.1154 (9)    0.460 (2)   0.8047 (8)   0.0241 (9)    0.462 (7)    
C2S   0.770 (2)     1.107 (6)   0.7747 (14)  0.0287 (9)    0.118 (7)    
HA    0.763429      1.250725  0.766630     0.034*       0.118 (7)    
C51B  1.0594 (5)    0.616 (2)   0.8184 (8)   0.0210 (7)    0.538 (7)    
C52B  1.0853 (6)    0.793 (2)   0.8456 (7)   0.0255 (12)   0.538 (7)    
C53B  1.1752 (6)    0.8393 (12)  0.8646 (5)   0.0287 (14)   0.538 (7)    
C54B  1.2393 (5)    0.7081 (11)  0.8564 (4)   0.0292 (14)   0.538 (7)    
C55B  1.2134 (7)    0.5311 (12)  0.8292 (5)   0.0280 (13)   0.538 (7)    
C56B  1.1235 (8)    0.4852 (18)  0.8102 (7)   0.0241 (9)    0.538 (7)    
F1B   1.0276 (8)    0.927 (2)   0.8547 (6)   0.0303 (14)   0.538 (7)    
F2B   1.2028 (4)    1.0127 (9)  0.8910 (3)   0.0382 (11)   0.538 (7)    
F3B   1.3255 (4)    0.7480 (10)  0.8757 (3)   0.0379 (10)   0.538 (7)    
F4B   1.2721 (5)    0.4032 (10)  0.8213 (3)   0.0363 (11)   0.538 (7)    
F5B   1.0987 (10)   0.315 (4)   0.7843 (8)   0.0289 (15)   0.538 (7)    
C101  0.80163 (15)  0.3017 (4)  0.56770 (10) 0.0137 (4)    
C102  0.88211 (16)  0.2099 (4)  0.56870 (10) 0.0148 (4)    
C103  0.90484 (16)  0.1527 (4)  0.52009 (11) 0.0158 (4)    

Acta Cryst. (2020). E76, 214-220
### Atomic displacement parameters (Å²)

|     | U₁₁    | U₂₂    | U₃₃    | U₁₂    | U₁₃    | U₂₃    |
|-----|--------|--------|--------|--------|--------|--------|
| C1  | 0.0128 (10) | 0.0167 (10) | 0.0107 (10) | −0.0011 (8) | 0.0027 (8) | 0.0002 (8) |
| Br1 | 0.02061 (12) | 0.03766 (15) | 0.01195 (10) | 0.00155 (11) | 0.00370 (9) | 0.00789 (11) |
| C2  | 0.0145 (11) | 0.0201 (11) | 0.0109 (10) | −0.0009 (8) | 0.0028 (8) | 0.0020 (8) |
| Br2 | 0.01293 (10) | 0.03700 (15) | 0.01655 (11) | 0.00356 (11) | −0.00026 (8) | 0.00561 (11) |
| C3  | 0.0113 (10) | 0.0210 (11) | 0.0123 (10) | −0.0005 (8) | 0.0005 (8) | 0.0023 (8) |
| Br3 | 0.01564 (11) | 0.03080 (14) | 0.01819 (12) | −0.01240 (10) | 0.00399 (9) | 0.00174 (10) |
| C4  | 0.0095 (9) | 0.0170 (10) | 0.0118 (10) | −0.0019 (8) | 0.0001 (8) | 0.0004 (8) |
| Br4 | 0.01723 (11) | 0.02391 (11) | 0.01397 (10) | −0.00445 (10) | 0.00694 (8) | 0.00244 (9) |
| C5  | 0.0118 (10) | 0.0193 (11) | 0.0127 (10) | −0.0041 (8) | 0.0037 (8) | −0.0012 (8) |
| Br5 | 0.02230 (12) | 0.02245 (12) | 0.02858 (13) | −0.00561 (10) | 0.01408 (10) | −0.01180 (10) |
| C6  | 0.0099 (9) | 0.0176 (10) | 0.0126 (10) | −0.0019 (8) | 0.0023 (8) | 0.0002 (8) |
| F6  | 0.0148 (6) | 0.0309 (8) | 0.0135 (7) | 0.0038 (7) | 0.0017 (5) | 0.0026 (6) |
| Br6 | 0.01599 (11) | 0.01999 (12) | 0.02962 (13) | −0.00840 (10) | 0.00952 (9) | −0.00895 (10) |
| C7  | 0.0120 (10) | 0.0205 (12) | 0.0142 (10) | −0.0053 (8) | 0.0041 (8) | 0.0001 (8) |
| F7  | 0.0154 (7) | 0.0339 (9) | 0.0219 (8) | 0.0074 (7) | 0.0103 (6) | 0.0043 (7) |
| Br7 | 0.01161 (10) | 0.01818 (10) | 0.01770 (11) | 0.00211 (9) | 0.00253 (8) | −0.00019 (9) |
| C8  | 0.0120 (10) | 0.0191 (11) | 0.0116 (10) | −0.0025 (8) | 0.0047 (8) | 0.0023 (8) |
| F8  | 0.0231 (8) | 0.0395 (10) | 0.0144 (7) | 0.0028 (7) | 0.0105 (6) | −0.0013 (6) |
| Br8 | 0.01666 (11) | 0.02534 (12) | 0.01741 (11) | 0.00252 (9) | 0.00493 (9) | −0.00738 (9) |
| C9  | 0.0100 (10) | 0.0175 (10) | 0.0128 (10) | −0.0026 (8) | 0.0037 (8) | −0.0001 (8) |
| F9  | 0.0191 (7) | 0.0417 (10) | 0.0109 (7) | 0.0042 (7) | −0.0004 (6) | 0.0005 (7) |
| C10 | 0.0106 (9) | 0.0192 (10) | 0.0121 (10) | −0.0008 (8) | 0.0035 (8) | 0.0003 (8) |
| F10 | 0.0136 (6) | 0.0366 (9) | 0.0176 (7) | 0.0075 (7) | 0.0026 (5) | 0.0001 (7) |
| C11 | 0.0114 (10) | 0.0179 (11) | 0.0119 (10) | −0.0016 (8) | 0.0033 (8) | −0.0017 (8) |
| F11 | 0.0190 (7) | 0.0339 (9) | 0.0147 (7) | −0.0046 (7) | 0.0046 (5) | 0.0036 (7) |
| C12 | 0.0127 (10) | 0.0191 (10) | 0.0141 (10) | −0.0018 (8) | 0.0049 (8) | −0.0044 (8) |
| Atom | U_{11}  | U_{22}  | U_{33}  | U_{12}  | U_{13}  | U_{23}  |
|------|---------|---------|---------|---------|---------|---------|
| F12  | 0.0194  | 0.0364  | 0.0186  | -0.0014 | -0.0059 | 0.0008  |
| C13  | 0.0113  | 0.0174  | 0.0163  | -0.0050 | 0.0037  | -0.0035 |
| F13  | 0.0092  | 0.0391  | 0.0436  | -0.0090 | -0.0150 | 0.0024  |
| C14  | 0.0106  | 0.0178  | 0.0108  | -0.0036 | 0.0029  | -0.0016 |
| F14  | 0.0167  | 0.0310  | 0.0364  | -0.0066 | 0.0128  | 0.0085  |
| C15  | 0.0100  | 0.0160  | 0.0117  | -0.0029 | 0.0024  | 0.0003  |
| F15  | 0.0187  | 0.0241  | 0.0160  | -0.0034 | 0.0044  | 0.0046  |
| C16  | 0.0094  | 0.0155  | 0.0132  | -0.0019 | 0.0035  | 0.0008  |
| F16  | 0.0387  | 0.0377  | 0.0246  | -0.0199 | 0.0130  | -0.0029 |
| C17  | 0.0121  | 0.0155  | 0.0142  | -0.0010 | 0.0043  | 0.0008  |
| F17  | 0.0390  | 0.0824  | 0.0253  | -0.0214 | 0.0179  | 0.0062  |
| C18  | 0.0129  | 0.0165  | 0.0142  | -0.0020 | 0.0061  | -0.0021 |
| F18  | 0.0316  | 0.0870  | 0.0141  | 0.0069  | 0.0110  | -0.0063 |
| C19  | 0.0115  | 0.0166  | 0.0119  | -0.0015 | 0.0031  | -0.0009 |
| F19  | 0.0423  | 0.0587  | 0.0298  | -0.0051 | 0.0096  | -0.0288 |
| C20  | 0.0130  | 0.0177  | 0.0114  | -0.0029 | 0.0030  | -0.0009 |
| F20  | 0.0314  | 0.0332  | 0.0319  | -0.0137 | 0.0119  | -0.0134 |
| N21  | 0.0085  | 0.0167  | 0.0105  | -0.0010 | 0.0008  | 0.0010  |
| N22  | 0.0103  | 0.0171  | 0.0109  | -0.0028 | 0.0022  | -0.0005 |
| N23  | 0.0102  | 0.0179  | 0.0137  | -0.0050 | 0.0048  | -0.0047 |
| Cl1S | 0.0756  | 0.0204  | 0.0025  | 0.0033  | 0.0072  | 0.0020  |
| F5   | 0.011   | 0.043   | 0.030   | 0.008   | 0.001   | 0.002   |
| F4   | 0.0166  | 0.053   | 0.041   | 0.007   | 0.0093  | 0.006   |
| F3   | 0.0136  | 0.066   | 0.0308  | -0.014  | -0.0003 | 0.008   |
| F2   | 0.030   | 0.051   | 0.0291  | -0.0231 | 0.0003  | -0.007  |
| F1   | 0.026   | 0.038   | 0.024   | -0.012  | 0.001   | -0.009  |
| C1S  | 0.044   | 0.0133  | 0.0273  | 0.0029  | 0.0058  | 0.0024  |
| Cl2S | 0.0507  | 0.0301  | 0.0534  | -0.0098 | -0.0100 | 0.0206  |
| Cl3S | 0.0720  | 0.0283  | 0.0474  | 0.0150  | 0.0326  | 0.0138  |
| N24  | 0.0100  | 0.0178  | 0.0117  | -0.0008 | 0.0027  | -0.0017 |
| C51  | 0.0106  | 0.038   | 0.0130  | -0.0062 | 0.0011  | 0.0038  |
| C52  | 0.014   | 0.047   | 0.0141  | -0.012  | 0.002   | 0.001   |
| C53  | 0.019   | 0.051   | 0.0150  | -0.016  | 0.0008  | 0.001   |
| C54  | 0.0128  | 0.055   | 0.0175  | -0.012  | -0.0001 | 0.007   |
| C55  | 0.0096  | 0.055   | 0.019   | -0.004  | 0.0040  | 0.008   |
| C56  | 0.0091  | 0.047   | 0.015   | -0.0042 | -0.0001 | 0.0050  |
| C2S  | 0.044   | 0.0133  | 0.0273  | 0.0029  | 0.0058  | 0.0024  |
| C51B | 0.0106  | 0.038   | 0.0130  | -0.0062 | 0.0011  | 0.0038  |
| C52B | 0.014   | 0.047   | 0.0141  | -0.012  | 0.000   | 0.001   |
| C53B | 0.019   | 0.051   | 0.0150  | -0.016  | 0.0008  | 0.001   |
| C54B | 0.0128  | 0.055   | 0.0175  | -0.012  | -0.0001 | 0.007   |
| C55B | 0.0096  | 0.055   | 0.019   | -0.004  | 0.0040  | 0.008   |
| C56B | 0.0091  | 0.047   | 0.015   | -0.0042 | -0.0001 | 0.0050  |
| F1B  | 0.026   | 0.038   | 0.024   | -0.012  | 0.001   | -0.009  |
| F2B  | 0.030   | 0.051   | 0.0291  | -0.0231 | 0.0003  | -0.007  |
| F3B  | 0.0136  | 0.066   | 0.0308  | -0.014  | -0.0003 | 0.008   |
| F4B  | 0.0166  | 0.053   | 0.041   | 0.007   | 0.0093  | 0.006   |
| F5B  | 0.011   | 0.043   | 0.030   | 0.008   | 0.001   | 0.002   |
C101  0.0109 (9)  0.0190 (10)  0.0118 (10)  −0.0015 (8)  0.0039 (8)  −0.0007 (8)
C102  0.0121 (10)  0.0187 (11)  0.0137 (10)  −0.0012 (8)  0.0036 (8)  0.0011 (8)
C103  0.0127 (10)  0.0203 (12)  0.0165 (11)  0.0011 (8)  0.0076 (8)  0.0024 (8)
C104  0.0187 (11)  0.0232 (11)  0.0139 (10)  −0.0010 (10)  0.0078 (8)  −0.0004 (10)
C105  0.0143 (11)  0.0254 (12)  0.0108 (10)  −0.0016 (9)  0.0017 (8)  0.0001 (9)
C106  0.0113 (10)  0.0204 (11)  0.0166 (11)  −0.0007 (8)  0.0047 (8)  −0.0008 (8)
C151  0.0095 (9)  0.0155 (10)  0.0134 (10)  −0.0018 (8)  0.0034 (8)  −0.0021 (8)
C152  0.0137 (10)  0.0190 (11)  0.0141 (10)  −0.0026 (9)  0.0036 (8)  −0.0008 (8)
C153  0.0146 (11)  0.0212 (12)  0.0178 (12)  0.0003 (9)  −0.0011 (9)  −0.0020 (9)
C154  0.0071 (9)  0.0209 (11)  0.0314 (13)  −0.0036 (9)  0.0003 (9)  −0.0004 (11)
C155  0.0119 (10)  0.0183 (12)  0.0284 (13)  −0.0023 (9)  0.0087 (9)  0.0030 (10)
C156  0.0129 (10)  0.0165 (11)  0.0161 (11)  −0.0003 (8)  0.0053 (8)  0.0005 (8)
C201  0.0119 (10)  0.0268 (12)  0.0109 (10)  −0.0014 (9)  0.0022 (8)  −0.0014 (9)
C202  0.0163 (11)  0.0328 (14)  0.0151 (11)  −0.0054 (10)  0.0040 (9)  0.0002 (10)
C203  0.0191 (13)  0.054 (2)  0.0155 (12)  −0.0046 (12)  0.0070 (10)  0.0053 (12)
C204  0.0180 (12)  0.063 (2)  0.0108 (11)  0.0059 (14)  0.0043 (9)  −0.0027 (13)
C205  0.0209 (13)  0.0445 (17)  0.0154 (12)  0.0019 (12)  0.0010 (10)  −0.0117 (12)
C206  0.0163 (11)  0.0303 (13)  0.0172 (12)  −0.0024 (10)  0.0045 (9)  −0.0051 (10)
Cl1S  0.123 (11)  0.048 (7)  0.037 (5)  0.045 (7)  0.024 (7)  0.015 (5)
Cl1S  0.123 (11)  0.044 (6)  0.097 (10)  −0.017 (5)  −0.003 (6)  0.036 (6)
Cl1S  0.075 (10)  0.025 (5)  0.032 (8)  −0.003 (7)  −0.015 (8)  −0.010 (6)

