# research communications

# Nitrosonium complexation by the tetraphosphonate cavitand 5,11,17,23-tetramethyl-6,10:12,16:-18,22:24,4-tetrakis(phenylphosphonato-$\kappa^2O,O$)-resorcin(4)arene

Roberta Pinalli and Chiara Massera*

Dipartimento di Scienze Chimiche, della Vita e della Sostenibilità Ambientale, Università di Parma, Parco Area delle Scienze 17/A, 43124 Parma, Italy. *Correspondence e-mail: chiara.massera@unipr.it

The crystal structure of a new supramolecular complex between the tetraphosphonate cavitand 5,11,17,23-tetramethyl-6,10:12,16:18,22:24,4-tetrakis(phenylphosphonato-$\kappa^2O,O'$)resorcin(4)arene and the nitrosyl cation $NO^+$, as the $BF_4^-$ salt, is reported. The complex, of general formula $[(C_{56}H_{44}P_4O_{12})(NO)]BF_4\cdot CH_2Cl_2$ or NO@Tiiii[H, $CH_3$, $C_6H_5$] $BF_4\cdot CH_2Cl_2$, crystallizes in the space group $P\bar{1}$. The nitrosyl cation is disordered over two equivalent positions, with occupancies of 0.503 (2) and 0.497 (2), and interacts with two adjacent P=O groups at the upper rim of the cavitand through dipole–charge interactions. In the lattice, the cavitands are connected through a series of C—H···π interactions involving the methyl and methylenic H atoms and the aromatic rings of the macrocycle. The structure is further stabilized by the presence of C—H···F interactions between the hydrogen atoms of the cavitands and the F atoms of the tetrafluoridoborate anion. As a result of the disorder, the lattice dichloromethane molecules could not be modelled in terms of atomic sites, and were treated using the *PLATON* SQUEEZE procedure [Spek (2015). *Acta Cryst.* C**71**, 9–18]. The complexation process has also been studied in solution through NMR titrations.

## 1. Chemical context

Cavitands (Cram, 1983; Cram & Cram, 1994) are synthetic organic compounds endowed with a rigid, pre-organized cavity that have been used extensively both in solution (Hooley & Rebek, 2009; Pochorovski *et al.*, 2012) and in the solid state (Riboni *et al.*, 2016) as molecular receptors for neutral molecules and cationic species (Pinalli & Dalcanale, 2013). This versatility stems from the possibility of decorating both the upper and the lower rim of the resorcinarene skeleton with desired functionalities.

In our group, we have been particularly interested in tetraphosphonate cavitands of the general formula Tiiii[$R$, $R_1$, $R_2$] ($R$ = lower rim substituents; $R_1$ = upper rim substituents; $R_2$ = substituents on the P atom) in which the upper rim of the macrocycle is functionalized with four P=O groups, all pointing inwards towards the cavity (Pinalli & Dalcanale, 2013). In this way, the π basicity of the cavity, useful for C—H···π recognition, is enriched with dipolar groups that can act both as hydrogen-bond acceptors and interact with cationic species through cation–dipole interactions.

The nitrosonium ion and its salts have been studied in the past to investigate similarities and differences with the $O_2^+$ ion in terms of size, ionization potential, electron affinity, oxida-

**1801**
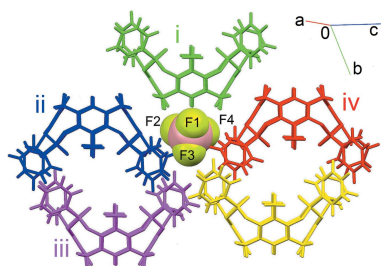
tion power *etc* (Mazej *et al.*, 2009). Moreover, the NO$^+$ cation can be used as a model for nitrogen oxides in molecular recognition phenomena. Indeed, the formation of stable, host–guest complexes between NO$^+$ cations and organic molecular receptors has been studied in solution with resorcinarenes (Botta *et al.*, 2007) or with calixarenes, both in solution (Zyryanov *et al.*, 2002, 2003) and in the solid state (Rathore *et al.*, 2000). In particular, nitrosonium hexachloroantimonate was shown to form an inclusion compound with tetramethoxy- and tetra-*n*-propoxycalix(4)arenes due to the interaction between the positive charge of the guest and the electron-rich aromatic cavity of the host (Rathore *et al.*, 2000). Inspired by this work, we decided to carry out a combined solution and solid-state study of the complexation properties of the rigid tetraphosphonate cavitand 5,11,17,23-tetramethyl-6,10:12,16:18,22:24,4-tetrakis(phenylphosphonato-*O,O′*)resorcin(4)arene (from now on indicated as Tiiii[H, CH$_3$, C$_6$H$_5$]) towards NOBF$_4$.
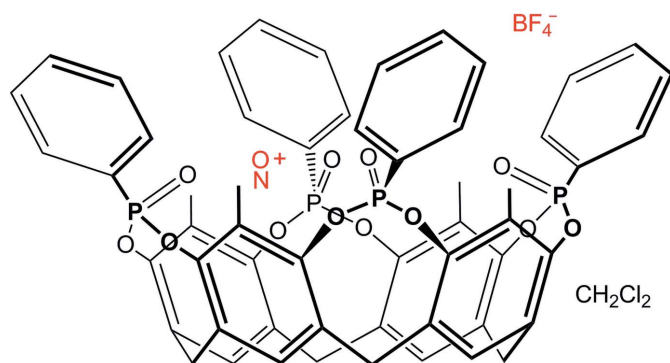


## 2. Studies in solution

Preliminary $^{31}$P and $^1$H NMR studies were performed to probe the complexation properties of the cavitand towards the nitrosonium ion in solution. To this purpose, we synthesized the cavitand Tiiii[C$_3$H$_7$, CH$_3$, C$_6$H$_5$], functionalized at the lower rim with four –C$_3$H$_7$ alkyl chains to enhance the cavitand solubility. The NMR tube was filled with 0.5 ml of a CDCl$_3$ solution containing the cavitand (1 mmol concentration). The NOBF$_4$ titrant solution was prepared by dissolving the guest in 0.4 ml (10 mmol) of the above-mentioned cavitand solution to keep the concentration of the host constant during the titration. Portions (0.25 eq., 22.5 µL) of the titrant were added by syringe to the NMR tube. During the titration, the phosphorous singlet of the cavitand shifted slightly downfield, from 6.01 (signal for the free host) to 7.42 ppm upon addition of an excess (2.5 eq.) of the guest (see Fig. S1 in the *Supporting information*), indicating the presence of cation–dipole interactions between the nitrosonium ion and the phosphonate groups at the upper rim. The broadening of the signal is due to the fast exchange (at the NMR time scale) of the guest inside the cavity.

In Fig. 1, the comparison between the $^1$H spectra recorded after each guest addition is reported. As can be seen, the

protons of the methyl group in the apical position of the cavitand skeleton (purple dot) are shifted up-field, increasing the guest concentration; this means that the presence of the NO$^+$ cation in proximity to the cavitand upper rim creates a change in the environment, which results in an overall shielding effect. On the contrary, the signals of the protons at the lower rim, namely the aromatic hydrogens (light-blue dot), the bridging methines (green dot) and the alkyl methylenic groups (red dot), are shifted downfield. This is due to the perturbation created by the BF$_4^-$ anion, which is likely positioned among the alkyl feet of the cavitand, as already observed for counter-anions in other crystal structures previously reported (Pinalli *et al.*, 2016). Also in this case, broadening of the signals was observed.

Following these results, solid-state studies were carried out to obtain an insight into the type, number, strength and geometry of the weak interactions taking place in the system.

## 3. Structural commentary

The molecular structure of NO@Tiiii[H, CH$_3$, C$_6$H$_5$]BF$_4$·CH$_2$Cl$_2$ is reported in Fig. 2. The complex crystallizes in the space group $P\bar{1}$, and the asymmetric unit comprises one cavitand, one molecule of NOBF$_4$ (with the cation disordered over two equivalent positions) and one disordered molecule of dichloromethane. The NO$^+$BF$_4^-$ ionic pair is separated, and the nitrosonium ion is located within the macrocycle, not deep inside the cavity, but lying in the mean plane passing through the four phosphonate oxygen atoms O3*A*, O3*B*, O3*C* and O3*D* (for detailed geometrical parameters, see Table 1). The nitrogen and oxygen atoms of the guest point towards the lower and the upper rims, respectively, and are held in place *via* cation–dipole interactions with two adjacent P=O groups. It is interesting to note that the NO$^+$ ion is disordered with 50% probability over two equivalent orientations [N1O1 with occupancy of 0.503 (2) and N2O2 with occupancy of 0.497 (2)], thus forming alternately an interaction with each of the two
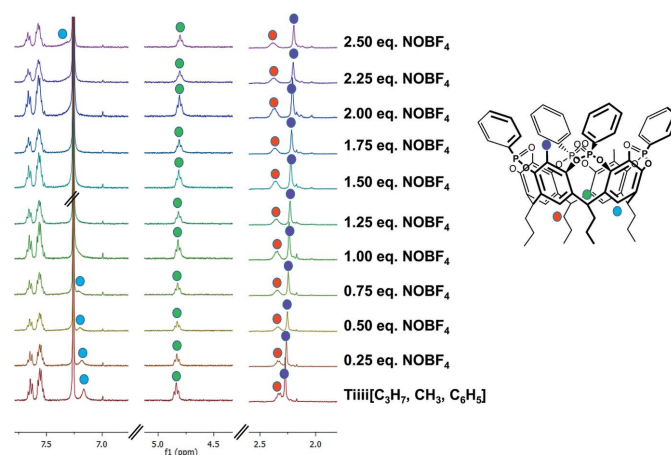


