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# Isostructural behaviour in ammonium and potassium salt forms of sulfonated azo dyes

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**CCDC references:** 2331925; 2331924; 2331923; 2331922; 2331921; 2331920; 2331919; 2331918; 2331917

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The structures of five ammonium salt forms of monosulfonated azo dyes, derivatives of 4-(2-phenyldiazen-1-yl)benzenesulfonate, with the general formula $[NH_4][O_3S(C_6H_4)NN(C_6H_3)RR']\cdot XH_2O$ [$R$ = OH, $NH_2$ or $N(C_2H_4OH)_2$; $R'$ = H or OH] are presented. All form simple layered structures with alternating hydrophobic (organic) and hydrophilic (cation, solvent and polar groups) layers. To assess for isostructural behaviour of the ammonium cation with $M^+$ ions, the packing of these structures is compared with literature examples. To aid this comparison, the corresponding structures of four potassium salt forms of the monosulfonated azo dyes are also presented herein. Of the five ammonium salts it is found that three have isostructural equivalents. In two cases this equivalent is a potassium salt form and in one case it is a rubidium salt form. The isostructurality of ion packing and of unit-cell symmetry and dimensions tolerates cases where the ammonium ions form somewhat different interaction types with coformer species than do the potassium or rubidium ions. No sodium salt forms are found to be isostructural with any ammonium equivalent. However, similarities in the anion packing within a single hydrophobic layer are found for a group that consists of the ammonium and rubidium salt forms of one azo anion species and the sodium and silver salt forms of a different azo species.

## 1. Introduction

The process of salt selection aims to choose the form of an active organic material that has the best properties for effectiveness and for commercialization. Salt selection is well studied in the area of pharmaceuticals (Stahl & Wermuth, 2008; Mahmood *et al.*, 2023; Bharate, 2021; Arlin *et al.*, 2011; Black *et al.*, 2007), but it is perhaps less well known that similar processes are used to select for material properties in other areas too. One example is the process of laking sulfonated azo colourants. A typical process here involves the substitution of an $M^+$ cation with an $M^{2+}$ cation, such as Ca or Ba, to switch from an aqueous-soluble dyestuff to an insoluble pigment (Christie & Mackay, 2008; Schmidt *et al.*, 2009; Kennedy *et al.*, 2012). As with pharmaceuticals, the material properties of pigments are dependant upon their crystal structures, and upon the intermolecular interactions present within the crystal (Hao & Iqbal, 1997). However, in the field of sulfonated azo colourants relatively few pigment structures are known due to the insoluble nature of the materials and to the highly anisotropic habits of many species. This means that a high percentage of the crystal structures that are known are derived from less common methods than standard single-crystal X-ray diffraction (*e.g.* structure from powder diffraction, from electron diffraction or through use of synchrotron radiation – see Schmidt *et al.*, 2009; Gorelik *et al.*, 2009; Kennedy *et al.*, 2000; Grzesiak-Nowak *et al.*, 2019). One strategy for understanding

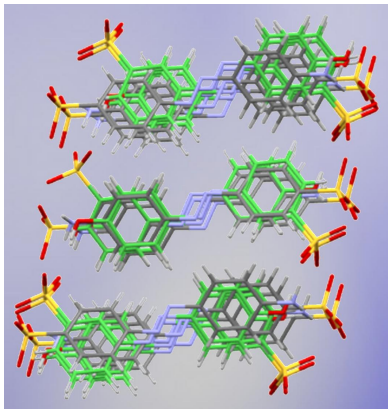


**Table 1**
Experimental details.

H atoms were treated by a mixture of independent and constrained refinement. The absorption correction was multi-scan for $NH_4$**1** (*CrysAlis PRO*; Rigaku OD, 2019) and K**1** (*SADABS*; Bruker, 2012).

| | $NH_4$**1** | $NH_4$**2** | $NH_4$**3** |
|---|---|---|---|
| Crystal data | | | |
| Chemical formula | $NH_4^+ \cdot C_{12}H_9N_2O_4S^-$ | $NH_4^+ \cdot C_{12}H_9N_2O_5S^- \cdot 2H_2O$ | $NH_4^+ \cdot C_{12}H_{10}N_3O_3S^- \cdot 1.5H_2O$ |
| $M_r$ | 295.31 | 347.34 | 321.35 |
| Crystal system, space group | Monoclinic, $P2_1$ | Triclinic, $P\overline{1}$ | Monoclinic, $C2/c$ |
| Temperature (K) | 100 | 123 | 123 |
| $a, b, c$ (Å) | 5.8163 (1), 6.9218 (1), 15.6295 (2) | 8.2876 (1), 10.6404 (1), 17.4834 (3) | 35.1636 (15), 7.8905 (3), 10.4972 (5) |
| $\alpha, \beta, \gamma$ (°) | 90, 93.354 (1), 90 | 89.734 (1), 84.793 (1), 86.146 (1) | 90, 100.091 (2), 90 |
| $V$ (Å$^3$) | 628.15 (2) | 1531.91 (4) | 2867.5 (2) |
| $Z$ | 2 | 4 | 8 |
| Radiation type | Cu $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 2.48 | 0.25 | 0.25 |
| Crystal size (mm) | 0.22 × 0.12 × 0.06 | 0.42 × 0.40 × 0.10 | 0.35 × 0.30 × 0.05 |
| Data collection | | | |
| Diffractometer | Rigaku Synergy-i | Enraf–Nonius KappaCCD | Enraf–Nonius KappaCCD |
| $T_{min}$, $T_{max}$ | 0.859, 1.000 | – | – |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 10144, 2345, 2336 | 15978, 8209, 6704 | 6133, 3271, 1857 |
| $R_{int}$ | 0.054 | 0.021 | 0.074 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.616 | 0.685 | 0.649 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.029, 0.084, 1.08 | 0.036, 0.094, 1.02 | 0.049, 0.105, 1.02 |
| No. of reflections | 2345 | 8209 | 3271 |
| No. of parameters | 203 | 495 | 231 |
| No. of restraints | 1 | 1 | 1 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.26, −0.33 | 0.58, −0.41 | 0.26, −0.46 |
| Absolute structure | Refined as an inversion twin. | – | – |
| Absolute structure parameter | 0.00 (2) | – | – |

| | $NH_4$**4** | $NH_4$**5** | K**1** |
|---|---|---|---|
| Crystal data | | | |
| Chemical formula | $NH_4^+ \cdot C_{16}H_{18}N_3O_5S^- \cdot H_2O$ | $NH_4^+ \cdot C_{12}H_9N_2O_4S^-$ | $[K(C_{12}H_9N_2O_4S)(H_2O)]$ |
| $M_r$ | 400.45 | 295.31 | 334.39 |
| Crystal system, space group | Triclinic, $P\overline{1}$ | Orthorhombic, $Pccn$ | Triclinic, $P\overline{1}$ |
| Temperature (K) | 123 | 123 | 150 |
| $a, b, c$ (Å) | 8.4933 (1), 13.0977 (2), 17.1657 (3) | 12.6592 (3), 28.3597 (7), 7.1268 (2) | 5.9620 (7), 7.2033 (11), 31.929 (5) |
| $\alpha, \beta, \gamma$ (°) | 90.970 (1), 103.180 (1), 95.132 (1) | 90, 90, 90 | 83.852 (14), 86.361 (15), 88.868 (15) |
| $V$ (Å$^3$) | 1850.43 (5) | 2558.60 (11) | 1360.5 (3) |
| $Z$ | 4 | 8 | 4 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ | Synchrotron, λ = 0.689 Å |
| $\mu$ (mm$^{-1}$) | 0.22 | 0.27 | 0.51 |
| Crystal size (mm) | 0.5 × 0.5 × 0.15 | 0.30 × 0.10 × 0.05 | 0.20 × 0.14 × 0.03 |
| Data collection | | | |
| Diffractometer | Enraf–Nonius KappaCCD | Enraf–Nonius KappaCCD | Bruker APEXII CCD |
| $T_{min}$, $T_{max}$ | – | – | 0.751, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 16736, 8841, 5911 | 5346, 2923, 1850 | 10143, 5691, 4405 |
| $R_{int}$ | 0.043 | 0.069 | 0.032 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.660 | 0.649 | 0.636 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.047, 0.118, 1.04 | 0.051, 0.118, 1.03 | 0.060, 0.180, 1.02 |
| No. of reflections | 8841 | 2923 | 5691 |
| No. of parameters | 550 | 201 | 403 |
| No. of restraints | 8 | 0 | 6 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.57, −0.42 | 0.29, −0.45 | 0.59, −0.67 |

| | K**3** | K**4** | K**5** |
|---|---|---|---|
| Crystal data | | | |
| Chemical formula | $[K(C_{12}H_{10}N_3O_3S)(H_2O)_2]$ | $[K(C_{16}H_{18}N_3O_5S)(H_2O)_2]$ | $[K(C_{12}H_9N_2O_4S)]$ |
| $M_r$ | 351.42 | 439.52 | 316.37 |
| Crystal system, space group | Triclinic, $P\overline{1}$ | Monoclinic, $P2_1/n$ | Orthorhombic, $Pccn$ |

**Table 1 (continued)**

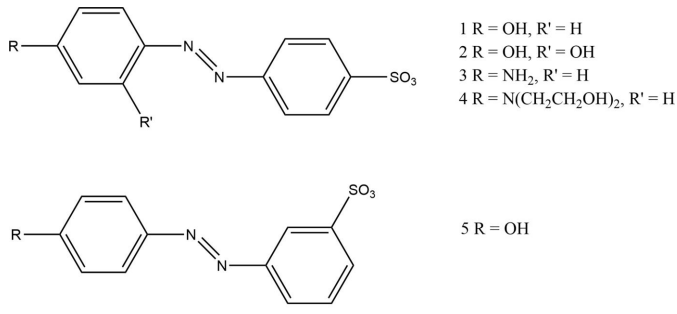
| | K3 | K4 | K5 |
|---|---|---|---|
| Temperature (K) | 123 | 123 | 123 |
| *a*, *b*, *c* (Å) | 13.3058 (2), 13.6247 (2), 18.4664 (3) | 9.4006 (2), 12.1583 (3), 34.4743 (9) | 12.5535 (2), 27.9698 (5), 6.9982 (1) |
| $\alpha$, $\beta$, $\gamma$ (°) | 88.373 (1), 73.971 (1), 66.313 (1) | 90, 95.496 (1), 90 | 90, 90, 90 |
| *V* (Å$^3$) | 2933.52 (8) | 3922.14 (16) | 2457.20 (7) |
| *Z* | 8 | 8 | 8 |
| Radiation type | Mo *K*$\alpha$ | Mo *K*$\alpha$ | Mo *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.53 | 0.42 | 0.62 |
| Crystal size (mm) | 0.7 × 0.3 × 0.02 | 0.20 × 0.18 × 0.08 | 0.70 × 0.08 × 0.04 |
| Data collection | | | |
| Diffractometer | Enraf–Nonius KappaCCD | Enraf–Nonius KappaCCD | Enraf–Nonius KappaCCD |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 26000, 13392, 10848 | 14501, 7971, 4387 | 5174, 2802, 1971 |
| $R_{int}$ | 0.023 | 0.084 | 0.056 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.649 | 0.628 | 0.648 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.036, 0.098, 1.03 | 0.060, 0.102, 1.01 | 0.042, 0.092, 1.05 |
| No. of reflections | 13392 | 7971 | 2802 |
| No. of parameters | 895 | 560 | 185 |
| No. of restraints | 24 | 112 | 1 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.80, −0.79 | 0.37, −0.34 | 0.39, −0.41 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *DENZO* and *COLLECT* (Otwinowski & Minor, 1997), *APEX2*, *SAINT* and *SADABS* (Bruker, 2012), *SHELXT* (Sheldrick, 2015*a*), *SIR92* (Altomare *et al.*, 1994) and *SHELXL2018* (Sheldrick, 2015*b*) in *WinGX* (Farrugia, 2012).

the structure of sulfonated azo pigments has been to study systematically the structures of similarly functionalized, but easier to manipulate, dyes and then to cross-check any structure-to-property relationships identified against those pigment structures that are known (Kennedy *et al.*, 2004, 2009, 2012).

Ammonium salt forms of sulfonated azo colourants are sometimes used in preference to alkali-metal salts, either in the finished product or as an intermediate prior to laking (Christie & Mackay, 2008; Al Isawi *et al.*, 2021; Gonzalez & Miksovska, 2014). Despite this, only two structures of ammonium salts of sulfonated azo colourants appear to have been determined, namely, those of diammonium Orange G tetrahydrate and of the nitrile-substituted $[NH_4][O_3S(C_6H_4)$-$NN(C_6H_4)NHCH_2CH_2CN]\cdot H_2O$ (Ojala *et al.*, 1994; Astbury *et al.*, 2013). The $NH_4^+$ ion is sometimes known as a pseudo-alkali metal due to it propensity to act in an isostructural manner with the heavier Group 1 metal ions. Its effective ionic radius has been estimated at 1.40 to 1.67 Å, depending on the coordination number (Sidey, 2016). Thus, as well as having the same charge as an alkali metal, it also has an ionic radius similar to those of $K^+$ and $Rb^+$. An obvious difference is that bonds from an alkali-metal ion to, for example, an O-atom donor are typically described as ionic *M*—O interactions, whereas $NH_4^+$ will interact with O *via* N—H···O hydrogen bonds. As there are four H-atom donors per ammonium ion this may limit $NH_4^+$ to lower coordination numbers than those typically seen for K or Rb. It seems to be this feature that is responsible for ammonium forming isostructural pairs with Na compounds, as well as with K and Rb compounds, despite the smaller ionic radius of $Na^+$ (*e.g.* Khan & Baur, 1972; Christov, 2003; Emerson *et al.*, 2014). Many examples of isostructurality between ammonium and Group 1 metal ions are for inorganic systems, but organic examples are also known. Of particular relevance to sulfonated azo species is the sweetener cyclamic acid. This is an $RSO_3^-$-containing organic species and its ammonium, Na, K and Rb salt forms are known to form an isostructural series (Leban *et al.*, 2007).

In order to investigate the structural relationships between ammonium salt forms of sulfonated azo dyes and their alkali-metal congeners, we herein present the crystal structures of ammonium salts of five azo anion species (Scheme 1). The structures of their Na-salt equivalents have already been reported (Kennedy *et al.*, 2001, 2020; Dodds *et al.*, 2017), as has the structure of one of their K equivalents (Kennedy *et al.*, 2004). In order to complete the comparison we report herein the crystal structures of the remaining four K-salt equivalents.




Scheme 1

## 2. Experimental

The Na salt of dye **3** was obtained from Fujifilm. The other dyes were synthesized as their Na salts using the well-known azo-coupling method (Alsantali *et al.*, 2022; Kennedy *et al.*, 2001). Na salts were converted to $NH_4$ or K salts by reaction with a slight excess of either $NH_4Cl$ or KCl in warm water. Solutions were filtered to give clear aqueous solutions and then allowed to evaporate for 2 to 7 d. This gave yellow or

yellow–orange crystals of the desired salt forms that were suitable for single-crystal diffraction analysis. FT–IR spectra were measured as KBr discs using a Nicolet Avatar 380 spectrometer. Raman data were measured from solids using a Reinshaw Ramascope with excitation at 785 nm. Aqueous UV–Vis spectra were measured using a Cary 300 Bio spectrophotometer and solid-state UV–Vis spectra were measured using a Phillips PU8749 spectrophotometer. Measurements on K**1** were made at Station 9.8 of the Daresbury SRS. All other measurements were made using standard Rigaku Synergy-i ($NH_4$**4**) or Enraf–Nonius KappaCCD (all other structures) laboratory diffractometers equipped with CCD detectors.

### 2.1. Spectroscopic data

$NH_4$**1**, FT–IR (cm $^{-1}$): 3421, 3267, 3088, 1598, 1501, 1280, 1193, 1040. Raman (cm$^{-1}$): 1120, 1147, 1185, 1435, 1459, 1594. UV–Vis ($\lambda_{max}$, nm): 348 (aq), 346 (solid).

$NH_4$**2**, FT–IR (cm $^{-1}$): 1629, 1588, 1419, 1337, 1198, 1116, 1034, 999, 840, 804. Raman (cm$^{-1}$): 239, 317, 348, 446, 836, 1036, 1122, 1161, 1197, 1324, 1375, 1419, 1591. UV–Vis ($\lambda_{max}$, nm): 417 (aq), 334 (solid).

$NH_4$**3**, FT–IR (cm $^{-1}$): 1634, 1603, 1501, 1398, 1383, 1275, 1193, 1116, 1034, 1004, 835. Raman (cm$^{-1}$): peaks masked by fluorescence. UV–Vis ($\lambda_{max}$, nm): 386 (aq), 332 (solid).

$NH_4$**4**, FT–IR (cm $^{-1}$): 1598, 1511, 1403, 1213, 1116, 1075, 1024, 844, 814, 691. Raman (cm$^{-1}$): 1034, 1115, 1143, 1197, 1315, 1356, 1387, 1419, 1441, 1589. UV–Vis ($\lambda_{max}$, nm): 459 (aq), 418 (solid).

$NH_4$**5**, FT–IR (cm $^{-1}$): 3405, 3190, 2366, 1597, 1495, 1423, 1177, 1136, 1034. Raman (cm$^{-1}$): 1138, 1183, 1422, 1452 (somewhat masked by fluorescence). UV–Vis ($\lambda_{max}$, nm): 348 (aq), 351 (solid).

K**1**, FT–IR (cm $^{-1}$): 1593, 1460, 1372, 1178, 850, 717. Raman (cm$^{-1}$): 628, 794, 922, 1102, 1121, 1147, 1158, 1309, 1396, 1435, 1590, 1607. UV–Vis ($\lambda_{max}$, nm): 348 (aq), 334 (solid).

K**2**, FT–IR (cm $^{-1}$): 3472, 3390, 1593, 1469, 1372, 1198, 1121, 1039, 839, 803. Raman (cm$^{-1}$): 1033, 1122, 1161, 1198, 1326, 1374, 1418, 1592. UV–Vis ($\lambda_{max}$, nm): 430 (aq), 336 (solid).

K**3**, FT–IR (cm $^{-1}$): 1644, 1460, 1372, 1193, 1116, 1029, 839, 716. Raman (cm$^{-1}$): 780, 921, 1036, 1120, 1152, 1427, 1588. UV–Vis ($\lambda_{max}$, nm): 387 (aq), 345 (solid).

K**4**, FT–IR (cm $^{-1}$): 1597, 1515, 1382, 1351, 1316, 1259, 1223, 1182, 1116, 1049, 1003. Raman (cm$^{-1}$): 739, 1032, 1117, 1143, 1195, 1315, 1355, 1392, 1421, 1440. UV–Vis ($\lambda_{max}$, nm): 458 (aq), 413 (solid).

K**5**, FT–IR (cm $^{-1}$): 3390, 3078, 1603, 1501, 1383, 1188, 1034, 845. Raman (cm$^{-1}$): 1136, 1169, 1185, 1424, 1454 (somewhat masked by fluorescence). UV–Vis ($\lambda_{max}$, nm): 347 (aq), 348 (solid).

### 2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. For K**4**, the azobenzene core of one of the two crystallographically independent dye anions was treated as disordered over two sites. In a similar way, one of the four independent $SO_3$ groups of K**3** was also treated as disordered over two sites, a rotation about the C—S bond giving alternative positions for the three O atoms. In both cases, appropriate restraints and constraints on the displacement parameters were added so as to ensure that the structures approximated normal behaviour. Finally, one water molecule of $NH_4$**4** was modelled as rotationally disordered about atom O2*W* so that the water molecule has three independent H-atom sites. Where possible, H atoms attached to O or to N atoms were positioned as found in difference syntheses and refined freely and isotropically. Where riding models were required, *X*—H bond lengths were set at 0.88 (1) Å. All H atoms bound to C atoms were included in riding models, with C—H = 0.95 or 0.99 Å for CH and $CH_2$ groups, respectively. For all H atoms in riding models, $U_{iso}$(H) values were set to $1.2U_{eq}$ of the parent atom.

## 3. Results and discussion

All structures discussed are of crystal samples obtained from aqueous recrystallizations. Aqueous conditions were chosen to best reflect normal synthesis and usage of sulfonated azo dyes. It is possible to dry the hydrate structures described to give anhydrous materials. However, no polymorph screen has been attempted and so the existence of other forms is entirely likely. Selected crystallographic and refinement parameters are given in Table 1 and selected geometric parameters are given in Tables 2 to 14. Representations of the crystal structures of the ammonium salt forms $NH_4$**1**, $NH_4$**2**, $NH_4$**3**, $NH_4$**4** and $NH_4$**5** are given in Figs. 1 to 5. Both the phenol derivatives $NH_4$**1** and $NH_4$**5** are simple anhydrous salts with an $NH_4^+$ cation and an azo anion in the asymmetric unit. The other species are all hydrated forms as shown in Table 15. Additionally, both $NH_4$**2** and $NH_4$**4** are $Z'$ = 2 structures with two cation/anion pairs per asymmetric unit, as well as their accompanying water molecules. The bond lengths about the azo chromophores are in good agreement with those described for *s*-block metal salts of similar azo anions (Kennedy *et al.*, 2001, 2020). The N═N bonds range from 1.254 (3) to 1.275 (2) Å, whilst the C—N bonds show ranges of 1.423 (2)–1.439 (2) and 1.396 (2)–1.425 (3) Å for the bonds

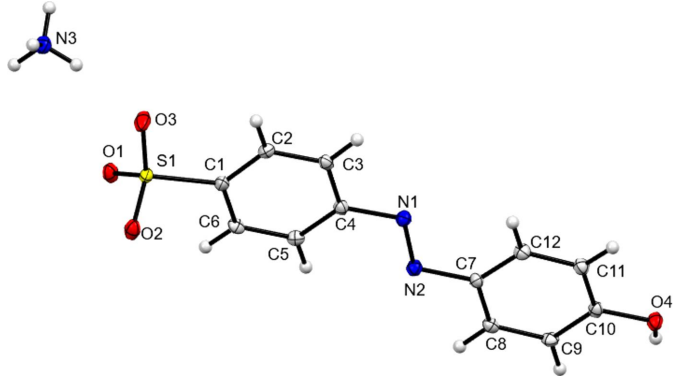



**Figure 1**
The asymmetric unit contents of $NH_4$**1**. Here and elsewhere displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as small spheres of arbitrary size.