Geometric parameters (Å, º)

| Bond or Angle | Distance/° |
|---------------|------------|
| C1—C2         | 1.442 (3)  |
| C1—C20        | 1.394 (3)  |
| C1—N21        | 1.371 (3)  |
| Br1—C2        | 1.861 (2)  |
| C2—C3         | 1.368 (3)  |
| Br2—C3        | 1.862 (2)  |
| C3—C4         | 1.439 (3)  |
| Br3—C7        | 1.868 (2)  |
| C4—C5         | 1.392 (3)  |
| C4—N21        | 1.375 (3)  |
| Br4—C8        | 1.862 (2)  |
| C5—C6         | 1.414 (3)  |
| C5—C51        | 1.510 (9)  |
| C5—C51B       | 1.504 (8)  |
| Br5—C12       | 1.864 (2)  |
| C6—C7         | 1.467 (3)  |
| C6—N22        | 1.357 (3)  |
| F6—C102       | 1.339 (3)  |
| Br6—C13       | 1.862 (2)  |
| C7—C8         | 1.352 (3)  |
| F7—C103       | 1.340 (3)  |
| Br7—C17       | 1.868 (2)  |
| C8—C9         | 1.458 (3)  |
| F8—C104       | 1.336 (3)  |
| Bond          | Distance (Å) | Bond          | Distance (Å) | Bond          | Distance (Å) |
|---------------|--------------|---------------|--------------|---------------|--------------|
| Br8—C18       | 1.867 (2)    | C51B—C52B     | 1.3900       |               |              |
| C9—C10        | 1.407 (3)    | C51B—C56B     | 1.3900       |               |              |
| C9—N22        | 1.366 (3)    | C52B—C53B     | 1.3900       |               |              |
| F9—C105       | 1.344 (3)    | C52B—F1B      | 1.335 (16)   |               |              |
| C10—C11       | 1.407 (3)    | C53B—C54B     | 1.3900       |               |              |
| C10—C101      | 1.484 (3)    | C53B—F2B      | 1.366 (10)   |               |              |
| F10—C106      | 1.343 (3)    | C54B—C55B     | 1.3900       |               |              |
| C11—C12       | 1.432 (3)    | C54B—F3B      | 1.330 (9)    |               |              |
| C11—N23       | 1.372 (3)    | C55B—C56B     | 1.3900       |               |              |
| F11—C152      | 1.336 (3)    | C55B—F4B      | 1.312 (11)   |               |              |
| C12—C13       | 1.371 (3)    | C56B—F5B      | 1.34 (2)     |               |              |
| F12—C153      | 1.344 (3)    | C101—C102     | 1.392 (3)    |               |              |
| C13—C14       | 1.430 (3)    | C101—C106     | 1.395 (3)    |               |              |
| F13—C154      | 1.335 (3)    | C102—C103     | 1.384 (3)    |               |              |
| C14—C15       | 1.412 (3)    | C103—C104     | 1.379 (3)    |               |              |
| C14—N23       | 1.372 (3)    | C104—C105     | 1.380 (4)    |               |              |
| F14—C155      | 1.337 (3)    | C105—C106     | 1.378 (3)    |               |              |
| C15—C16       | 1.405 (3)    | C151—C152     | 1.397 (3)    |               |              |
| C15—C151      | 1.484 (3)    | C151—C156     | 1.395 (3)    |               |              |
| F15—C156      | 1.331 (3)    | C152—C153     | 1.383 (3)    |               |              |
| C16—C17       | 1.460 (3)    | C153—C154     | 1.380 (4)    |               |              |
| C16—N24       | 1.374 (3)    | C154—C155     | 1.378 (4)    |               |              |
| F16—C202      | 1.336 (3)    | C155—C156     | 1.389 (3)    |               |              |
| C17—C18       | 1.354 (3)    | C201—C202     | 1.389 (4)    |               |              |
| F17—C203      | 1.338 (4)    | C201—C206     | 1.391 (4)    |               |              |
| C18—C19       | 1.456 (3)    | C202—C203     | 1.385 (4)    |               |              |
| F18—C204      | 1.336 (3)    | C203—C204     | 1.369 (5)    |               |              |
| C19—C20       | 1.420 (3)    | C204—C205     | 1.385 (5)    |               |              |
| C19—N24       | 1.358 (3)    | C205—C206     | 1.378 (4)    |               |              |
| C20—C1—C2     | 130.6 (2)    | F3—C54—C55    | 121.0 (6)    |               |              |
| N21—C1—C2     | 105.2 (2)    | C55—C54—C53   | 120.0        |               |              |
| N21—C1—C20    | 124.1 (2)    | F4—C55—C54    | 117.6 (8)    |               |              |
| C1—C2—Br1     | 127.12 (18)  | F4—C55—C56    | 122.4 (8)    |               |              |
| C3—C2—C1      | 108.4 (2)    | C54—C55—C56   | 120.0        |               |              |
| C3—C2—Br1     | 123.99 (19)  | F5—C56—C51    | 122.3 (14)   |               |              |
| C2—C3—Br2     | 123.52 (19)  | F5—C56—C55    | 117.7 (14)   |               |              |
| C2—C3—C4      | 108.6 (2)    | C55—C56—C51   | 120.0        |               |              |
| C4—C3—Br2     | 127.57 (19)  | C14S—C2S—HA   | 114.7        |               |              |
| C5—C4—C3      | 130.8 (2)    | C14S—C2S—C15S | 109 (2)      |               |              |
| N21—C4—C3     | 105.2 (2)    | C14S—C2S—C16S | 102 (2)      |               |              |
| N21—C4—C5     | 124.0 (2)    | C15S—C2S—HA   | 114.7        |               |              |
| C4—C5—C6      | 123.8 (2)    | C16S—C2S—HA   | 114.7        |               |              |
| C4—C5—C51     | 118.4 (10)   | C16S—C2S—C15S | 100 (2)      |               |              |
| C4—C5—C51B    | 118.3 (8)    | C52B—C51B—C5  | 118.1 (9)    |               |              |
| C6—C5—C51     | 117.8 (10)   | C52B—C51B—C56B| 120.0        |               |              |
| C6—C5—C51B    | 117.9 (8)    | C56B—C51B—C5  | 121.9 (9)    |               |              |
| C5—C6—C7      | 126.6 (2)    | C53B—C52B—C51B| 120.0        |               |              |
N22—C6—C5 122.6 (2) F1B—C52B—C51B 123.4 (10)
N22—C6—C7 110.5 (2) F1B—C52B—C53B 116.6 (10)
C6—C7—Br3 129.58 (18) C52B—C53B—C54B 120.0
C8—C7—Br3 123.96 (18) F2B—C53B—C52B 121.4 (6)
C8—C7—C6 106.0 (2) F2B—C53B—C54B 118.6 (6)
C7—C8—Br4 125.40 (18) C53B—C54B—C55B 120.0
C7—C8—C9 106.7 (2) F3B—C54B—C53B 120.5 (5)
C9—C8—Br4 127.57 (18) F3B—C54B—C55B 119.5 (5)
C10—C9—C8 126.2 (2) C54B—C55B—C56B 120.0