**Figure 1**
Selected portions of the $^1$H NMR (400 MHz, CDCl$_3$, 298 K) spectra recorded during the titration of the cavitand with increasing equivalents of NOBF$_4$.
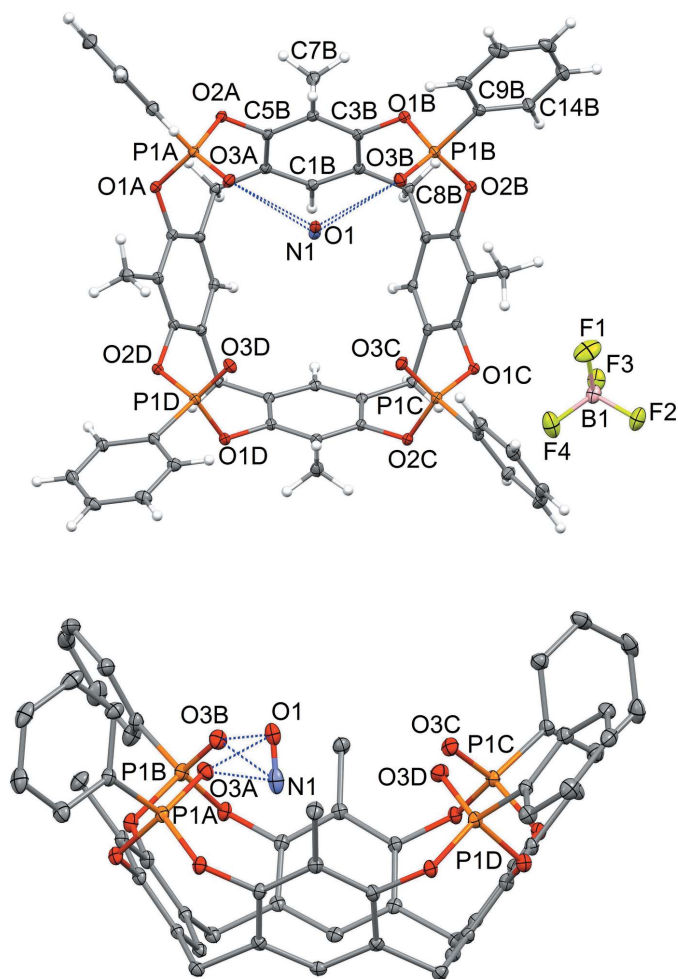
**Table 1**
Host–guest interactions (Å) in NO@Tiiii[H, CH₃, C₆H₅]BF₄.

| | | | |
|---|---|---|---|
| O3A···O1 | 2.621 (5) | O3D···O2 | 2.604 (4) |
| O3A···N1 | 2.661 (6) | O3D···N2 | 2.650 (4) |
| O3B···O1 | 2.609 (3) | O1···PL | 0.471 (4) |
| O3B···N1 | 2.664 (5) | N1···PL | 0.492 (6) |
| O3C···O2 | 2.621 (4) | O2···PL | 0.466 (4) |
| O3C···N2 | 2.625 (7) | N2···PL | 0.416 (6) |

PL is the mean plane passing through the four phosphonate oxygen atoms, O3A, O3B, O3C and O3D.

opposite $P{=}O$ groups (Fig. 2; the second orientation is not shown), namely $P1A{=}O3A$ and $P1B{=}O3B$ for N1O1 and $P1C{=}O3C$ and $P1D{=}O3D$ with N2O2 [O3A···O1, 2.621 (5); O3A···N1, 2.661 (6); O3B···O1, 2.609 (3); O3B···N1, 2.664 (5); O3C···O2, 2.621 (4); O3C···N2, 2.625 (7); O3D···O2, 2.604 (4); O3D···N2, 2.650 (4) Å]. This phenomenon has already been observed in the solid state with

**Table 2**
Hydrogen-bond geometry (Å, °).

Cg1, Cg2 and Cg3 are the centroids of the aromatic rings C9B–C14B, C9D–C14D and C1A–C6A, respectively.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| C1B[i]—H1B[i]···F1 | 0.95 | 2.41 | 3.344 (3) | 169 |
| C14B[ii]—H14B[ii]···F2 | 0.95 | 2.57 | 3.357 (3) | 140 |
| C7C[ii]—H7C3[ii]···F2 | 0.98 | 2.62 | 3.484 (2) | 147 |
| C8C[i]—H8C1[i]···F2 | 0.98 | 2.49 | 3.379 (3) | 150 |
| C1D[i]—H1D[i]···F2 | 0.95 | 2.60 | 3.439 (2) | 147 |
| C11A[iii]—H11A[iii]···F3 | 0.95 | 2.45 | 3.254 (2) | 142 |
| C7C[ii]—H7C3[ii]···F3 | 0.98 | 2.64 | 3.569 (3) | 160 |
| C11C—H11C···F4 | 0.95 | 2.53 | 3.447 (3) | 162 |
| C1D[i]—H1D[i]···F4 | 0.95 | 2.65 | 3.509 (3) | 150 |
| C14D[iv]—H14D[iv]···F4 | 0.95 | 2.63 | 3.336 (4) | 131 |
| C7D—H7D1···Cg1[v] | 0.98 | 2.80 | 3.524 (4) | 131 |
| C7B—H7B1···Cg2[vi] | 0.98 | 2.88 | 3.530 (4) | 124 |
| C8D—H8D2···Cg3[vii] | 0.98 | 2.87 | 3.594 (3) | 131 |

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

phosphonate cavitands hosting methanol and ethanol molecules (Melegari *et al.*, 2008) and confirms that, for these systems, the stability of the host–guest complex is entropic in origin, since the guest can choose from two up to four energetically and geometrically equivalent interaction modes with the host. In this case, the $NO^+$ cation forms two sets of strong interactions with two adjacent $P{=}O$ groups, which results in a better stabilizing effect than four weaker interactions with all the phosphonate moieties of the upper rim. The $BF_4^-$ ion is outside the cavity, forming weak $C-H\cdots F$ interactions with the cavitands (see *Section 4* for details).

The dichloromethane solvent molecule is heavily disordered and could not be modelled, but its residual electron density, occupying a void of 312 Å³ (Spek, 2015) is located in the hydrophobic pockets among the cavitands.

## 4. Supramolecular features

In the lattice, the cavitands form a supramolecular ribbon along the *a*-axis direction through a series of $C-H\cdots\pi$ interactions between the H atoms of the methyl groups at the upper rim and the phenyl rings of the phosphonato moieties. In particular, each cavitand interacts with two adjacent ones acting simultaneously as a donor to two methyl groups and as an acceptor to two aromatic rings (see Table 2 and Fig. 3; the centroids involved are Cg1 and Cg2, represented as red and green spheres, respectively). Moreover, pairs of centrosymmetric cavitands form another set of $C-H\cdots\pi$ interactions involving the methylenic hydrogen atoms at the lower rim and the aromatic walls of the macrocycle (see Table 2 and Fig. 3, Cg3, blue centroids). The structure is further stabilized by the presence of $C-H\cdots F$ interactions between the hydrogen atoms of the cavitands and the fluorine atoms of the tetrafluoridoborate anion. More precisely, each $BF_4^-$ is surrounded by five cavitands (Fig. 4), with $C-H\cdots F$ distances ranging from 2.408 (2) to 2.653 (2) Å (Table 2).



**Figure 2**
Top and side views of the title compound, NO@Tiiii[H, CH₃, C₆H₅], with a partial atom-labelling scheme. Displacement ellipsoids are drawn at the 20% probability level. Only one of the two disordered $NO^+$ ions is shown. In the side view, the hydrogen atoms and the $BF_4^-$ counter-ion are not shown for clarity. Cation–dipole interactions are represented as blue dashed lines.
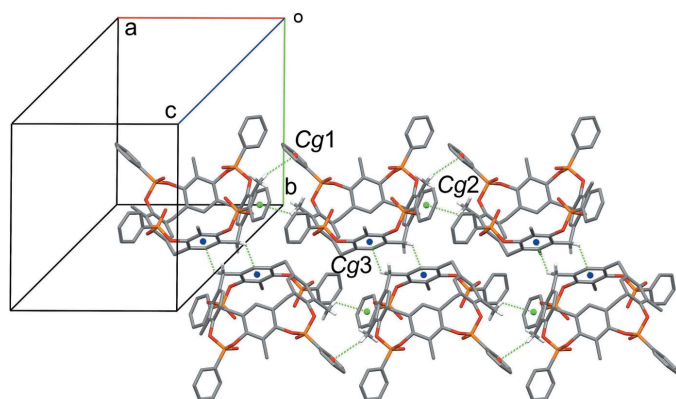
# research communications



**Figure 3**
C—H···π interactions (green dashed lines) forming a ribbon along the *a*-axis direction of the unit cell. Centroids *Cg*1 (C9B–C14B), *Cg*2 (C9D–C14D) and *Cg*3 (C1A–C6A) are represented as red, green and blue spheres, respectively.