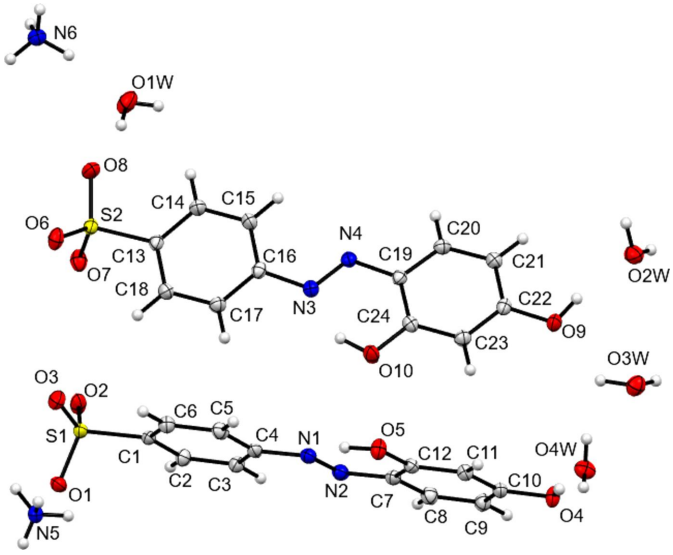



**Figure 2**
The asymmetric unit contents of $NH_4$**2**.

**Table 2**
Hydrogen-bond geometry (Å, °) for $NH_4$**1**.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H4N···O2$^{i}$ | 0.91 (5) | 2.30 (5) | 2.940 (4) | 127 (4) |
| N3—H4N···O1$^{ii}$ | 0.91 (5) | 2.21 (5) | 2.969 (3) | 141 (4) |
| N3—H3N···O3 | 0.86 (5) | 1.96 (5) | 2.808 (3) | 170 (4) |
| N3—H1N···O1$^{iii}$ | 0.86 (4) | 2.03 (5) | 2.889 (4) | 176 (4) |
| N3—H2N···O4$^{iv}$ | 0.88 (4) | 2.10 (4) | 2.946 (3) | 159 (4) |
| O4—H1$H$···O2$^{v}$ | 0.87 (5) | 1.95 (5) | 2.802 (3) | 167 (4) |

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

involving the C atoms of the sulfonated and nonsulfonated rings, respectively. The generally shorter bonds of the latter are due to resonance with the OH and amine substituents. It is noteworthy that it is $NH_4$**2** that displays both the longest N═N bond and the shortest N—C bond. The intramolecular O—H···N hydrogen bonding of $NH_4$**2** raises the possibility of tautomerism in this species and, although the electron density shows that the compound clearly exists largely as the azo tautomer, the bond lengths observed tend to reflect a small contribution from the alternative hydrazone tautomer. [See Kennedy *et al.* (2020) for a detailed discussion of factors that influence bond lengths and hence colour in similar azo dyes,

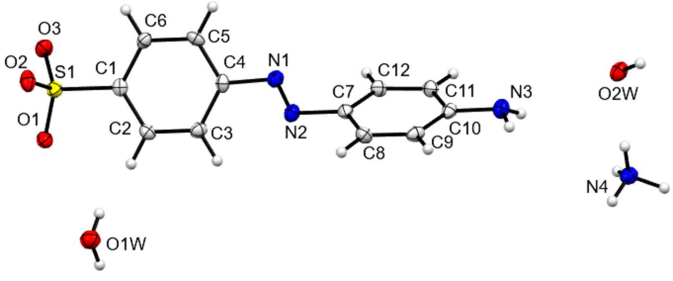



**Figure 3**
The asymmetric unit contents of $NH_4$**3**. A twofold rotation axis passes through atom O2$W$.

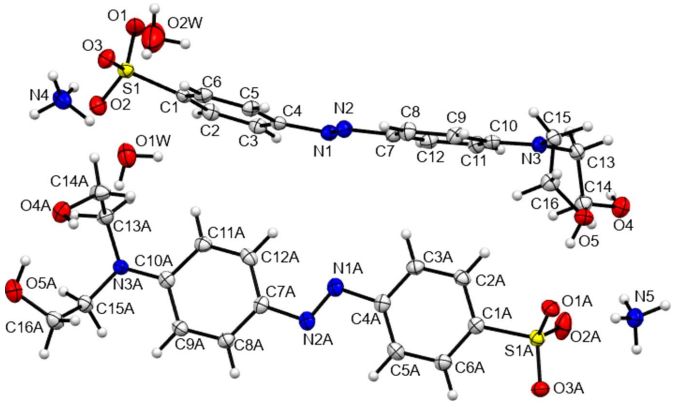



**Figure 4**
The asymmetric unit contents of $NH_4$**4**. The water molecule labelled O2$W$ is disordered so as to give three independent H-atom sites.

**Table 3**
Hydrogen-bond geometry (Å, °) for $NH_4$**2**.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H1$H$···O4$W$ | 0.84 (2) | 1.81 (2) | 2.6357 (17) | 170 (2) |
| O5—H2$H$···N1 | 0.98 (3) | 1.65 (3) | 2.5535 (16) | 151 (2) |
| O9—H3$H$···O2$W$ | 0.85 (2) | 1.83 (2) | 2.6673 (16) | 168 (2) |
| O10—H4$H$···N3 | 0.93 (3) | 1.69 (3) | 2.5420 (16) | 149 (2) |
| N5—H1N···O1$^{i}$ | 0.80 (2) | 2.16 (2) | 2.8858 (17) | 151.1 (18) |
| N5—H1N···O1$W^{ii}$ | 0.80 (2) | 2.627 (19) | 3.034 (2) | 113.3 (15) |
| N5—H2N···O1 | 0.92 (2) | 1.94 (2) | 2.8403 (18) | 168 (2) |
| N5—H3N···O3$W^{iii}$ | 0.94 (2) | 1.91 (2) | 2.8488 (17) | 172 (2) |
| N5—H4N···O6$^{iv}$ | 0.89 (2) | 2.01 (2) | 2.8425 (16) | 156 (2) |
| N6—H5N···S2$^{v}$ | 0.90 (2) | 2.99 (2) | 3.7547 (15) | 143.5 (17) |
| N6—H5N···O7$^{v}$ | 0.90 (2) | 1.99 (2) | 2.8804 (18) | 168 (2) |
| N6—H6N···O1$W$ | 0.92 (2) | 1.90 (2) | 2.8027 (18) | 169.7 (19) |
| N6—H7N···O3$W^{vi}$ | 0.92 (2) | 1.96 (2) | 2.8733 (19) | 170.4 (19) |
| N6—H8N···O2$^{vii}$ | 0.84 (2) | 2.42 (2) | 2.9090 (18) | 117.8 (18) |
| N6—H8N···O3$^{v}$ | 0.84 (2) | 2.36 (2) | 2.9153 (17) | 123.8 (19) |
| O1$W$—H2$W$···O4$^{viii}$ | 0.84 (3) | 2.01 (3) | 2.8150 (16) | 162 (2) |
| O1$W$—H1$W$···O8 | 0.82 (3) | 1.98 (3) | 2.7858 (17) | 166 (2) |
| O2$W$—H3$W$···O3$^{viii}$ | 0.83 (3) | 2.04 (3) | 2.8639 (16) | 171 (2) |
| O2$W$—H4$W$···O8$^{vi}$ | 0.87 (1) | 1.94 (1) | 2.7918 (17) | 169 (2) |
| O3$W$—H5$W$···O7$^{viii}$ | 0.88 (3) | 1.83 (3) | 2.7106 (16) | 179 (2) |
| O3$W$—H6$W$···O9 | 0.95 (3) | 2.04 (3) | 2.8347 (15) | 140 (2) |
| O4$W$—H7$W$···O2$^{ix}$ | 0.81 (2) | 1.94 (2) | 2.7482 (17) | 175 (2) |
| O4$W$—H8$W$···O6$^{ix}$ | 0.81 (3) | 2.01 (3) | 2.8128 (16) | 171 (2) |

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

and Yatsenko *et al.* (2024) for a discussion on azo/hydrazone tautomerization in solid-state arylazo compounds.] Most of

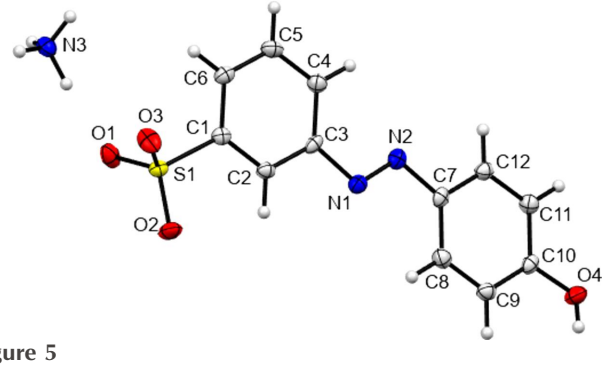



**Figure 5**
The asymmetric unit contents of $NH_4$**5**.

**Table 4**
Hydrogen-bond geometry (Å, °) for $NH_4$**3**.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N4—H1N···O2$W$ | 0.90 (3) | 2.00 (3) | 2.850 (3) | 157 (2) |
| N4—H2N···O1$^{i}$ | 0.94 (3) | 1.93 (3) | 2.862 (3) | 167 (3) |
| N4—H2N···O1$W^{i}$ | 0.94 (3) | 2.57 (3) | 3.039 (3) | 111 (2) |
| N4—H3N···O2$^{ii}$ | 0.86 (3) | 2.14 (3) | 2.963 (3) | 161 (3) |
| N4—H3N···O3$^{ii}$ | 0.86 (3) | 2.66 (3) | 3.351 (3) | 139 (3) |
| N4—H4N···O3$^{iii}$ | 0.98 (4) | 2.07 (4) | 2.987 (3) | 155 (3) |
| N3—H5N···O1$W^{i}$ | 0.85 (3) | 2.23 (3) | 3.067 (3) | 168 (2) |
| N3—H6N···O2$^{ii}$ | 0.86 (3) | 2.17 (3) | 3.015 (3) | 165 (2) |
| O1$W$—H1$W$···N3$^{ii}$ | 0.83 (3) | 2.21 (3) | 3.011 (3) | 162 (3) |
| O1$W$—H2$W$···O3$^{iv}$ | 0.88 (3) | 1.99 (3) | 2.861 (3) | 169 (3) |
| O2$W$—H3$W$···O1$^{v}$ | 0.87 (1) | 1.97 (1) | 2.792 (3) | 159 (3) |

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+2$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $x, -y+1, z+\frac{1}{2}$; (v) $x-\frac{1}{2}, y-\frac{1}{2}, z$.

**Table 5**
Hydrogen-bond geometry (Å, °) for $NH_4$**4**.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$W$···O1$^{i}$ | 0.88 (1) | 1.93 (1) | 2.802 (2) | 176 (3) |
| O1$W$—H2$W$···O5$^{ii}$ | 0.88 (1) | 1.84 (1) | 2.711 (2) | 168 (2) |
| O2$W$—H3$W$···O1$A^{ii}$ | 0.91 (1) | 2.05 (1) | 2.945 (2) | 172 (3) |
| O2$W$—H4$W$···O2$W^{iii}$ | 0.89 (1) | 2.29 (4) | 3.031 (5) | 141 (6) |
| O4—H1$H$···O3$A^{iv}$ | 0.84 (3) | 2.05 (3) | 2.885 (2) | 169 (3) |
| O5—H2$H$···O1$A$ | 0.81 (3) | 1.90 (3) | 2.698 (2) | 169 (3) |
| O4$A$—H3$H$···O1$^{i}$ | 0.80 (3) | 1.96 (3) | 2.756 (2) | 177 (3) |
| O5$A$—H4$H$···O4$A$ | 0.97 (3) | 1.79 (3) | 2.734 (2) | 165 (3) |
| N4—H1N···O3 | 0.87 (3) | 2.09 (3) | 2.957 (3) | 176 (2) |
| N4—H2N···O2$^{v}$ | 0.95 (3) | 1.92 (3) | 2.841 (3) | 163 (2) |
| N4—H3N···O3$A^{vi}$ | 0.89 (3) | 2.05 (3) | 2.923 (3) | 167 (2) |
| N4—H4N···O1$W$ | 1.13 (4) | 1.69 (4) | 2.818 (3) | 176 (3) |
| N5—H5N···O2$A$ | 0.91 (3) | 1.89 (3) | 2.784 (3) | 164 (2) |
| N5—H6N···O5$A^{vii}$ | 0.89 (4) | 2.21 (4) | 2.935 (3) | 138 (3) |
| N5—H6N···O1$W^{viii}$ | 0.89 (4) | 2.51 (4) | 3.094 (3) | 123 (3) |
| N5—H7N···O3$^{ix}$ | 0.96 (4) | 2.03 (4) | 2.952 (3) | 159 (3) |
| N5—H8N···O4$^{x}$ | 0.96 (3) | 2.00 (3) | 2.943 (3) | 168 (3) |

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

**Table 6**
Hydrogen-bond geometry (Å, °) for $NH_4$**5**.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H1N···O2$^{i}$ | 0.90 (4) | 2.48 (3) | 3.002 (4) | 118 (3) |
| N3—H1N···O4$^{ii}$ | 0.90 (4) | 2.35 (3) | 2.913 (4) | 121 (3) |
| N3—H1N···O4$^{iii}$ | 0.90 (4) | 2.50 (4) | 3.162 (4) | 131 (3) |
| N3—H2N···O1 | 0.96 (4) | 1.94 (4) | 2.893 (3) | 171 (3) |
| N3—H3N···O1$^{iv}$ | 0.89 (3) | 1.97 (3) | 2.822 (3) | 159 (3) |
| N3—H4N···O3$^{v}$ | 0.90 (4) | 1.97 (4) | 2.831 (3) | 160 (3) |
| O4—H1$H$···O2$^{vi}$ | 0.82 (4) | 2.00 (4) | 2.707 (3) | 144 (4) |

Symmetry codes: (i) $x-\frac{1}{2}, -y+1, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, y-\frac{1}{2}, -z$; (iii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y, z+\frac{1}{2}$; (v) $-x+\frac{3}{2}, y, z-\frac{1}{2}$; (vi) $-x+\frac{5}{2}, -y+\frac{3}{2}, z$.

**Table 7**
Selected bond lengths (Å) for **K1**.

| | | | |
|---|---|---|---|
| K1—O1 | 2.642 (2) | K2—O5 | 2.625 (2) |
| K1—O2$W^{i}$ | 2.688 (3) | K2—O2$W$ | 2.713 (2) |
| K1—O2$^{ii}$ | 2.728 (2) | K2—O7$^{ii}$ | 2.736 (2) |
| K1—O6 | 2.829 (3) | K2—O1$^{ii}$ | 2.769 (2) |
| K1—O8$^{iii}$ | 2.957 (2) | K2—O8$^{v}$ | 2.904 (2) |
| K1—O7 | 3.027 (2) | K2—O3$^{ii}$ | 3.020 (2) |
| K1—O4$^{iv}$ | 3.116 (2) | K2—O1$W$ | 3.050 (3) |
| K1—O1$W^{i}$ | 3.149 (2) | | |

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

the azo anions have planar conformations, with angles between the planes of the aromatic rings ranging from 2.23 (9) to 12.32 (11)°. Despite this, the azo group does form a small step between the two parallel ring planes [*e.g.* in $NH_4$**1**, atom N2 lies 0.473 (5) Å out of the plane defined by atoms C1–C6]. The exception is the anion of $NH_4$**3**, which adopts a twisted conformation with an angle of 58.76 (11)° between the planes of its two aromatic rings. This out-of-plane twist in the solid state should alter the resonance through the azobenzene fragment and may contribute to the relatively large difference found between the solution-state and solid-state $\lambda_{max}$ values (386 *versus* 332 nm). It has previously been shown for *s*-block metal salt forms that the planar or twisted conformation of *ortho*-sulfonated azo anions correlates with the packing motifs observed. Thus, twisted azo species gave structures with a simple alternating layer structure, *i.e.* layers of organic anions alternating with hydrophilic layers containing the cations and water molecules. In contrast, planar azo anions gave structures with organic bilayers (Kennedy *et al.*, 2009). This is not observed here; all the ammonium salts of the *para*- and *meta*-sulfonated anions **1** to **5** give simple layering structures with no bilayers, irrespective of the planarity of the anion (see Figs. 6 and 7 for examples).

Each H atom of every $NH_4^+$ cation acts as a hydrogen-bond donor to at least one O atom. There are no $NH_4$-to-N hydrogen-bonding interactions. From Tables 2 to 6 and the summary Table 15, it can be seen that of the 28 independent N—H donors of the ammonium groups, 10 form bifurcated

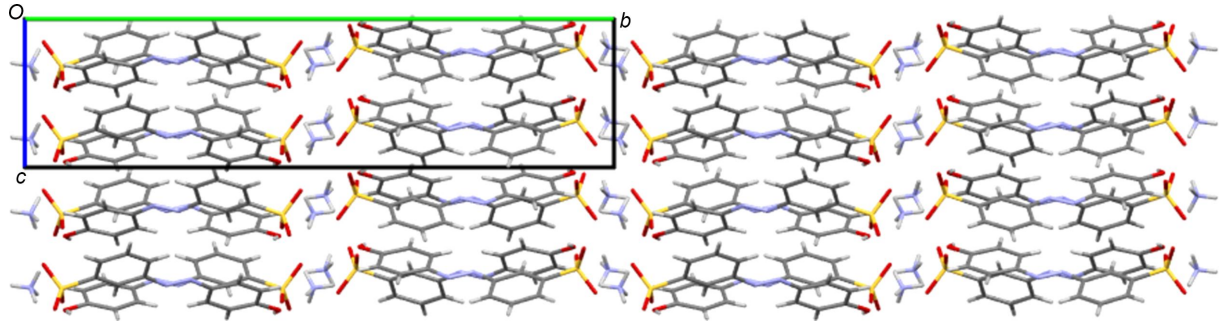



**Figure 6**
Packed structure of $NH_4$**5** viewed along the *a* axis. Note the simple layering structure with alternating hydrophobic (arylazo) and hydrophilic (cation) layers parallel to the *ac* plane.

**Table 8**
Hydrogen-bond geometry (Å, °) for K**1**.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4*H*···O1*W*$^{vi}$ | 0.88 (1) | 1.83 (2) | 2.676 (3) | 160 (3) |
| O8—H8*H*···O6$^{vii}$ | 0.88 (1) | 1.93 (3) | 2.748 (3) | 155 (5) |
| O1*W*—H1*W*···O3$^{ii}$ | 0.87 (1) | 1.95 (2) | 2.801 (3) | 165 (4) |
| O1*W*—H2*W*···O2$^{viii}$ | 0.88 (1) | 1.93 (1) | 2.803 (4) | 177 (5) |
| O2*W*—H3*W*···O3$^{ix}$ | 0.88 (1) | 2.20 (3) | 2.866 (3) | 133 (4) |
| O2*W*—H3*W*···O4$^{vi}$ | 0.88 (1) | 2.29 (3) | 2.905 (3) | 127 (3) |
| O2*W*—H4*W*···O5$^{ii}$ | 0.88 (1) | 1.92 (2) | 2.778 (3) | 165 (4) |

Symmetry codes: (ii) $x+1, y, z$; (vi) $-x+2, -y-1, -z+1$; (vii) $-x+2, -y, -z+2$; (viii) $x+1, y-1, z$; (ix) $x+2, y, z$.

**Table 9**
Selected bond lengths (Å) for K**3**.

| | | | |
|---|---|---|---|
| K1—O2 | 2.6531 (14) | K3—O3*C* | 2.649 (2) |
| K1—O1*A*$^{i}$ | 2.6570 (14) | K3—O1*B* | 2.7285 (16) |
| K1—O6*W*$^{ii}$ | 2.7388 (15) | K3—O5*W*$^{iv}$ | 2.8713 (16) |
| K1—O7*W*$^{ii}$ | 2.7669 (15) | K3—O4*W* | 2.8932 (16) |
| K1—O1*W* | 2.7806 (15) | K3—O5*W* | 2.9166 (15) |
| K1—O1*A* | 2.8284 (14) | K3—O3*B* | 3.2009 (19) |
| K1—O2*A* | 2.9928 (15) | K3—O4*W*$^{iv}$ | 3.2971 (17) |
| K2—O3 | 2.7462 (14) | K4—O2*B*$^{v}$ | 2.6854 (15) |
| K2—O1*C* | 2.7536 (17) | K4—O6*W* | 2.7164 (15) |
| K2—O2*A* | 2.7929 (14) | K4—O2*B* | 2.7701 (15) |
| K2—O2*W* | 2.8559 (16) | K4—O8*W* | 2.8338 (16) |
| K2—O3*W*$^{iii}$ | 2.8666 (15) | K4—O7*W* | 2.8395 (16) |
| K2—O3*W* | 2.8901 (16) | K4—O3*C* | 2.954 (2) |
| K2—O2 | 3.0708 (15) | K4—O2*C* | 3.097 (2) |
| K2—O3*A* | 3.1446 (16) | K4—O1*B* | 3.1503 (16) |
| K3—O3 | 2.6206 (14) | | |

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

hydrogen bonds and two (atoms H8N of $NH_4$**2** and H1N of $NH_4$**5**) interact with three separate O-atom acceptors. Most ammonium cations interact with six O atoms, but one (that containing atom N4 of structure $NH_4$**4**) has a coordination number of 5 and one (containing atom N3 of $NH_4$**5**) has a coordination number of 7. In all five structures, the majority of the ammonium cation hydrogen-bond interactions are with the O atoms of the formally negatively charged $SO_3$ groups. For the anhydrous species $NH_4$**1** and $NH_4$**5**, the ammonium ions also donate hydrogen bonds to the phenol OH groups; thus, hydrogen bonding from ammonium to both the $SO_3$ head and the OH tail of the azo anions leads to the anions bridging between the inorganic/hydrophilic layers of the packing structures (Fig. 6). The hydrates $NH_4$**2** and $NH_4$**3** have no direct ammonium-to-tail-group hydrogen-bond interactions; instead, the water molecules act as intermediaries or bridges and accept/donate hydrogen bonds from both ammonium and tail groups. Thus, for these hydrate species, interactions between the hydrophobic and hydrophilic layers of the packing structure is *via* the water molecules. Despite featuring a lower cation coordination number, in hydrate $NH_4$**4**, the cations donate hydrogen bonds to all of the different types of acceptor groups available, *i.e.* to the $SO_3$ heads, to the OH groups of the tail and to bridging water molecules.

As they are situated at the centre of the hydrophobic layers, none of the –N=N– chromophore units take part in intermolecular hydrogen bonding. Indeed, the azo groups of $NH_4$**1**, $NH_4$**3** and $NH_4$**4** have no intermolecular contacts less than the sum of the van der Waals radii. $NH_4$**5** is the only species of the five to show face-to-face $\pi$-contacts between the azobenzene units, with closest N2···C4 and C7···C3 contact distances of 3.160 (4) and 3.352 (4) Å, respectively. This forms the stacking motif seen extending parallel to the *c* axis in Fig. 8. In $NH_4$**2**, the *ortho*-OH substituent of one of the independent azo anions of the asymmetric unit approaches the azo group of the other anion [O10···N1 = 3.052 (2) Å], creating dimeric pairs of anions (Fig. 9).