N22—C9—C10 110.1 (2) F4B—C55B—C54B 121.6 (6)
N22—C9—C10 122.9 (2) F4B—C55B—C56B 118.4 (7)
C9—C10—C101 118.8 (2) C55B—C56B—C51B 120.0
C11—C10—C9 123.0 (2) F5B—C56B—C51B 120.0 (11)
C11—C10—C101 118.0 (2) C102—C101—C10 121.4 (2)
C10—C11—C12 131.3 (2) C102—C101—C106 116.5 (2)
N23—C11—C12 123.0 (2) C102—C101—C10 122.1 (2)
N23—C11—C12 105.6 (2) C106—C101—C10 122.1 (2)
C11—C12—Br5 126.88 (18) F6—C102—C101 119.9 (2)
C13—C12—Br5 124.21 (19) F6—C102—C103 117.7 (2)
C13—C12—C11 108.3 (2) C103—C102—C101 122.4 (2)
C12—C13—Br6 124.44 (19) F7—C103—C102 120.7 (2)
C12—C13—C14 108.5 (2) F7—C103—C104 120.0 (2)
C14—C13—Br6 126.40 (18) C104—C103—C102 119.3 (2)
C15—C14—C13 131.5 (2) F8—C104—C103 120.6 (2)
N23—C14—C15 105.6 (2) F8—C104—C105 119.5 (2)
N23—C14—C15 122.9 (2) C103—C104—C105 119.9 (2)
C14—C15—C151 117.8 (2) F9—C105—C104 119.2 (2)
C16—C15—C14 121.8 (2) F9—C105—C106 120.8 (2)
C16—C15—C151 120.4 (2) C106—C105—C106 119.9 (2)
C15—C16—C17 127.5 (2) F10—C106—C101 119.4 (2)
N24—C16—C15 122.1 (2) F10—C106—C105 118.6 (2)
N24—C16—C17 110.2 (2) C105—C106—C101 122.0 (2)
C16—C17—Br7 127.80 (18) F11—C105—C101 121.6 (2)
C18—C17—Br7 125.52 (19) C152—C151—C15 121.6 (2)
C18—C17—C16 106.3 (2) C156—C151—C15 121.6 (2)
C17—C18—Br8 125.01 (18) C156—C151—C152 116.8 (2)
C17—C18—C19 106.6 (2) F11—C152—C151 120.2 (2)
C19—C18—Br8 127.61 (17) F11—C152—C153 118.1 (2)
C20—C19—C18 126.2 (2) C153—C152—C151 121.7 (2)
N24—C19—C18 110.6 (2) F12—C153—C152 120.4 (2)
N24—C19—C20 123.0 (2) F12—C153—C154 119.6 (2)
C1—C20—C19 123.4 (2) C154—C153—C152 120.0 (2)
C1—C20—C201 119.6 (2) F13—C154—C153 119.9 (2)
C19—C20—C201 117.0 (2) C155—C154—C153 120.0 (2)
C1—N21—C4 112.39 (19) F14—C155—C154 119.9 (2)
C1—N21—H21 123.8 F14—C155—C156 120.5 (2)
C4—N21—H21 123.8 C154—C155—C156 119.6 (2)
C6—N22—C9 106.16 (19) F15—C156—C151 119.9 (2)
C11—N23—H23 124.1  F15—C156—C155 118.3 (2)
C14—N23—C11 111.9 (2)  C155—C156—C151 121.9 (2)
C14—N23—H23 124.1  C202—C201—C20 120.6 (2)
C11S—C1S—H 108.0  C202—C201—C206 116.6 (2)
C11S—C1S—C12S 111.8 (3)  C206—C201—C20 122.8 (2)
C11S—C1S—C13S 110.9 (3)  F16—C202—C201 119.9 (2)
C12S—C1S—H 108.0  F16—C202—C203 117.9 (3)
C12S—C1S—C13S 110.1 (3)  C203—C202—C201 122.2 (3)
C13S—C1S—H 108.0  F17—C203—C202 120.5 (3)
C19—N24—C16 105.90 (19)  F17—C203—C206 119.8 (3)
C52—C51—C5 122.6 (11)  C204—C203—C202 119.7 (3)
C52—C51—C56 120.0  F18—C204—C203 120.4 (3)
C56—C51—C5 117.4 (11)  C18—C19—C20—C1 −158.9 (3)
C56—C51—C56 111.8 (3)  C18—C19—C20—C201 22.2 (4)
F1—C52—C51 119.4 (12)  C18—C19—N24—C16 −6.5 (3)
F1—C52—C53 120.5 (11)  C20—C19—C20—C19 15.5 (4)
C53—C52—C51 120.0  C20—C19—C20—C201 −165.6 (2)
C2—C1—C20—C19 172.97 (18)  N21—C4—C5—C6 −15.0 (4)
C2—C1—C20—C201 −117.1 (3)  N21—C4—C5—C51 167.0 (8)
C1—C20—C201—C202 −117.1 (3)  N21—C4—C5—C51B 163.1 (7)
Br1—C2—C3—Br1 −0.6 (3)  N22—C6—C7—Br3 169.45 (19)
Br1—C2—C3—C4 173.4 (3)  N22—C6—C7—C8 −2.7 (3)
C2—C3—C4—C5 −13.4 (4)  Br4—C8—C9—C10 22.4 (4)
C2—C3—C4—N21 −3.2 (3)  Br4—C8—C9—N22 −167.51 (18)
C2—C3—C4—C5 173.4 (3)  
Br2—C3—C4—C5 170.02 (19)  
Br2—C3—C4—N21 −12.9 (8)  
C3—C4—C5—C6 169.0 (3)  
C3—C4—C5—C51B 177.3 (3)  
C3—C4—C5—C51B 177.3 (3)  
C3—C4—N21—C1 176.7 (3)  
Br3—C7—C8—Br4 −0.7 (3)  
Br3—C7—C8—C9 174.94 (18)  
C4—C5—C6—C7 165.5 (2)  
C4—C5—C6—N22 21.1 (4)  
C4—C5—C51—C52 165.5 (2)  
C4—C5—C51—C56 113.2 (9)  
C4—C5—C51B—C52B 112.2 (8)  
Br4—C8—C9—C10 22.4 (4)  
Br4—C8—C9—N22 −167.51 (18)  

Acta Cryst. (2020). E76, 214-220
C5—C4—N21—C1  -171.6 (2)  N22—C9—C10—C101  -153.4 (2)
C5—C6—C7—Br3  -16.5 (4)  N23—C11—C12—Br5  -168.58 (18)
C5—C6—C7—C8  171.4 (2)  N23—C11—C12—C13  2.6 (3)
C5—C6—N22—C9  -167.7 (2)  N23—C14—C15—C16  -25.9 (4)
C5—C51—C52—F1  3.7 (15)  N23—C14—C15—C151  155.8 (2)
C5—C51—C52—C53  -178.5 (18)  F4—C55—C56—F5  -4 (2)
C5—C51—C56—F5  -1.1 (18)  F4—C55—C56—C51  176.7 (13)
C5—C51—C56—C55  178.6 (17)  F3—C54—C55—F4  0.9 (14)
C5—C51B—C52B—C53B -178.7 (15)  F3—C54—C55—C56  177.8 (12)
C5—C51B—C52B—F1B  1.0 (13)  N23—C14—C15—C151  155.8 (2)
C5—C51B—C56B—C55B -178.6 (15)  F2—C53—C54—F3  2.1 (13)
C5—C51B—C56B—F5B  -1.6 (15)  F2—C53—C54—C55  179.9 (13)
Br5—C12—C13—Br6  0.4 (3)  F1—C52—C53—C54  177.8 (17)
Br5—C12—C13—C14  171.48 (18)  F1—C52—C53—F2  -179.9 (12)
C6—C5—C51—C52  113.6 (10)  C51—C5—C6—C7  -16.5 (9)
C6—C5—C51—C56  -64.9 (12)  C51—C5—C6—N22  160.8 (7)
C6—C5—C51B—C52B  109.1 (8)  C51B—C5—C6—C7  -12.6 (8)
C6—C5—C51B—C56B  -69.6 (11)  C51B—C5—C6—N22  160.8 (7)
C6—C7—C8—Br4  171.98 (18)  C51—C5—C6—C7  -16.5 (9)
C6—C7—C8—C9  -2.2 (3)  C51—C5—C6—N22  160.8 (7)
F6—C102—C103—F7  -0.4 (4)  C51—C5—C6—C7  -16.5 (9)
F6—C102—C103—C104  178.5 (2)  C51—C5—C6—N22  160.8 (7)
Br6—C13—C14—C15  -14.4 (4)  C51—C5—C6—C7  -16.5 (9)
Br6—C13—C14—N23  168.31 (18)  C51—C5—C6—N22  160.8 (7)
C7—C6—N22—C9  6.7 (3)  C51—C5—C6—C7  -16.5 (9)
C7—C8—C9—C10  -163.6 (2)  C51—C5—C6—N22  160.8 (7)
C7—C8—C9—N22  6.5 (3)  C51—C5—C6—C7  -16.5 (9)
F7—C103—C104—F8  1.7 (4)  C51—C5—C6—N22  160.8 (7)
F7—C103—C104—C105  -179.5 (2)  C51—C5—C6—C7  -16.5 (9)
Br7—C17—C18—Br8  -3.3 (3)  C51—C5—C6—N22  160.8 (7)
Br7—C17—C18—C19  -174.12 (18)  C51—C5—C6—C7  -16.5 (9)
C8—C9—C10—C11  -168.6 (2)  C51—C5—C6—N22  160.8 (7)
C8—C9—C10—C101  15.5 (4)  C51—C5—C6—C7  -12.6 (8)
C8—C9—N22—C6  -8.1 (3)  C51B—C5—C6—N22  160.8 (7)
F8—C104—C105—F9  -1.0 (4)  C51B—C5—C6—C7  -12.6 (8)
F8—C104—C105—C106  178.2 (2)  C52—C51—C56—F5  0.0
Br8—C18—C19—C20  20.1 (4)  C52—C51—C56—C55  0.0
Br8—C18—C19—N24  -165.68 (18)  C52—C51—C56—C55  0.0
C9—C10—C11—C12  -158.5 (3)  C52—C51—C56—F5B  179.8 (17)
C9—C10—C11—N23  25.2 (4)  C52—C51—C56—C55  0.0
C9—C10—C101—C102  54.8 (3)  C52—C51—C56—F5B  179.8 (17)
C9—C10—C101—C106  -125.1 (3)  C52—C51—C56—C55  0.0
F9—C105—C106—F10  -2.3 (4)  C52—C51—C56—F5B  179.8 (17)
F9—C105—C106—C101  178.6 (2)  C52—C51—C56—C55  0.0
C10—C9—N22—C6  162.4 (2)  C52—C51—C56—F5B  179.8 (17)
C10—C11—C12—Br5  14.6 (4)  F1B—C52B—C53B—C54B  -179.7 (14)
C10—C11—C12—C13  -174.2 (3)  F1B—C52B—C53B—F2B  -0.2 (14)
C10—C11—N23—C14  172.7 (2)  F2B—C53B—C54B—C55B  -179.5 (10)