## 5. Database survey

A search in the Cambridge Structural Database (Version 5.38, update May 2017; Groom *et al.*, 2016) for structures containing the isolated NO fragment, with no restrictions on the charge or on the type of bond connecting nitrogen and oxygen, yielded 65 species which are, of course, very different in nature. Meaningful comparisons with our complex are only possible with the series of calixarene-based, host–guest complexes already cited in the introduction, namely GOTCAT, GOTDEY, GOTGEB, GOTHAY and GOTHAY01 (Rathore *et al.*, 2000) and with a cationic radical calixarene derivative capable of binding neutral nitric oxide (JAHFOO; Rathore *et al.*, 2004). In particular, in GOTCAT, the NO$^+$ cation is buried deep inside the cavity, where it interacts with two distal aromatic groups of the calixarene guest. Since the calixarene is in the 1,3-alternate conformation, two sets of co-facial benzene rings are present, and the NO$^+$ ion is equally distributed between them (see Fig. 5, one pair of rings is shown
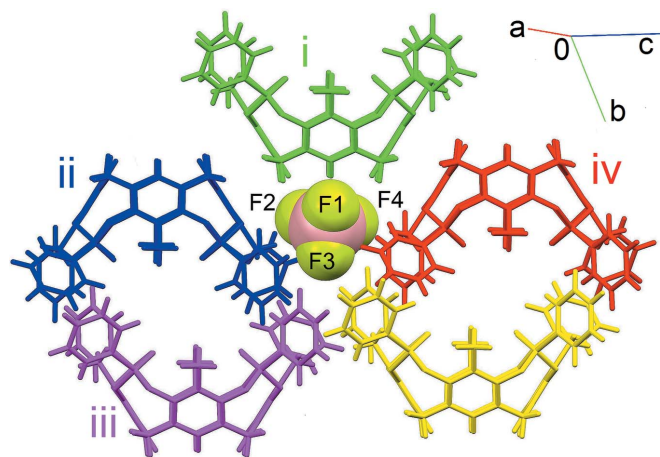
in space-filling model, the other one in capped-stick mode). The electron-rich pocket formed by the co-facial pair is essential for the complexation, and the NO$^+$ ion is not bound by a single aromatic ring alone (see, for instance, GOTDEY and GOTGEB). In the case of JAHFOO, the calixarene has been oxidized to carry an overall positive charge on its core, in order to make it a good receptor for an electron rich-guest such as nitric oxide. Nevertheless, the interaction mode is similar to that observed for GOTCAT, with two disordered NO molecules buried between two distinct pairs of distal aromatic rings (Fig. 5). Also, in the title complex the guest is disordered over two equivalent positions, but its interaction with the electron-rich cavity is negligible due to the presence of the dipolar phosphonate groups which 'hold' the NO$^+$ ion at the brim of the upper rim (Fig. 5).

## 6. Synthesis and crystallization

$^1$H NMR spectra were obtained using a Bruker AMX-400 (400 MHz) spectrometer. All chemical shifts ($\delta$) were reported in ppm relative to the proton resonances resulting from incomplete deuteration of the NMR solvents. $^{31}$P NMR spectra were obtained using a Bruker AMX-400 (162 MHz) spectrometer. All chemical shifts ($\delta$) were recorded in ppm relative to external 85% H$_3$PO$_4$ at 0.00 ppm. All commercial reagents were ACS reagent grade and used as received. The cavitands Tiiii[H, CH$_3$, C$_6$H$_5$] and Tiiii[C$_3$H$_7$, CH$_3$, C$_6$H$_5$] were prepared following published procedures (Tonezzer *et al.*, 2008; Menozzi *et al.*, 2015).

NO@Tiiii[H, CH$_3$, C$_6$H$_5$]BF$_4$·CH$_2$Cl$_2$ was obtained by mixing a dichloromethane solution of Tiiii[H, CH$_3$, C$_6$H$_5$]



**Figure 4**
View of the BF$_4^-$ ion surrounded by the five closest cavitands through C—H···F interactions. [Symmetry codes: (i) *x*, *y* − 1, *z*; (ii) −*x* + 1, −*y* + 1, −*z*; (iii) *x*, *y*, *z* − 1; (iv) −*x*, −*y* + 1, −*z* + 1.]
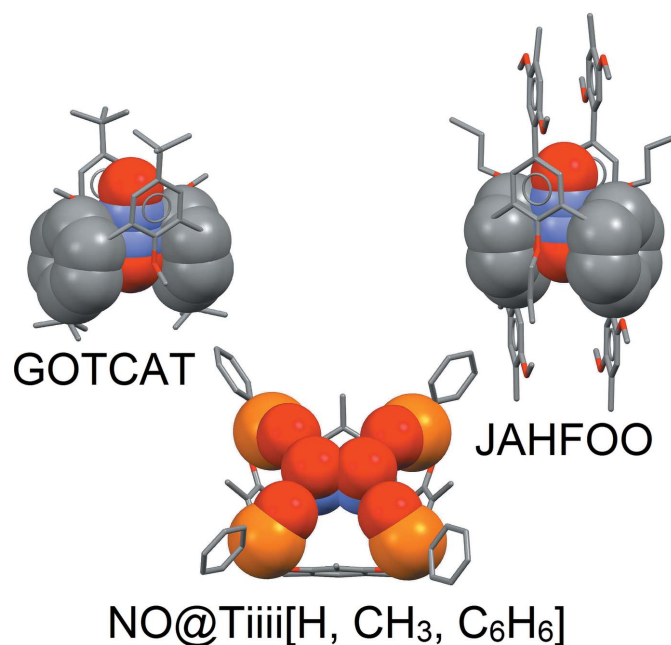


**Figure 5**
Comparison of the interaction modes of GOTCAT, JAHFOO (side view), and of the title compound, NO@Tiiii[H, CH$_3$, C$_6$H$_5$] (top view), highlighting the disorder of the guest over two equivalent positions. The space-filling view is only partial for reasons of clarity.

**Table 3**
Experimental details.

| | |
|---|---|
| **Crystal data** | |
| Chemical formula | $C_{56}H_{44}P_4O_{12}\cdot NO^+\cdot BF_4^-\cdot CH_2Cl_2$ |
| $M_r$ | 1234.54 |
| Crystal system, space group | Triclinic, $P\bar{1}$ |
| Temperature (K) | 190 |
| $a$, $b$, $c$ (Å) | 13.856 (1), 14.909 (2), 16.357 (2) |
| $\alpha$, $\beta$, $\gamma$ (°) | 63.224 (2), 73.137 (2), 88.093 (2) |
| $V$ (Å$^3$) | 2868.2 (6) |
| $Z$ | 2 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.30 |
| Crystal size (mm) | $0.16 \times 0.13 \times 0.10$ |
| | |
| **Data collection** | |
| Diffractometer | Bruker SMART BREEZE CCD area-detector |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2008) |
| $T_{min}$, $T_{max}$ | 0.812, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 36384, 14109, 9478 |
| $R_{int}$ | 0.033 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.690 |
| | |
| **Refinement** | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.042, 0.128, 1.00 |
| No. of reflections | 14109 |
| No. of parameters | 735 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.63, $-0.41$ |

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SIR97* (Altomare *et al.*, 1999), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008), *WinGX* (Farrugia, 2012), *PARST* (Nardelli, 1995) and *publCIF* (Westrip, 2010).

(1 eq.) with a dichloromethane solution of NOBF$_4$ (1 eq.). The mixture was left to evaporate to yield colourless single crystals of the 1:1 complex that were suitable for X-ray diffraction analysis.

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The nitrosonium ion was found to be disordered over two positions, with a refined occupancy ratio of 0.503 (2):0.497 (2). The C-bound H atoms were placed in calculated positions and refined using a riding model: C—H = 0.95-0.98 Å with $U_{iso}(H) = 1.5U_{eq}$(C-methyl) and $1.2U_{eq}$(C) for other H atoms.

As a result of severe disorder, the CH$_2$Cl$_2$ solvent could not be sensibly modelled in terms of atomic sites, and was treated using the *PLATON* SQUEEZE procedure (Spek, 2015); the solvent contribution to the diffraction pattern was removed and modified $F_o^2$ written to a new HKL file. The number of electrons corresponding to the solvent molecules were included in the formula, formula weight, calculated density, $\mu$ and $F(000)$.

## 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.

Botta, B., D'Acquarica, I., Delle Monache, G., Nevola, L., Tullo, D., Ugozzoli, F. & Pierini, M. (2007). *J. Am. Chem. Soc.* **129**, 11202–11212.

Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cram, D. J. (1983). *Science*, **219**, 1177–1183.

Cram, D. J. & Cram, J. M. (1994). *Container Molecules and their Guests, Monographs in Supramolecular Chemistry*, edited by J. F. Stoddart, vol. 4. Royal Society of Chemistry, Cambridge, UK.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Hooley, R. J. & Rebek, J. Jr (2009). *Chem. Biol.* **16**, 255–264.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Mazej, Z., Ponikvar-Svet, M., Liebman, J. F., Passmore, J. & Jenkins, H. D. B. (2009). *J. Fluor. Chem.* **130**, 788–791.

Melegari, M., Suman, M., Pirondini, L., Moiani, D., Massera, C., Ugozzoli, F., Kalenius, E., Vainiotalo, P., Mulatier, J.-C., Dutasta, J.-P. & Dalcanale, E. (2008). *Chem. Eur. J.* **14**, 5772–5779.

Menozzi, D., Pinalli, R., Massera, C., Maffei, F. & Dalcanale, E. (2015). *Molecules*, **20**, 4460–4472.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Pinalli, R., Brancatelli, G., Pedrini, A., Menozzi, D., Hernández, D., Ballester, P., Geremia, S. & Dalcanale, E. (2016). *J. Am. Chem. Soc.* **138**, 8569–8580.

Pinalli, R. & Dalcanale, E. (2013). *Acc. Chem. Res.* **46**, 399–411.

Pochorovski, I., Ebert, M.-O., Gisselbrecht, J.-P., Boudon, C., Schweizer, W. B. & Diederich, F. (2012). *J. Am. Chem. Soc.* **134**, 14702–14705.

Rathore, R., Abdelwahed, S. H. & Guzei, I. A. (2004). *J. Am. Chem. Soc.* **126**, 13582–13583.

Rathore, R., Lindeman, S. V., Rao, K. S. S. P., Sun, D. & Kochi, J. K. (2000). *Angew. Chem. Int. Ed.* **39**, 2123–2127.

Riboni, N., Trzcinski, J. W., Bianchi, F., Massera, C., Pinalli, R., Sidisky, L., Dalcanale, E. & Careri, M. (2016). *Anal. Chim. Acta*, **905**, 79–84.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2015). *Acta Cryst.* C**71**, 9–18.

Tonezzer, M., Melegari, M., Maggioni, G., Milan, R., Della Mea, G. & Dalcanale, E. (2008). *Chem. Mater.* **20**, 6535–6542.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Zyryanov, G. V., Kang, Y. & Rudkevich, D. M. (2003). *J. Am. Chem. Soc.* **125**, 2997–3007.