The structure of K**2** was reported by Kennedy *et al.* (2004). Here, we report the remaining K-salt structures of azo anions **1** to **5**. Figs. 9 to 13 show the fundamental features of these structures and selected geometric parameters are given in

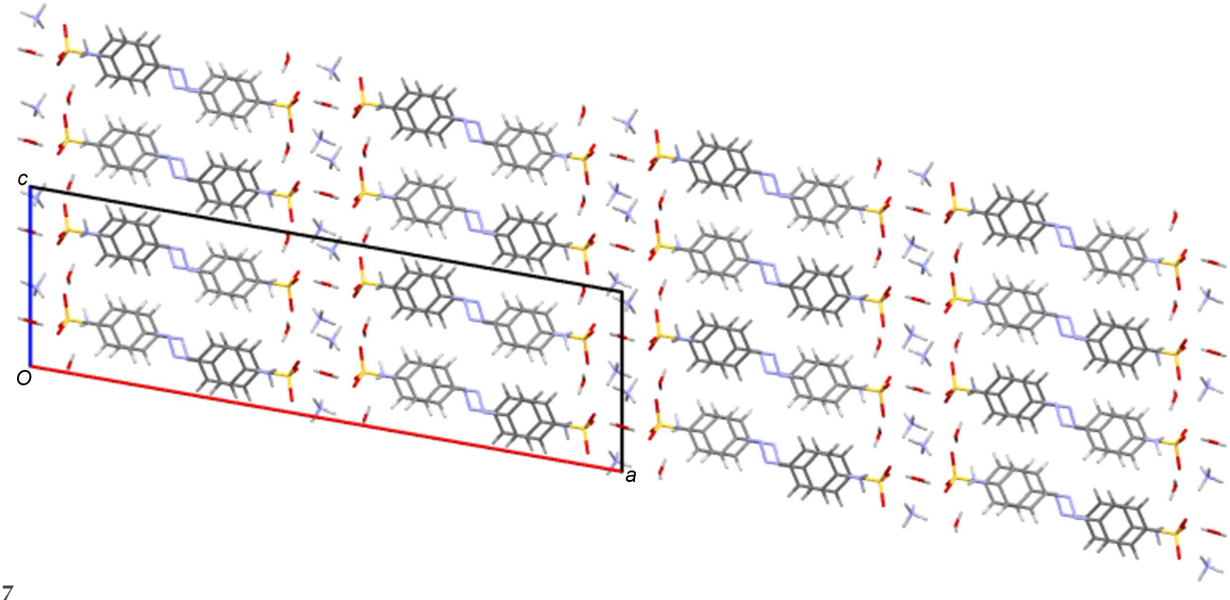



**Figure 7**
Packed structure of $NH_4$**3** viewed along the *b* axis. Despite the twisted nature of the azo anion, this structure has a similar simple layering structure with alternating hydrophobic (arylazo) and hydrophilic (cation and water) layers as the planar species. Here the layers lie parallel to the *bc* plane.

**Table 10**
Hydrogen-bond geometry (Å, °) for K**3**.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H1N···O3$B^{vi}$ | 0.92 (3) | 2.35 (3) | 3.210 (2) | 155 (2) |
| N3—H2N···O2$W^{vi}$ | 0.81 (3) | 2.40 (3) | 3.147 (2) | 154 (2) |
| N3$A$—H3N···O8$W^{vii}$ | 0.82 (3) | 2.27 (3) | 3.016 (2) | 153 (3) |
| N3$A$—H4N···O1$^{viii}$ | 0.88 (3) | 2.27 (3) | 3.115 (2) | 161 (2) |
| N3$B$—H5N···O2$C^{ix}$ | 0.90 (3) | 2.50 (3) | 3.384 (3) | 167 (2) |
| N3$B$—H5N···O5$C^{ix}$ | 0.90 (3) | 2.26 (3) | 3.078 (14) | 151 (2) |
| N3$B$—H6N···O1$W^{vi}$ | 0.88 (3) | 2.21 (3) | 3.065 (2) | 164 (2) |
| N3$C$—H7N···O4$W^{viii}$ | 0.86 (3) | 2.38 (3) | 3.149 (2) | 149 (2) |
| N3$C$—H8N···O3$A^{vii}$ | 0.87 (3) | 2.53 (3) | 3.330 (2) | 154 (2) |
| O1$W$—H1$W$···N3$C^{x}$ | 0.87 (1) | 2.62 (2) | 3.428 (2) | 156 (2) |
| O1$W$—H2$W$···O1$B^{ii}$ | 0.87 (1) | 1.94 (1) | 2.808 (2) | 174 (2) |
| O2$W$—H3$W$···O1$C^{iii}$ | 0.87 (1) | 2.14 (1) | 2.963 (3) | 160 (3) |
| O2$W$—H3$W$···O5$C^{iii}$ | 0.87 (1) | 2.24 (3) | 2.973 (18) | 143 (2) |
| O2$W$—H4$W$···N3$B^{vi}$ | 0.87 (1) | 2.56 (2) | 3.322 (2) | 148 (2) |
| O3$W$—H5$W$···N3$^{vi}$ | 0.87 (1) | 2.25 (2) | 3.036 (2) | 151 (3) |
| O3$W$—H6$W$···O3$B$ | 0.87 (1) | 2.15 (2) | 2.906 (2) | 145 (2) |
| O4$W$—H7$W$···O1 | 0.87 (1) | 2.03 (1) | 2.879 (2) | 166 (3) |
| O4$W$—H8$W$···O3$C^{iv}$ | 0.87 (1) | 2.02 (1) | 2.837 (2) | 156 (2) |
| O4$W$—H8$W$···O6$C^{iv}$ | 0.87 (1) | 2.27 (2) | 3.126 (16) | 170 (3) |
| O5$W$—H9$W$···O3$^{iv}$ | 0.87 (1) | 2.66 (2) | 3.376 (2) | 141 (2) |
| O5$W$—H10$W$···O3$A^{iv}$ | 0.89 (1) | 1.93 (1) | 2.807 (2) | 169 (2) |
| O6$W$—H11$W$···O5$W$ | 0.87 (1) | 2.11 (1) | 2.924 (2) | 158 (2) |
| O6$W$—H12$W$···O1$^{iv}$ | 0.86 (1) | 1.95 (1) | 2.7885 (19) | 164 (3) |
| O7$W$—H13$W$···O2$C^{v}$ | 0.87 (1) | 2.02 (1) | 2.896 (2) | 177 (3) |
| O7$W$—H13$W$···O5$C^{v}$ | 0.87 (1) | 1.88 (2) | 2.698 (12) | 156 (3) |
| O7$W$—H14$W$···O3$W^{v}$ | 0.88 (1) | 2.22 (1) | 3.085 (2) | 169 (2) |
| O8$W$—H15$W$···O2$A^{xi}$ | 0.88 (1) | 1.94 (1) | 2.808 (2) | 168 (2) |

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

**Table 11**
Selected bond lengths (Å) for K**4**.

| | | | |
|---|---|---|---|
| K1—O2$W^{i}$ | 2.738 (3) | K2—O5$^{v}$ | 2.794 (3) |
| K1—O1$A^{ii}$ | 2.771 (3) | K2—O1$A$ | 2.800 (2) |
| K1—O1$W$ | 2.772 (3) | K2—O2$A^{ii}$ | 2.821 (2) |
| K1—O2$A$ | 2.786 (2) | K2—O1 | 2.862 (2) |
| K1—O3$W^{iii}$ | 2.798 (3) | K2—O1$W^{ii}$ | 2.898 (3) |
| K1—O4$A^{iv}$ | 2.820 (3) | K2—O3$A$ | 2.947 (2) |
| K1—O3$A$ | 2.919 (2) | K2—O2 | 3.073 (3) |
| K2—O2$W^{i}$ | 2.753 (3) | | |

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $-x+1, -y+1, -z$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Table 7 to 14. K**5** is anhydrous and a $Z'$ = 1 structure, but all other K salts were isolated as hydrates and have $Z'$ = 2 (or 4 for K**3**); see Table 15 for a summary of these and other structural features. Values for the disordered anion of K**4** have been excluded from the geometric discussion below. The azo N═N bond lengths range from 1.258 (3) to 1.276 (3) Å, with the long value again found for the salt of **2** with its intramolecular O—H···N hydrogen bond and the possibility of tautomerization. The C—N bonds range from 1.420 (3) to 1.442 (4) and from 1.397 (3) to 1.432 (4) Å for the bonds to the sulfonated heads and to the phenol/amine tails, respectively. Again, it is K**2** that presents the most different bond lengths. All these bond lengths are similar to those found for the $NH_4$ salts, above, and for related sulfonated azo salt forms (Kennedy *et al.*, 2020). Most azo anions have planar geometries [angles between ring planes = 0.93 (7) to 8.15 (22)°], with the exception of K**1**, where the two independent azo anions are both twisted [angles between ring planes = 43.15 (7) and 40.48 (8)°]. This contrasts with the ammonium salt structures, where all species were planar except for the twisted $NH_4$**3**. As with the $NH_4$ species, all five K structures both planar and twisted have simple layered packing systems with layers of hydrophilic/inorganic species (K and water) alternating with layers of a hydrophobic/organic nature (azobenzene) (see Fig. 14 for an example).

Each K centre has a coordination number of 7 or 8 (see Table 15), with all bonds from K being to O atoms. All K centres interact with $SO_3$ groups and, where present, with water molecules. In K**1**, K**4** and the anhydrous K**5**, there are also bonds formed between K and the OH groups of the tails of the azo anions. No such K-to-tail bonds are formed to the OH groups of K**2** or to the $NH_2$ groups of K**3**, instead these form only hydrogen bonds (Table 10). This throws up a similarity to the $NH_4$ structures, where no direct $NH_4$-to-tail hydrogen bond was found for $NH_4$**2** or $NH_4$**3**, despite such bonds existing in all other compounds. As would be expected from the positions of the sulfonate groups and the nature of the cation, all five potassium salts form structures described elsewhere as class three 'higher connectivity' azo colourant structures (Kennedy *et al.*, 2004). Structures K**1** and K**5** both form three-dimensional coordination polymers, with K—O bonds (K-to-$SO_3$ in K**5** and both K-to-$SO_3$ and K-to-$OH_2$ in K**1**) forming bridges between K centres and allowing propagation of the polymer in two dimensions, whilst the third dimension features bridging between K centres through the body of the azo ion and hence utilizing both the head and tail functional groups of the azo anions (Fig. 14). Despite also

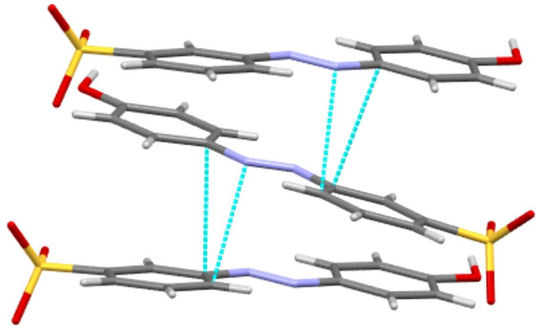


**Figure 8**
Part of azo stack propagating parallel to the *c* axis in the structure of $NH_4$**5**.

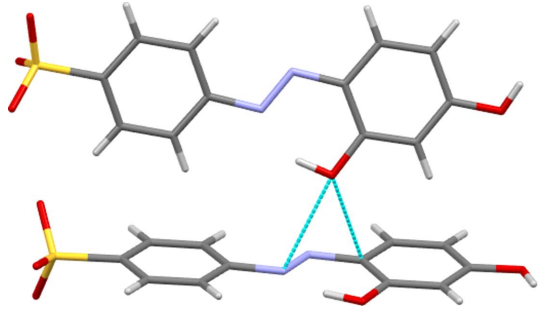


**Figure 9**
Dimeric close contact between the two azo anions of the asymmetric unit of $NH_4$**2**.

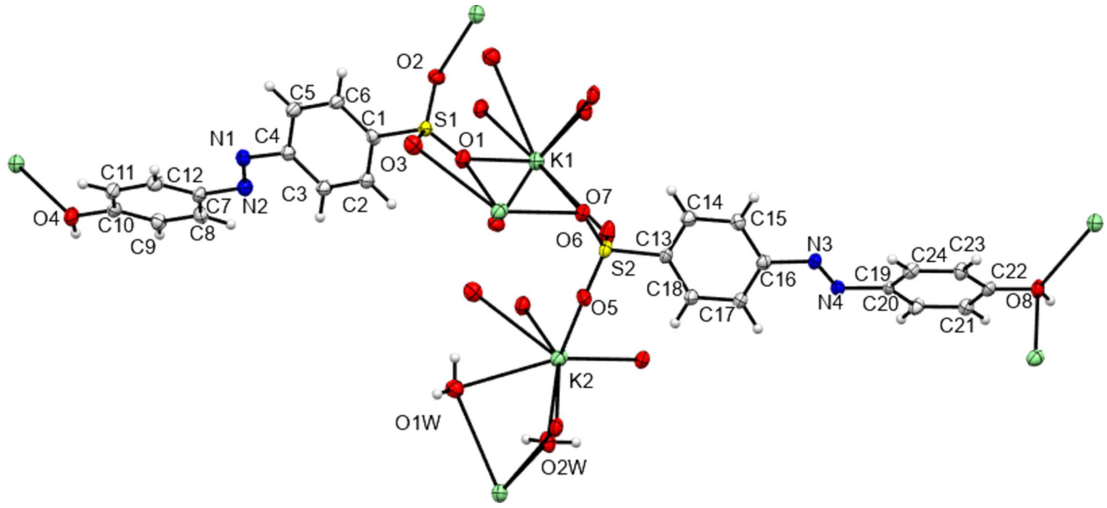



**Figure 10**
The contents of the asymmetric unit of K**1** extended so as to show the coordination geometry about each independent K centre.

**Table 12**
Hydrogen-bond geometry (Å, °) for K**4**.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H1*H*···O4*W* | 0.87 (1) | 1.84 (1) | 2.703 (4) | 171 (4) |
| O5—H2*H*···O4 | 0.88 (1) | 1.85 (1) | 2.719 (3) | 173 (4) |
| O4*A*—H3*H*···O5*A* | 0.87 (1) | 1.86 (2) | 2.695 (3) | 161 (3) |
| O5*A*—H4*H*···O3*W* | 0.88 (1) | 1.93 (2) | 2.738 (3) | 154 (3) |
| O1*W*—H1*W*···O5*A*$^{iii}$ | 0.87 (1) | 2.22 (2) | 3.017 (3) | 151 (3) |
| O1*W*—H2*W*···O4$^{iv}$ | 0.87 (1) | 2.08 (1) | 2.906 (3) | 158 (3) |
| O2*W*—H3*W*···O5$^{v}$ | 0.87 (1) | 1.93 (1) | 2.791 (4) | 170 (4) |
| O2*W*—H4*W*···O2 | 0.87 (1) | 1.99 (2) | 2.795 (3) | 153 (3) |
| O3*W*—H5*W*···O3$^{vi}$ | 0.87 (1) | 1.93 (1) | 2.786 (3) | 167 (3) |
| O3*W*—H6*W*···O4*W*$^{vii}$ | 0.88 (1) | 1.99 (1) | 2.821 (4) | 158 (3) |
| O4*W*—H7*W*···O4*A*$^{viii}$ | 0.88 (1) | 1.94 (1) | 2.789 (3) | 163 (3) |
| O4*W*—H8*W*···O1$^{vi}$ | 0.88 (1) | 1.86 (1) | 2.738 (3) | 180 (4) |

Symmetry codes: (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (vi) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vii) $-x+1, -y+1, -z+1$; (viii) $x+1, y, z$.

**Table 13**
Selected bond lengths (Å) for K**5**.

| | | | |
|---|---|---|---|
| K1—O2$^{i}$ | 2.7117 (18) | K1—O1 | 2.863 (2) |
| K1—O3$^{ii}$ | 2.7511 (18) | K1—O2 | 2.970 (2) |
| K1—O4$^{iii}$ | 2.7954 (19) | K1—O4$^{v}$ | 3.0023 (19) |
| K1—O3$^{iv}$ | 2.815 (2) | | |

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $x+\frac{1}{2}, -y+1, -z+\frac{1}{2}$; (v) $-x+\frac{3}{2}, -y+\frac{1}{2}, z$.

featuring bridges between K centres *via* head-to-tail contacts with the azo anion, K**4** is only a two-dimensional coordination polymer. Here, $SO_3$ and $OH_2$ bridges between K centres lead only to discrete $K_4$ tetramers and it is only the through-azo head-to-tail interactions that allow the coordination polymer to propagate parallel to the *b* and *c* directions (Fig. 15). With

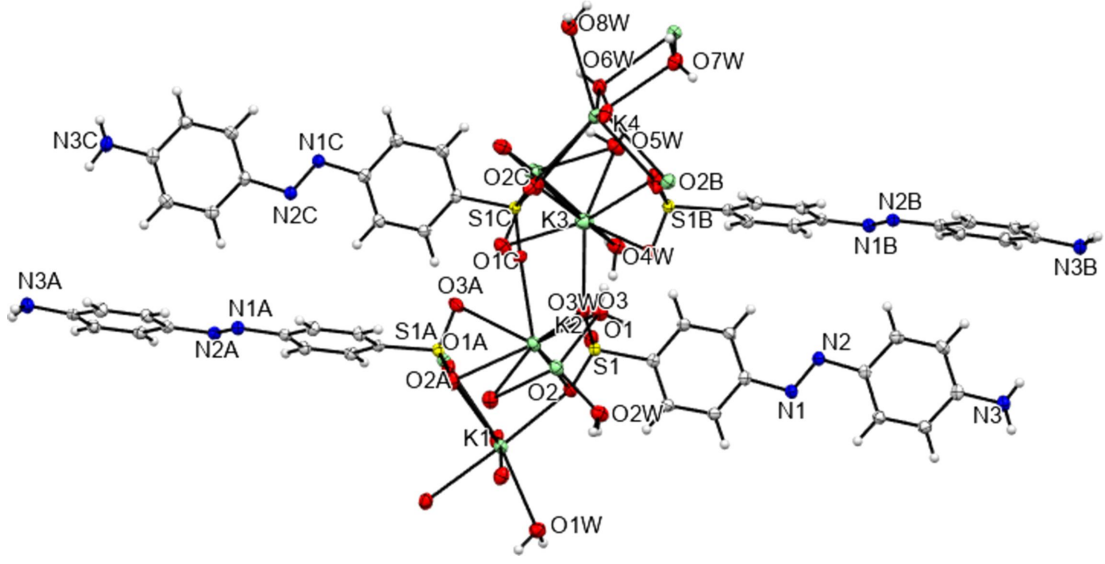



**Figure 11**
The contents of the asymmetric unit of K**3** extended so as to show the coordination geometry about each independent K centre. Disorder in the $SO_3$ group of atom S1*C* is not shown for clarity.

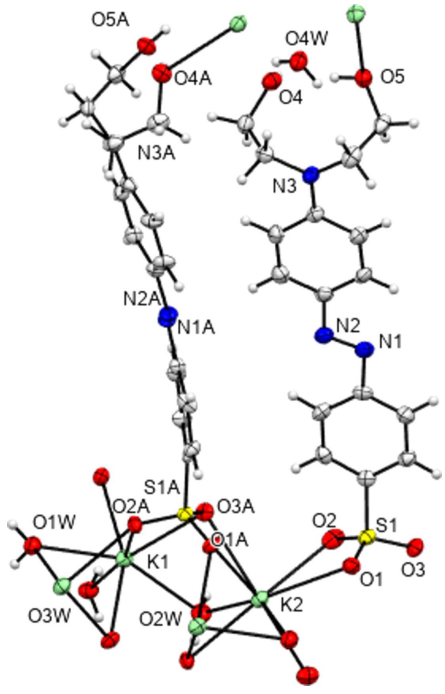



**Figure 12**
The contents of the asymmetric unit of K**4** extended so as to show the coordination geometry about each independent K centre. Disorder in the azo ion of atom N1*A* is not shown for clarity.

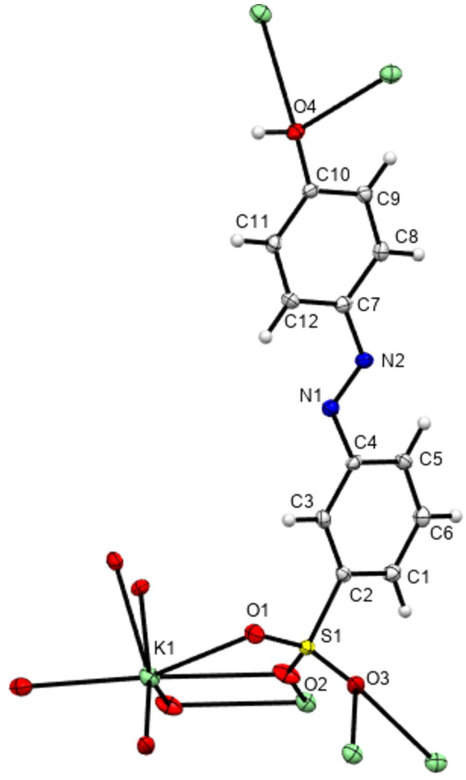



**Figure 13**
The contents of the asymmetric unit of K**5** extended so as to show the coordination geometry about the K centre.

**Table 14**
Hydrogen-bond geometry (Å, °) for K**5**.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H1*H*···O1$^{v}$ | 0.87 (1) | 2.02 (3) | 2.732 (3) | 139 (3) |

Symmetry code: (v) $-x+\frac{3}{2}, -y+\frac{1}{2}, z$.

no interaction with the amine tail of the azo anion, K**3** displays a two-dimensional coordination polymer structure based solely on $SO_3$ and $OH_2$ bridges between K centres. Here, the polymeric structure propagates parallel to the *a* and *b* directions (Fig. 16). K**2** also does not feature any K-to-tail interactions and here only a one-dimensional coordination polymer is formed. $SO_3$ and $OH_2$ bridges between K centres leads to a polymer that propagates parallel to the crystallographic *a* direction (Fig. 17).

The structures of the five Na salt equivalents of **1** to **5** crystallized from water solutions are all available from the literature (Kennedy *et al.*, 2001, 2020; Dodds *et al.*, 2017). As summarized in Table 15, Na**1** forms a one-dimensional coordination polymer, whilst the other four Na structures all form

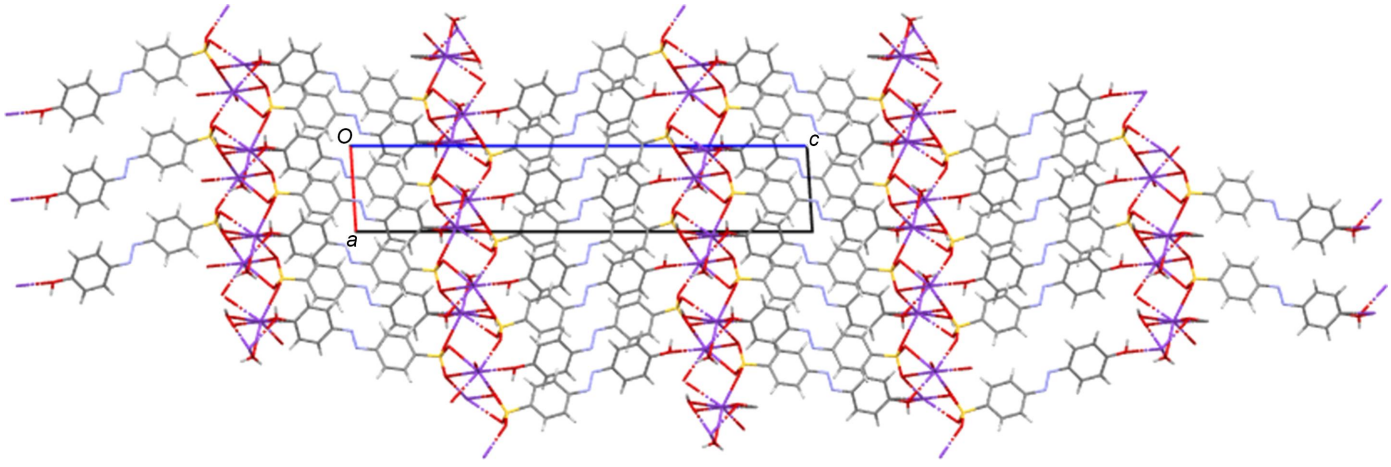



**Figure 14**
Packing diagram of K**1** as viewed along the *b* axis. Note the alternating hydrophobic and hydrophilic layers that propagate parallel to the *ab* plane. The three-dimensional coordination polymer propagates parallel to *a* and *b* (and throughout the hydrophilic layers) *via* sulfonate and water bridges between the K centres. The coordination polymer propagates parallel to *c* *via* K bonding to both the sulfonate head and the OH tail of the azo anions.