Acta Cryst. (2020). E76, 214-220
| Bond                  | Angle (°) | Torsion (°) |
|-----------------------|-----------|-------------|
| C10—C101—C102—F6     | 0.5 (4)   |             |
| C10—C101—C102—C103  | −179.3 (2)|             |
| C10—C101—C106—F10   | 1.5 (4)   |             |
| C10—C101—C106—C105  | −179.5 (2)|             |
| C11—C10—C101—C102   | −121.3 (3)|             |
| C11—C10—C101—C106   | 58.8 (3)  |             |
| C11—C12—C13—Br6     | −171.15 (18)|          |
| C11—C12—C13—C14     | 0.0 (3)   |             |
| F11—C152—C153—F12   | 2.7 (4)   |             |
| F11—C152—C153—C154  | −179.0 (2)|             |
| C12—C11—N23—C14     | −4.4 (3)  |             |
| C12—C13—C14—C15     | 174.7 (3) |             |
| C12—C13—C14—N23     | −2.6 (3)  |             |
| F12—C153—C154—F13   | −0.6 (4)  |             |
| F12—C153—C154—C155  | 178.9 (3) |             |
| C13—C14—C15—C16     | 157.2 (3) |             |
| C13—C14—C15—C151    | −21.1 (4) |             |
| C13—C14—N23—C11     | 4.4 (3)   |             |
| F13—C154—C155—F14   | 0.1 (4)   |             |
| F13—C154—C155—C156  | 178.1 (2) |             |
| C14—C15—C16—C17     | 162.8 (2) |             |
| C14—C15—C16—N24     | −23.7 (4) |             |
| C14—C15—C151—C152   | −53.8 (3) |             |
| C14—C15—C151—C156   | 125.3 (3) |             |
| F14—C155—C156—F15   | −0.9 (4)  |             |
| F14—C155—C156—C151  | 178.8 (2) |             |
| C15—C14—N23—C11     | −173.2 (2)|             |
| C15—C16—C17—Br7     | −16.0 (4) |             |
| C15—C16—C17—C18     | 171.1 (2) |             |
| C15—C16—N24—C19     | −168.6 (2)|             |
| C15—C151—C152—F11   | −2.5 (4)  |             |
| C15—C151—C152—C153  | 177.2 (2) |             |
| C15—C151—C156—F15   | 1.2 (4)   |             |
| C15—C151—C156—C155  | −178.5 (2)|             |
| C16—C15—C151—C152   | 127.9 (3) |             |
| C16—C15—C151—C156   | −53.1 (3) |             |
| C16—C17—C18—Br8     | 169.77 (17)|          |
| C16—C17—C18—C19     | −1.0 (3)  |             |
| F16—C202—C203—F17   | 0.2 (4)   |             |
| F16—C202—C203—C204  | 179.8 (3) |             |
| C17—C16—N24—C19     | 5.9 (3)   |             |
| C17—C18—C19—C20     | −169.4 (2)|             |
| C17—C18—C19—N24     | 4.8 (3)   |             |
| F17—C203—C204—F18   | −0.5 (5)  |             |
| F17—C203—C204—C205  | 179.1 (3) |             |
Hydrogen-bond geometry (Å, °)

|     | D—H  | H···A  | D···A  | D—H···A   |
|-----|------|-------|-------|-----------|
| N21—H21···N22 | 0.88  | 2.34  | 2.866 (3) | 119        |
| N21—H21···N24 | 0.88  | 2.42  | 2.928 (3) | 117        |
| N23—H23···N22 | 0.88  | 2.48  | 2.931 (3) | 113        |
| N23—H23···N24 | 0.88  | 2.46  | 2.895 (3) | 111        |

2,3,7,8,12,13,17,18-Octabromo-5,10,15,20-tetrakis(pentafluorophenyl)porphyrin tetrahydrofuran monosolvate (1_THF)

Crystal data

- C₄₄H₂Br₈F₂₀N₄·C₄H₈O
- Mr = 1677.88
- Monoclinic, P2₁/n
- a = 21.524 (9) Å
- b = 9.545 (4) Å
- c = 26.020 (11) Å
- β = 107.426 (7)°
- V = 5101 (4) Å³
- Z = 4
- F(000) = 3176
- Dᵓ = 2.185 Mg m⁻³
- Mo Kα radiation, λ = 0.71073 Å
- θ = 2.3°–28.3°
- µ = 6.41 mm⁻¹
- T = 100 K
- Block fragment, blue
- 0.42 × 0.11 × 0.09 mm

Data collection

- Bruker APEXII CCD diffractometer
- Radiation source: microfocus sealed X-ray tube, graphite-monochromated
- Detector resolution: 7.9 pixels mm⁻¹
- φ and ω scans
- Absorption correction: multi-scan (SADABS; Bruker, 2016)
- T.min = 0.455, T.max = 0.746
- 148293 measured reflections
- 12689 independent reflections
- 10079 reflections with I > 2σ(I)
- R(int) = 0.082
- θ.max = 28.3°, θ.min = 1.5°
- h = −28→28
- k = −12→12
- l = −34→34

Refinement

- Refinement on F²
- Least-squares matrix: full
- R[F² > 2σ(F²)] = 0.054
- wR(F²) = 0.095
- S = 1.21
- 12689 reflections
- 908 parameters
- 718 restraints
- Hydrogen site location: mixed
- H atoms treated by a mixture of independent and constrained refinement
- w = 1/[σ²(Fo²) + 36.9739P]
- where P = (Fo² + 2Fc²)/3
- (Δ/σ)max = 0.001
- Δρ max = 1.63 e Å⁻³
- Δρ min = −0.98 e Å⁻³

Special details

- Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.
- Refinement. Two perfluoro rings were modelled as disordered over two locations with restraints (SADI, SIMU) and constraints (EXYZ, EADP for ipso carbons C12/C12a and C21/C21a) with occupancies of 75:25 and 70:30%. Pyrrole hydrogens located on the difference map and refined using restraints (DFIX). There is a short intermolecular HL···HL contact Br5···Br5 of 3.23 Angstroms (symmetry code = 1-x,-y,1-z = 3_656). See ALERT B
Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2)