Zyryanov, G. V., Kang, Y., Stampp, S. P. & Rudkevich, D. M. (2002). *Chem. Commun.* pp. 2792–2793.

# Nitrosonium complexation by the tetraphosphonate cavitand 5,11,17,23-tetramethyl-6,10:12,16:18,22:24,4-tetrakis(phenylphosphonato-$\kappa^2O,O$)resorcin(4)arene

**Roberta Pinalli and Chiara Massera**

**Computing details**

Data collection: *APEX2* (Bruker, 2008); cell refinement: *APEX2* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012), *PARST* (Nardelli, 1995) and *publCIF* (Westrip, 2010).

**Nitrosonium tetrafluoridoborate–5,11,17,23-tetramethyl-6,10:12,16:18,22:24,4-tetrakis(phenylphosphonato-$\kappa^2O,O$)resorcin(4)arene–dichloromethane (1/1/1)**

*Crystal data*

| | |
|---|---|
| $C_{56}H_{44}P_4O_{12}{\cdot}NO^+{\cdot}BF_4^-{\cdot}CH_2Cl_2$ | $Z = 2$ |
| $M_r = 1234.54$ | $F(000) = 1268$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.429$ Mg m$^{-3}$ |
| $a = 13.856$ (1) Å | Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å |
| $b = 14.909$ (2) Å | Cell parameters from 250 reflections |
| $c = 16.357$ (2) Å | $\theta = 1.5–29.4°$ |
| $\alpha = 63.224$ (2)° | $\mu = 0.30$ mm$^{-1}$ |
| $\beta = 73.137$ (2)° | $T = 190$ K |
| $\gamma = 88.093$ (2)° | Prismatic, colourless |
| $V = 2868.2$ (6) Å$^3$ | $0.16 \times 0.13 \times 0.10$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART BREEZE CCD area-detector diffractometer | 36384 measured reflections |
| Radiation source: fine-focus sealed tube | 14109 independent reflections |
| Graphite monochromator | 9478 reflections with $I > 2\sigma(I)$ |
| $\omega$ scan | $R_{int} = 0.033$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2008) | $\theta_{max} = 29.4°$, $\theta_{min} = 1.5°$ |
| | $h = -18{\rightarrow}18$ |
| | $k = -20{\rightarrow}20$ |
| $T_{min} = 0.812$, $T_{max} = 1.000$ | $l = -22{\rightarrow}22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 14109 reflections |
| Least-squares matrix: full | 735 parameters |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | 0 restraints |
| $wR(F^2) = 0.128$ | Primary atom site location: structure-invariant direct methods |
| $S = 1.00$ | |

| | | |
|---|---|---|
| Secondary atom site location: difference Fourier map | $w = 1/[\sigma^2(F_o^2) + (0.0693P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ | |
| Hydrogen site location: inferred from neighbouring sites | $(\Delta/\sigma)_{max} = 0.001$ | |
| H-atom parameters constrained | $\Delta\rho_{max} = 0.63$ e Å$^{-3}$ | |
| | $\Delta\rho_{min} = -0.41$ e Å$^{-3}$ | |

*Special details*

**Experimental**. The calculated molar mass, density and absorption coefficient include two disordered dichloromethane molecules per cell which do not appear in the final files because of the refinements carried out with data subjected to SQUEEZE.

**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 ($Å^2$)*

| | *x* | *y* | *z* | $U_{iso}*/U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.3415 (3) | 0.7159 (4) | 0.4179 (3) | 0.0386 (10) | 0.503 (2) |
| O1 | 0.3391 (2) | 0.6435 (3) | 0.4543 (2) | 0.0307 (8) | 0.503 (2) |
| N2 | 0.2020 (3) | 0.7031 (4) | 0.3846 (3) | 0.0426 (11) | 0.497 (2) |
| O2 | 0.1903 (2) | 0.6363 (3) | 0.4165 (2) | 0.0266 (7) | 0.497 (2) |
| B1 | 0.2882 (2) | 0.2304 (2) | 0.12591 (19) | 0.0410 (6) | |
| F1 | 0.34181 (17) | 0.16872 (13) | 0.18534 (15) | 0.0875 (6) | |
| F2 | 0.28773 (14) | 0.19718 (12) | 0.05981 (11) | 0.0638 (4) | |
| F3 | 0.33203 (12) | 0.32872 (10) | 0.08112 (10) | 0.0553 (4) | |
| F4 | 0.19008 (14) | 0.22111 (13) | 0.18451 (14) | 0.0790 (6) | |
| P1A | 0.35319 (4) | 0.75850 (4) | 0.63016 (3) | 0.02411 (12) | |
| P1B | 0.60883 (4) | 0.73524 (4) | 0.25305 (4) | 0.02623 (12) | |
| P1C | 0.19872 (4) | 0.71498 (4) | 0.14576 (3) | 0.02352 (11) | |
| P1D | −0.05960 (4) | 0.73655 (4) | 0.52317 (4) | 0.02430 (12) | |
| O1A | 0.26652 (10) | 0.82864 (10) | 0.64092 (9) | 0.0264 (3) | |
| O2A | 0.45495 (10) | 0.83412 (10) | 0.56545 (9) | 0.0256 (3) | |
| O3A | 0.33168 (11) | 0.69279 (11) | 0.59071 (10) | 0.0315 (3) | |
| O1B | 0.64868 (10) | 0.81454 (11) | 0.27975 (10) | 0.0286 (3) | |
| O2B | 0.60145 (10) | 0.80000 (11) | 0.14780 (9) | 0.0275 (3) | |
| O3B | 0.51418 (10) | 0.67359 (11) | 0.32481 (10) | 0.0344 (3) | |
| O1C | 0.29952 (10) | 0.78374 (10) | 0.06637 (9) | 0.0250 (3) | |
| O2C | 0.11212 (10) | 0.78858 (10) | 0.13782 (9) | 0.0260 (3) | |
| O3C | 0.20873 (11) | 0.66232 (11) | 0.24296 (10) | 0.0310 (3) | |
| O1D | −0.08197 (10) | 0.80541 (10) | 0.42444 (9) | 0.0265 (3) | |
| O2D | −0.03700 (9) | 0.81178 (10) | 0.56143 (9) | 0.0251 (3) | |
| O3D | 0.02187 (10) | 0.67259 (11) | 0.51314 (10) | 0.0331 (3) | |
| C1A | 0.19513 (14) | 0.99115 (14) | 0.42444 (13) | 0.0239 (4) | |
| H1A | 0.2222 | 1.0501 | 0.3648 | 0.029* | |
| C2A | 0.25267 (14) | 0.95390 (14) | 0.48748 (13) | 0.0233 (4) | |
| C3A | 0.21105 (14) | 0.86730 (15) | 0.57387 (13) | 0.0239 (4) | |
| C4A | 0.11502 (14) | 0.81716 (14) | 0.60086 (14) | 0.0246 (4) | |
| C5A | 0.06242 (14) | 0.85866 (14) | 0.53393 (13) | 0.0235 (4) | |

| | | | |
|---|---|---|---|
| C6A | 0.09877 (14) | 0.94465 (14) | 0.44598 (13) | 0.0227 (4) |
| C7A | 0.07040 (16) | 0.72542 (16) | 0.69608 (15) | 0.0345 (5) |
| H7A1 | 0.1199 | 0.7067 | 0.7317 | 0.052* |
| H7A2 | 0.0534 | 0.6692 | 0.6853 | 0.052* |
| H7A3 | 0.0088 | 0.7405 | 0.7334 | 0.052* |
| C8A | 0.35871 (14) | 1.00412 (15) | 0.46023 (14) | 0.0253 (4) |
| H8A1 | 0.3721 | 0.9976 | 0.5188 | 0.030* |
| H8A2 | 0.3630 | 1.0771 | 0.4160 | 0.030* |
| C9A | 0.36897 (14) | 0.69707 (15) | 0.74569 (14) | 0.0265 (4) |
| C10A | 0.35124 (16) | 0.59170 (16) | 0.79585 (15) | 0.0338 (5) |
| H10A | 0.3299 | 0.5545 | 0.7686 | 0.041* |
| C11A | 0.36511 (18) | 0.54167 (19) | 0.88606 (16) | 0.0443 (6) |
| H11A | 0.3528 | 0.4700 | 0.9208 | 0.053* |
| C12A | 0.39638 (18) | 0.5952 (2) | 0.92518 (16) | 0.0463 (6) |
| H12A | 0.4063 | 0.5604 | 0.9866 | 0.056* |
| C13A | 0.41351 (19) | 0.6994 (2) | 0.87595 (17) | 0.0473 (6) |
| H13A | 0.4349 | 0.7360 | 0.9037 | 0.057* |
| C14A | 0.39949 (17) | 0.75105 (18) | 0.78587 (15) | 0.0378 (5) |
| H14A | 0.4108 | 0.8228 | 0.7522 | 0.045* |
| C1B | 0.46749 (14) | 0.99163 (14) | 0.31262 (13) | 0.0244 (4) |
| H1B | 0.4392 | 1.0488 | 0.2751 | 0.029* |
| C2B | 0.53674 (14) | 0.94606 (14) | 0.26635 (13) | 0.0241 (4) |
| C3B | 0.57720 (14) | 0.86308 (15) | 0.32371 (14) | 0.0250 (4) |
| C4B | 0.55347 (14) | 0.82460 (15) | 0.42299 (14) | 0.0263 (4) |
| C5B | 0.48240 (14) | 0.87296 (15) | 0.46416 (13) | 0.0246 (4) |
| C6B | 0.43862 (14) | 0.95615 (14) | 0.41178 (13) | 0.0232 (4) |
| C7B | 0.60064 (17) | 0.73656 (17) | 0.48175 (15) | 0.0365 (5) |
| H7B1 | 0.6048 | 0.7425 | 0.5379 | 0.055* |
| H7B2 | 0.6690 | 0.7360 | 0.4425 | 0.055* |
| H7B3 | 0.5590 | 0.6735 | 0.5033 | 0.055* |
| C8B | 0.56298 (15) | 0.98464 (15) | 0.15868 (13) | 0.0267 (4) |
| H8B1 | 0.6338 | 0.9735 | 0.1331 | 0.032* |
| H8B2 | 0.5584 | 1.0583 | 0.1275 | 0.032* |
| C9B | 0.71418 (15) | 0.66821 (16) | 0.23315 (15) | 0.0296 (4) |
| C10B | 0.72732 (17) | 0.58210 (17) | 0.31116 (18) | 0.0373 (5) |
| H10B | 0.6810 | 0.5605 | 0.3739 | 0.045* |
| C11B | 0.80909 (18) | 0.52802 (18) | 0.2962 (2) | 0.0459 (6) |
| H11B | 0.8188 | 0.4697 | 0.3489 | 0.055* |
| C12B | 0.87546 (18) | 0.5590 (2) | 0.2052 (2) | 0.0470 (6) |
| H12B | 0.9313 | 0.5223 | 0.1955 | 0.056* |
| C13B | 0.86170 (18) | 0.6421 (2) | 0.1286 (2) | 0.0497 (7) |
| H13B | 0.9075 | 0.6620 | 0.0659 | 0.060* |
| C14B | 0.78131 (17) | 0.6980 (2) | 0.14123 (17) | 0.0434 (6) |
| H14B | 0.7724 | 0.7560 | 0.0877 | 0.052* |
| C1C | 0.40529 (14) | 0.97320 (14) | 0.11292 (13) | 0.0241 (4) |
| H1C | 0.3904 | 1.0354 | 0.1140 | 0.029* |
| C2C | 0.33983 (14) | 0.92483 (14) | 0.09105 (12) | 0.0227 (4) |
| C3C | 0.36369 (14) | 0.83356 (14) | 0.09052 (13) | 0.0232 (4) |