**Table 15**
Selected crystallographic parameters and bonding characteristics.

| | $NH_4$**1** | Na**1** | K**1** | $NH_4$**2** | Na**2** | K**2** | $NH_4$**3** | Na**3** | K**3** |
|---|---|---|---|---|---|---|---|---|---|
| CN | 6 | 6 | 8, 7 | 6, 6 | 6 | 7, 7 | 6 | 6 | 7,8,8,8 |
| CPD | | 1 | 3 | | 2 | 1 | | 2 | 2 |
| $H_2O$ | 0 | 2 | 1 | 2 | 2.5 | 2 | 1.5 | 2 | 2 |
| Cation-to-$SO_3$ interaction | yes | yes | yes | yes | yes | yes | yes | yes | yes |
| Cation-to-tail interaction | yes | no | yes | no | no | no | no | yes | no |
| Cation-to-water interaction | no | yes | yes | yes | yes | yes | yes | yes | yes |
| Space group | $P2_1$ | *Pbcn* | $P\overline{1}$ | $P\overline{1}$ | *C*2/*c* | $P\overline{1}$ | *C*2/*c* | $P2_1$ | $P\overline{1}$ |
| *a* (Å) | 5.8163 (1) | 14.383 | 5.9620 (7) | 8.2876 (1) | 35.036 | 8.177 | 35.1636 (15) | 7.829 | 13.3058 (2) |
| *b* (Å) | 6.9218 (1) | 5.813 | 7.2033 (11) | 10.6404 (1) | 5.410 | 10.529 | 7.8905 (3) | 5.784 | 13.6247 (2) |
| *c* (Å) | 15.6295 (2) | 32.891 | 31.929 (5) | 17.4834 (3) | 15.978 | 17.483 | 10.4972 (5) | 16.486 | 18.4664 (3) |
| $\alpha$ (°) | 90 | 90 | 83.852 (14) | 89.734 (1) | 90 | 89.82 | 90 | 90 | 88.373 (1) |
| $\beta$ (°) | 93.354 (1) | 90 | 86.361 (15) | 84.793 (1) | 98.05 | 85.49 | 100.091 (2) | 98.62 | 73.971 (1) |
| $\gamma$ (°) | 90 | 90 | 88.868 (15) | 86.146 (1) | 90 | 86.81 | 90 | 90 | 66.313 (1) |
| *V*/*Z* (Å$^3$) | 314.07 | 343.75 | 340.12 | 382.98 | 374.85 | 374.57 | 358.43 | 369.05 | 366.69 |

| | $NH_4$**4** | Na**4** | K**4** | $NH_4$**5** | Na**5** | K**5** |
|---|---|---|---|---|---|---|
| CN | 5, 6 | 7,7 | 7, 8 | 7 | 6 | 7 |
| CPD | | 2 | 2 | | 2 | 3 |
| $H_2O$ | 1 | 1.5 | 2 | 0 | 2 | 0 |
| Cation-to-$SO_3$ interaction | yes | yes | yes | yes | yes | yes |
| Cation-to-tail interaction | yes | yes | yes | yes | yes | yes |
| Cation-to-water interaction | yes | yes | yes | no | yes | no |
| Space group | $P\overline{1}$ | $P\overline{1}$ | $P2_1/n$ | *Pccn* | *Pbca* | *Pccn* |
| *a* (Å) | 8.4930 (1) | 9.691 | 9.4006 (2) | 12.6590 (2) | 7.123 | 12.5535 (2) |
| *b* (Å) | 13.0980 (2) | 11.487 | 12.1583 (3) | 28.3600 (3) | 11.940 | 27.9698 (5) |
| *c* (Å) | 17.1660 (3) | 16.605 | 34.4743 (9) | 7.1270 (7) | 32.561 | 6.9982 (1) |
| $\alpha$ (°) | 90.970 (1) | 93.73 | 90 | 90 | 90 | 90 |
| $\beta$ (°) | 103.180 (1) | 92.76 | 95.496 (1) | 90 | 90 | 90 |
| $\gamma$ (°) | 95.132 (1) | 95.37 | 90 | 90 | 90 | 90 |
| *V*/*Z* (Å$^3$) | 462.61 | 458.04 | 490.27 | 319.82 | 346.14 | 307.15 |

Notes: CN = coordination number; CPD = coordination polymer dimensionality; *V*/*Z* = unit-cell volume divided by *Z*.

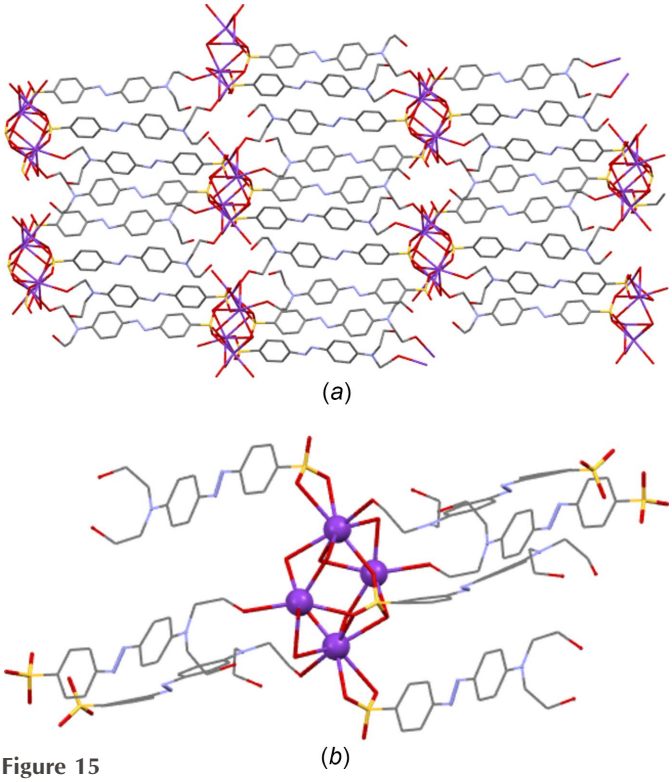



**Figure 15**
(*a*) View of K**4** illustrating that here K tetramers link into a two-dimensional coordination polymer only *via* through-azo interactions with the head and tail groups of the azo anions. (*b*) Detail of one $K_4$ tetrameric unit.

two-dimensional coordination polymers. All Na centres are six-coordinate, except for those in Na**4**, which are seven-coordinate. All Na centres bond to O atoms of the sulfonate groups and to water ligands. Na**1** and Na**2** do not form bonds from Na to the tail phenol groups, but the other three structures do form Na-to-tail bonds to N or to O atoms. The azo anions that form bonds to Na with their tail groups are thus

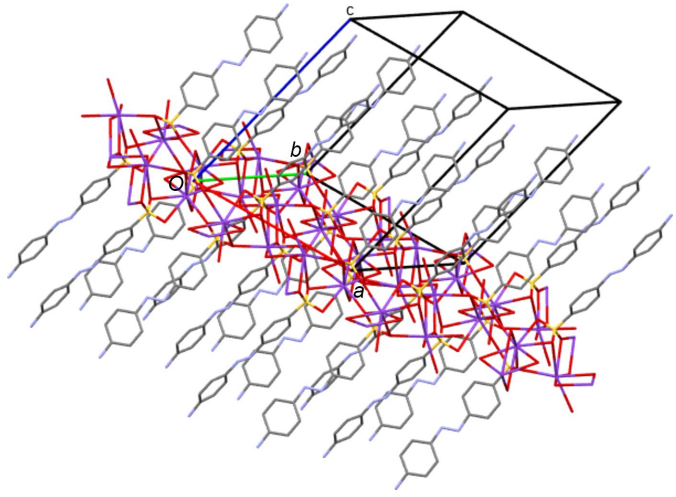



**Figure 16**
The two-dimensional coordination polymer of K**3** propagates only *via* sulfonate groups and water groups that bridge between K centres – there is no head-to-tail through-azo bridge.

**Table 16**
Unit-cell dimensions of selected salt forms of Orange G (OG).

Data taken from Ojala *et al.* (1994) and from Kennedy *et al.* (2006).

| | $NH_4$OG | AgOG | NaOG | Na/KOG | Na/RbOG |
|---|---|---|---|---|---|
| Space group | $P\bar{1}$ | $P\bar{1}$ | $P\bar{1}$ | $P\bar{1}$ | $P\bar{1}$ |
| $a$ (Å) | 9.165 | 8.870 | 8.900 | 8.991 | 9.025 |
| $b$ (Å) | 10.149 | 10.678 | 10.470 | 10.401 | 10.654 |
| $c$ (Å) | 12.623 | 13.273 | 13.735 | 14.996 | 15.077 |
| $\alpha$ (°) | 87.43 | 73.56 | 73.49 | 83.35 | 82.30 |
| $\beta$ (°) | 88.07 | 77.19 | 79.29 | 85.05 | 84.92 |
| $\gamma$ (°) | 71.00 | 71.66 | 69.50 | 70.23 | 69.39 |

different from those that interact with K or with $NH_4$, as described above. The generally lower coordination numbers of the Na salts (6 or 7) as compared to the K salts (7 or 8) seems to be reflected in a similarly generally lower dimensionality of coordination polymers (one- or two-dimensional for Na, and one- to three-dimensional for K), but this is not a hard and fast rule. This is shown by comparing Na**2** with K**2**, where Na has a coordination number of 6 *versus* 7 for K, but where the Na compound is a two-dimensional coordination polymer and the K compound only one-dimensional.

Comparing the unit-cell dimensions given in Table 15, it can clearly be seen that there are two isostructural pairs. The space group and unit-cell dimensions of $NH_4$**2** match those of K**2** and there is a similar match between $NH_4$**5** and K**5**. Based on the unit-cell dimensions, none of the Na species are isostructural with any $NH_4$ or K salt form. For the $NH_4$**5** and K**5** pair, all generalized structural descriptors (such as $Z'$, hydration state and the coordination number of the cation) are identical, as would be expected for a truly isostructural pair. However, the descriptors given for the $NH_4$**2** and K**2** pair in Table 15, whilst mostly identical, do differ with respect to the coordination number of the cation. The $NH_4$ ions are given as making six interactions each, whilst the K centres are given as each making seven interactions. Investigation shows that this

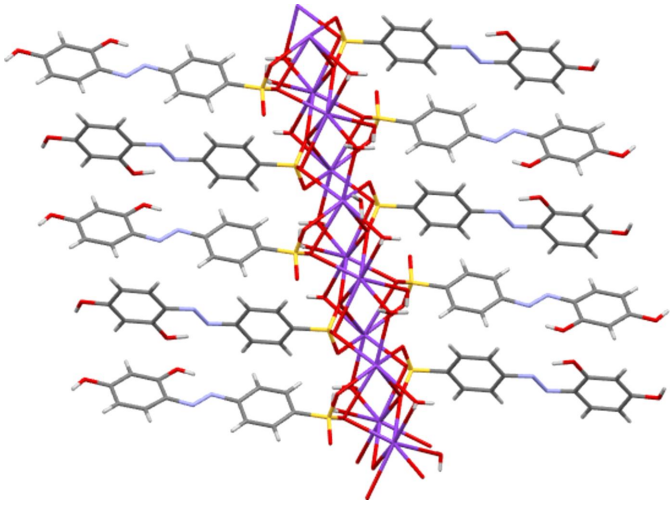


**Figure 17**
The one-dimensional coordination polymer structure of K**2**, with the coordination chain propagating parallel to the *a* direction (Kennedy *et al.*, 2004).

difference is not just a case of an inappropriate cut-off distance being used to calculate potential hydrogen-bonding interactions. For example, the 'extra' seventh bond for atom K1 is an interaction with a sulfonate O atom. The closest sulfonate O atom to the equivalent ammonium ion in $NH_4$**2**, that has not already been accounted for as a hydrogen-bonding interaction, gives an N···O distance of over 4.2 Å, which is clearly too long for a hydrogen bond; see Table 3 for genuine N···O hydrogen-bond distances. Thus, although $NH_4$**2** and K**2** have similar unit-cell dimensions and similar compositions, small changes in the orientations of the sulfonate groups and the cations allow the ammonium and potassium cations to make somewhat different interactions from each other. The ability of isostructural structures to tolerate small changes within a given packing motif has been discussed recently by Bombicz (2024).

Expanding from the 15 structures of Table 15 to other literature structures, of the five azo anions studied here, only **3** has had the structure of its Rb salt form reported (Kennedy *et al.*, 2004). The structure of Rb**3** is found to be isostructural with that of $NH_4$**3** (compare *C*2/*c*, 35.256, 7.738, 10.674 Å and 99.88° with the values given for $NH_4$**3** in Table 1). As with $NH_4$**2** and K**2** above, all structural descriptors match, except for the Rb cation having a higher coordination number than the ammonium cation (8 *versus* 6). Other sulfonated monoazo $NH_4$ salt form structures available are those of diammonium Orange G tetrahydrate and of the nitrile-substituted $[NH_4][O_3S(C_6H_4)NN(C_6H_4)NHCH_2CH_2CN]\cdot H_2O$ (Ojala *et al.*, 1994; Astbury *et al.*, 2013). For these azo anions, the only available comparisons for the ammonium salts are the Na and mixed Na/K and Na/Rb salt forms of Orange G and the Na salt of the nitrile (Kennedy *et al.*, 2006; Astbury *et al.*, 2013). Based on unit-cell dimensions, the nitrile shows no isostructurality. There are however similarities, if not exact matches, between the unit cells of the Orange G structures of Table 16; see comments below for a fuller discussion of these features.

The 'crystal packing similarity' tool available within *Mercury* (Macrae *et al.*, 2020) can be used to find packing similarity regardless of any matches in the unit-cell dimension. For instance, it has been used to find similarities in the packing behaviour of dopamine and tyramine fragments in the salt and hydrate forms of these active pharmaceutical ingredients (APIs), regardless of counter-ions, solvents and the identity and protonation state of the phenylethylamine fragment itself (Kennedy *et al.*, 2023). This tool was applied to the 15 structures of Table 15 and to the other relevant $NH_4$, Na, K, Rb, Cs and Ag salt structures found in the Cambridge Structural Database (CSD; Groom *et al.*, 2016). The packing analysis was set up to examine packed fragments consisting of 15 sulfonated azo molecular units. Other components (cations and solvent molecules) were ignored. Indeed, in order to simplify the definition of a 'molecular fragment', all metal ions were deleted from the CIF files used for these analyses. Only the three pairs previously identified as being isostructural from their unit-cell dimensions (*i.e.* $NH_4$**2** and K**2**, $NH_4$**5** and K**5**, and $NH_4$**3** and Rb**3**) had azo anion packing that matched at a 15/15 level with a 20% tolerance allowed. However, it was

found that $NH_4$**3** also matched at a 13/15 level with the isostructural pair of structures having CSD refcodes BAHNAC and BAHNIK (Dodds *et al.*, 2017). Surprisingly, these two structures are not forms of the *para*-$NH_2$-substituted anion **3**, but correspond to the Na and Ag salt forms of the *meta*-OH-substituted anion **5**. Further investigation of these structures reveals that it is their layered natures that lead to this partial match. Those azo fragments that lie within a single hydrophobic layer of $NH_4$**3** match well with their equivalents in Na**5** and Ag**5**, despite the differences in $SO_3$ position and the different chemical natures of the $NH_2$/OH tail groups. However, the azo fragments within the neighbouring hydrophobic layers do not match, being rotated with respect to one another (Fig. 18). A similar situation was found for the Orange G salts of Table 16. As is perhaps suggested by the differences in the unit-cell parameters, none of the metal salt forms were truly isostructural with the ammonium salt of Orange G. However, both the mixed Na/K and Na/Rb species matched the ammonium salt at a 12/15 level and the pure Na species matched at a 9/15 level. As with the case for **3** above, the matches within one organic layer are reasonable, but the structures do not match well when neighbouring layers are considered. Finally, examining the structures of Table 15 it can be seen that $NH_4$**1** and K**1** have somewhat similar *a* and *b* dimensions, whilst *c* is approximately doubled. Despite this, investigations showed that these structures did not show any significant similarity in the packing for the azo anions.

## 4. Summary

Five new structures of ammonium salt forms of sulfonated azo dyes have been presented and compared to the equivalent Na and K salt structures. Despite being based on ammonium-to-sulfonate hydrogen bonds rather than metal-ion-to-sulfonate bonds, the ammonium salt forms are found to have structural types that are in many ways similar to those found for heavier alkali-metal ions (Na, K and Rb). The ammonium structures have the same simple alternating hydrophobic/hydrophilic layer structures described earlier for *s*-block metal salt forms of *para*- and *meta*-sulfonated azo dyes (Kennedy *et al.*, 2004) and do not show the more complicated layering motifs seen elsewhere (Kennedy *et al.*, 2009). Two of the five ammonium structures ($NH_4$**2** and $NH_4$**5**) show isostructurality with their equivalent potassium structures. For anion **3**, the ammonium and Rb salt forms are also found to be isostructural, although unfortunately no other Rb salt structures are available for comparison. For the isostructural pair of **5**, all structural descriptors are very similar, but for the **2** and **3** pairs there are small differences in, for example, the rotation of the sulfonate groups that allow the ammonium cations to make less formal contacts than do the K or Rb ions. No sodium salt is found to be isostructural with an ammonium salt, but use of the 'crystal packing similarity' tool within *Mercury* did highlight that the azo anions of $NH_4$**3** do adopt similar packing to the chemically different azo anions of both Na**5** and Ag**5**, although this similarity only holds within a single hydrophobic layer, with neighbouring layers behaving differently. The ammonium salt of Orange G shows similar single-layer-matching behaviour with its Na and mixed Na/K and Na/Rb salt forms. Overall there is thus a high propensity for ammonium salt forms of sulfonated azo dyes to be isostructural with the equivalent K or Rb forms, a propensity which is aided by small amounts of flexibility in the structures that allows for the different coordinating abilities of the cations. Little evidence is found for isostructural relationships between the salt forms of sulfonated azo dyes of ammonium salts and the equivalent sodium salts. Here, the smaller radius of the $Na^+$ ion is perhaps hard to offset against the lower coordination number of sodium. However, the similarity of anion packing found, for instance, within single layers for $NH_4$**3** (and hence Rb**3**) with Na**5** and

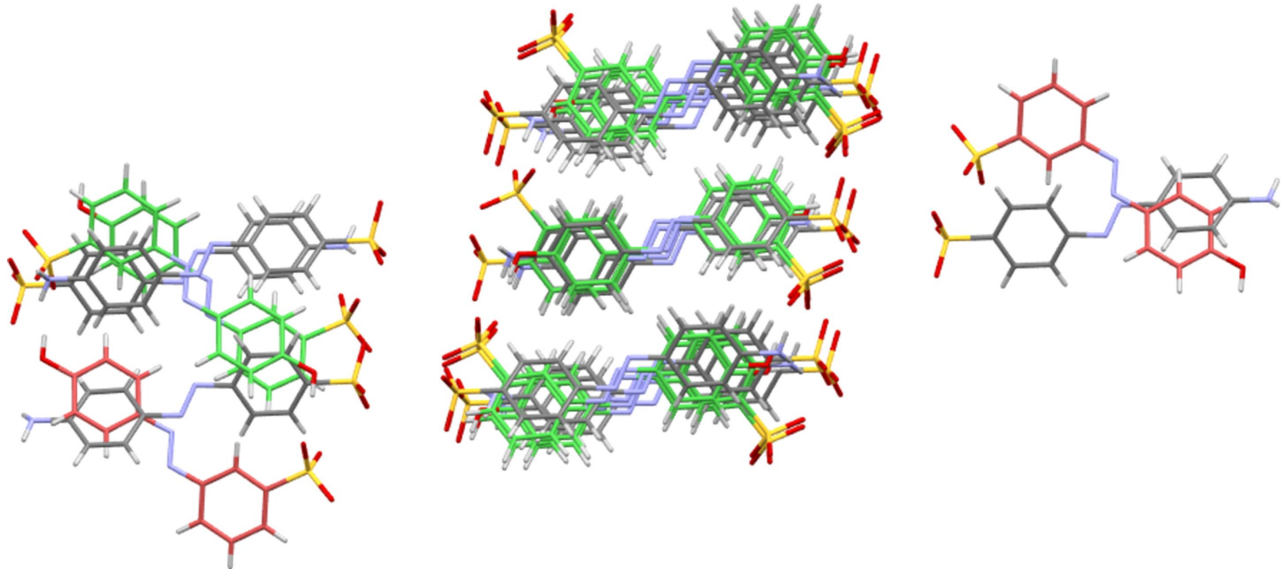


**Figure 18**
15-fragment overlay of the structure of $NH_4$**3** (multicoloured) with that of BAHNIK (Ag**5**, green and red; Dodds *et al.*, 2017). The anionic azo fragments in the central layer give reasonable packing matches for the azobenzene units, but the anions of the neighbouring layers do not match well.

Ag**5** may indicate that isostructurality is possible and may be found if a larger sample of structures was available.