| Atom | x      | y      | z      | Uiso* | Ueq  | Occ. (<1) |
|------|--------|--------|--------|-------|------|-----------|
| Br1  | 0.52232(2) | 0.99320(5) | 0.17627(2) | 0.01967(11) | 0.306(17) |
| Br2  | 0.65964(2) | 0.99488(5) | 0.28686(2) | 0.01897(11) | 0.306(17) |
| Br3  | 0.82447(2) | 0.44087(5) | 0.35875(2) | 0.01795(11) | 0.306(17) |
| Br4  | 0.77566(3) | 0.15494(6) | 0.41730(2) | 0.02278(12) | 0.306(17) |
| Br5  | 0.49322(3) | 0.11413(6) | 0.45284(2) | 0.02566(13) | 0.306(17) |
| Br6  | 0.35528(3) | 0.08167(6) | 0.34382(2) | 0.02386(12) | 0.306(17) |
| Br7  | 0.35141(3) | 0.15311(6) | 0.10022(2) | 0.02311(12) | 0.306(17) |
| Br8  | 0.39813(3) | 0.44804(6) | 0.04830(2) | 0.02922(14) | 0.306(17) |
| F1B  | 0.3598(14) | 0.762(2) | 0.1217(9) | 0.024(3) | 0.306(17) |
| F2B  | 0.3219(10) | 0.893(2) | 0.0271(8) | 0.034(3) | 0.306(17) |
| F3B  | 0.4084(10) | 0.924(2) | −0.0300(7) | 0.037(3) | 0.306(17) |
| F4B  | 0.5320(10) | 0.819(2) | 0.0078(7) | 0.033(3) | 0.306(17) |
| F5B  | 0.5669(13) | 0.674(3) | 0.1029(10) | 0.022(3) | 0.306(17) |
| F6   | 0.73871(14) | 0.7001(3) | 0.23590(11) | 0.0205(7) | 0.306(17) |
| F7   | 0.85729(15) | 0.8205(3) | 0.25703(12) | 0.0241(7) | 0.306(17) |
| F8   | 0.92243(15) | 0.9035(4) | 0.35882(14) | 0.0313(8) | 0.306(17) |
| F9   | 0.86678(15) | 0.8738(3) | 0.43919(12) | 0.0255(7) | 0.306(17) |
| F10  | 0.75021(15) | 0.7465(3) | 0.41955(11) | 0.0222(7) | 0.306(17) |
| F11B | 0.6146(14) | −0.081(4) | 0.3942(12) | 0.025(3) | 0.252(18) |
| F12B | 0.6698(11) | −0.231(3) | 0.4832(9) | 0.030(3) | 0.252(18) |
| F13B | 0.7274(11) | −0.093(3) | 0.5776(9) | 0.032(3) | 0.252(18) |
| F14B | 0.7159(12) | 0.188(3) | 0.5826(10) | 0.033(3) | 0.252(18) |
| F15B | 0.6587(12) | 0.339(3) | 0.4930(12) | 0.028(3) | 0.252(18) |
| F16  | 0.40450(15) | −0.0892(3) | 0.23781(13) | 0.0266(7) | 0.306(10) |
| F17  | 0.28883(18) | −0.2265(4) | 0.20717(15) | 0.0360(9) | 0.306(10) |
| F18  | 0.17533(17) | −0.0789(4) | 0.17611(17) | 0.0441(10) | 0.306(10) |
| F19  | 0.17774(15) | 0.2053(4) | 0.17564(16) | 0.0395(9) | 0.306(10) |
| F20  | 0.29175(15) | 0.3428(3) | 0.20559(13) | 0.0261(7) | 0.306(10) |
| N21  | 0.57239(19) | 0.6076(4) | 0.25003(16) | 0.0159(9) | 0.306(10) |
| N22  | 0.63177(19) | 0.4305(4) | 0.34616(16) | 0.0153(9) | 0.306(10) |
| H22  | 0.5882(12) | 0.470(5) | 0.339(2) | 0.015(14)* | 0.306(10) |
| N23  | 0.5165(2) | 0.2459(4) | 0.30937(16) | 0.0159(9) | 0.306(10) |
| N24  | 0.4668(2) | 0.4152(5) | 0.21131(16) | 0.0163(9) | 0.306(10) |
| H24  | 0.492(2) | 0.442(6) | 0.2476(10) | 0.032(17)* | 0.306(10) |
| C1   | 0.4597(2) | 0.4950(6) | 0.16626(19) | 0.0154(10) | 0.306(10) |
| C2   | 0.4854(2) | 0.6308(5) | 0.1660(2) | 0.0170(10) | 0.306(10) |
| C3   | 0.5348(2) | 0.6877(5) | 0.2093(2) | 0.0164(10) | 0.306(10) |
| C4   | 0.5587(2) | 0.8328(5) | 0.21547(19) | 0.0147(10) | 0.306(10) |
| C5   | 0.6130(2) | 0.8340(5) | 0.2584(2) | 0.0150(10) | 0.306(10) |
| C6   | 0.6230(2) | 0.6905(5) | 0.27870(19) | 0.0148(10) | 0.306(10) |
| C7   | 0.6794(2) | 0.6364(5) | 0.31605(19) | 0.0149(10) | 0.306(10) |
| C8   | 0.6838(2) | 0.5035(5) | 0.33991(19) | 0.0154(10) | 0.306(10) |
| C9   | 0.7404(2) | 0.4180(5) | 0.36414(19) | 0.0143(10) | 0.306(10) |
| C10  | 0.7210(2) | 0.3030(5) | 0.3861(2) | 0.0154(10) | 0.306(10) |
| C11  | 0.6514(2) | 0.3098(5) | 0.37521(19) | 0.0158(10) | 0.306(10) |
| Atom   | x       | y       | z       | U(eq)  |
|--------|---------|---------|---------|--------|
| C12    | 0.6088  | 0.2197  | 0.3909  | 0.0181 |
| C12A   | 0.6362  | 0.1253  | 0.4380  | 0.0225 |
| C12F   | 0.6624  | 0.1997  | 0.4855  | 0.0246 |
| C12E   | 0.6914  | 0.129   | 0.5333  | 0.0259 |
| C12D   | 0.6943  | -0.017  | 0.5336  | 0.0258 |
| C12C   | 0.6680  | -0.0913 | 0.4861  | 0.0250 |
| C12B   | 0.6390  | -0.0202 | 0.4383  | 0.0234 |
| C13    | 0.5419  | 0.2086  | 0.3621  | 0.0176 |
| C14    | 0.4905  | 0.1467  | 0.3813  | 0.0166 |
| C15    | 0.4364  | 0.1415  | 0.3390  | 0.0190 |
| C16    | 0.4536  | 0.2014  | 0.2931  | 0.0145 |
| C17    | 0.4142  | 0.2103  | 0.2392  | 0.0151 |
| C18    | 0.4276  | 0.2986  | 0.2003  | 0.0159 |
| C19    | 0.3984  | 0.2980  | 0.1429  | 0.0160 |
| C20    | 0.4176  | 0.4162  | 0.1218  | 0.0175 |
| C21B   | 0.4602  | 0.7128  | 0.1155  | 0.0194 |
| C22B   | 0.4009  | 0.775   | 0.0944  | 0.0222 |
| C23B   | 0.3830  | 0.846   | 0.0440  | 0.0258 |
| C24B   | 0.4034  | 0.854   | 0.0173  | 0.0268 |
| C25B   | 0.4895  | 0.802   | 0.0375  | 0.0259 |
| C26B   | 0.5064  | 0.729   | 0.0845  | 0.0226 |
| C71    | 0.7412  | 0.7171  | 0.3271  | 0.0149 |
| C72    | 0.7695  | 0.7413  | 0.2864  | 0.0159 |
| C73    | 0.8303  | 0.8037  | 0.2964  | 0.0175 |
| C74    | 0.8631  | 0.8463  | 0.3483  | 0.0195 |
| C75    | 0.8353  | 0.8286  | 0.3890  | 0.0176 |
| C76    | 0.7754  | 0.7640  | 0.3785  | 0.0177 |
| C71    | 0.3519  | 0.1309  | 0.2218  | 0.0165 |
| C72    | 0.3489  | -0.0143 | 0.2218  | 0.0210 |
| C73    | 0.2904  | -0.0867 | 0.2065  | 0.0271 |
| C74    | 0.2327  | -0.0110 | 0.1905  | 0.0315 |
| C75    | 0.2339  | 0.1325  | 0.1905  | 0.0252 |
| C76    | 0.2929  | 0.2012  | 0.2059  | 0.0207 |
| F1A    | 0.3574  | 0.7361  | 0.1329  | 0.0236 |
| F2A    | 0.3068  | 0.8801  | 0.0399  | 0.0346 |
| F3A    | 0.3808  | 0.9315  | -0.0262 | 0.0399 |
| F4A    | 0.5055  | 0.8370  | 0.0000  | 0.0370 |
| F5A    | 0.5569  | 0.6956  | 0.0924  | 0.0267 |
| F11A   | 0.6002  | -0.0773 | 0.3832  | 0.0261 |
| F12A   | 0.6504  | -0.2514 | 0.4669  | 0.0321 |
| F13A   | 0.7155  | -0.1454 | 0.5655  | 0.0349 |
| F14A   | 0.7237  | 0.1351  | 0.5815  | 0.0362 |
| F15A   | 0.6742  | 0.3116  | 0.4979  | 0.0303 |
| O25    | 0.5156  | 0.5565  | 0.3555  | 0.0452 |
| C26    | 0.4822  | 0.6868  | 0.3386  | 0.0428 |
| H26A   | 0.4400  | 0.670382 | 0.310628 | 0.051* |
| H26B   | 0.508874 | 0.750268 | 0.323698 | 0.051* |
| C27    | 0.4714  | 0.7495  | 0.3890  | 0.0485 |
### Atomic displacement parameters ($\AA^2$)