| | | | |
|---|---|---|---|
| C4C | 0.44987 (14) | 0.78914 (15) | 0.10945 (13) | 0.0246 (4) |
| C5C | 0.51171 (14) | 0.84139 (15) | 0.13116 (13) | 0.0241 (4) |
| C6C | 0.49252 (14) | 0.93202 (14) | 0.13327 (13) | 0.0240 (4) |
| C7C | 0.47466 (16) | 0.69088 (16) | 0.10745 (16) | 0.0317 (5) |
| H7C1 | 0.4156 | 0.6587 | 0.1053 | 0.048* |
| H7C2 | 0.4923 | 0.6459 | 0.1655 | 0.048* |
| H7C3 | 0.5322 | 0.7041 | 0.0503 | 0.048* |
| C8C | 0.24356 (14) | 0.96932 (14) | 0.07192 (13) | 0.0241 (4) |
| H8C1 | 0.2544 | 1.0439 | 0.0441 | 0.029* |
| H8C2 | 0.2276 | 0.9529 | 0.0247 | 0.029* |
| C9C | 0.16854 (14) | 0.63680 (15) | 0.09894 (14) | 0.0251 (4) |
| C10C | 0.17014 (16) | 0.53322 (16) | 0.14934 (16) | 0.0331 (5) |
| H10C | 0.1867 | 0.5055 | 0.2078 | 0.040* |
| C11C | 0.14744 (17) | 0.47044 (18) | 0.11403 (19) | 0.0418 (6) |
| H11C | 0.1495 | 0.3996 | 0.1478 | 0.050* |
| C12C | 0.12199 (17) | 0.5105 (2) | 0.0301 (2) | 0.0457 (6) |
| H12C | 0.1054 | 0.4670 | 0.0067 | 0.055* |
| C13C | 0.12048 (19) | 0.6130 (2) | −0.02015 (19) | 0.0462 (6) |
| H13C | 0.1030 | 0.6400 | −0.0781 | 0.055* |
| C14C | 0.14423 (17) | 0.67743 (18) | 0.01321 (16) | 0.0368 (5) |
| H14C | 0.1439 | 0.7484 | −0.0220 | 0.044* |
| C1D | 0.13485 (14) | 0.97464 (14) | 0.22290 (13) | 0.0239 (4) |
| H1D | 0.1751 | 1.0356 | 0.2027 | 0.029* |
| C2D | 0.05730 (14) | 0.93516 (14) | 0.31036 (13) | 0.0231 (4) |
| C3D | −0.00030 (14) | 0.84668 (15) | 0.33686 (13) | 0.0242 (4) |
| C4D | 0.01450 (14) | 0.79593 (15) | 0.28118 (14) | 0.0255 (4) |
| C5D | 0.09407 (14) | 0.83894 (14) | 0.19564 (13) | 0.0232 (4) |
| C6D | 0.15514 (14) | 0.92744 (14) | 0.16438 (13) | 0.0228 (4) |
| C7D | −0.05138 (17) | 0.70142 (17) | 0.31159 (16) | 0.0365 (5) |
| H7D1 | −0.0530 | 0.6961 | 0.2544 | 0.055* |
| H7D2 | −0.1204 | 0.7041 | 0.3481 | 0.055* |
| H7D3 | −0.0237 | 0.6424 | 0.3521 | 0.055* |
| C8D | 0.03903 (14) | 0.98661 (14) | 0.37464 (13) | 0.0235 (4) |
| H8D1 | 0.0594 | 1.0602 | 0.3342 | 0.028* |
| H8D2 | −0.0343 | 0.9763 | 0.4101 | 0.028* |
| C9D | −0.18126 (14) | 0.67358 (15) | 0.60172 (14) | 0.0258 (4) |
| C10D | −0.21742 (16) | 0.59045 (16) | 0.59617 (15) | 0.0319 (5) |
| H10D | −0.1763 | 0.5680 | 0.5530 | 0.038* |
| C11D | −0.31361 (16) | 0.54133 (16) | 0.65406 (16) | 0.0367 (5) |
| H11D | −0.3388 | 0.4852 | 0.6505 | 0.044* |
| C12D | −0.37320 (16) | 0.57426 (17) | 0.71741 (16) | 0.0386 (5) |
| H12D | −0.4393 | 0.5406 | 0.7567 | 0.046* |
| C13D | −0.33707 (16) | 0.65558 (18) | 0.72374 (16) | 0.0388 (5) |
| H13D | −0.3781 | 0.6770 | 0.7678 | 0.047* |
| C14D | −0.24110 (16) | 0.70591 (16) | 0.66583 (15) | 0.0321 (5) |
| H14D | −0.2163 | 0.7620 | 0.6698 | 0.039* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.026 (2) | 0.068 (3) | 0.030 (2) | 0.002 (2) | −0.0062 (16) | −0.031 (2) |
| O1 | 0.0231 (15) | 0.0457 (19) | 0.0267 (16) | −0.0007 (14) | −0.0038 (12) | −0.0216 (15) |
| N2 | 0.025 (2) | 0.084 (4) | 0.036 (3) | 0.012 (2) | −0.0103 (18) | −0.041 (3) |
| O2 | 0.0266 (16) | 0.0343 (17) | 0.0239 (16) | 0.0087 (14) | −0.0083 (12) | −0.0176 (15) |
| B1 | 0.0572 (18) | 0.0266 (13) | 0.0331 (14) | 0.0055 (12) | −0.0109 (13) | −0.0106 (11) |
| F1 | 0.1356 (18) | 0.0472 (10) | 0.0887 (13) | 0.0254 (11) | −0.0712 (13) | −0.0180 (10) |
| F2 | 0.0914 (12) | 0.0594 (10) | 0.0434 (9) | −0.0021 (9) | −0.0133 (8) | −0.0296 (8) |
| F3 | 0.0762 (11) | 0.0290 (7) | 0.0436 (8) | −0.0007 (7) | −0.0064 (7) | −0.0092 (6) |
| F4 | 0.0738 (12) | 0.0646 (11) | 0.0825 (13) | −0.0121 (9) | 0.0162 (10) | −0.0432 (10) |
| P1A | 0.0246 (3) | 0.0281 (3) | 0.0190 (2) | −0.0001 (2) | −0.0069 (2) | −0.0100 (2) |
| P1B | 0.0213 (2) | 0.0333 (3) | 0.0237 (3) | 0.0047 (2) | −0.0060 (2) | −0.0135 (2) |
| P1C | 0.0246 (3) | 0.0281 (3) | 0.0211 (2) | 0.0035 (2) | −0.0079 (2) | −0.0136 (2) |
| P1D | 0.0200 (2) | 0.0283 (3) | 0.0246 (3) | 0.00258 (19) | −0.0042 (2) | −0.0137 (2) |
| O1A | 0.0255 (7) | 0.0335 (8) | 0.0207 (7) | 0.0036 (6) | −0.0088 (6) | −0.0119 (6) |
| O2A | 0.0254 (7) | 0.0322 (7) | 0.0175 (6) | −0.0013 (6) | −0.0063 (5) | −0.0100 (6) |
| O3A | 0.0361 (8) | 0.0342 (8) | 0.0264 (7) | −0.0018 (6) | −0.0103 (6) | −0.0151 (6) |
| O1B | 0.0234 (7) | 0.0387 (8) | 0.0259 (7) | 0.0058 (6) | −0.0076 (6) | −0.0170 (6) |
| O2B | 0.0219 (7) | 0.0378 (8) | 0.0237 (7) | 0.0060 (6) | −0.0073 (6) | −0.0150 (6) |
| O3B | 0.0269 (8) | 0.0400 (9) | 0.0297 (8) | 0.0012 (6) | −0.0030 (6) | −0.0139 (7) |
| O1C | 0.0237 (7) | 0.0313 (7) | 0.0241 (7) | 0.0020 (6) | −0.0074 (5) | −0.0161 (6) |
| O2C | 0.0258 (7) | 0.0333 (7) | 0.0261 (7) | 0.0062 (6) | −0.0090 (6) | −0.0193 (6) |
| O3C | 0.0372 (8) | 0.0351 (8) | 0.0227 (7) | 0.0052 (6) | −0.0116 (6) | −0.0136 (6) |
| O1D | 0.0211 (7) | 0.0349 (8) | 0.0232 (7) | 0.0012 (6) | −0.0036 (5) | −0.0149 (6) |
| O2D | 0.0198 (6) | 0.0314 (7) | 0.0241 (7) | 0.0003 (5) | −0.0042 (5) | −0.0143 (6) |
| O3D | 0.0259 (7) | 0.0353 (8) | 0.0372 (8) | 0.0081 (6) | −0.0053 (6) | −0.0190 (7) |
| C1A | 0.0292 (10) | 0.0212 (9) | 0.0206 (9) | 0.0029 (8) | −0.0048 (8) | −0.0107 (8) |
| C2A | 0.0234 (9) | 0.0245 (10) | 0.0243 (10) | 0.0027 (7) | −0.0053 (8) | −0.0143 (8) |
| C3A | 0.0238 (10) | 0.0295 (10) | 0.0215 (9) | 0.0051 (8) | −0.0089 (8) | −0.0133 (8) |
| C4A | 0.0227 (9) | 0.0280 (10) | 0.0220 (9) | 0.0019 (8) | −0.0044 (8) | −0.0121 (8) |
| C5A | 0.0190 (9) | 0.0285 (10) | 0.0245 (10) | 0.0017 (7) | −0.0038 (7) | −0.0152 (8) |
| C6A | 0.0260 (10) | 0.0237 (9) | 0.0234 (9) | 0.0071 (8) | −0.0084 (8) | −0.0150 (8) |
| C7A | 0.0294 (11) | 0.0365 (12) | 0.0269 (11) | −0.0024 (9) | −0.0079 (9) | −0.0058 (9) |
| C8A | 0.0268 (10) | 0.0260 (10) | 0.0242 (10) | −0.0005 (8) | −0.0072 (8) | −0.0127 (8) |
| C9A | 0.0207 (9) | 0.0348 (11) | 0.0206 (9) | 0.0016 (8) | −0.0049 (8) | −0.0107 (8) |
| C10A | 0.0332 (11) | 0.0338 (11) | 0.0283 (11) | 0.0051 (9) | −0.0065 (9) | −0.0112 (9) |
| C11A | 0.0400 (13) | 0.0418 (13) | 0.0295 (12) | 0.0120 (11) | −0.0036 (10) | −0.0030 (10) |
| C12A | 0.0374 (13) | 0.0701 (18) | 0.0227 (11) | 0.0156 (12) | −0.0118 (10) | −0.0132 (12) |
| C13A | 0.0482 (15) | 0.0689 (18) | 0.0307 (12) | 0.0020 (13) | −0.0183 (11) | −0.0237 (13) |
| C14A | 0.0420 (13) | 0.0430 (13) | 0.0264 (11) | −0.0031 (10) | −0.0121 (10) | −0.0129 (10) |
| C1B | 0.0251 (10) | 0.0230 (9) | 0.0226 (9) | −0.0024 (7) | −0.0081 (8) | −0.0075 (8) |
| C2B | 0.0227 (9) | 0.0259 (10) | 0.0211 (9) | −0.0051 (7) | −0.0048 (8) | −0.0092 (8) |
| C3B | 0.0194 (9) | 0.0331 (11) | 0.0229 (10) | 0.0016 (8) | −0.0048 (8) | −0.0142 (8) |
| C4B | 0.0234 (10) | 0.0331 (11) | 0.0243 (10) | 0.0020 (8) | −0.0095 (8) | −0.0134 (9) |
| C5B | 0.0239 (9) | 0.0310 (10) | 0.0171 (9) | −0.0041 (8) | −0.0050 (7) | −0.0100 (8) |
| C6B | 0.0214 (9) | 0.0242 (10) | 0.0235 (9) | −0.0032 (7) | −0.0063 (8) | −0.0106 (8) |