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# supporting information

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## Isostructural behaviour in ammonium and potassium salt forms of sulfonated azo dyes

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### Computing details

#### Ammonium 4-[2-(4-hydroxyphenyl)diazen-1-yl]benzenesulfonate (NH41)

*Crystal data*

$NH_4^+ \cdot C_{12}H_9N_2O_4S^-$
$M_r$ = 295.31
Monoclinic, $P2_1$
$a$ = 5.8163 (1) Å
$b$ = 6.9218 (1) Å
$c$ = 15.6295 (2) Å
$\beta$ = 93.354 (1)°
$V$ = 628.15 (2) Å$^3$
$Z$ = 2
$F(000)$ = 308
$D_x$ = 1.561 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 12251 reflections
$\theta$ = 2.8–71.4°
$\mu$ = 2.48 mm$^{-1}$
$T$ = 100 K
Broken tablet, yellow
0.22 × 0.12 × 0.06 mm

*Data collection*

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Rigaku OD, 2019)
$T_{min}$ = 0.859, $T_{max}$ = 1.000
10144 measured reflections
2345 independent reflections
2336 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.054
$\theta_{max}$ = 71.8°, $\theta_{min}$ = 2.8°
$h$ = −7→7
$k$ = −8→8
$l$ = −18→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.084
$S$ = 1.08
2345 reflections
203 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.3416P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$
Extinction correction: SHELXL2018 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0059 (10)
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.00 (2)

*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**. Refined as a 2-component inversion twin.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.35361 (10) | 0.29158 (10) | 0.12661 (3) | 0.0123 (2) |
| O1 | 0.4130 (4) | 0.4665 (3) | 0.08089 (13) | 0.0173 (5) |
| O2 | 0.4932 (4) | 0.1276 (3) | 0.10136 (13) | 0.0182 (5) |
| O3 | 0.1084 (3) | 0.2481 (4) | 0.12008 (12) | 0.0230 (6) |
| O4 | 1.1582 (3) | 0.3583 (3) | 0.85733 (13) | 0.0162 (5) |
| N1 | 0.6429 (4) | 0.3448 (4) | 0.49689 (15) | 0.0138 (5) |
| N2 | 0.8571 (4) | 0.3609 (4) | 0.51273 (15) | 0.0138 (5) |
| N3 | −0.2044 (4) | 0.3231 (4) | −0.01967 (16) | 0.0159 (6) |
| C1 | 0.4303 (5) | 0.3311 (4) | 0.23627 (17) | 0.0134 (6) |
| C2 | 0.2832 (4) | 0.2720 (5) | 0.29837 (16) | 0.0127 (5) |
| H2 | 0.134187 | 0.223460 | 0.282173 | 0.015* |
| C3 | 0.3571 (4) | 0.2850 (5) | 0.38439 (15) | 0.0127 (5) |
| H3 | 0.256899 | 0.248354 | 0.427438 | 0.015* |
| C4 | 0.5780 (5) | 0.3516 (4) | 0.40746 (18) | 0.0130 (6) |
| C5 | 0.7218 (5) | 0.4191 (4) | 0.34458 (17) | 0.0126 (6) |
| H5 | 0.868874 | 0.471605 | 0.360730 | 0.015* |
| C6 | 0.6473 (5) | 0.4084 (4) | 0.25935 (18) | 0.0130 (6) |
| H6 | 0.743184 | 0.453318 | 0.216382 | 0.016* |
| C7 | 0.9258 (5) | 0.3512 (4) | 0.60152 (17) | 0.0123 (6) |
| C8 | 1.1468 (5) | 0.4181 (4) | 0.62395 (17) | 0.0132 (6) |
| H8 | 1.243125 | 0.460308 | 0.580605 | 0.016* |
| C9 | 1.2272 (5) | 0.4236 (4) | 0.70920 (17) | 0.0135 (6) |
| H9 | 1.376389 | 0.472961 | 0.724518 | 0.016* |
| C10 | 1.0875 (5) | 0.3560 (4) | 0.77230 (17) | 0.0127 (6) |
| C11 | 0.8704 (4) | 0.2800 (5) | 0.74958 (16) | 0.0142 (5) |
| H11 | 0.778425 | 0.229619 | 0.792613 | 0.017* |
| C12 | 0.7882 (4) | 0.2777 (5) | 0.66469 (16) | 0.0139 (5) |
| H12 | 0.639877 | 0.226653 | 0.649335 | 0.017* |
| H1H | 1.268 (8) | 0.444 (8) | 0.861 (3) | 0.037 (12)* |
| H1N | −0.260 (7) | 0.214 (7) | −0.038 (2) | 0.018 (9)* |
| H2N | −0.114 (7) | 0.362 (6) | −0.060 (3) | 0.025 (10)* |
| H3N | −0.122 (8) | 0.301 (8) | 0.027 (3) | 0.043 (12)* |
| H4N | −0.312 (9) | 0.414 (8) | −0.008 (3) | 0.051 (14)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0123 (3) | 0.0150 (3) | 0.0093 (3) | 0.0001 (3) | −0.0010 (2) | 0.0009 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O1 | 0.0233 (10) | 0.0151 (11) | 0.0137 (9) | 0.0022 (9) | 0.0038 (8) | 0.0041 (8) |
| O2 | 0.0259 (11) | 0.0153 (11) | 0.0131 (10) | 0.0039 (9) | −0.0018 (8) | −0.0026 (8) |
| O3 | 0.0152 (10) | 0.0391 (16) | 0.0142 (9) | −0.0059 (10) | −0.0029 (7) | 0.0036 (10) |
| O4 | 0.0157 (10) | 0.0205 (11) | 0.0121 (9) | −0.0028 (9) | −0.0014 (7) | −0.0014 (8) |
| N1 | 0.0132 (11) | 0.0146 (13) | 0.0133 (11) | −0.0003 (9) | −0.0003 (9) | −0.0014 (9) |
| N2 | 0.0137 (11) | 0.0146 (11) | 0.0130 (11) | −0.0006 (10) | −0.0003 (9) | −0.0005 (9) |
| N3 | 0.0156 (11) | 0.0176 (16) | 0.0142 (12) | −0.0015 (10) | −0.0007 (10) | 0.0003 (10) |
| C1 | 0.0140 (12) | 0.0154 (16) | 0.0108 (12) | 0.0033 (10) | 0.0000 (9) | −0.0001 (10) |
| C2 | 0.0114 (11) | 0.0126 (14) | 0.0139 (12) | 0.0005 (12) | −0.0011 (9) | −0.0001 (12) |
| C3 | 0.0139 (11) | 0.0117 (12) | 0.0128 (11) | 0.0023 (13) | 0.0034 (9) | 0.0006 (12) |
| C4 | 0.0151 (13) | 0.0130 (13) | 0.0109 (12) | 0.0015 (11) | 0.0014 (10) | −0.0009 (10) |
| C5 | 0.0118 (12) | 0.0107 (14) | 0.0154 (13) | −0.0002 (11) | 0.0006 (10) | −0.0009 (11) |
| C6 | 0.0124 (12) | 0.0119 (14) | 0.0150 (13) | −0.0017 (11) | 0.0036 (10) | 0.0010 (11) |
| C7 | 0.0130 (12) | 0.0127 (14) | 0.0112 (12) | 0.0012 (11) | 0.0002 (10) | −0.0019 (10) |
| C8 | 0.0131 (12) | 0.0122 (14) | 0.0144 (13) | 0.0007 (11) | 0.0026 (10) | −0.0006 (11) |
| C9 | 0.0094 (12) | 0.0152 (15) | 0.0158 (13) | −0.0012 (11) | 0.0002 (10) | −0.0016 (12) |
| C10 | 0.0162 (13) | 0.0114 (13) | 0.0105 (12) | 0.0017 (11) | −0.0001 (10) | −0.0021 (10) |
| C11 | 0.0144 (12) | 0.0151 (14) | 0.0135 (11) | −0.0001 (13) | 0.0025 (9) | 0.0008 (13) |
| C12 | 0.0132 (12) | 0.0137 (14) | 0.0147 (12) | −0.0017 (13) | 0.0007 (9) | −0.0017 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O3 | 1.456 (2) | C3—C4 | 1.392 (4) |
| S1—O1 | 1.457 (2) | C3—H3 | 0.9500 |
| S1—O2 | 1.463 (2) | C4—C5 | 1.407 (4) |
| S1—C1 | 1.767 (3) | C5—C6 | 1.379 (4) |
| O4—C10 | 1.368 (3) | C5—H5 | 0.9500 |
| O4—H1H | 0.87 (5) | C6—H6 | 0.9500 |
| N1—N2 | 1.261 (3) | C7—C8 | 1.392 (4) |
| N1—C4 | 1.427 (4) | C7—C12 | 1.402 (4) |
| N2—C7 | 1.423 (4) | C8—C9 | 1.386 (4) |
| N3—H1N | 0.86 (4) | C8—H8 | 0.9500 |
| N3—H2N | 0.88 (4) | C9—C10 | 1.395 (4) |
| N3—H3N | 0.86 (5) | C9—H9 | 0.9500 |
| N3—H4N | 0.91 (5) | C10—C11 | 1.394 (4) |
| C1—C2 | 1.392 (4) | C11—C12 | 1.384 (3) |
| C1—C6 | 1.399 (4) | C11—H11 | 0.9500 |
| C2—C3 | 1.390 (3) | C12—H12 | 0.9500 |
| C2—H2 | 0.9500 | | |

| | | | |
|---|---|---|---|
| O3—S1—O1 | 113.38 (13) | C5—C4—N1 | 124.1 (3) |
| O3—S1—O2 | 112.19 (14) | C6—C5—C4 | 119.4 (3) |
| O1—S1—O2 | 111.15 (12) | C6—C5—H5 | 120.3 |
| O3—S1—C1 | 106.90 (12) | C4—C5—H5 | 120.3 |
| O1—S1—C1 | 107.07 (13) | C5—C6—C1 | 119.8 (3) |
| O2—S1—C1 | 105.62 (12) | C5—C6—H6 | 120.1 |
| C10—O4—H1H | 105 (3) | C1—C6—H6 | 120.1 |
| N2—N1—C4 | 113.0 (2) | C8—C7—C12 | 120.0 (2) |

| | | | |
|---|---|---|---|
| N1—N2—C7 | 113.9 (2) | C8—C7—N2 | 115.6 (2) |
| H1N—N3—H2N | 105 (4) | C12—C7—N2 | 124.4 (2) |
| H1N—N3—H3N | 108 (4) | C9—C8—C7 | 120.4 (2) |
| H2N—N3—H3N | 109 (4) | C9—C8—H8 | 119.8 |
| H1N—N3—H4N | 115 (4) | C7—C8—H8 | 119.8 |
| H2N—N3—H4N | 112 (4) | C8—C9—C10 | 119.5 (3) |
| H3N—N3—H4N | 107 (4) | C8—C9—H9 | 120.2 |
| C2—C1—C6 | 121.0 (2) | C10—C9—H9 | 120.2 |
| C2—C1—S1 | 120.0 (2) | O4—C10—C11 | 117.9 (2) |
| C6—C1—S1 | 118.8 (2) | O4—C10—C9 | 121.9 (3) |
| C3—C2—C1 | 119.2 (2) | C11—C10—C9 | 120.2 (2) |
| C3—C2—H2 | 120.4 | C12—C11—C10 | 120.4 (2) |
| C1—C2—H2 | 120.4 | C12—C11—H11 | 119.8 |
| C2—C3—C4 | 120.0 (2) | C10—C11—H11 | 119.8 |
| C2—C3—H3 | 120.0 | C11—C12—C7 | 119.4 (2) |
| C4—C3—H3 | 120.0 | C11—C12—H12 | 120.3 |
| C3—C4—C5 | 120.4 (3) | C7—C12—H12 | 120.3 |
| C3—C4—N1 | 115.5 (2) | | |

| | | | |
|---|---|---|---|
| C4—N1—N2—C7 | −179.0 (2) | C4—C5—C6—C1 | 0.1 (4) |
| O3—S1—C1—C2 | 15.9 (3) | C2—C1—C6—C5 | 2.8 (4) |
| O1—S1—C1—C2 | 137.7 (2) | S1—C1—C6—C5 | −172.3 (2) |
| O2—S1—C1—C2 | −103.8 (2) | N1—N2—C7—C8 | −163.7 (3) |
| O3—S1—C1—C6 | −169.0 (2) | N1—N2—C7—C12 | 17.3 (4) |
| O1—S1—C1—C6 | −47.2 (3) | C12—C7—C8—C9 | −3.9 (4) |
| O2—S1—C1—C6 | 71.3 (3) | N2—C7—C8—C9 | 177.0 (3) |
| C6—C1—C2—C3 | −2.0 (4) | C7—C8—C9—C10 | 1.9 (4) |
| S1—C1—C2—C3 | 173.0 (2) | C8—C9—C10—O4 | −179.9 (3) |
| C1—C2—C3—C4 | −1.7 (5) | C8—C9—C10—C11 | 1.3 (4) |
| C2—C3—C4—C5 | 4.5 (5) | O4—C10—C11—C12 | 178.7 (3) |
| C2—C3—C4—N1 | −175.2 (3) | C9—C10—C11—C12 | −2.4 (5) |
| N2—N1—C4—C3 | 163.8 (3) | C10—C11—C12—C7 | 0.4 (5) |
| N2—N1—C4—C5 | −16.0 (4) | C8—C7—C12—C11 | 2.7 (4) |
| C3—C4—C5—C6 | −3.8 (4) | N2—C7—C12—C11 | −178.3 (3) |
| N1—C4—C5—C6 | 176.0 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H4*N*···O2$^{i}$ | 0.91 (5) | 2.30 (5) | 2.940 (4) | 127 (4) |
| N3—H4*N*···O1$^{ii}$ | 0.91 (5) | 2.21 (5) | 2.969 (3) | 141 (4) |
| N3—H3*N*···O3 | 0.86 (5) | 1.96 (5) | 2.808 (3) | 170 (4) |
| N3—H1*N*···O1$^{iii}$ | 0.86 (4) | 2.03 (5) | 2.889 (4) | 176 (4) |
| N3—H2*N*···O4$^{iv}$ | 0.88 (4) | 2.10 (4) | 2.946 (3) | 159 (4) |
| O4—H1*H*···O2$^{v}$ | 0.87 (5) | 1.95 (5) | 2.802 (3) | 167 (4) |

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

**Ammonium 4-[2-(2,4-dihydroxyphenyl)diazen-1-yl]benzenesulfonate dihydrate (NH42)**

*Crystal data*

$NH_4^+ \cdot C_{12}H_9N_2O_5S^- \cdot 2H_2O$
$M_r = 347.34$
Triclinic, $P\overline{1}$
$a = 8.2876$ (1) Å
$b = 10.6404$ (1) Å
$c = 17.4834$ (3) Å
$\alpha = 89.734$ (1)°
$\beta = 84.793$ (1)°
$\gamma = 86.146$ (1)°
$V = 1531.91$ (4) Å$^3$
$Z = 4$
$F(000) = 728$
$D_x = 1.506$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8081 reflections
$\theta = 1.0$–$29.1°$
$\mu = 0.25$ mm$^{-1}$
$T = 123$ K
Rhomb, yellow-orange
0.42 × 0.40 × 0.10 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
phi and $\omega$ scans
15978 measured reflections
8209 independent reflections
6704 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 29.1°$, $\theta_{min} = 1.2°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -23 \rightarrow 23$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.094$
$S = 1.02$
8209 reflections
495 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.8626P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.58$ e Å$^{-3}$
$\Delta\rho_{min} = -0.41$ 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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.62192 (4) | 0.04619 (3) | 0.10240 (2) | 0.01709 (8) |
| S2 | 0.09636 (4) | 0.38246 (3) | 0.12364 (2) | 0.01713 (8) |
| O1 | 0.76850 (13) | −0.01171 (10) | 0.06027 (6) | 0.0214 (2) |
| O2 | 0.48499 (13) | −0.03156 (11) | 0.09970 (6) | 0.0271 (2) |
| O3 | 0.58719 (14) | 0.17567 (10) | 0.07836 (6) | 0.0260 (2) |
| O4 | 0.98516 (14) | 0.14681 (10) | 0.75672 (6) | 0.0219 (2) |
| O5 | 0.71395 (13) | −0.04078 (10) | 0.56317 (6) | 0.0222 (2) |
| O6 | 0.07572 (14) | 0.25449 (10) | 0.09790 (6) | 0.0242 (2) |
| O7 | 0.22672 (14) | 0.43876 (10) | 0.07644 (6) | 0.0250 (2) |
| O8 | −0.05487 (13) | 0.46083 (11) | 0.12893 (6) | 0.0270 (2) |
| O9 | 0.52842 (13) | 0.41871 (10) | 0.76776 (6) | 0.0218 (2) |

| | | | | |
|---|---|---|---|---|
| O10 | 0.51487 (14) | 0.23392 (10) | 0.52554 (6) | 0.0245 (2) |
| O1W | −0.16671 (16) | 0.71210 (13) | 0.15040 (8) | 0.0322 (3) |
| O2W | 0.38515 (14) | 0.56714 (12) | 0.88010 (7) | 0.0275 (2) |
| O3W | 0.71337 (14) | 0.32285 (11) | 0.88635 (7) | 0.0260 (2) |
| O4W | 0.84097 (16) | −0.00710 (11) | 0.85537 (7) | 0.0255 (2) |
| N1 | 0.76041 (15) | 0.05814 (11) | 0.43032 (7) | 0.0187 (2) |
| N2 | 0.85430 (15) | 0.13819 (11) | 0.45120 (7) | 0.0193 (2) |
| N3 | 0.33504 (14) | 0.35053 (11) | 0.43444 (7) | 0.0176 (2) |
| N4 | 0.28336 (14) | 0.43660 (11) | 0.48284 (7) | 0.0174 (2) |
| N5 | 0.93365 (17) | 0.16781 (13) | −0.03240 (7) | 0.0194 (2) |
| N6 | −0.39612 (17) | 0.72023 (14) | 0.04186 (8) | 0.0233 (3) |
| C1 | 0.66330 (17) | 0.05333 (13) | 0.19978 (7) | 0.0162 (3) |
| C2 | 0.77120 (17) | 0.13898 (13) | 0.22148 (8) | 0.0192 (3) |
| H1 | 0.819914 | 0.193701 | 0.184416 | 0.023* |
| C3 | 0.80712 (18) | 0.14393 (13) | 0.29746 (8) | 0.0193 (3) |
| H2 | 0.880801 | 0.201593 | 0.312773 | 0.023* |
| C4 | 0.73336 (17) | 0.06287 (13) | 0.35112 (8) | 0.0179 (3) |
| C5 | 0.62652 (18) | −0.02228 (14) | 0.32902 (8) | 0.0201 (3) |
| H3 | 0.577284 | −0.076857 | 0.366013 | 0.024* |
| C6 | 0.59143 (18) | −0.02792 (14) | 0.25305 (8) | 0.0196 (3) |
| 4H | 0.519150 | −0.086608 | 0.237650 | 0.023* |
| C7 | 0.88081 (17) | 0.13237 (13) | 0.52941 (8) | 0.0181 (3) |
| C8 | 0.98383 (18) | 0.21938 (14) | 0.55419 (8) | 0.0213 (3) |
| H5 | 1.030580 | 0.276831 | 0.518260 | 0.026* |
| C9 | 1.01927 (18) | 0.22375 (14) | 0.62985 (8) | 0.0207 (3) |
| H6 | 1.090060 | 0.283074 | 0.645886 | 0.025* |
| C10 | 0.94882 (17) | 0.13902 (13) | 0.68248 (8) | 0.0180 (3) |
| C11 | 0.84685 (17) | 0.05103 (13) | 0.65983 (8) | 0.0184 (3) |
| H8 | 0.800804 | −0.005918 | 0.696292 | 0.022* |
| C12 | 0.81178 (17) | 0.04618 (13) | 0.58317 (8) | 0.0175 (3) |
| C13 | 0.15837 (16) | 0.37139 (13) | 0.21753 (8) | 0.0166 (3) |
| C14 | 0.08726 (18) | 0.45338 (14) | 0.27459 (8) | 0.0219 (3) |
| H10 | 0.001186 | 0.512467 | 0.263835 | 0.026* |
| C15 | 0.14229 (18) | 0.44866 (14) | 0.34716 (8) | 0.0221 (3) |
| H11 | 0.094605 | 0.504738 | 0.386260 | 0.027* |
| C16 | 0.26783 (17) | 0.36126 (13) | 0.36236 (8) | 0.0169 (3) |
| C17 | 0.33736 (18) | 0.27771 (13) | 0.30555 (8) | 0.0195 (3) |
| H12 | 0.421320 | 0.217035 | 0.316760 | 0.023* |
| C18 | 0.28372 (17) | 0.28334 (13) | 0.23264 (8) | 0.0195 (3) |
| H13 | 0.331939 | 0.227749 | 0.193401 | 0.023* |
| C19 | 0.34935 (16) | 0.42642 (13) | 0.55348 (8) | 0.0162 (3) |
| C20 | 0.29766 (17) | 0.52133 (13) | 0.60747 (8) | 0.0188 (3) |
| H14 | 0.222998 | 0.587727 | 0.594106 | 0.023* |
| C21 | 0.35318 (18) | 0.52007 (14) | 0.67955 (8) | 0.0196 (3) |
| H15 | 0.314923 | 0.583618 | 0.716005 | 0.023* |
| C22 | 0.46707 (17) | 0.42355 (13) | 0.69833 (8) | 0.0172 (3) |
| C23 | 0.52292 (18) | 0.32983 (13) | 0.64569 (8) | 0.0189 (3) |
| H17 | 0.601645 | 0.266207 | 0.658768 | 0.023* |