|       | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-------|-----------|-----------|-----------|-----------|-----------|-----------|
| Br1   | 0.0205 (2) | 0.0128 (2) | 0.0221 (3) | 0.0018 (2) | 0.00074 (19) | 0.0039 (2) |
| Br2   | 0.0225 (2) | 0.0116 (2) | 0.0198 (2) | −0.0023 (2) | 0.00176 (19) | −0.0003 (2) |
| Br3   | 0.0163 (2) | 0.0166 (2) | 0.0195 (2) | 0.0006 (2)  | 0.00298 (19) | −0.0003 (2) |
| Br4   | 0.0204 (3) | 0.0165 (3) | 0.0274 (3) | 0.0033 (2)  | 0.0011 (2)  | 0.0070 (2)  |
| Br5   | 0.0309 (3) | 0.0302 (3) | 0.0177 (3) | −0.0009 (2) | 0.0100 (2)  | 0.0056 (2)  |
| Br6   | 0.0197 (3) | 0.0276 (3) | 0.0268 (3) | −0.0009 (2) | 0.0107 (2)  | 0.0072 (2)  |
| Br7   | 0.0259 (3) | 0.0183 (3) | 0.0216 (3) | −0.0048 (2) | 0.0017 (2)  | −0.0021 (2) |
| Br8   | 0.0433 (3) | 0.0249 (3) | 0.0145 (2) | −0.0085 (3) | 0.0011 (2)  | 0.0009 (2)  |
| F1B   | 0.024 (4)   | 0.021 (5)   | 0.022 (5)   | 0.004 (4)   | −0.001 (5)   | 0.008 (4)   |
| F2B   | 0.037 (5)   | 0.022 (4)   | 0.029 (5)   | 0.001 (4)   | −0.012 (4)   | 0.006 (4)   |
| F3B   | 0.048 (6)   | 0.028 (5)   | 0.022 (4)   | −0.002 (6)   | −0.008 (5)   | 0.009 (4)   |
| F4B   | 0.044 (6)   | 0.032 (5)   | 0.021 (5)   | −0.007 (5)   | 0.007 (5)   | 0.010 (4)   |
| F5B   | 0.027 (6)   | 0.022 (6)   | 0.018 (6)   | −0.001 (5)   | 0.008 (5)   | 0.011 (5)   |
| F6    | 0.0234 (16) | 0.0227 (17) | 0.0141 (14) | −0.0004 (13) | 0.0034 (12) | −0.0023 (12) |
| F7    | 0.0283 (17) | 0.0228 (17) | 0.0242 (16) | −0.0036 (14) | 0.0126 (13) | 0.0019 (14)  |
| F8    | 0.0222 (16) | 0.033 (2)   | 0.0364 (19) | −0.0134 (15) | 0.0055 (14) | −0.0040 (16) |
| F9    | 0.0270 (17) | 0.0245 (18) | 0.0193 (15) | −0.0054 (14) | −0.0019 (13) | −0.0059 (13) |
| F10   | 0.0260 (16) | 0.0264 (17) | 0.0151 (15) | −0.0026 (14) | 0.0073 (12) | −0.0020 (13) |
| F11B  | 0.022 (6)   | 0.025 (5)   | 0.026 (6)   | 0.000 (5)    | 0.002 (5)    | 0.010 (5)    |
| F12B  | 0.030 (5)   | 0.030 (5)   | 0.031 (5)   | 0.008 (5)    | 0.008 (4)    | 0.015 (5)    |
| F13B  | 0.028 (5)   | 0.039 (6)   | 0.028 (5)   | 0.013 (5)    | 0.008 (4)    | 0.019 (5)    |
| F14B  | 0.034 (5)   | 0.041 (6)   | 0.019 (5)   | 0.003 (6)    | −0.001 (4)   | 0.008 (6)    |
| F15B  | 0.025 (6)   | 0.031 (6)   | 0.024 (5)   | 0.002 (5)    | −0.001 (5)   | 0.003 (5)    |
| F16   | 0.0236 (16) | 0.0167 (17) | 0.0383 (19) | 0.0016 (13) | 0.0075 (14) | 0.0057 (14) |
| Atom | U_{11}  | U_{22}  | U_{33}  | U_{12}  | U_{13}  | U_{23}  |
|------|---------|---------|---------|---------|---------|---------|
| F17  | 0.040   | 0.0193  | 0.049   | -0.0109 | 0.0139  | -0.0005 |
| F18  | 0.0234  | 0.040   | 0.064   | -0.0194 | 0.0056  | -0.002  |
| F19  | 0.0142  | 0.042   | 0.058   | 0.0033  | 0.0050  | 0.0043  |
| F20  | 0.0218  | 0.0203  | 0.0354  | 0.0020  | 0.0074  | 0.0020  |
| N21  | 0.015   | 0.014   | 0.014   | -0.0024 | 0.0015  | 0.0022  |
| N22  | 0.013   | 0.013   | 0.018   | -0.0050 | 0.0025  | 0.0034  |
| N23  | 0.016   | 0.016   | 0.014   | -0.0011 | 0.0011  | 0.0026  |
| N24  | 0.018   | 0.018   | 0.0109  | -0.0022 | 0.0011  | 0.0020  |
| C1   | 0.015   | 0.016   | 0.014   | 0.004   | 0.0034  | 0.003  |
| C2   | 0.017   | 0.018   | 0.014   | 0.003   | 0.0029  | 0.003  |
| C3   | 0.015   | 0.016   | 0.017   | -0.001  | 0.0032  | 0.004  |
| C4   | 0.015   | 0.013   | 0.017   | 0.0019  | 0.0045  | 0.003  |
| C5   | 0.018   | 0.009   | 0.017   | 0.0016  | 0.0039  | 0.0008  |
| C6   | 0.015   | 0.016   | 0.014   | 0.0005  | 0.0052  | 0.0027  |
| C7   | 0.019   | 0.011   | 0.013   | 0.0016  | 0.0036  | -0.001  |
| C8   | 0.017   | 0.015   | 0.012   | -0.002  | 0.0022  | 0.000  |
| C9   | 0.015   | 0.012   | 0.014   | 0.0000  | 0.0006  | -0.002  |
| C10  | 0.017   | 0.011   | 0.016   | 0.0024  | 0.0005  | 0.0025  |
| C11  | 0.020   | 0.013   | 0.012   | 0.0000  | 0.0015  | 0.0008  |
| C12  | 0.024   | 0.015   | 0.014   | -0.001  | 0.004   | 0.002  |
| C12A | 0.0202  | 0.0260  | 0.0209  | -0.0001 | 0.0055  | 0.0079  |
| C12F | 0.023   | 0.028   | 0.021   | 0.000   | 0.004   | 0.009  |
| C12E | 0.025   | 0.030   | 0.021   | 0.001   | 0.005   | 0.010  |
| C12D | 0.024   | 0.030   | 0.023   | 0.004   | 0.007   | 0.011  |
| C12C | 0.023   | 0.027   | 0.025   | 0.004   | 0.008   | 0.010  |
| C12B | 0.021   | 0.026   | 0.023   | 0.002   | 0.007   | 0.008  |
| C13  | 0.024   | 0.012   | 0.016   | -0.002  | 0.006   | 0.002  |
| C14  | 0.024   | 0.019   | 0.007   | 0.000   | 0.0043  | 0.0037  |
| C15  | 0.018   | 0.015   | 0.025   | -0.003  | 0.008   | 0.002  |
| C16  | 0.017   | 0.010   | 0.016   | -0.0008 | 0.0046  | 0.0028  |
| C17  | 0.013   | 0.015   | 0.017   | 0.0013  | 0.0045  | 0.002  |
| C18  | 0.013   | 0.014   | 0.019   | 0.0030  | 0.0004  | 0.002  |
| C19  | 0.016   | 0.018   | 0.014   | 0.000   | 0.0031  | -0.001 |
| C20  | 0.019   | 0.019   | 0.011   | 0.002   | 0.0000  | 0.002  |
| C21B | 0.0267  | 0.0146  | 0.0141  | -0.0026 | 0.0018  | 0.0026  |
| C22B | 0.029   | 0.016   | 0.017   | -0.001  | 0.000   | 0.003  |
| C23B | 0.032   | 0.019   | 0.019   | -0.002  | -0.003  | 0.005  |
| C24B | 0.035   | 0.020   | 0.019   | -0.003  | -0.001  | 0.005  |
| C25B | 0.034   | 0.021   | 0.019   | -0.003  | 0.001   | 0.005  |
| C26B | 0.030   | 0.018   | 0.017   | -0.003  | 0.002   | 0.005  |
| C71  | 0.018   | 0.006   | 0.017   | -0.0018 | 0.0010  | 0.0012  |
| C72  | 0.020   | 0.011   | 0.014   | 0.002   | 0.0007  | -0.0003 |
| C73  | 0.020   | 0.012   | 0.022   | 0.001   | 0.009   | 0.003  |
| C74  | 0.016   | 0.010   | 0.029   | -0.003  | 0.003   | -0.001 |
| C75  | 0.019   | 0.013   | 0.017   | -0.002  | -0.001  | -0.003 |
| C76  | 0.021   | 0.013   | 0.019   | 0.003   | 0.005   | 0.000  |
| C171 | 0.019   | 0.018   | 0.013   | -0.002  | 0.0061  | 0.001  |
| C172 | 0.020   | 0.019   | 0.024   | -0.001  | 0.006   | 0.003  |
### Geometric parameters (Å, °)

| Bond                  | Length (Å) | Angle (°)    |
|-----------------------|------------|-------------|
| Br1—C4                | 1.876 (5)  |             |
| Br2—C5                | 1.862 (5)  |             |
| Br3—C9                | 1.869 (5)  |             |
| Br4—C10               | 1.863 (5)  |             |
| Br5—C14               | 1.869 (5)  |             |
| Br6—C15               | 1.877 (5)  |             |
| Br7—C19               | 1.870 (5)  |             |
| Br8—C20               | 1.858 (5)  |             |
| F1B—C22B              | 1.33 (3)   |             |
| F2B—C23B              | 1.33 (3)   |             |
| F3B—C24B              | 1.35 (3)   |             |
| F4B—C25B              | 1.37 (3)   |             |
| F5B—C26B              | 1.35 (3)   |             |
| F6—C72                | 1.343 (5)  |             |