| | | | | | |
|---|---|---|---|---|---|
| C7B | 0.0389 (12) | 0.0436 (13) | 0.0253 (11) | 0.0145 (10) | −0.0136 (9) | −0.0128 (10) |
| C8B | 0.0277 (10) | 0.0282 (10) | 0.0194 (9) | −0.0032 (8) | −0.0052 (8) | −0.0079 (8) |
| C9B | 0.0232 (10) | 0.0376 (11) | 0.0345 (11) | 0.0060 (8) | −0.0112 (9) | −0.0207 (10) |
| C10B | 0.0312 (11) | 0.0327 (12) | 0.0453 (13) | 0.0029 (9) | −0.0123 (10) | −0.0155 (10) |
| C11B | 0.0396 (13) | 0.0287 (12) | 0.0712 (18) | 0.0076 (10) | −0.0239 (13) | −0.0206 (12) |
| C12B | 0.0305 (12) | 0.0490 (15) | 0.081 (2) | 0.0129 (11) | −0.0206 (13) | −0.0453 (15) |
| C13B | 0.0350 (13) | 0.0731 (19) | 0.0529 (16) | 0.0167 (13) | −0.0103 (12) | −0.0414 (15) |
| C14B | 0.0358 (13) | 0.0615 (16) | 0.0351 (12) | 0.0169 (11) | −0.0117 (10) | −0.0243 (12) |
| C1C | 0.0290 (10) | 0.0238 (9) | 0.0155 (9) | 0.0007 (8) | −0.0039 (8) | −0.0074 (7) |
| C2C | 0.0221 (9) | 0.0264 (10) | 0.0136 (8) | 0.0009 (7) | −0.0020 (7) | −0.0064 (7) |
| C3C | 0.0231 (9) | 0.0285 (10) | 0.0174 (9) | −0.0005 (8) | −0.0053 (7) | −0.0104 (8) |
| C4C | 0.0244 (10) | 0.0279 (10) | 0.0185 (9) | 0.0014 (8) | −0.0032 (7) | −0.0102 (8) |
| C5C | 0.0200 (9) | 0.0311 (10) | 0.0178 (9) | 0.0029 (8) | −0.0045 (7) | −0.0091 (8) |
| C6C | 0.0254 (10) | 0.0269 (10) | 0.0140 (8) | −0.0035 (8) | −0.0029 (7) | −0.0061 (8) |
| C7C | 0.0295 (11) | 0.0348 (11) | 0.0355 (11) | 0.0076 (9) | −0.0103 (9) | −0.0201 (10) |
| C8C | 0.0261 (10) | 0.0251 (10) | 0.0188 (9) | 0.0033 (8) | −0.0062 (8) | −0.0086 (8) |
| C9C | 0.0220 (9) | 0.0308 (10) | 0.0269 (10) | 0.0032 (8) | −0.0063 (8) | −0.0179 (9) |
| C10C | 0.0302 (11) | 0.0330 (11) | 0.0360 (12) | 0.0052 (9) | −0.0082 (9) | −0.0172 (10) |
| C11C | 0.0346 (12) | 0.0349 (12) | 0.0553 (15) | 0.0012 (10) | −0.0030 (11) | −0.0266 (12) |
| C12C | 0.0331 (12) | 0.0603 (17) | 0.0659 (17) | 0.0036 (11) | −0.0112 (12) | −0.0499 (15) |
| C13C | 0.0460 (14) | 0.0673 (18) | 0.0474 (14) | 0.0100 (12) | −0.0237 (12) | −0.0396 (14) |
| C14C | 0.0420 (13) | 0.0415 (13) | 0.0356 (12) | 0.0095 (10) | −0.0180 (10) | −0.0218 (10) |
| C1D | 0.0249 (10) | 0.0225 (9) | 0.0236 (10) | 0.0046 (7) | −0.0092 (8) | −0.0092 (8) |
| C2D | 0.0243 (9) | 0.0256 (10) | 0.0231 (9) | 0.0078 (8) | −0.0102 (8) | −0.0129 (8) |
| C3D | 0.0200 (9) | 0.0319 (10) | 0.0204 (9) | 0.0037 (8) | −0.0050 (7) | −0.0125 (8) |
| C4D | 0.0227 (9) | 0.0314 (10) | 0.0262 (10) | 0.0034 (8) | −0.0081 (8) | −0.0161 (9) |
| C5D | 0.0235 (9) | 0.0302 (10) | 0.0229 (9) | 0.0078 (8) | −0.0093 (8) | −0.0171 (8) |
| C6D | 0.0227 (9) | 0.0258 (10) | 0.0201 (9) | 0.0067 (7) | −0.0089 (7) | −0.0096 (8) |
| C7D | 0.0336 (12) | 0.0433 (13) | 0.0365 (12) | −0.0068 (10) | −0.0026 (10) | −0.0258 (11) |
| C8D | 0.0250 (10) | 0.0243 (9) | 0.0232 (9) | 0.0067 (8) | −0.0077 (8) | −0.0126 (8) |
| C9D | 0.0212 (9) | 0.0282 (10) | 0.0238 (10) | 0.0027 (8) | −0.0066 (8) | −0.0088 (8) |
| C10D | 0.0305 (11) | 0.0316 (11) | 0.0324 (11) | 0.0034 (9) | −0.0093 (9) | −0.0139 (9) |
| C11D | 0.0326 (12) | 0.0286 (11) | 0.0417 (13) | −0.0010 (9) | −0.0120 (10) | −0.0095 (10) |
| C12D | 0.0242 (11) | 0.0371 (12) | 0.0360 (12) | 0.0006 (9) | −0.0053 (9) | −0.0035 (10) |
| C13D | 0.0289 (11) | 0.0463 (14) | 0.0326 (12) | 0.0053 (10) | −0.0007 (9) | −0.0165 (11) |
| C14D | 0.0289 (11) | 0.0363 (12) | 0.0296 (11) | 0.0034 (9) | −0.0048 (9) | −0.0163 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—O1 | 0.966 (5) | C7B—H7B2 | 0.9800 |
| N2—O2 | 0.885 (5) | C7B—H7B3 | 0.9800 |
| B1—F3 | 1.374 (3) | C8B—C6C | 1.524 (3) |
| B1—F2 | 1.379 (3) | C8B—H8B1 | 0.9900 |
| B1—F1 | 1.382 (3) | C8B—H8B2 | 0.9900 |
| B1—F4 | 1.387 (3) | C9B—C14B | 1.392 (3) |
| P1A—O3A | 1.4716 (14) | C9B—C10B | 1.397 (3) |
| P1A—O1A | 1.5894 (14) | C10B—C11B | 1.397 (3) |
| P1A—O2A | 1.5950 (14) | C10B—H10B | 0.9500 |