| | | | | |
|---|---|---|---|---|
| C24 | 0.46361 (17) | 0.32899 (13) | 0.57373 (8) | 0.0172 (3) |
| H1H | 0.939 (3) | 0.092 (2) | 0.7835 (12) | 0.037 (6)* |
| H2H | 0.708 (3) | −0.022 (2) | 0.5086 (15) | 0.062 (7)* |
| H3H | 0.477 (3) | 0.473 (2) | 0.7984 (13) | 0.042 (6)* |
| H4H | 0.464 (3) | 0.254 (2) | 0.4811 (15) | 0.060 (7)* |
| H1N | 1.013 (2) | 0.1347 (18) | −0.0556 (11) | 0.023 (5)* |
| H2N | 0.871 (3) | 0.109 (2) | −0.0084 (13) | 0.042 (6)* |
| H3N | 0.868 (3) | 0.218 (2) | −0.0635 (13) | 0.045 (6)* |
| H4N | 0.966 (3) | 0.216 (2) | 0.0036 (14) | 0.047 (6)* |
| H5N | −0.354 (3) | 0.663 (2) | 0.0065 (13) | 0.040 (6)* |
| H6N | −0.329 (3) | 0.7249 (19) | 0.0805 (13) | 0.040 (6)* |
| H7N | −0.495 (3) | 0.697 (2) | 0.0640 (13) | 0.042 (6)* |
| H8N | −0.412 (3) | 0.790 (2) | 0.0202 (13) | 0.043 (6)* |
| H1W | −0.123 (3) | 0.640 (3) | 0.1495 (14) | 0.055 (7)* |
| H2W | −0.131 (3) | 0.754 (2) | 0.1849 (15) | 0.055 (7)* |
| H3W | 0.403 (3) | 0.641 (2) | 0.8893 (14) | 0.050 (7)* |
| H4W | 0.2803 (12) | 0.566 (2) | 0.8824 (13) | 0.048 (6)* |
| H5W | 0.733 (3) | 0.400 (2) | 0.8992 (14) | 0.055 (7)* |
| H6W | 0.691 (3) | 0.330 (3) | 0.8338 (17) | 0.078 (9)* |
| H7W | 0.744 (3) | 0.0001 (19) | 0.8695 (12) | 0.031 (5)* |
| H8W | 0.868 (3) | −0.080 (2) | 0.8637 (13) | 0.048 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.01834 (17) | 0.01888 (16) | 0.01370 (15) | 0.00414 (13) | −0.00344 (12) | −0.00218 (12) |
| S2 | 0.01813 (17) | 0.01791 (16) | 0.01587 (16) | −0.00119 (12) | −0.00427 (12) | −0.00132 (12) |
| O1 | 0.0217 (5) | 0.0244 (5) | 0.0167 (5) | 0.0061 (4) | 0.0000 (4) | −0.0011 (4) |
| O2 | 0.0231 (6) | 0.0357 (6) | 0.0236 (5) | −0.0038 (5) | −0.0060 (4) | −0.0062 (5) |
| O3 | 0.0384 (6) | 0.0202 (5) | 0.0191 (5) | 0.0095 (5) | −0.0100 (5) | −0.0017 (4) |
| O4 | 0.0276 (6) | 0.0249 (5) | 0.0145 (5) | −0.0060 (4) | −0.0055 (4) | −0.0014 (4) |
| O5 | 0.0265 (6) | 0.0235 (5) | 0.0178 (5) | −0.0075 (4) | −0.0045 (4) | 0.0006 (4) |
| O6 | 0.0331 (6) | 0.0195 (5) | 0.0216 (5) | −0.0045 (4) | −0.0096 (4) | −0.0020 (4) |
| O7 | 0.0296 (6) | 0.0279 (6) | 0.0180 (5) | −0.0084 (5) | −0.0002 (4) | 0.0000 (4) |
| O8 | 0.0236 (6) | 0.0316 (6) | 0.0261 (6) | 0.0074 (5) | −0.0099 (4) | −0.0045 (5) |
| O9 | 0.0244 (5) | 0.0248 (5) | 0.0166 (5) | 0.0030 (4) | −0.0066 (4) | −0.0026 (4) |
| O10 | 0.0310 (6) | 0.0225 (5) | 0.0192 (5) | 0.0092 (4) | −0.0058 (4) | −0.0052 (4) |
| O1W | 0.0351 (7) | 0.0257 (6) | 0.0388 (7) | −0.0025 (5) | −0.0192 (6) | −0.0029 (5) |
| O2W | 0.0237 (6) | 0.0269 (6) | 0.0318 (6) | −0.0001 (5) | −0.0027 (5) | −0.0081 (5) |
| O3W | 0.0290 (6) | 0.0232 (6) | 0.0265 (6) | −0.0016 (5) | −0.0067 (5) | 0.0022 (4) |
| O4W | 0.0249 (6) | 0.0235 (6) | 0.0275 (6) | −0.0009 (5) | 0.0009 (5) | 0.0017 (4) |
| N1 | 0.0186 (6) | 0.0194 (6) | 0.0173 (6) | 0.0027 (5) | −0.0010 (5) | −0.0013 (4) |
| N2 | 0.0188 (6) | 0.0196 (6) | 0.0189 (6) | 0.0018 (5) | −0.0002 (5) | 0.0001 (4) |
| N3 | 0.0187 (6) | 0.0179 (6) | 0.0162 (5) | −0.0015 (5) | −0.0015 (4) | −0.0005 (4) |
| N4 | 0.0164 (6) | 0.0188 (6) | 0.0172 (5) | −0.0022 (4) | −0.0011 (4) | −0.0008 (4) |
| N5 | 0.0207 (6) | 0.0213 (6) | 0.0163 (6) | −0.0015 (5) | −0.0026 (5) | −0.0031 (5) |
| N6 | 0.0210 (7) | 0.0251 (7) | 0.0237 (7) | 0.0024 (5) | −0.0043 (5) | −0.0014 (6) |
| C1 | 0.0167 (6) | 0.0186 (6) | 0.0129 (6) | 0.0039 (5) | −0.0027 (5) | −0.0013 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0205 (7) | 0.0207 (7) | 0.0162 (6) | 0.0001 (5) | −0.0024 (5) | 0.0014 (5) |
| C3 | 0.0196 (7) | 0.0202 (7) | 0.0184 (6) | 0.0009 (5) | −0.0043 (5) | −0.0015 (5) |
| C4 | 0.0193 (7) | 0.0198 (7) | 0.0137 (6) | 0.0069 (5) | −0.0024 (5) | −0.0001 (5) |
| C5 | 0.0209 (7) | 0.0213 (7) | 0.0172 (6) | 0.0013 (6) | 0.0004 (5) | 0.0025 (5) |
| C6 | 0.0200 (7) | 0.0203 (7) | 0.0185 (7) | −0.0007 (5) | −0.0022 (5) | −0.0007 (5) |
| C7 | 0.0179 (7) | 0.0200 (7) | 0.0158 (6) | 0.0041 (5) | −0.0016 (5) | −0.0012 (5) |
| C8 | 0.0221 (7) | 0.0219 (7) | 0.0196 (7) | −0.0006 (6) | −0.0011 (5) | 0.0003 (5) |
| C9 | 0.0222 (7) | 0.0201 (7) | 0.0202 (7) | −0.0014 (6) | −0.0045 (6) | −0.0021 (5) |
| C10 | 0.0181 (7) | 0.0200 (7) | 0.0158 (6) | 0.0031 (5) | −0.0041 (5) | −0.0037 (5) |
| C11 | 0.0187 (7) | 0.0196 (7) | 0.0167 (6) | 0.0001 (5) | −0.0015 (5) | −0.0016 (5) |
| C12 | 0.0156 (6) | 0.0176 (6) | 0.0190 (6) | 0.0018 (5) | −0.0020 (5) | −0.0047 (5) |
| C13 | 0.0158 (6) | 0.0196 (6) | 0.0150 (6) | −0.0034 (5) | −0.0025 (5) | −0.0011 (5) |
| C14 | 0.0207 (7) | 0.0253 (7) | 0.0192 (7) | 0.0051 (6) | −0.0040 (5) | −0.0027 (6) |
| C15 | 0.0234 (7) | 0.0254 (7) | 0.0169 (6) | 0.0043 (6) | −0.0022 (6) | −0.0052 (5) |
| C16 | 0.0169 (6) | 0.0179 (6) | 0.0163 (6) | −0.0038 (5) | −0.0026 (5) | 0.0005 (5) |
| C17 | 0.0191 (7) | 0.0186 (7) | 0.0210 (7) | 0.0021 (5) | −0.0048 (5) | −0.0015 (5) |
| C18 | 0.0191 (7) | 0.0196 (7) | 0.0197 (7) | 0.0008 (5) | −0.0029 (5) | −0.0045 (5) |
| C19 | 0.0152 (6) | 0.0181 (6) | 0.0157 (6) | −0.0022 (5) | −0.0022 (5) | 0.0004 (5) |
| C20 | 0.0173 (7) | 0.0187 (7) | 0.0202 (7) | 0.0015 (5) | −0.0027 (5) | −0.0004 (5) |
| C21 | 0.0197 (7) | 0.0204 (7) | 0.0183 (6) | 0.0009 (5) | −0.0016 (5) | −0.0028 (5) |
| C22 | 0.0174 (6) | 0.0197 (6) | 0.0151 (6) | −0.0042 (5) | −0.0028 (5) | 0.0010 (5) |
| C23 | 0.0205 (7) | 0.0172 (6) | 0.0190 (6) | 0.0020 (5) | −0.0039 (5) | 0.0003 (5) |
| C24 | 0.0188 (7) | 0.0162 (6) | 0.0164 (6) | −0.0017 (5) | 0.0002 (5) | −0.0015 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.4529 (12) | C1—C6 | 1.389 (2) |
| S1—O3 | 1.4564 (11) | C1—C2 | 1.396 (2) |
| S1—O1 | 1.4624 (10) | C2—C3 | 1.3896 (19) |
| S1—C1 | 1.7700 (13) | C2—H1 | 0.9500 |
| S2—O8 | 1.4539 (11) | C3—C4 | 1.399 (2) |
| S2—O7 | 1.4586 (11) | C3—H2 | 0.9500 |
| S2—O6 | 1.4617 (11) | C4—C5 | 1.388 (2) |
| S2—C13 | 1.7653 (14) | C5—C6 | 1.3875 (19) |
| O4—C10 | 1.3629 (16) | C5—H3 | 0.9500 |
| O4—H1H | 0.84 (2) | C6—4H | 0.9500 |
| O5—C12 | 1.3383 (17) | C7—C8 | 1.397 (2) |
| O5—H2H | 0.98 (3) | C7—C12 | 1.420 (2) |
| O9—C22 | 1.3574 (16) | C8—C9 | 1.382 (2) |
| O9—H3H | 0.85 (2) | C8—H5 | 0.9500 |
| O10—C24 | 1.3413 (16) | C9—C10 | 1.402 (2) |
| O10—H4H | 0.93 (3) | C9—H6 | 0.9500 |
| O1W—H1W | 0.82 (3) | C10—C11 | 1.385 (2) |
| O1W—H2W | 0.84 (3) | C11—C12 | 1.3990 (19) |
| O2W—H3W | 0.83 (3) | C11—H8 | 0.9500 |
| O2W—H4W | 0.867 (10) | C13—C14 | 1.3913 (19) |
| O3W—H5W | 0.88 (3) | C13—C18 | 1.3940 (19) |
| O3W—H6W | 0.95 (3) | C14—C15 | 1.3863 (19) |

| | | | |
|---|---|---|---|
| O4W—H7W | 0.81 (2) | C14—H10 | 0.9500 |
| O4W—H8W | 0.81 (3) | C15—C16 | 1.391 (2) |
| N1—N2 | 1.2680 (17) | C15—H11 | 0.9500 |
| N1—C4 | 1.4227 (17) | C16—C17 | 1.3943 (19) |
| N2—C7 | 1.4050 (17) | C17—C18 | 1.3874 (19) |
| N3—N4 | 1.2756 (16) | C17—H12 | 0.9500 |
| N3—C16 | 1.4244 (17) | C18—H13 | 0.9500 |
| N4—C19 | 1.3963 (17) | C19—C20 | 1.4041 (19) |
| N5—H1N | 0.80 (2) | C19—C24 | 1.4221 (19) |
| N5—H2N | 0.92 (2) | C20—C21 | 1.3799 (19) |
| N5—H3N | 0.94 (2) | C20—H14 | 0.9500 |
| N5—H4N | 0.89 (2) | C21—C22 | 1.4059 (19) |
| N6—H5N | 0.90 (2) | C21—H15 | 0.9500 |
| N6—H6N | 0.92 (2) | C22—C23 | 1.3874 (19) |
| N6—H7N | 0.92 (2) | C23—C24 | 1.3919 (19) |
| N6—H8N | 0.84 (2) | C23—H17 | 0.9500 |

| | | | |
|---|---|---|---|
| O2—S1—O3 | 113.29 (7) | C8—C7—N2 | 115.77 (13) |
| O2—S1—O1 | 111.57 (6) | C8—C7—C12 | 119.15 (13) |
| O3—S1—O1 | 111.92 (7) | N2—C7—C12 | 125.08 (13) |
| O2—S1—C1 | 106.74 (7) | C9—C8—C7 | 121.46 (14) |
| O3—S1—C1 | 106.36 (6) | C9—C8—H5 | 119.3 |
| O1—S1—C1 | 106.44 (6) | C7—C8—H5 | 119.3 |
| O8—S2—O7 | 112.77 (7) | C8—C9—C10 | 118.78 (14) |
| O8—S2—O6 | 112.41 (7) | C8—C9—H6 | 120.6 |
| O7—S2—O6 | 110.95 (7) | C10—C9—H6 | 120.6 |
| O8—S2—C13 | 106.48 (6) | O4—C10—C11 | 121.49 (13) |
| O7—S2—C13 | 106.47 (6) | O4—C10—C9 | 117.21 (13) |
| O6—S2—C13 | 107.32 (6) | C11—C10—C9 | 121.31 (13) |
| C10—O4—H1H | 110.2 (15) | C10—C11—C12 | 119.88 (13) |
| C12—O5—H2H | 101.5 (15) | C10—C11—H8 | 120.1 |
| C22—O9—H3H | 110.6 (15) | C12—C11—H8 | 120.1 |
| C24—O10—H4H | 103.8 (15) | O5—C12—C11 | 118.27 (13) |
| H1W—O1W—H2W | 109 (2) | O5—C12—C7 | 122.31 (12) |
| H3W—O2W—H4W | 105 (2) | C11—C12—C7 | 119.42 (13) |
| H5W—O3W—H6W | 104 (2) | C14—C13—C18 | 120.74 (13) |
| H7W—O4W—H8W | 104 (2) | C14—C13—S2 | 120.09 (11) |
| N2—N1—C4 | 114.84 (12) | C18—C13—S2 | 119.11 (10) |
| N1—N2—C7 | 114.40 (12) | C15—C14—C13 | 119.86 (13) |
| N4—N3—C16 | 114.71 (11) | C15—C14—H10 | 120.1 |
| N3—N4—C19 | 114.81 (11) | C13—C14—H10 | 120.1 |
| H1N—N5—H2N | 111.0 (18) | C14—C15—C16 | 119.56 (13) |
| H1N—N5—H3N | 113.3 (19) | C14—C15—H11 | 120.2 |
| H2N—N5—H3N | 108.3 (19) | C16—C15—H11 | 120.2 |
| H1N—N5—H4N | 108.0 (19) | C15—C16—C17 | 120.60 (13) |
| H2N—N5—H4N | 107.7 (19) | C15—C16—N3 | 123.87 (12) |
| H3N—N5—H4N | 108.4 (19) | C17—C16—N3 | 115.52 (12) |
| H5N—N6—H6N | 110.5 (18) | C18—C17—C16 | 119.88 (13) |

| | | | |
|---|---|---|---|
| H5N—N6—H7N | 110.3 (19) | C18—C17—H12 | 120.1 |
| H6N—N6—H7N | 107.0 (18) | C16—C17—H12 | 120.1 |
| H5N—N6—H8N | 109 (2) | C17—C18—C13 | 119.34 (13) |
| H6N—N6—H8N | 112 (2) | C17—C18—H13 | 120.3 |
| H7N—N6—H8N | 108 (2) | C13—C18—H13 | 120.3 |
| C6—C1—C2 | 120.97 (12) | N4—C19—C20 | 116.23 (12) |
| C6—C1—S1 | 119.83 (11) | N4—C19—C24 | 125.04 (12) |
| C2—C1—S1 | 119.18 (11) | C20—C19—C24 | 118.73 (12) |
| C3—C2—C1 | 119.78 (13) | C21—C20—C19 | 121.30 (13) |
| C3—C2—H1 | 120.1 | C21—C20—H14 | 119.3 |
| C1—C2—H1 | 120.1 | C19—C20—H14 | 119.3 |
| C2—C3—C4 | 119.12 (13) | C20—C21—C22 | 119.15 (13) |
| C2—C3—H2 | 120.4 | C20—C21—H15 | 120.4 |
| C4—C3—H2 | 120.4 | C22—C21—H15 | 120.4 |
| C5—C4—C3 | 120.73 (13) | O9—C22—C23 | 117.27 (12) |
| C5—C4—N1 | 114.64 (13) | O9—C22—C21 | 121.83 (12) |
| C3—C4—N1 | 124.62 (13) | C23—C22—C21 | 120.89 (12) |
| C6—C5—C4 | 120.20 (13) | C22—C23—C24 | 120.02 (13) |
| C6—C5—H3 | 119.9 | C22—C23—H17 | 120.0 |
| C4—C5—H3 | 119.9 | C24—C23—H17 | 120.0 |
| C5—C6—C1 | 119.19 (13) | O10—C24—C23 | 118.42 (12) |
| C5—C6—4H | 120.4 | O10—C24—C19 | 121.70 (12) |
| C1—C6—4H | 120.4 | C23—C24—C19 | 119.87 (12) |

| | | | |
|---|---|---|---|
| C4—N1—N2—C7 | −179.73 (11) | N2—C7—C12—C11 | −179.40 (12) |
| C16—N3—N4—C19 | −179.87 (11) | O8—S2—C13—C14 | −14.43 (14) |
| O2—S1—C1—C6 | 12.29 (13) | O7—S2—C13—C14 | 106.13 (13) |
| O3—S1—C1—C6 | 133.52 (11) | O6—S2—C13—C14 | −135.01 (12) |
| O1—S1—C1—C6 | −106.99 (12) | O8—S2—C13—C18 | 168.28 (12) |
| O2—S1—C1—C2 | −169.23 (11) | O7—S2—C13—C18 | −71.16 (13) |
| O3—S1—C1—C2 | −47.99 (13) | O6—S2—C13—C18 | 47.70 (13) |
| O1—S1—C1—C2 | 71.50 (12) | C18—C13—C14—C15 | 0.6 (2) |
| C6—C1—C2—C3 | −0.3 (2) | S2—C13—C14—C15 | −176.62 (12) |
| S1—C1—C2—C3 | −178.79 (10) | C13—C14—C15—C16 | −0.4 (2) |
| C1—C2—C3—C4 | −0.3 (2) | C14—C15—C16—C17 | −0.7 (2) |
| C2—C3—C4—C5 | 0.5 (2) | C14—C15—C16—N3 | 178.73 (14) |
| C2—C3—C4—N1 | 179.66 (12) | N4—N3—C16—C15 | −7.1 (2) |
| N2—N1—C4—C5 | −178.08 (12) | N4—N3—C16—C17 | 172.30 (13) |
| N2—N1—C4—C3 | 2.70 (19) | C15—C16—C17—C18 | 1.5 (2) |
| C3—C4—C5—C6 | 0.0 (2) | N3—C16—C17—C18 | −178.00 (13) |
| N1—C4—C5—C6 | −179.29 (12) | C16—C17—C18—C13 | −1.2 (2) |
| C4—C5—C6—C1 | −0.6 (2) | C14—C13—C18—C17 | 0.1 (2) |
| C2—C1—C6—C5 | 0.8 (2) | S2—C13—C18—C17 | 177.42 (11) |
| S1—C1—C6—C5 | 179.23 (11) | N3—N4—C19—C20 | 178.63 (12) |
| N1—N2—C7—C8 | 179.98 (12) | N3—N4—C19—C24 | −0.9 (2) |
| N1—N2—C7—C12 | 0.06 (19) | N4—C19—C20—C21 | 179.16 (13) |
| N2—C7—C8—C9 | 179.75 (13) | C24—C19—C20—C21 | −1.2 (2) |
| C12—C7—C8—C9 | −0.3 (2) | C19—C20—C21—C22 | 1.7 (2) |

| | | | |
|---|---|---|---|
| C7—C8—C9—C10 | −0.4 (2) | C20—C21—C22—O9 | 178.72 (13) |
| C8—C9—C10—O4 | −179.37 (13) | C20—C21—C22—C23 | −0.4 (2) |
| C8—C9—C10—C11 | 0.7 (2) | O9—C22—C23—C24 | 179.45 (13) |
| O4—C10—C11—C12 | 179.74 (12) | C21—C22—C23—C24 | −1.4 (2) |
| C9—C10—C11—C12 | −0.4 (2) | C22—C23—C24—O10 | −177.09 (13) |
| C10—C11—C12—O5 | 179.41 (12) | C22—C23—C24—C19 | 1.9 (2) |
| C10—C11—C12—C7 | −0.3 (2) | N4—C19—C24—O10 | −2.1 (2) |
| C8—C7—C12—O5 | −179.05 (13) | C20—C19—C24—O10 | 178.33 (13) |
| N2—C7—C12—O5 | 0.9 (2) | N4—C19—C24—C23 | 178.99 (13) |
| C8—C7—C12—C11 | 0.7 (2) | C20—C19—C24—C23 | −0.6 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H1*H*···O4*W* | 0.84 (2) | 1.81 (2) | 2.6357 (17) | 170 (2) |
| O5—H2*H*···N1 | 0.98 (3) | 1.65 (3) | 2.5535 (16) | 151 (2) |
| O9—H3*H*···O2*W* | 0.85 (2) | 1.83 (2) | 2.6673 (16) | 168 (2) |
| O10—H4*H*···N3 | 0.93 (3) | 1.69 (3) | 2.5420 (16) | 149 (2) |
| N5—H1*N*···O1$^{i}$ | 0.80 (2) | 2.16 (2) | 2.8858 (17) | 151.1 (18) |
| N5—H1*N*···O1*W*$^{ii}$ | 0.80 (2) | 2.627 (19) | 3.034 (2) | 113.3 (15) |
| N5—H2*N*···O1 | 0.92 (2) | 1.94 (2) | 2.8403 (18) | 168 (2) |
| N5—H3*N*···O3*W*$^{iii}$ | 0.94 (2) | 1.91 (2) | 2.8488 (17) | 172 (2) |
| N5—H4*N*···O6$^{iv}$ | 0.89 (2) | 2.01 (2) | 2.8425 (16) | 156 (2) |
| N6—H5*N*···S2$^{v}$ | 0.90 (2) | 2.99 (2) | 3.7547 (15) | 143.5 (17) |
| N6—H5*N*···O7$^{v}$ | 0.90 (2) | 1.99 (2) | 2.8804 (18) | 168 (2) |
| N6—H6*N*···O1*W* | 0.92 (2) | 1.90 (2) | 2.8027 (18) | 169.7 (19) |
| N6—H7*N*···O3*W*$^{vi}$ | 0.92 (2) | 1.96 (2) | 2.8733 (19) | 170.4 (19) |
| N6—H8*N*···O2$^{vii}$ | 0.84 (2) | 2.42 (2) | 2.9090 (18) | 117.8 (18) |
| N6—H8*N*···O3$^{v}$ | 0.84 (2) | 2.36 (2) | 2.9153 (17) | 123.8 (19) |
| O1*W*—H2*W*···O4$^{viii}$ | 0.84 (3) | 2.01 (3) | 2.8150 (16) | 162 (2) |
| O1*W*—H1*W*···O8 | 0.82 (3) | 1.98 (3) | 2.7858 (17) | 166 (2) |
| O2*W*—H3*W*···O3$^{viii}$ | 0.83 (3) | 2.04 (3) | 2.8639 (16) | 171 (2) |
| O2*W*—H4*W*···O8$^{vi}$ | 0.87 (1) | 1.94 (1) | 2.7918 (17) | 169 (2) |
| O3*W*—H5*W*···O7$^{viii}$ | 0.88 (3) | 1.83 (3) | 2.7106 (16) | 179 (2) |
| O3*W*—H6*W*···O9 | 0.95 (3) | 2.04 (3) | 2.8347 (15) | 140 (2) |
| O4*W*—H7*W*···O2$^{ix}$ | 0.81 (2) | 1.94 (2) | 2.7482 (17) | 175 (2) |
| O4*W*—H8*W*···O6$^{ix}$ | 0.81 (3) | 2.01 (3) | 2.8128 (16) | 171 (2) |

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

## Ammonium 4-[2-(4-aminophenyl)diazen-1-yl]benzenesulfonate sesquihydrate (NH43)

*Crystal data*

$NH_4^+ \cdot C_{12}H_{10}N_3O_3S^- \cdot 1.5(H_2O)$
$M_r$ = 321.35
Monoclinic, *C*2/*c*
*a* = 35.1636 (15) Å
*b* = 7.8905 (3) Å
*c* = 10.4972 (5) Å
*β* = 100.091 (2)°
*V* = 2867.5 (2) Å$^3$
*Z* = 8
*F*(000) = 1352
$D_x$ = 1.489 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.7107 Å

Cell parameters from 3419 reflections
$\theta$ = 1.0–27.5°
$\mu$ = 0.25 mm$^{-1}$
$T$ = 123 K
Plate, yellow
0.35 × 0.30 × 0.05 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
phi and $\omega$ scans
6133 measured reflections
3271 independent reflections
1857 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.074
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 2.4°
$h$ = −45→45
$k$ = −10→10
$l$ = −13→13