*Acta Cryst.* (2020). *E*76, 214-220
F7—C73  1.329 (6)  C71—C72  1.389 (7)
F8—C74  1.339 (6)  C71—C76  1.392 (7)
F9—C75  1.350 (5)  C72—C73  1.389 (7)
F10—C76  1.346 (6)  C73—C74  1.386 (7)
F11B—C12B  1.25 (3)  C74—C75  1.373 (8)
F12B—C12C  1.34 (2)  C75—C76  1.380 (7)
F13B—C12D  1.36 (2)  C171—C172  1.388 (7)
F14B—C12E  1.36 (3)  C171—C176  1.385 (7)
F15B—C12F  1.35 (3)  C172—C173  1.390 (9)
F16—C172  1.349 (6)  C173—C174  1.370 (9)
F17—C173  1.335 (7)  C174—C175  1.378 (7)
F18—C174  1.344 (6)  C175—C176  1.378 (7)
F19—C175  1.346 (6)  F1A—C22A  1.354 (15)
F20—C176  1.352 (6)  F2A—C23A  1.348 (12)
N21—C3  1.358 (6)  F3A—C24A  1.334 (12)
N21—C6  1.371 (6)  F4A—C25A  1.337 (13)
N22—H22  0.974 (10)  F5A—C26A  1.344 (15)
N22—C8  1.369 (6)  F11A—C122  1.356 (12)
N22—C11  1.373 (6)  F12A—C123  1.331 (11)
N23—C13  1.363 (6)  F13A—C124  1.343 (9)
N23—C16  1.361 (6)  F14A—C125  1.334 (11)
N24—H24  0.975 (10)  F15A—C126  1.333 (11)
N24—C1  1.368 (6)  O25—C26  1.439 (8)
N24—C18  1.374 (6)  O25—C29  1.459 (9)
C1—C2  1.410 (7)  C26—H26A  0.9900
C1—C20  1.448 (7)  C26—H26B  0.9900
C2—C3  1.407 (7)  C26—C27  1.521 (10)
C2—C21B  1.487 (7)  C27—H27A  0.9900
C2—C21A  1.487 (7)  C27—H27B  0.9900
C3—C4  1.469 (7)  C27—C28  1.522 (11)
C4—C5  1.354 (7)  C28—H28A  0.9900
C5—C6  1.461 (7)  C28—H28B  0.9900
C6—C7  1.407 (7)  C28—C29  1.481 (10)
C7—C8  1.403 (7)  C29—H29A  0.9900
C7—C71  1.490 (7)  C29—H29B  0.9900
C8—C9  1.446 (7)  C21A—C22A  1.400 (18)
C9—C10  1.361 (7)  C21A—C26A  1.370 (19)
C10—C11  1.440 (7)  C22A—C23A  1.385 (15)
C11—C12  1.404 (7)  C23A—C24A  1.385 (14)
C12—C12A  1.493 (7)  C24A—C25A  1.377 (13)
C12—C12C  1.414 (7)  C25A—C26A  1.384 (16)
C12—C12D  1.493 (7)  C121—C122  1.395 (11)
C12A—C12F  1.3900  C121—C126  1.405 (10)
C12A—C12B  1.3900  C122—C123  1.396 (10)
C12F—C12E  1.3900  C123—C124  1.385 (11)
C12E—C12D  1.3900  C124—C125  1.362 (12)
C12D—C12C  1.3900  C125—C126  1.389 (11)
C12C—C12B  1.3900
C13—C12—C121 118.6 (5) C27—C26—H26B 110.6
C12F—C12A—C12 112.2 (7) C26—C27—H27A 111.1
C12F—C12A—C12B 120.0 C26—C27—H27B 111.1
C12B—C12A—C12 127.8 (7) C26—C27—C28 103.3 (6)
F15B—C12F—C12A 127.2 (14) H27A—C27—H27B 109.1
F15B—C12F—C12E 112.5 (14) C28—C27—H27A 111.1
C12E—C12F—C12A 120.0 C28—C27—H27B 111.1
F14B—C12E—C12F 125.8 (14) C27—C28—H28A 111.3
F14B—C12E—C12D 114.2 (14) C27—C28—H28B 111.3
C12F—C12E—C12D 123.0 (15) C29—C28—H28A 111.3
C12E—C12F—C12C 120.0 C29—C28—H28B 111.3
F13B—C12D—C12C 123.2 (14) O25—C29—C28 109.1 (6)
F13B—C12D—C12B 116.8 (14) O25—C29—H29A 109.9
C12B—C12D—C12C 120.0 O25—C29—H29B 109.9
F11B—C12B—C12A 116.8 (18) C28—C29—H29A 109.9
F11B—C12B—C12C 116.7 (15) C29—C28—H28B 111.3
C12E—C12D—C12C 120.0
F12B—C12C—C12D 123.2 (14) O25—C29—C28 109.1 (6)
F12B—C12C—C12B 116.8 (14) O25—C29—H29A 109.9
C12B—C12C—C12D 120.0 O25—C29—H29B 109.9
F11B—C12B—C12A 116.8 (18) C28—C29—H29A 109.9
F11B—C12B—C12C 123.2 (18) C28—C29—H29B 109.9
C12C—C12B—C12A 120.0 H29A—C29—H29B 108.3
N23—C13—C12 123.4 (5) C22A—C21A—C2 118.6 (6)
N23—C13—C14 109.5 (4) C26A—C21A—C2 124.3 (6)
C12—C13—C14 126.9 (5) C26A—C21A—C22A 117.1 (7)
C13—C14—Br5 127.2 (4) F1A—C22A—C21A 121.1 (9)
C15—C14—Br5 124.8 (4) F1A—C22A—C23A 117.6 (13)
C15—C14—C13 106.9 (4) C23A—C22A—C21A 121.3 (10)
C14—C15—Br6 123.5 (4) F2A—C23A—C22A 121.3 (10)
C14—C15—C16 106.6 (4) F2A—C23A—C24A 119.1 (8)
C16—C15—Br6 129.7 (4) C24A—C23A—C22A 119.7 (10)
N23—C16—C15 109.8 (4) F3A—C24A—C23A 120.4 (9)
N23—C16—C17 122.5 (4) F3A—C24A—C25A 119.7 (10)
C17—C16—C15 127.7 (4) C25A—C24A—C23A 119.9 (9)
C16—C17—C18 123.9 (5) F4A—C25A—C24A 120.5 (10)
C16—C17—C171 119.4 (4) F4A—C25A—C26A 120.1 (10)
C18—C17—C171 116.6 (4) C24A—C25A—C26A 119.4 (10)
N24—C18—C17 125.5 (5) F5A—C26A—C21A 119.1 (10)
N24—C18—C19 105.6 (4) F5A—C26A—C25A 118.1 (13)
C17—C18—C19 128.5 (5) C21A—C26A—C25A 122.7 (10)
C18—C19—Br7 127.7 (4) C122—C121—C12 120.3 (6)
C20—C19—Br7 123.0 (4) C122—C121—C126 115.9 (6)
C20—C19—C18 108.8 (4) C126—C121—C12 123.7 (6)
C1—C20—Br8 129.1 (4) F11A—C122—C121 120.7 (8)
C19—C20—Br8 123.0 (4) F11A—C122—C123 116.2 (8)
C19—C20—C1 107.5 (4) C121—C122—C12 123.1 (8)
C22B—C21B—C2 129.4 (13) C12A—C123—C122 121.7 (8)
C22B—C21B—C26B 116.1 (17) F12A—C123—C124 120.4 (7)
C26B—C21B—C2 114.5 (12) C124—C123—C122 117.9 (8)
F1B—C22B—C21B 116 (2) F13A—C124—C123 119.6 (8)
F1B—C22B—C23B 121 (3) F13A—C124—C125 118.9 (8)
| Bond | Angle (°) | Error (°) | Bond | Angle (°) | Error (°) |
|------|----------|-----------|------|----------|-----------|
| C21B—C22B—C23B | 123 (3) | | C125—C124—C123 | 121.5 (7) |
| F2B—C23B—C22B | 116 (2) | | F14A—C125—C124 | 121.5 (7) |
| F2B—C23B—C24B | 128 (2) | | F14A—C125—C126 | 118.9 (8) |
| C24B—C23B—C22B | 116 (2) | | C124—C125—C126 | 119.6 (7) |
| F3B—C24B—C23B | 112 (2) | | F15A—C126—C121 | 117.8 (7) |
| C25B—C24B—F3B | 125 (2) | | F15A—C126—C125 | 120.1 (8) |
| C25B—C24B—C23B | 123 (2) | | C125—C126—C121 | 122.0 (8) |
| C24B—C25B—F4B | 117 (2) | | | |

| Bond | Angle (°) | Error (°) | Bond | Angle (°) | Error (°) |
|------|----------|-----------|------|----------|-----------|
| Br1—C4—C5—Br2 | 1.5 (7) | | C12A—C12—C13—N23 | −154.4 (5) |
| Br1—C4—C5—C6 | 177.4 (3) | | C12A—C12—C13—C14 | 20.2 (8) |
| Br2—C5—C6—C7 | −17.4 (8) | | C12A—C12F—C12E—F14B | −177 (3) |
| Br3—C9—C10—Br4 | 3.2 (6) | | C12F—C12A—C12B—F11B | −180 (2) |
| Br3—C9—C10—C11 | −170.2 (3) | | C12F—C12A—C12B—C12C | 0.0 |
| Br4—C10—C11—N22 | −172.6 (4) | | C12F—C12E—C12D—F13B | 173 (2) |
| Br4—C10—C11—C12 | 9.5 (8) | | C12F—C12E—C12D—C12C | 0.0 |
| Br5—C14—C15—Br6 | −6.7 (7) | | C12E—C12D—C12C—F12B | 179 (3) |
| Br5—C14—C15—C16 | 168.6 (4) | | C12E—C12D—C12C—C12B | 0.0 |
| Br6—C15—C16—N23 | 172.5 (4) | | C12D—C12C—C12B—F11B | 180 (3) |
| Br6—C15—C16—C17 | −9.6 (8) | | C12D—C12C—C12B—C12A | 0.0 |
| Br7—C19—C20—Br8 | 1.0 (6) | | C12B—C12A—C12F—F15B | −174 (3) |
| Br7—C19—C20—C1 | −172.6 (4) | | C12B—C12A—C12F—C12E | 0.0 |
| F1B—C22B—C23B—F2B | −2 (4) | | C13—N23—C16—C15 | 4.3 (6) |
| F1B—C22B—C23B—C24B | −179 (3) | | C13—N23—C16—C17 | −173.7 (5) |
| F2B—C23B—C24B—F3B | 3 (4) | | C13—C12—C12A—C12F | −120.0 (10) |
| F2B—C23B—C24B—C25B | −179 (3) | | C13—C12—C12A—C12B | 61.6 (13) |
| F3B—C24B—C25B—F4B | 0 (4) | | C13—C12—C12C—F12B | 179 (3) |
| F3B—C24B—C25B—C26B | −178 (3) | | C13—C12—C12C—C12B | 0.0 |
| F4B—C25B—C26B—F5B | 1 (4) | | C13—C14—C15—Br6 | −175.7 (4) |
| F4B—C25B—C26B—C21B | 180 (2) | | C13—C14—C15—C16 | −0.5 (6) |
| F6—C72—C73—F7 | −0.8 (7) | | C14—C15—C16—N23 | −2.4 (6) |
| F6—C72—C73—C74 | −179.6 (4) | | C14—C15—C16—C17 | 175.6 (5) |
| F7—C73—C74—C75 | −0.5 (7) | | C15—N23—C16—C17 | 163.5 (5) |
| F7—C73—C74—F8 | −179.8 (5) | | C15—C16—C17—C171 | −11.8 (8) |
| F8—C74—C75—F9 | 2.5 (8) | | C16—N23—C13—C12 | 170.8 (5) |
| F8—C74—C75—C76 | −177.2 (5) | | C16—N23—C13—C14 | −4.6 (6) |
| F9—C75—C76—F10 | −0.8 (7) | | C16—C17—C18—N24 | −20.9 (8) |
| F9—C75—C76—C71 | 179.4 (5) | | C16—C17—C18—C19 | 167.4 (5) |
| F12B—C12C—C12B—F11B | 1 (2) | | C16—C17—C171—C172 | −64.6 (7) |
| F12B—C12C—C12B—C12A | −179 (2) | | C16—C17—C171—C176 | 113.9 (6) |
| F13B—C12D—C12C—F12B | 5 (3) | | C17—C18—C19—Br7 | −18.5 (8) |
| F13B—C12D—C12C—C12B | −173.8 (19) | | C17—C18—C19—C20 | 169.5 (5) |
| F14B—C12E—C12D—F13B | −10 (3) | | C17—C171—C172—F16 | −0.3 (8) |
| F14B—C12E—C12D—C12C | 177 (2) | | C17—C171—C172—C173 | 178.8 (5) |
| F15B—C12F—C12E—F14B | −2 (2) | | C17—C171—C176—F20 | 0.5 (8) |
| F15B—C12F—C12E—C12D | 175 (2) | | C17—C171—C176—C175 | −178.8 (5) |
| F16—C172—C173—F17 | −0.4 (8) | | C18—N24—C1—C2 | 169.7 (5) |
F16—C172—C173—C174 179.2 (5) C18—N24—C1—C20 6.1 (6)
F17—C173—C174—F18 0.4 (9) C18—C17—C171—C172 119.7 (6)
F17—C173—C174—C175 179.1 (6) C18—C17—C171—C176 61.8 (7)
F18—C174—C175—F19 0.4 (9) C18—C19—C20—Br8 173.3 (4)
F18—C174—C175—C176 179.2 (5) C18—C19—C20—Br8 0.2 (6)
F19—C175—C176—F20 0.1 (8) C20—C1—C2—C3 168.4 (5)
F19—C175—C176—C171 179.5 (5) C20—C1—C2—C21B 8.4 (8)
N21—C3—C4—Br1 173.3 (4) C21B—C2—C3—N21 8.4 (8)
N21—C3—C4—C5 3.7 (6) C21B—C2—C3—C4 11.7 (8)
N21—C6—C7—C8 −19.1 (8) C21B—C2—C3—C4 11.7 (8)
N21—C6—C7—C71 153.7 (5) C21B—C2—C3—C4 11.7 (8)
N22—C8—C9—Br3 168.2 (4) C21B—C2—C3—C4 11.7 (8)
N22—C8—C9—C10 −3.2 (5) C22B—C21B—C22B—F1B 178 (3)
N22—C11—C12—C12A −157.2 (5) C22B—C21B—C22B—C23B 175 (3)
N22—C11—C12—C13 26.5 (8) C22B—C21B—C22B—C23B 175 (3)
N23—C13—C14—Br5 −165.5 (4) C22B—C21B—C22B—C23B 175 (3)
N23—C13—C14—C15 3.2 (6) C22B—C21B—C22B—C23B 175 (3)
N23—C16—C17—C18 −18.8 (8) C22B—C21B—C22B—C23B 175 (3)
N23—C16—C17—C171 165.9 (5) C22B—C21B—C22B—C23B 175 (3)
N24—C1—C2—C3 16.8 (8) C22B—C21B—C22B—C23B 175 (3)
N24—C1—C2—C21B −166.5 (5) C22—C21B—C22B—C23B 175 (3)
N24—C1—C2—C21A 166.5 (5) C22—C21B—C22B—C23B 175 (3)
N24—C1—C20—Br8 −169.3 (4) C71—C7—C8—N22 162.3 (5)
N24—C1—C20—C19 3.8 (6) C71—C7—C8—C9 15.1 (8)
N24—C18—C19—Br7 168.5 (4) C71—C7—C8—C9 15.1 (8)
N24—C18—C19—C20 −3.4 (6) C71—C7—C8—C9 15.1 (8)
C1—N24—C18—C17 −167.2 (5) C71—C7—C8—C9 15.1 (8)
C1—N24—C18—C19 6.0 (6) C71—C7—C8—C9 15.1 (8)
C1—C2—C3—N21 15.3 (8) C71—C7—C8—C9 15.1 (8)
C1—C2—C3—C4 −171.6 (5) C71—C7—C8—C9 15.1 (8)
C1—C2—C21B—C22B 73 (2) C73—C74—C75—F9 178.2 (5)
C1—C2—C21B—C26B −107.6 (17) C73—C74—C75—C76 2.1 (8)
C1—C2—C21A—C22A 69.4 (9) C74—C75—C76—F10 178.9 (5)
C1—C2—C21A—C26A −108.8 (10) C74—C75—C76—C71 −0.9 (8)
C2—C1—C20—Br8 15.1 (8) C76—C71—C72—F6 −179.2 (4)
C2—C1—C20—C19 −171.9 (5) C76—C71—C72—C73 2.8 (7)
C2—C3—C4—Br1 12.9 (8) C171—C17—C18—N24 154.5 (5)
C2—C3—C4—C5 −170.1 (5) C171—C17—C18—C19 15.1 (8)
C2—C21B—C22B—F1B 0 (4) C171—C17—C18—C19 15.1 (8)
C2—C21B—C22B—C23B −177.5 (18) C171—C17—C18—C19 15.1 (8)
C2—C21B—C26B—F5B −1 (4) C172—C171—C176—C175 −0.2 (8)
C2—C21B—C26B—C25B 180 (2) C172—C171—C176—C175 −0.2 (8)
C2—C21A—C22A—F1A 2.4 (16) C172—C173—C174—F18 −179.2 (5)
C2—C21A—C22A—C23A −178.5 (9) C172—C173—C174—C175 −0.5 (9)
C2—C21A—C26A—F5A 0.4 (17) C173—C174—C175—F19 −179.1 (5)
C2—C21A—C26A—C25A 177.9 (10) C173—C174—C175—C176 0.6 (10)
C3—N21—C6—C5 6.2 (5) C174—C175—C176—F20 −179.5 (5)
C174—C175—C176—C171 −0.2 (9)