| | | | |
|---|---|---|---|
| P1A—C9A | 1.768 (2) | C11B—C12B | 1.374 (4) |
| P1B—O3B | 1.4688 (15) | C11B—H11B | 0.9500 |
| P1B—O2B | 1.5825 (14) | C12B—C13B | 1.366 (4) |
| P1B—O1B | 1.5943 (15) | C12B—H12B | 0.9500 |
| P1B—C9B | 1.776 (2) | C13B—C14B | 1.392 (3) |
| P1C—O3C | 1.4695 (14) | C13B—H13B | 0.9500 |
| P1C—O1C | 1.5911 (14) | C14B—H14B | 0.9500 |
| P1C—O2C | 1.5921 (14) | C1C—C2C | 1.391 (3) |
| P1C—C9C | 1.7727 (19) | C1C—C6C | 1.396 (3) |
| P1D—O3D | 1.4738 (14) | C1C—H1C | 0.9500 |
| P1D—O2D | 1.5867 (14) | C2C—C3C | 1.393 (3) |
| P1D—O1D | 1.5916 (14) | C2C—C8C | 1.521 (3) |
| P1D—C9D | 1.7708 (19) | C3C—C4C | 1.392 (3) |
| O1A—C3A | 1.420 (2) | C4C—C5C | 1.392 (3) |
| O2A—C5B | 1.418 (2) | C4C—C7C | 1.506 (3) |
| O1B—C3B | 1.425 (2) | C5C—C6C | 1.382 (3) |
| O2B—C5C | 1.412 (2) | C7C—H7C1 | 0.9800 |
| O1C—C3C | 1.418 (2) | C7C—H7C2 | 0.9800 |
| O2C—C5D | 1.418 (2) | C7C—H7C3 | 0.9800 |
| O1D—C3D | 1.422 (2) | C8C—C6D | 1.518 (3) |
| O2D—C5A | 1.418 (2) | C8C—H8C1 | 0.9900 |
| C1A—C2A | 1.392 (3) | C8C—H8C2 | 0.9900 |
| C1A—C6A | 1.399 (3) | C9C—C10C | 1.388 (3) |
| C1A—H1A | 0.9500 | C9C—C14C | 1.394 (3) |
| C2A—C3A | 1.389 (3) | C10C—C11C | 1.386 (3) |
| C2A—C8A | 1.520 (3) | C10C—H10C | 0.9500 |
| C3A—C4A | 1.397 (3) | C11C—C12C | 1.377 (4) |
| C4A—C5A | 1.391 (3) | C11C—H11C | 0.9500 |
| C4A—C7A | 1.502 (3) | C12C—C13C | 1.374 (4) |
| C5A—C6A | 1.388 (3) | C12C—H12C | 0.9500 |
| C6A—C8D | 1.519 (3) | C13C—C14C | 1.387 (3) |
| C7A—H7A1 | 0.9800 | C13C—H13C | 0.9500 |
| C7A—H7A2 | 0.9800 | C14C—H14C | 0.9500 |
| C7A—H7A3 | 0.9800 | C1D—C6D | 1.389 (3) |
| C8A—C6B | 1.521 (3) | C1D—C2D | 1.393 (3) |
| C8A—H8A1 | 0.9900 | C1D—H1D | 0.9500 |
| C8A—H8A2 | 0.9900 | C2D—C3D | 1.385 (3) |
| C9A—C14A | 1.385 (3) | C2D—C8D | 1.522 (3) |
| C9A—C10A | 1.396 (3) | C3D—C4D | 1.395 (3) |
| C10A—C11A | 1.391 (3) | C4D—C5D | 1.389 (3) |
| C10A—H10A | 0.9500 | C4D—C7D | 1.502 (3) |
| C11A—C12A | 1.370 (4) | C5D—C6D | 1.391 (3) |
| C11A—H11A | 0.9500 | C7D—H7D1 | 0.9800 |
| C12A—C13A | 1.380 (4) | C7D—H7D2 | 0.9800 |
| C12A—H12A | 0.9500 | C7D—H7D3 | 0.9800 |
| C13A—C14A | 1.391 (3) | C8D—H8D1 | 0.9900 |
| C13A—H13A | 0.9500 | C8D—H8D2 | 0.9900 |
| C14A—H14A | 0.9500 | C9D—C14D | 1.394 (3) |

| | | | |
|---|---|---|---|
| C1B—C6B | 1.391 (3) | C9D—C10D | 1.402 (3) |
| C1B—C2B | 1.398 (3) | C10D—C11D | 1.385 (3) |
| C1B—H1B | 0.9500 | C10D—H10D | 0.9500 |
| C2B—C3B | 1.392 (3) | C11D—C12D | 1.390 (3) |
| C2B—C8B | 1.515 (3) | C11D—H11D | 0.9500 |
| C3B—C4B | 1.392 (3) | C12D—C13D | 1.384 (3) |
| C4B—C5B | 1.396 (3) | C12D—H12D | 0.9500 |
| C4B—C7B | 1.498 (3) | C13D—C14D | 1.387 (3) |
| C5B—C6B | 1.391 (3) | C13D—H13D | 0.9500 |
| C7B—H7B1 | 0.9800 | C14D—H14D | 0.9500 |
| | | | |
| F3—B1—F2 | 111.3 (2) | H8B1—C8B—H8B2 | 107.9 |
| F3—B1—F1 | 109.5 (2) | C14B—C9B—C10B | 119.8 (2) |
| F2—B1—F1 | 108.9 (2) | C14B—C9B—P1B | 121.19 (17) |
| F3—B1—F4 | 110.4 (2) | C10B—C9B—P1B | 118.96 (16) |
| F2—B1—F4 | 110.1 (2) | C11B—C10B—C9B | 119.5 (2) |
| F1—B1—F4 | 106.6 (2) | C11B—C10B—H10B | 120.3 |
| O3A—P1A—O1A | 113.25 (8) | C9B—C10B—H10B | 120.3 |
| O3A—P1A—O2A | 113.39 (8) | C12B—C11B—C10B | 120.1 (2) |
| O1A—P1A—O2A | 105.21 (7) | C12B—C11B—H11B | 119.9 |
| O3A—P1A—C9A | 116.42 (9) | C10B—C11B—H11B | 119.9 |
| O1A—P1A—C9A | 103.86 (8) | C13B—C12B—C11B | 120.5 (2) |
| O2A—P1A—C9A | 103.48 (8) | C13B—C12B—H12B | 119.7 |
| O3B—P1B—O2B | 114.65 (8) | C11B—C12B—H12B | 119.7 |
| O3B—P1B—O1B | 112.61 (8) | C12B—C13B—C14B | 120.8 (2) |
| O2B—P1B—O1B | 105.36 (8) | C12B—C13B—H13B | 119.6 |
| O3B—P1B—C9B | 116.20 (10) | C14B—C13B—H13B | 119.6 |
| O2B—P1B—C9B | 102.10 (9) | C9B—C14B—C13B | 119.3 (2) |
| O1B—P1B—C9B | 104.63 (8) | C9B—C14B—H14B | 120.3 |
| O3C—P1C—O1C | 113.22 (8) | C13B—C14B—H14B | 120.3 |
| O3C—P1C—O2C | 113.28 (8) | C2C—C1C—C6C | 121.17 (18) |
| O1C—P1C—O2C | 105.45 (7) | C2C—C1C—H1C | 119.4 |
| O3C—P1C—C9C | 116.01 (9) | C6C—C1C—H1C | 119.4 |
| O1C—P1C—C9C | 103.90 (8) | C1C—C2C—C3C | 117.94 (17) |
| O2C—P1C—C9C | 103.83 (8) | C1C—C2C—C8C | 120.41 (17) |
| O3D—P1D—O2D | 113.92 (8) | C3C—C2C—C8C | 121.62 (17) |
| O3D—P1D—O1D | 112.87 (8) | C4C—C3C—C2C | 123.55 (17) |
| O2D—P1D—O1D | 105.77 (7) | C4C—C3C—O1C | 117.27 (16) |
| O3D—P1D—C9D | 116.93 (9) | C2C—C3C—O1C | 119.14 (16) |
| O2D—P1D—C9D | 103.46 (8) | C5C—C4C—C3C | 115.43 (17) |
| O1D—P1D—C9D | 102.56 (8) | C5C—C4C—C7C | 121.96 (18) |
| C3A—O1A—P1A | 120.78 (12) | C3C—C4C—C7C | 122.60 (18) |
| C5B—O2A—P1A | 118.31 (11) | C6C—C5C—C4C | 124.08 (18) |
| C3B—O1B—P1B | 119.32 (12) | C6C—C5C—O2B | 118.99 (17) |
| C5C—O2B—P1B | 121.91 (11) | C4C—C5C—O2B | 116.88 (17) |
| C3C—O1C—P1C | 120.00 (11) | C5C—C6C—C1C | 117.82 (17) |
| C5D—O2C—P1C | 118.16 (11) | C5C—C6C—C8B | 121.32 (18) |
| C3D—O1D—P1D | 119.51 (12) | C1C—C6C—C8B | 120.85 (17) |