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.105
$S$ = 1.02
3271 reflections
231 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.43663 (2) | 0.11499 (8) | 0.70251 (6) | 0.01953 (19) |
| O1 | 0.45304 (4) | 0.2492 (2) | 0.79049 (15) | 0.0232 (4) |
| O2 | 0.45060 (5) | −0.0525 (2) | 0.74823 (16) | 0.0254 (4) |
| O3 | 0.44203 (5) | 0.1429 (2) | 0.56905 (15) | 0.0258 (4) |
| O1W | 0.43599 (5) | 0.5179 (3) | 0.9712 (2) | 0.0288 (5) |
| O2W | 0.000000 | 0.0222 (3) | 0.750000 | 0.0255 (6) |
| N1 | 0.26669 (5) | 0.1179 (2) | 0.69356 (18) | 0.0206 (5) |
| N2 | 0.25653 (6) | 0.1029 (2) | 0.80233 (19) | 0.0209 (5) |
| N3 | 0.09692 (6) | 0.1286 (3) | 0.8002 (2) | 0.0229 (5) |
| N4 | 0.01011 (8) | 0.2779 (3) | 0.9447 (2) | 0.0231 (5) |
| C1 | 0.38640 (6) | 0.1194 (3) | 0.7015 (2) | 0.0158 (5) |
| C2 | 0.37175 (7) | 0.1937 (3) | 0.8030 (2) | 0.0178 (6) |
| H3 | 0.388827 | 0.245602 | 0.872118 | 0.021* |
| C3 | 0.33251 (7) | 0.1922 (3) | 0.8037 (2) | 0.0199 (6) |
| H4 | 0.322609 | 0.241477 | 0.873737 | 0.024* |
| C4 | 0.30749 (7) | 0.1180 (3) | 0.7009 (2) | 0.0182 (6) |
| C5 | 0.32184 (7) | 0.0481 (3) | 0.5976 (2) | 0.0193 (6) |
| H1 | 0.304641 | 0.001030 | 0.526474 | 0.023* |
| C6 | 0.36131 (7) | 0.0470 (3) | 0.5983 (2) | 0.0189 (6) |
| H2 | 0.371235 | −0.002996 | 0.528569 | 0.023* |

| | | | | |
|---|---|---|---|---|
| C7 | 0.21591 (7) | 0.1143 (3) | 0.7977 (2) | 0.0189 (6) |
| C8 | 0.20127 (7) | 0.0432 (3) | 0.9007 (2) | 0.0217 (6) |
| H7 | 0.218270 | −0.007777 | 0.970567 | 0.026* |
| C9 | 0.16185 (7) | 0.0469 (3) | 0.9011 (2) | 0.0204 (6) |
| H8 | 0.151853 | −0.005480 | 0.969770 | 0.024* |
| C10 | 0.13691 (7) | 0.1264 (3) | 0.8022 (2) | 0.0177 (6) |
| C11 | 0.15198 (7) | 0.2021 (3) | 0.7018 (2) | 0.0211 (6) |
| H6 | 0.135167 | 0.259073 | 0.634717 | 0.025* |
| C12 | 0.19107 (7) | 0.1952 (3) | 0.6988 (2) | 0.0197 (6) |
| H5 | 0.200951 | 0.245721 | 0.629153 | 0.024* |
| H1N | 0.0074 (8) | 0.178 (4) | 0.902 (3) | 0.046 (9)* |
| H2N | 0.0243 (9) | 0.255 (4) | 1.028 (3) | 0.068 (11)* |
| H3N | 0.0210 (9) | 0.348 (4) | 0.900 (3) | 0.065 (12)* |
| H4N | −0.0141 (10) | 0.327 (4) | 0.963 (3) | 0.081 (12)* |
| H5N | 0.0911 (8) | 0.092 (3) | 0.870 (3) | 0.036 (9)* |
| H6N | 0.0856 (7) | 0.223 (4) | 0.775 (2) | 0.038 (9)* |
| H1W | 0.4271 (8) | 0.525 (4) | 0.892 (3) | 0.040 (10)* |
| H2W | 0.4346 (9) | 0.622 (4) | 1.000 (3) | 0.065 (12)* |
| H3W | −0.0192 (6) | −0.044 (3) | 0.757 (3) | 0.049 (10)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0178 (3) | 0.0194 (3) | 0.0217 (4) | −0.0012 (3) | 0.0044 (3) | −0.0010 (3) |
| O1 | 0.0187 (10) | 0.0243 (10) | 0.0269 (10) | −0.0035 (8) | 0.0046 (7) | −0.0055 (9) |
| O2 | 0.0190 (10) | 0.0197 (10) | 0.0370 (11) | 0.0025 (8) | 0.0038 (8) | 0.0025 (8) |
| O3 | 0.0229 (10) | 0.0360 (12) | 0.0202 (9) | −0.0035 (9) | 0.0087 (7) | 0.0004 (8) |
| O1W | 0.0314 (12) | 0.0306 (13) | 0.0240 (13) | 0.0036 (10) | 0.0038 (9) | 0.0001 (10) |
| O2W | 0.0249 (17) | 0.0245 (16) | 0.0294 (15) | 0.000 | 0.0109 (13) | 0.000 |
| N1 | 0.0201 (12) | 0.0199 (12) | 0.0226 (12) | −0.0003 (10) | 0.0058 (9) | −0.0003 (11) |
| N2 | 0.0186 (12) | 0.0218 (12) | 0.0234 (12) | −0.0003 (10) | 0.0066 (9) | 0.0005 (10) |
| N3 | 0.0201 (13) | 0.0218 (14) | 0.0276 (14) | 0.0016 (12) | 0.0064 (10) | 0.0020 (12) |
| N4 | 0.0245 (14) | 0.0216 (15) | 0.0237 (15) | −0.0005 (12) | 0.0058 (12) | −0.0015 (13) |
| C1 | 0.0166 (13) | 0.0121 (13) | 0.0188 (13) | 0.0001 (12) | 0.0039 (10) | 0.0060 (12) |
| C2 | 0.0186 (14) | 0.0163 (13) | 0.0178 (13) | −0.0021 (12) | 0.0011 (10) | 0.0012 (11) |
| C3 | 0.0213 (14) | 0.0211 (14) | 0.0186 (13) | −0.0011 (12) | 0.0068 (11) | −0.0005 (12) |
| C4 | 0.0158 (13) | 0.0173 (13) | 0.0215 (14) | 0.0019 (12) | 0.0037 (10) | 0.0048 (12) |
| C5 | 0.0225 (15) | 0.0195 (14) | 0.0140 (13) | −0.0016 (12) | −0.0022 (11) | 0.0027 (11) |
| C6 | 0.0199 (14) | 0.0205 (14) | 0.0173 (14) | 0.0022 (12) | 0.0064 (11) | −0.0002 (12) |
| C7 | 0.0168 (14) | 0.0176 (13) | 0.0219 (14) | 0.0007 (12) | 0.0023 (11) | −0.0041 (12) |
| C8 | 0.0214 (15) | 0.0242 (14) | 0.0184 (14) | 0.0013 (12) | 0.0008 (11) | 0.0004 (12) |
| C9 | 0.0246 (15) | 0.0199 (14) | 0.0184 (14) | −0.0031 (12) | 0.0085 (11) | −0.0006 (12) |
| C10 | 0.0185 (14) | 0.0141 (13) | 0.0214 (14) | 0.0002 (12) | 0.0057 (11) | −0.0016 (12) |
| C11 | 0.0217 (14) | 0.0199 (14) | 0.0204 (14) | 0.0044 (12) | 0.0004 (11) | −0.0009 (12) |
| C12 | 0.0204 (14) | 0.0197 (14) | 0.0195 (14) | −0.0026 (12) | 0.0043 (11) | −0.0001 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O1 | 1.4564 (17) | C1—C6 | 1.394 (3) |
| S1—O2 | 1.4612 (17) | C2—C3 | 1.381 (3) |
| S1—O3 | 1.4630 (16) | C2—H3 | 0.9500 |
| S1—C1 | 1.765 (2) | C3—C4 | 1.396 (3) |
| O1W—H1W | 0.83 (3) | C3—H4 | 0.9500 |
| O1W—H2W | 0.88 (3) | C4—C5 | 1.389 (3) |
| O2W—H3W | 0.867 (10) | C5—C6 | 1.387 (3) |
| O2W—H3W$^{i}$ | 0.867 (10) | C5—H1 | 0.9500 |
| N1—N2 | 1.260 (2) | C6—H2 | 0.9500 |
| N1—C4 | 1.423 (3) | C7—C12 | 1.390 (3) |
| N2—C7 | 1.423 (3) | C7—C8 | 1.394 (3) |
| N3—C10 | 1.403 (3) | C8—C9 | 1.387 (3) |
| N3—H5N | 0.85 (3) | C8—H7 | 0.9500 |
| N3—H6N | 0.86 (3) | C9—C10 | 1.387 (3) |
| N4—H1N | 0.90 (3) | C9—H8 | 0.9500 |
| N4—H2N | 0.94 (3) | C10—C11 | 1.394 (3) |
| N4—H3N | 0.86 (3) | C11—C12 | 1.382 (3) |
| N4—H4N | 0.98 (4) | C11—H6 | 0.9500 |
| C1—C2 | 1.391 (3) | C12—H5 | 0.9500 |
| | | | |
| O1—S1—O2 | 112.10 (10) | C5—C4—C3 | 120.3 (2) |
| O1—S1—O3 | 113.23 (10) | C5—C4—N1 | 117.3 (2) |
| O2—S1—O3 | 110.97 (10) | C3—C4—N1 | 122.4 (2) |
| O1—S1—C1 | 105.93 (10) | C6—C5—C4 | 119.9 (2) |
| O2—S1—C1 | 107.36 (10) | C6—C5—H1 | 120.0 |
| O3—S1—C1 | 106.79 (10) | C4—C5—H1 | 120.0 |
| H1W—O1W—H2W | 104 (3) | C5—C6—C1 | 119.9 (2) |
| H3W—O2W—H3W$^{i}$ | 106 (4) | C5—C6—H2 | 120.1 |
| N2—N1—C4 | 113.20 (18) | C1—C6—H2 | 120.1 |
| N1—N2—C7 | 113.92 (19) | C12—C7—C8 | 119.7 (2) |
| C10—N3—H5N | 112.2 (18) | C12—C7—N2 | 123.3 (2) |
| C10—N3—H6N | 114.9 (18) | C8—C7—N2 | 116.9 (2) |
| H5N—N3—H6N | 113 (3) | C9—C8—C7 | 119.9 (2) |
| H1N—N4—H2N | 106 (3) | C9—C8—H7 | 120.0 |
| H1N—N4—H3N | 108 (3) | C7—C8—H7 | 120.0 |
| H2N—N4—H3N | 115 (3) | C10—C9—C8 | 120.6 (2) |
| H1N—N4—H4N | 115 (3) | C10—C9—H8 | 119.7 |
| H2N—N4—H4N | 103 (2) | C8—C9—H8 | 119.7 |
| H3N—N4—H4N | 110 (3) | C9—C10—C11 | 119.0 (2) |
| C2—C1—C6 | 119.9 (2) | C9—C10—N3 | 121.1 (2) |
| C2—C1—S1 | 120.53 (17) | C11—C10—N3 | 119.8 (2) |
| C6—C1—S1 | 119.54 (18) | C12—C11—C10 | 120.8 (2) |
| C3—C2—C1 | 120.3 (2) | C12—C11—H6 | 119.6 |
| C3—C2—H3 | 119.8 | C10—C11—H6 | 119.6 |
| C1—C2—H3 | 119.8 | C11—C12—C7 | 119.9 (2) |
| C2—C3—C4 | 119.6 (2) | C11—C12—H5 | 120.1 |

| | | | |
|---|---|---|---|
| C2—C3—H4 | 120.2 | C7—C12—H5 | 120.1 |
| C4—C3—H4 | 120.2 | | |
| | | | |
| C4—N1—N2—C7 | 175.90 (19) | C4—C5—C6—C1 | −1.4 (4) |
| O1—S1—C1—C2 | −21.1 (2) | C2—C1—C6—C5 | −0.5 (3) |
| O2—S1—C1—C2 | 98.82 (19) | S1—C1—C6—C5 | 179.20 (18) |
| O3—S1—C1—C2 | −142.10 (19) | N1—N2—C7—C12 | −23.1 (3) |
| O1—S1—C1—C6 | 159.19 (18) | N1—N2—C7—C8 | 158.6 (2) |
| O2—S1—C1—C6 | −80.9 (2) | C12—C7—C8—C9 | 2.9 (4) |
| O3—S1—C1—C6 | 38.2 (2) | N2—C7—C8—C9 | −178.7 (2) |
| C6—C1—C2—C3 | 1.7 (3) | C7—C8—C9—C10 | −2.4 (4) |
| S1—C1—C2—C3 | −178.02 (18) | C8—C9—C10—C11 | 0.3 (4) |
| C1—C2—C3—C4 | −1.0 (4) | C8—C9—C10—N3 | 178.9 (2) |
| C2—C3—C4—C5 | −0.9 (4) | C9—C10—C11—C12 | 1.4 (3) |
| C2—C3—C4—N1 | −177.6 (2) | N3—C10—C11—C12 | −177.2 (2) |
| N2—N1—C4—C5 | 147.6 (2) | C10—C11—C12—C7 | −1.0 (3) |
| N2—N1—C4—C3 | −35.6 (3) | C8—C7—C12—C11 | −1.2 (3) |
| C3—C4—C5—C6 | 2.1 (4) | N2—C7—C12—C11 | −179.5 (2) |
| N1—C4—C5—C6 | 179.0 (2) | | |

Symmetry code: (i) −*x*, *y*, −*z*+3/2.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4—H1*N*···O2*W* | 0.90 (3) | 2.00 (3) | 2.850 (3) | 157 (2) |
| N4—H2*N*···O1$^{ii}$ | 0.94 (3) | 1.93 (3) | 2.862 (3) | 167 (3) |
| N4—H2*N*···O1*W*$^{ii}$ | 0.94 (3) | 2.57 (3) | 3.039 (3) | 111 (2) |
| N4—H3*N*···O2$^{iii}$ | 0.86 (3) | 2.14 (3) | 2.963 (3) | 161 (3) |
| N4—H3*N*···O3$^{iii}$ | 0.86 (3) | 2.66 (3) | 3.351 (3) | 139 (3) |
| N4—H4*N*···O3$^{iv}$ | 0.98 (4) | 2.07 (4) | 2.987 (3) | 155 (3) |
| N3—H5*N*···O1*W*$^{ii}$ | 0.85 (3) | 2.23 (3) | 3.067 (3) | 168 (2) |
| N3—H6*N*···O2$^{iii}$ | 0.86 (3) | 2.17 (3) | 3.015 (3) | 165 (2) |
| O1*W*—H1*W*···N3$^{iii}$ | 0.83 (3) | 2.21 (3) | 3.011 (3) | 162 (3) |
| O1*W*—H2*W*···O3$^{v}$ | 0.88 (3) | 1.99 (3) | 2.861 (3) | 169 (3) |
| O2*W*—H3*W*···O1$^{vi}$ | 0.87 (1) | 1.97 (1) | 2.792 (3) | 159 (3) |

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

## Ammonium 4-(2-{4-[bis(2-hydroxyethyl)amino]phenyl}diazen-1-yl)benzenesulfonate monohydrate (NH44)

*Crystal data*

$NH_4^+ \cdot C_{16}H_{18}N_3O_5S^- \cdot H_2O$
$M_r$ = 400.45
Triclinic, $P\overline{1}$
*a* = 8.4933 (1) Å
*b* = 13.0977 (2) Å
*c* = 17.1657 (3) Å
*α* = 90.970 (1)°
*β* = 103.180 (1)°
*γ* = 95.132 (1)°
*V* = 1850.43 (5) Å$^3$
*Z* = 4
*F*(000) = 848
$D_x$ = 1.437 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 8751 reflections
*θ* = 1.0–27.9°

$\mu$ = 0.22 mm$^{-1}$
$T$ = 123 K

Cut plate, yellow orange
0.5 × 0.5 × 0.15 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
phi and $\omega$ scans
16736 measured reflections
8841 independent reflections

5911 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043
$\theta_{max}$ = 28.0°, $\theta_{min}$ = 1.2°
$h$ = −11→11
$k$ = −17→17
$l$ = −22→22

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.118
$S$ = 1.04
8841 reflections
550 parameters
8 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.4114P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.57 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.42 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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | −0.17962 (6) | 0.40832 (4) | 0.11851 (3) | 0.02084 (13) | |
| S1A | 0.89947 (6) | 0.10864 (4) | 0.81730 (3) | 0.02110 (13) | |
| O1 | −0.35205 (17) | 0.38875 (12) | 0.11656 (9) | 0.0329 (4) | |
| O1A | 0.88697 (18) | −0.00026 (10) | 0.79522 (9) | 0.0297 (4) | |
| O2 | −0.12780 (18) | 0.51596 (10) | 0.11604 (9) | 0.0303 (4) | |
| O2A | 0.7922 (2) | 0.13162 (13) | 0.86831 (9) | 0.0399 (4) | |
| O3 | −0.13347 (18) | 0.34429 (11) | 0.05857 (8) | 0.0275 (3) | |
| O3A | 1.06745 (17) | 0.14814 (11) | 0.85103 (9) | 0.0270 (3) | |
| O4 | 0.3424 (2) | 0.03149 (12) | 0.90342 (9) | 0.0310 (4) | |
| O4A | 0.3842 (2) | 0.50130 (13) | 0.07514 (9) | 0.0298 (4) | |
| O5 | 0.6302 (2) | −0.14218 (13) | 0.77988 (9) | 0.0299 (4) | |
| O5A | 0.5451 (2) | 0.69228 (13) | 0.07851 (9) | 0.0338 (4) | |
| N1 | 0.0781 (2) | 0.23905 (13) | 0.43773 (10) | 0.0220 (4) | |
| N1A | 0.6312 (2) | 0.32623 (13) | 0.52115 (10) | 0.0239 (4) | |
| N2 | 0.1444 (2) | 0.15696 (13) | 0.43423 (10) | 0.0236 (4) | |
| N2A | 0.7053 (2) | 0.41022 (13) | 0.50930 (10) | 0.0230 (4) | |
| N3 | 0.3017 (2) | −0.08822 (12) | 0.69994 (10) | 0.0213 (4) | |
| N3A | 0.4234 (2) | 0.61579 (12) | 0.23547 (10) | 0.0204 (4) | |
| N4 | 0.1309 (3) | 0.30779 (17) | −0.02165 (13) | 0.0305 (5) | |
| N5 | 0.6587 (3) | 0.15278 (17) | 1.00084 (13) | 0.0289 (4) | |
| C1 | −0.0800 (2) | 0.36624 (14) | 0.21287 (12) | 0.0187 (4) | |

| | | | | |
|---|---|---|---|---|
| C1A | 0.8318 (2) | 0.17296 (15) | 0.72803 (12) | 0.0200 (4) |
| C2 | −0.0972 (3) | 0.41491 (15) | 0.28276 (12) | 0.0228 (5) |
| H2 | −0.149957 | 0.476152 | 0.279943 | 0.027* |
| C2A | 0.6835 (3) | 0.13752 (15) | 0.67789 (12) | 0.0227 (5) |
| H2A | 0.624361 | 0.077229 | 0.690080 | 0.027* |
| C3 | −0.0375 (2) | 0.37424 (15) | 0.35638 (12) | 0.0222 (5) |
| H3 | −0.047185 | 0.408354 | 0.404098 | 0.027* |
| C3A | 0.6224 (3) | 0.19002 (15) | 0.61039 (12) | 0.0235 (5) |
| H3A | 0.520852 | 0.165819 | 0.576214 | 0.028* |
| C4 | 0.0369 (2) | 0.28326 (15) | 0.36042 (12) | 0.0198 (4) |
| C4A | 0.7085 (3) | 0.27817 (15) | 0.59206 (12) | 0.0219 (5) |
| C5 | 0.0585 (2) | 0.23671 (15) | 0.29092 (12) | 0.0214 (4) |
| H5 | 0.112535 | 0.175969 | 0.293915 | 0.026* |
| C5A | 0.8571 (3) | 0.31410 (16) | 0.64225 (13) | 0.0248 (5) |
| H5A | 0.915888 | 0.374439 | 0.629883 | 0.030* |
| C6 | 0.0020 (2) | 0.27789 (15) | 0.21743 (12) | 0.0220 (4) |
| H6 | 0.018872 | 0.246300 | 0.170152 | 0.026* |
| C6A | 0.9192 (3) | 0.26163 (15) | 0.71037 (12) | 0.0239 (5) |
| H6A | 1.020451 | 0.285888 | 0.744758 | 0.029* |
| C7 | 0.1810 (2) | 0.10273 (15) | 0.50505 (12) | 0.0223 (5) |
| C7A | 0.6255 (3) | 0.45964 (15) | 0.44071 (12) | 0.0215 (4) |
| C8 | 0.2579 (3) | 0.01469 (16) | 0.49867 (13) | 0.0253 (5) |
| H8 | 0.281847 | −0.002980 | 0.448998 | 0.030* |
| C8A | 0.7089 (3) | 0.54725 (15) | 0.41917 (12) | 0.0226 (5) |
| H8A | 0.811905 | 0.572131 | 0.451636 | 0.027* |
| C9 | 0.3005 (3) | −0.04780 (16) | 0.56259 (12) | 0.0245 (5) |
| H9 | 0.355253 | −0.106573 | 0.556626 | 0.029* |
| C9A | 0.6442 (2) | 0.59843 (15) | 0.35140 (12) | 0.0217 (4) |
| H9A | 0.704469 | 0.657026 | 0.337306 | 0.026* |
| C10 | 0.2635 (2) | −0.02522 (15) | 0.63652 (12) | 0.0205 (4) |
| C10A | 0.4899 (2) | 0.56516 (14) | 0.30270 (12) | 0.0195 (4) |
| C11 | 0.1840 (2) | 0.06468 (15) | 0.64191 (12) | 0.0231 (5) |
| H11 | 0.158480 | 0.082624 | 0.691148 | 0.028* |
| C11A | 0.4039 (3) | 0.47813 (15) | 0.32746 (12) | 0.0221 (4) |
| H11A | 0.298707 | 0.454379 | 0.296832 | 0.026* |
| C12 | 0.1430 (2) | 0.12635 (15) | 0.57798 (12) | 0.0225 (5) |
| H12 | 0.088398 | 0.185410 | 0.583295 | 0.027* |
| C12A | 0.4703 (3) | 0.42788 (15) | 0.39475 (12) | 0.0233 (5) |
| H12A | 0.409623 | 0.370507 | 0.410364 | 0.028* |
| C13 | 0.2643 (3) | −0.06521 (16) | 0.77689 (12) | 0.0240 (5) |
| H13A | 0.258991 | −0.129575 | 0.805996 | 0.029* |
| H13B | 0.155624 | −0.039534 | 0.766994 | 0.029* |
| C13A | 0.2716 (3) | 0.57465 (16) | 0.18036 (12) | 0.0241 (5) |
| H13C | 0.231958 | 0.628891 | 0.143451 | 0.029* |
| H13D | 0.189095 | 0.557588 | 0.211785 | 0.029* |
| C14 | 0.3863 (3) | 0.01328 (16) | 0.82977 (12) | 0.0257 (5) |
| H14A | 0.495397 | −0.011879 | 0.840483 | 0.031* |
| H14B | 0.391470 | 0.078372 | 0.801570 | 0.031* |