**Acta Cryst. (2020). E76, 214-220**

sup-22
| Bond         | Distance (Å) | Bond         | Distance (Å) |
|-------------|-------------|-------------|-------------|
| C3—N21—C6—C7 | 165.2 (5)   | C176—C171—C172—F16 | 178.8 (4) |
| C3—C2—C21B—C22B | 110.2 (2) | C176—C171—C172—C173 | 0.2 (8)  |
| C3—C2—C21B—C26B | 92.2 (17)  | F1A—C22A—C23A—F2A | -0.3 (17) |
| C3—C2—C21A—C22A | 113.8 (9)  | F1A—C22A—C23A—C24A | 179.8 (10) |
| C3—C2—C21A—C26A | 68.0 (11)  | F2A—C23A—C24A—F3A | -0.3 (14) |
| C3—C4—C5—Br2  | -175.6 (4) | F2A—C23A—C24A—C25A | 179.5 (10) |
| C3—C4—C5—C6   | 0.2 (6)     | F3A—C24A—C25A—F4A | -2.5 (15) |
| C4—C5—C6—N21  | -4.0 (6)    | F3A—C24A—C25A—C26A | 180.0 (11) |
| C4—C5—C6—C7   | 167.1 (5)   | F4A—C25A—C26A—F5A | 0.3 (19)  |
| C5—C6—C7—C8   | 170.9 (5)   | F4A—C25A—C26A—C21A | -177.2 (11) |
| C5—C6—C7—C71  | -16.2 (8)   | F11A—C122—C123—C124 | 178.6 (9)  |
| C6—N21—C3—C2  | 168.1 (5)   | F11A—C122—C123—C124 | 0.9 (12)  |
| C6—N21—C3—C4  | -6.1 (6)    | F12A—C123—C124—F13A | -177.8 (9) |
| C6—C7—C8—N22  | -24.6 (8)   | F12A—C123—C124—C125 | 179.0 (8)  |
| C6—C7—C8—C9   | 157.9 (5)   | F13A—C124—C125—F14A | 1.8 (12)  |
| C6—C7—C71—C72 | -62.6 (7)   | F13A—C124—C125—C126 | 179.6 (9)  |
| C6—C7—C71—C76 | 121.4 (5)   | F14A—C125—C126—F15A | 1.5 (12)  |
| C7—C8—C9—Br3  | -14.0 (8)   | F14A—C125—C126—C121 | -177.6 (9) |
| C7—C8—C9—C10  | 174.6 (5)   | O25—C26—C27—C28 | -32.3 (7)  |
| C7—C71—C72—F6  | 4.6 (7)     | C26—O25—C29—C28 | 3.1 (10)   |
| C7—C71—C72—C73 | -173.4 (5)  | C26—C27—C28—C29 | 33.1 (8)   |
| C7—C71—C76—F10 | -5.2 (7)    | C27—C28—C29—O25 | -23.0 (10) |
| C7—C71—C76—C75 | 174.6 (5)   | C29—O25—C26—C27 | 18.4 (8)   |
| C8—N22—C11—C10 | -2.7 (6)    | C21A—C2—C3—N21 | -161.3 (5) |
| C8—N22—C11—C12 | 175.5 (5)   | C21A—C2—C3—C4 | 11.7 (8)   |
| C8—C7—C71—C72 | 110.8 (5)   | C21A—C22A—C23A—F2A | -179.5 (10) |
| C8—C7—C71—C76 | -65.1 (6)   | C21A—C22A—C23A—C24A | 0.6 (17)  |
| C8—C9—C10—Br4  | 175.0 (4)   | C22A—C21A—C26A—F5A | -177.8 (12) |
| C8—C9—C10—C11 | 1.7 (6)     | C22A—C21A—C26A—C25A | -0.3 (18)  |
| C9—C10—C11—N22 | 0.5 (6)     | C22A—C23A—C24A—F3A | 179.6 (10) |
| C9—C10—C11—C12 | -177.5 (5)  | C22A—C23A—C24A—C25A | -0.6 (15)  |
| C10—C11—C12—C12A | 20.4 (8)   | C23A—C24A—C25A—F4A | 177.7 (10) |
| C10—C11—C12—C13 | -155.8 (5)  | C23A—C24A—C25A—C26A | 0.2 (16)   |
| C10—C11—C12—C121 | 20.4 (8)   | C24A—C25A—C26A—F5A | 177.8 (11) |
| C11—N22—C8—C7   | -174.3 (5)  | C24A—C25A—C26A—C21A | 0.3 (19)   |
| C11—N22—C8—C9   | 3.6 (5)     | C26A—C21A—C22A—F1A | -179.3 (12) |
| C11—C12—C12A—C12F | 63.7 (11)  | C26A—C21A—C22A—C23A | -0.2 (17)  |
| C11—C12—C12A—C12B | -114.7 (12) | C121—C12—C13—N23 | -154.4 (5) |
| C11—C12—C13—N23 | 21.8 (8)    | C121—C12—C13—C14 | 20.2 (8)   |
| C11—C12—C13—C14 | -163.6 (5)  | C121—C122—C123—F12A | -180.0 (9) |
| C11—C12—C121—C122 | -119.7 (7) | C121—C122—C123—C124 | 0.2 (11)   |
| C11—C12—C121—C126 | 60.7 (9)   | C122—C121—C126—F15A | -179.5 (9) |
| C12—C12A—C12F—F15B | 8.3 (3)    | C122—C121—C126—C125 | 1.8 (11)   |
| C12—C12A—C12F—C12E | -178.5 (7) | C122—C123—C124—F13A | -179.3 (8) |
| C12—C12A—C12B—F11B | -2.3 (3)   | C122—C123—C124—C125 | 2.0 (12)   |
| C12—C12A—C12B—C12C | 178.3 (8)  | C123—C124—C125—F14A | 177.6 (9)  |
| C12—C13—C14—Br5  | 19.3 (8)    | C123—C124—C125—C126 | -2.3 (12)  |
| C12—C13—C14—C15 | -172.1 (5)  | C124—C125—C126—F15A | -178.3 (9) |
Hydrogen-bond geometry (Å, °)

| D—H···A            | D—H  | H···A  | D···A  | D—H···A |
|--------------------|------|--------|--------|---------|
| N22—H22···O25      | 0.97 (1) | 1.92 (2) | 2.849 (6) | 158 (5) |

C12—C121—C122—F11A  0.0 (12)  C124—C125—C126—C121  0.3 (12)
C12—C121—C122—C123  178.3 (6)  C126—C121—C122—F11A  179.6 (9)
C12—C121—C126—F15A  0.0 (12)  C126—C121—C122—C123  −2.1 (11)
C12—C121—C126—C125  −178.6 (6)