| | | | |
|---|---|---|---|
| C5A—O2D—P1D | 121.38 (11) | C4C—C7C—H7C1 | 109.5 |
| C2A—C1A—C6A | 122.21 (17) | C4C—C7C—H7C2 | 109.5 |
| C2A—C1A—H1A | 118.9 | H7C1—C7C—H7C2 | 109.5 |
| C6A—C1A—H1A | 118.9 | C4C—C7C—H7C3 | 109.5 |
| C3A—C2A—C1A | 117.45 (17) | H7C1—C7C—H7C3 | 109.5 |
| C3A—C2A—C8A | 121.68 (17) | H7C2—C7C—H7C3 | 109.5 |
| C1A—C2A—C8A | 120.82 (17) | C6D—C8C—C2C | 110.63 (15) |
| C2A—C3A—C4A | 123.67 (18) | C6D—C8C—H8C1 | 109.5 |
| C2A—C3A—O1A | 118.98 (16) | C2C—C8C—H8C1 | 109.5 |
| C4A—C3A—O1A | 117.30 (16) | C6D—C8C—H8C2 | 109.5 |
| C5A—C4A—C3A | 115.46 (17) | C2C—C8C—H8C2 | 109.5 |
| C5A—C4A—C7A | 121.91 (17) | H8C1—C8C—H8C2 | 108.1 |
| C3A—C4A—C7A | 122.63 (18) | C10C—C9C—C14C | 120.11 (19) |
| C6A—C5A—C4A | 124.41 (17) | C10C—C9C—P1C | 118.53 (16) |
| C6A—C5A—O2D | 118.99 (17) | C14C—C9C—P1C | 121.36 (16) |
| C4A—C5A—O2D | 116.51 (16) | C11C—C10C—C9C | 119.7 (2) |
| C5A—C6A—C1A | 116.79 (17) | C11C—C10C—H10C | 120.2 |
| C5A—C6A—C8D | 122.56 (17) | C9C—C10C—H10C | 120.2 |
| C1A—C6A—C8D | 120.65 (17) | C12C—C11C—C10C | 120.1 (2) |
| C4A—C7A—H7A1 | 109.5 | C12C—C11C—H11C | 119.9 |
| C4A—C7A—H7A2 | 109.5 | C10C—C11C—H11C | 119.9 |
| H7A1—C7A—H7A2 | 109.5 | C13C—C12C—C11C | 120.4 (2) |
| C4A—C7A—H7A3 | 109.5 | C13C—C12C—H12C | 119.8 |
| H7A1—C7A—H7A3 | 109.5 | C11C—C12C—H12C | 119.8 |
| H7A2—C7A—H7A3 | 109.5 | C12C—C13C—C14C | 120.5 (2) |
| C2A—C8A—C6B | 111.10 (15) | C12C—C13C—H13C | 119.8 |
| C2A—C8A—H8A1 | 109.4 | C14C—C13C—H13C | 119.8 |
| C6B—C8A—H8A1 | 109.4 | C13C—C14C—C9C | 119.2 (2) |
| C2A—C8A—H8A2 | 109.4 | C13C—C14C—H14C | 120.4 |
| C6B—C8A—H8A2 | 109.4 | C9C—C14C—H14C | 120.4 |
| H8A1—C8A—H8A2 | 108.0 | C6D—C1D—C2D | 121.92 (18) |
| C14A—C9A—C10A | 120.16 (19) | C6D—C1D—H1D | 119.0 |
| C14A—C9A—P1A | 121.45 (16) | C2D—C1D—H1D | 119.0 |
| C10A—C9A—P1A | 118.38 (16) | C3D—C2D—C1D | 117.33 (17) |
| C11A—C10A—C9A | 119.4 (2) | C3D—C2D—C8D | 121.87 (17) |
| C11A—C10A—H10A | 120.3 | C1D—C2D—C8D | 120.79 (17) |
| C9A—C10A—H10A | 120.3 | C2D—C3D—C4D | 124.00 (17) |
| C12A—C11A—C10A | 120.4 (2) | C2D—C3D—O1D | 119.04 (16) |
| C12A—C11A—H11A | 119.8 | C4D—C3D—O1D | 116.93 (17) |
| C10A—C11A—H11A | 119.8 | C5D—C4D—C3D | 115.41 (17) |
| C11A—C12A—C13A | 120.4 (2) | C5D—C4D—C7D | 122.33 (17) |
| C11A—C12A—H12A | 119.8 | C3D—C4D—C7D | 122.26 (18) |
| C13A—C12A—H12A | 119.8 | C4D—C5D—C6D | 123.81 (17) |
| C12A—C13A—C14A | 120.2 (2) | C4D—C5D—O2C | 116.92 (16) |
| C12A—C13A—H13A | 119.9 | C6D—C5D—O2C | 119.27 (16) |
| C14A—C13A—H13A | 119.9 | C1D—C6D—C5D | 117.52 (17) |
| C9A—C14A—C13A | 119.5 (2) | C1D—C6D—C8C | 120.72 (17) |
| C9A—C14A—H14A | 120.3 | C5D—C6D—C8C | 121.72 (17) |

| | | | |
|---|---|---|---|
| C13A—C14A—H14A | 120.3 | C4D—C7D—H7D1 | 109.5 |
| C6B—C1B—C2B | 122.22 (18) | C4D—C7D—H7D2 | 109.5 |
| C6B—C1B—H1B | 118.9 | H7D1—C7D—H7D2 | 109.5 |
| C2B—C1B—H1B | 118.9 | C4D—C7D—H7D3 | 109.5 |
| C3B—C2B—C1B | 117.18 (17) | H7D1—C7D—H7D3 | 109.5 |
| C3B—C2B—C8B | 122.31 (18) | H7D2—C7D—H7D3 | 109.5 |
| C1B—C2B—C8B | 120.48 (18) | C6A—C8D—C2D | 111.24 (15) |
| C4B—C3B—C2B | 123.83 (18) | C6A—C8D—H8D1 | 109.4 |
| C4B—C3B—O1B | 116.91 (17) | C2D—C8D—H8D1 | 109.4 |
| C2B—C3B—O1B | 119.24 (16) | C6A—C8D—H8D2 | 109.4 |
| C3B—C4B—C5B | 115.61 (18) | C2D—C8D—H8D2 | 109.4 |
| C3B—C4B—C7B | 122.11 (18) | H8D1—C8D—H8D2 | 108.0 |
| C5B—C4B—C7B | 122.28 (18) | C14D—C9D—C10D | 120.33 (18) |
| C6B—C5B—C4B | 123.92 (17) | C14D—C9D—P1D | 121.92 (16) |
| C6B—C5B—O2A | 119.10 (17) | C10D—C9D—P1D | 117.74 (15) |
| C4B—C5B—O2A | 116.97 (17) | C11D—C10D—C9D | 119.5 (2) |
| C5B—C6B—C1B | 117.21 (18) | C11D—C10D—H10D | 120.3 |
| C5B—C6B—C8A | 122.02 (17) | C9D—C10D—H10D | 120.3 |
| C1B—C6B—C8A | 120.71 (17) | C10D—C11D—C12D | 119.9 (2) |
| C4B—C7B—H7B1 | 109.5 | C10D—C11D—H11D | 120.0 |
| C4B—C7B—H7B2 | 109.5 | C12D—C11D—H11D | 120.0 |
| H7B1—C7B—H7B2 | 109.5 | C13D—C12D—C11D | 120.6 (2) |
| C4B—C7B—H7B3 | 109.5 | C13D—C12D—H12D | 119.7 |
| H7B1—C7B—H7B3 | 109.5 | C11D—C12D—H12D | 119.7 |
| H7B2—C7B—H7B3 | 109.5 | C12D—C13D—C14D | 120.1 (2) |
| C2B—C8B—C6C | 112.07 (15) | C12D—C13D—H13D | 119.9 |
| C2B—C8B—H8B1 | 109.2 | C14D—C13D—H13D | 119.9 |
| C6C—C8B—H8B1 | 109.2 | C13D—C14D—C9D | 119.5 (2) |
| C2B—C8B—H8B2 | 109.2 | C13D—C14D—H14D | 120.2 |
| C6C—C8B—H8B2 | 109.2 | C9D—C14D—H14D | 120.2 |

*Hydrogen-bond geometry (Å, º)*

$Cg1$, $Cg2$ and $Cg3$ are the centroids of the aromatic rings C9B–C14B, C9D–C14D and C1A–C6A, respectively.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C1B[i]—H1B[i]···F1 | 0.95 | 2.41 | 3.344 (3) | 169 |
| C14B[ii]—H14B[ii]···F2 | 0.95 | 2.57 | 3.357 (3) | 140 |
| C7C[ii]—H7C3[ii]···F2 | 0.98 | 2.62 | 3.484 (2) | 147 |
| C8C[i]—H8C1[i]···F2 | 0.98 | 2.49 | 3.379 (3) | 150 |
| C1D[i]—H1D[i]···F2 | 0.95 | 2.60 | 3.439 (2) | 147 |
| C11A[iii]—H11A[iii]···F3 | 0.95 | 2.45 | 3.254 (2) | 142 |
| C7C[ii]—H7C3[ii]···F3 | 0.98 | 2.64 | 3.569 (3) | 160 |
| C11C—H11C···F4 | 0.95 | 2.53 | 3.447 (3) | 162 |
| C1D[i]—H1D[i]···F4 | 0.95 | 2.65 | 3.509 (3) | 150 |
| C14D[iv]—H14D[iv]···F4 | 0.95 | 2.63 | 3.336 (4) | 131 |
| C7D—H7D1···Cg1[v] | 0.98 | 2.80 | 3.524 (4) | 131 |

| | | | | |
|---|---|---|---|---|
| C7*B*—H7*B*1···*Cg*2[vi] | 0.98 | 2.88 | 3.530 (4) | 124 |
| C8*D*—H8*D*2···*Cg*3[vii] | 0.98 | 2.87 | 3.594 (3) | 131 |

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