| | | | | | |
|---|---|---|---|---|---|
| C14A | 0.2853 (3) | 0.48032 (17) | 0.13115 (13) | 0.0300 (5) | |
| H14C | 0.332100 | 0.427377 | 0.167557 | 0.036* | |
| H14D | 0.175268 | 0.452262 | 0.101761 | 0.036* | |
| C15 | 0.3707 (3) | −0.18485 (15) | 0.69021 (13) | 0.0237 (5) | |
| H15A | 0.318931 | −0.215719 | 0.636409 | 0.028* | |
| H15B | 0.343701 | −0.232829 | 0.730219 | 0.028* | |
| C15A | 0.5049 (3) | 0.71091 (15) | 0.21544 (12) | 0.0232 (5) | |
| H15C | 0.565540 | 0.747437 | 0.265797 | 0.028* | |
| H15D | 0.421297 | 0.754838 | 0.188657 | 0.028* | |
| C16 | 0.5525 (3) | −0.17382 (16) | 0.69951 (13) | 0.0262 (5) | |
| H16A | 0.588976 | −0.240219 | 0.686007 | 0.031* | |
| H16B | 0.582318 | −0.122439 | 0.662418 | 0.031* | |
| C16A | 0.6218 (3) | 0.69837 (17) | 0.16185 (13) | 0.0277 (5) | |
| H16C | 0.706454 | 0.757198 | 0.172350 | 0.033* | |
| H16D | 0.676385 | 0.635262 | 0.175844 | 0.033* | |
| O1W | 0.3971 (2) | 0.22906 (13) | 0.08102 (10) | 0.0413 (4) | |
| H1W | 0.473 (2) | 0.2803 (14) | 0.0933 (14) | 0.050* | |
| H2W | 0.393 (3) | 0.2095 (18) | 0.1295 (8) | 0.050* | |
| O2W | 0.0195 (3) | 0.09867 (17) | 0.05014 (14) | 0.0781 (7) | |
| H3W | 0.043 (4) | 0.073 (3) | 0.0998 (10) | 0.094* | |
| H4W | −0.040 (7) | 0.043 (3) | 0.025 (2) | 0.094* | 0.5 |
| H5W | 0.114 (5) | 0.085 (6) | 0.038 (3) | 0.094* | 0.5 |
| H1H | 0.260 (4) | 0.065 (2) | 0.8945 (19) | 0.074 (11)* | |
| H2H | 0.704 (4) | −0.099 (2) | 0.7780 (17) | 0.053 (9)* | |
| H3H | 0.461 (4) | 0.469 (2) | 0.0886 (18) | 0.068 (11)* | |
| H4H | 0.482 (4) | 0.626 (2) | 0.0679 (17) | 0.062 (9)* | |
| H1N | 0.050 (3) | 0.3194 (18) | −0.0005 (15) | 0.035 (7)* | |
| H2N | 0.145 (3) | 0.373 (2) | −0.0441 (15) | 0.047 (8)* | |
| H3N | 0.095 (3) | 0.260 (2) | −0.0613 (17) | 0.046 (8)* | |
| H4N | 0.234 (5) | 0.274 (3) | 0.021 (2) | 0.104 (12)* | |
| H5N | 0.703 (3) | 0.133 (2) | 0.9597 (17) | 0.051 (8)* | |
| H6N | 0.561 (5) | 0.176 (3) | 0.985 (2) | 0.089 (12)* | |
| H7N | 0.724 (4) | 0.209 (3) | 1.0321 (19) | 0.076 (10)* | |
| H8N | 0.642 (4) | 0.094 (2) | 1.0311 (18) | 0.068 (9)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0168 (3) | 0.0223 (3) | 0.0236 (3) | 0.0022 (2) | 0.0046 (2) | 0.0025 (2) |
| S1A | 0.0208 (3) | 0.0229 (3) | 0.0204 (3) | 0.0026 (2) | 0.0060 (2) | 0.0013 (2) |
| O1 | 0.0154 (8) | 0.0475 (10) | 0.0362 (9) | 0.0040 (7) | 0.0054 (7) | 0.0123 (7) |
| O1A | 0.0321 (9) | 0.0203 (8) | 0.0336 (9) | −0.0030 (6) | 0.0027 (7) | 0.0030 (6) |
| O2 | 0.0352 (9) | 0.0209 (8) | 0.0338 (9) | 0.0033 (7) | 0.0052 (7) | 0.0063 (6) |
| O2A | 0.0398 (10) | 0.0631 (12) | 0.0252 (9) | 0.0237 (9) | 0.0168 (8) | 0.0112 (8) |
| O3 | 0.0302 (9) | 0.0301 (8) | 0.0225 (8) | 0.0062 (7) | 0.0054 (7) | −0.0005 (6) |
| O3A | 0.0230 (8) | 0.0249 (8) | 0.0293 (8) | −0.0019 (6) | −0.0002 (6) | 0.0015 (6) |
| O4 | 0.0336 (10) | 0.0337 (9) | 0.0266 (9) | 0.0067 (8) | 0.0072 (7) | 0.0019 (7) |
| O4A | 0.0287 (9) | 0.0379 (9) | 0.0254 (9) | 0.0094 (8) | 0.0091 (7) | 0.0008 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O5 | 0.0239 (9) | 0.0340 (9) | 0.0295 (9) | −0.0038 (7) | 0.0041 (7) | 0.0016 (7) |
| O5A | 0.0416 (10) | 0.0343 (10) | 0.0299 (9) | 0.0067 (8) | 0.0155 (8) | 0.0070 (7) |
| N1 | 0.0200 (9) | 0.0224 (9) | 0.0235 (9) | 0.0030 (7) | 0.0042 (7) | 0.0002 (7) |
| N1A | 0.0293 (11) | 0.0220 (9) | 0.0219 (9) | 0.0034 (8) | 0.0084 (8) | −0.0004 (7) |
| N2 | 0.0241 (10) | 0.0210 (9) | 0.0248 (10) | 0.0033 (8) | 0.0036 (8) | 0.0017 (7) |
| N2A | 0.0285 (10) | 0.0212 (9) | 0.0216 (9) | 0.0043 (8) | 0.0098 (8) | −0.0005 (7) |
| N3 | 0.0216 (10) | 0.0212 (9) | 0.0217 (9) | 0.0042 (7) | 0.0051 (7) | 0.0028 (7) |
| N3A | 0.0193 (9) | 0.0204 (9) | 0.0209 (9) | −0.0010 (7) | 0.0047 (7) | 0.0007 (7) |
| N4 | 0.0379 (13) | 0.0289 (12) | 0.0270 (11) | −0.0040 (10) | 0.0154 (10) | −0.0051 (10) |
| N5 | 0.0321 (13) | 0.0323 (12) | 0.0232 (11) | 0.0018 (10) | 0.0088 (9) | 0.0012 (9) |
| C1 | 0.0148 (10) | 0.0180 (10) | 0.0231 (11) | −0.0012 (8) | 0.0050 (8) | 0.0019 (8) |
| C1A | 0.0208 (11) | 0.0208 (10) | 0.0201 (11) | 0.0046 (8) | 0.0076 (9) | −0.0018 (8) |
| C2 | 0.0229 (12) | 0.0175 (10) | 0.0286 (12) | 0.0041 (8) | 0.0061 (9) | 0.0003 (9) |
| C2A | 0.0249 (12) | 0.0192 (10) | 0.0240 (11) | −0.0007 (9) | 0.0071 (9) | −0.0014 (8) |
| C3 | 0.0227 (11) | 0.0202 (11) | 0.0243 (11) | 0.0002 (9) | 0.0078 (9) | −0.0038 (8) |
| C3A | 0.0236 (12) | 0.0227 (11) | 0.0234 (11) | 0.0013 (9) | 0.0044 (9) | −0.0026 (9) |
| C4 | 0.0160 (10) | 0.0201 (10) | 0.0223 (11) | −0.0017 (8) | 0.0037 (8) | 0.0003 (8) |
| C4A | 0.0254 (12) | 0.0216 (11) | 0.0200 (11) | 0.0055 (9) | 0.0073 (9) | −0.0012 (8) |
| C5 | 0.0168 (11) | 0.0217 (11) | 0.0257 (11) | 0.0054 (8) | 0.0038 (9) | 0.0004 (9) |
| C5A | 0.0258 (12) | 0.0215 (11) | 0.0287 (12) | 0.0004 (9) | 0.0101 (9) | 0.0030 (9) |
| C6 | 0.0170 (11) | 0.0249 (11) | 0.0246 (11) | 0.0030 (9) | 0.0053 (9) | −0.0027 (9) |
| C6A | 0.0215 (11) | 0.0252 (11) | 0.0249 (11) | 0.0005 (9) | 0.0056 (9) | −0.0008 (9) |
| C7 | 0.0211 (11) | 0.0233 (11) | 0.0208 (11) | 0.0024 (9) | 0.0015 (9) | 0.0007 (8) |
| C7A | 0.0248 (12) | 0.0219 (11) | 0.0185 (11) | 0.0055 (9) | 0.0055 (9) | −0.0011 (8) |
| C8 | 0.0299 (13) | 0.0243 (11) | 0.0224 (11) | 0.0040 (9) | 0.0074 (9) | −0.0016 (9) |
| C8A | 0.0212 (11) | 0.0238 (11) | 0.0224 (11) | 0.0031 (9) | 0.0042 (9) | −0.0008 (9) |
| C9 | 0.0276 (12) | 0.0218 (11) | 0.0240 (11) | 0.0051 (9) | 0.0049 (9) | −0.0024 (9) |
| C9A | 0.0225 (11) | 0.0195 (10) | 0.0237 (11) | −0.0010 (8) | 0.0078 (9) | −0.0006 (8) |
| C10 | 0.0160 (11) | 0.0218 (11) | 0.0222 (11) | −0.0002 (8) | 0.0022 (8) | −0.0010 (8) |
| C10A | 0.0218 (11) | 0.0189 (10) | 0.0201 (11) | 0.0041 (8) | 0.0091 (9) | −0.0021 (8) |
| C11 | 0.0219 (11) | 0.0276 (11) | 0.0210 (11) | 0.0029 (9) | 0.0073 (9) | −0.0019 (9) |
| C11A | 0.0224 (11) | 0.0215 (11) | 0.0222 (11) | 0.0002 (9) | 0.0058 (9) | −0.0026 (8) |
| C12 | 0.0198 (11) | 0.0222 (11) | 0.0249 (11) | 0.0040 (9) | 0.0031 (9) | −0.0014 (9) |
| C12A | 0.0296 (13) | 0.0178 (10) | 0.0253 (11) | −0.0003 (9) | 0.0132 (9) | −0.0013 (8) |
| C13 | 0.0253 (12) | 0.0234 (11) | 0.0237 (11) | 0.0000 (9) | 0.0077 (9) | 0.0031 (9) |
| C13A | 0.0209 (11) | 0.0288 (12) | 0.0218 (11) | 0.0010 (9) | 0.0044 (9) | −0.0014 (9) |
| C14 | 0.0289 (13) | 0.0244 (11) | 0.0233 (12) | 0.0011 (9) | 0.0056 (9) | −0.0009 (9) |
| C14A | 0.0258 (13) | 0.0357 (13) | 0.0278 (12) | −0.0045 (10) | 0.0081 (10) | −0.0087 (10) |
| C15 | 0.0260 (12) | 0.0177 (10) | 0.0266 (12) | 0.0018 (9) | 0.0046 (9) | 0.0022 (8) |
| C15A | 0.0233 (12) | 0.0209 (11) | 0.0257 (12) | 0.0041 (9) | 0.0055 (9) | 0.0031 (9) |
| C16 | 0.0278 (13) | 0.0239 (11) | 0.0271 (12) | 0.0039 (9) | 0.0064 (10) | −0.0012 (9) |
| C16A | 0.0239 (12) | 0.0262 (12) | 0.0343 (13) | 0.0024 (9) | 0.0091 (10) | 0.0046 (10) |
| O1W | 0.0530 (12) | 0.0355 (10) | 0.0374 (10) | 0.0040 (8) | 0.0141 (9) | 0.0100 (8) |
| O2W | 0.114 (2) | 0.0569 (14) | 0.0507 (14) | −0.0083 (15) | −0.0009 (13) | 0.0128 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.4431 (15) | C5—H5 | 0.9500 |
| S1—O1 | 1.4566 (15) | C5A—C6A | 1.389 (3) |
| S1—O3 | 1.4592 (15) | C5A—H5A | 0.9500 |
| S1—C1 | 1.770 (2) | C6—H6 | 0.9500 |
| S1A—O2A | 1.4462 (16) | C6A—H6A | 0.9500 |
| S1A—O3A | 1.4559 (15) | C7—C8 | 1.390 (3) |
| S1A—O1A | 1.4579 (15) | C7—C12 | 1.399 (3) |
| S1A—C1A | 1.765 (2) | C7A—C8A | 1.394 (3) |
| O4—C14 | 1.420 (3) | C7A—C12A | 1.397 (3) |
| O4—H1H | 0.84 (3) | C8—C9 | 1.381 (3) |
| O4A—C14A | 1.429 (3) | C8—H8 | 0.9500 |
| O4A—H3H | 0.80 (3) | C8A—C9A | 1.380 (3) |
| O5—C16 | 1.424 (3) | C8A—H8A | 0.9500 |
| O5—H2H | 0.81 (3) | C9—C10 | 1.408 (3) |
| O5A—C16A | 1.428 (3) | C9—H9 | 0.9500 |
| O5A—H4H | 0.97 (3) | C9A—C10A | 1.411 (3) |
| N1—N2 | 1.264 (2) | C9A—H9A | 0.9500 |
| N1—C4 | 1.439 (2) | C10—C11 | 1.421 (3) |
| N1A—N2A | 1.261 (2) | C10A—C11A | 1.422 (3) |
| N1A—C4A | 1.430 (3) | C11—C12 | 1.373 (3) |
| N2—C7 | 1.405 (2) | C11—H11 | 0.9500 |
| N2A—C7A | 1.416 (3) | C11A—C12A | 1.371 (3) |
| N3—C10 | 1.374 (2) | C11A—H11A | 0.9500 |
| N3—C13 | 1.460 (3) | C12—H12 | 0.9500 |
| N3—C15 | 1.464 (3) | C12A—H12A | 0.9500 |
| N3A—C10A | 1.373 (2) | C13—C14 | 1.518 (3) |
| N3A—C15A | 1.455 (2) | C13—H13A | 0.9900 |
| N3A—C13A | 1.467 (3) | C13—H13B | 0.9900 |
| N4—H1N | 0.87 (3) | C13A—C14A | 1.513 (3) |
| N4—H2N | 0.95 (3) | C13A—H13C | 0.9900 |
| N4—H3N | 0.89 (3) | C13A—H13D | 0.9900 |
| N4—H4N | 1.13 (4) | C14—H14A | 0.9900 |
| N5—H5N | 0.91 (3) | C14—H14B | 0.9900 |
| N5—H6N | 0.89 (4) | C14A—H14C | 0.9900 |
| N5—H7N | 0.96 (4) | C14A—H14D | 0.9900 |
| N5—H8N | 0.96 (3) | C15—C16 | 1.509 (3) |
| C1—C2 | 1.392 (3) | C15—H15A | 0.9900 |
| C1—C6 | 1.397 (3) | C15—H15B | 0.9900 |
| C1A—C2A | 1.389 (3) | C15A—C16A | 1.516 (3) |
| C1A—C6A | 1.397 (3) | C15A—H15C | 0.9900 |
| C2—C3 | 1.384 (3) | C15A—H15D | 0.9900 |
| C2—H2 | 0.9500 | C16—H16A | 0.9900 |
| C2A—C3A | 1.379 (3) | C16—H16B | 0.9900 |
| C2A—H2A | 0.9500 | C16A—H16C | 0.9900 |
| C3—C4 | 1.393 (3) | C16A—H16D | 0.9900 |
| C3—H3 | 0.9500 | O1W—H1W | 0.875 (10) |

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|---|---|---|---|
| C3A—C4A | 1.390 (3) | O1W—H2W | 0.882 (9) |
| C3A—H3A | 0.9500 | O2W—H3W | 0.907 (10) |
| C4—C5 | 1.387 (3) | O2W—H4W | 0.889 (10) |
| C4A—C5A | 1.393 (3) | O2W—H5W | 0.899 (10) |
| C5—C6 | 1.380 (3) | | |

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|---|---|---|---|
| O2—S1—O1 | 112.96 (9) | C9—C8—H8 | 119.1 |
| O2—S1—O3 | 113.85 (9) | C7—C8—H8 | 119.1 |
| O1—S1—O3 | 111.27 (9) | C9A—C8A—C7A | 121.04 (19) |
| O2—S1—C1 | 107.12 (9) | C9A—C8A—H8A | 119.5 |
| O1—S1—C1 | 104.63 (9) | C7A—C8A—H8A | 119.5 |
| O3—S1—C1 | 106.25 (9) | C8—C9—C10 | 120.62 (19) |
| O2A—S1A—O3A | 112.58 (10) | C8—C9—H9 | 119.7 |
| O2A—S1A—O1A | 112.66 (10) | C10—C9—H9 | 119.7 |
| O3A—S1A—O1A | 111.19 (9) | C8A—C9A—C10A | 121.12 (18) |
| O2A—S1A—C1A | 105.65 (9) | C8A—C9A—H9A | 119.4 |
| O3A—S1A—C1A | 107.50 (9) | C10A—C9A—H9A | 119.4 |
| O1A—S1A—C1A | 106.79 (9) | N3—C10—C9 | 121.13 (18) |
| C14—O4—H1H | 109 (2) | N3—C10—C11 | 121.97 (18) |
| C14A—O4A—H3H | 106 (2) | C9—C10—C11 | 116.90 (18) |
| C16—O5—H2H | 107 (2) | N3A—C10A—C9A | 121.74 (17) |
| C16A—O5A—H4H | 106.9 (17) | N3A—C10A—C11A | 121.25 (18) |
| N2—N1—C4 | 111.31 (16) | C9A—C10A—C11A | 116.99 (18) |
| N2A—N1A—C4A | 113.71 (17) | C12—C11—C10 | 121.86 (19) |
| N1—N2—C7 | 116.79 (17) | C12—C11—H11 | 119.1 |
| N1A—N2A—C7A | 113.68 (17) | C10—C11—H11 | 119.1 |
| C10—N3—C13 | 121.50 (17) | C12A—C11A—C10A | 121.17 (19) |
| C10—N3—C15 | 120.47 (17) | C12A—C11A—H11A | 119.4 |
| C13—N3—C15 | 117.88 (16) | C10A—C11A—H11A | 119.4 |
| C10A—N3A—C15A | 120.69 (17) | C11—C12—C7 | 120.38 (19) |
| C10A—N3A—C13A | 121.44 (16) | C11—C12—H12 | 119.8 |
| C15A—N3A—C13A | 117.84 (16) | C7—C12—H12 | 119.8 |
| H1N—N4—H2N | 98 (2) | C11A—C12A—C7A | 121.10 (19) |
| H1N—N4—H3N | 108 (2) | C11A—C12A—H12A | 119.5 |
| H2N—N4—H3N | 109 (2) | C7A—C12A—H12A | 119.5 |
| H1N—N4—H4N | 114 (2) | N3—C13—C14 | 114.18 (17) |
| H2N—N4—H4N | 122 (2) | N3—C13—H13A | 108.7 |
| H3N—N4—H4N | 105 (2) | C14—C13—H13A | 108.7 |
| H5N—N5—H6N | 114 (3) | N3—C13—H13B | 108.7 |
| H5N—N5—H7N | 111 (3) | C14—C13—H13B | 108.7 |
| H6N—N5—H7N | 104 (3) | H13A—C13—H13B | 107.6 |
| H5N—N5—H8N | 108 (2) | N3A—C13A—C14A | 114.69 (17) |
| H6N—N5—H8N | 106 (3) | N3A—C13A—H13C | 108.6 |
| H7N—N5—H8N | 114 (3) | C14A—C13A—H13C | 108.6 |
| C2—C1—C6 | 119.76 (18) | N3A—C13A—H13D | 108.6 |
| C2—C1—S1 | 119.93 (15) | C14A—C13A—H13D | 108.6 |
| C6—C1—S1 | 119.99 (16) | H13C—C13A—H13D | 107.6 |
| C2A—C1A—C6A | 120.26 (18) | O4—C14—C13 | 110.98 (17) |

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|---|---|---|---|
| C2A—C1A—S1A | 118.50 (15) | O4—C14—H14A | 109.4 |
| C6A—C1A—S1A | 121.11 (16) | C13—C14—H14A | 109.4 |
| C3—C2—C1 | 120.12 (18) | O4—C14—H14B | 109.4 |
| C3—C2—H2 | 119.9 | C13—C14—H14B | 109.4 |
| C1—C2—H2 | 119.9 | H14A—C14—H14B | 108.0 |
| C3A—C2A—C1A | 119.88 (19) | O4A—C14A—C13A | 112.62 (18) |
| C3A—C2A—H2A | 120.1 | O4A—C14A—H14C | 109.1 |
| C1A—C2A—H2A | 120.1 | C13A—C14A—H14C | 109.1 |
| C2—C3—C4 | 119.95 (19) | O4A—C14A—H14D | 109.1 |
| C2—C3—H3 | 120.0 | C13A—C14A—H14D | 109.1 |
| C4—C3—H3 | 120.0 | H14C—C14A—H14D | 107.8 |
| C2A—C3A—C4A | 120.4 (2) | N3—C15—C16 | 114.16 (17) |
| C2A—C3A—H3A | 119.8 | N3—C15—H15A | 108.7 |
| C4A—C3A—H3A | 119.8 | C16—C15—H15A | 108.7 |
| C5—C4—C3 | 119.75 (18) | N3—C15—H15B | 108.7 |
| C5—C4—N1 | 123.30 (17) | C16—C15—H15B | 108.7 |
| C3—C4—N1 | 116.82 (18) | H15A—C15—H15B | 107.6 |
| C3A—C4A—C5A | 119.94 (18) | N3A—C15A—C16A | 115.20 (17) |
| C3A—C4A—N1A | 114.97 (18) | N3A—C15A—H15C | 108.5 |
| C5A—C4A—N1A | 125.08 (18) | C16A—C15A—H15C | 108.5 |
| C6—C5—C4 | 120.56 (18) | N3A—C15A—H15D | 108.5 |
| C6—C5—H5 | 119.7 | C16A—C15A—H15D | 108.5 |
| C4—C5—H5 | 119.7 | H15C—C15A—H15D | 107.5 |
| C6A—C5A—C4A | 119.94 (19) | O5—C16—C15 | 109.56 (18) |
| C6A—C5A—H5A | 120.0 | O5—C16—H16A | 109.8 |
| C4A—C5A—H5A | 120.0 | C15—C16—H16A | 109.8 |
| C5—C6—C1 | 119.72 (19) | O5—C16—H16B | 109.8 |
| C5—C6—H6 | 120.1 | C15—C16—H16B | 109.8 |
| C1—C6—H6 | 120.1 | H16A—C16—H16B | 108.2 |
| C5A—C6A—C1A | 119.6 (2) | O5A—C16A—C15A | 113.46 (18) |
| C5A—C6A—H6A | 120.2 | O5A—C16A—H16C | 108.9 |
| C1A—C6A—H6A | 120.2 | C15A—C16A—H16C | 108.9 |
| C8—C7—C12 | 118.44 (18) | O5A—C16A—H16D | 108.9 |
| C8—C7—N2 | 114.54 (18) | C15A—C16A—H16D | 108.9 |
| C12—C7—N2 | 126.97 (18) | H16C—C16A—H16D | 107.7 |
| C8A—C7A—C12A | 118.49 (18) | H1W—O1W—H2W | 99.8 (17) |
| C8A—C7A—N2A | 116.70 (18) | H3W—O2W—H4W | 97 (2) |
| C12A—C7A—N2A | 124.81 (18) | H3W—O2W—H5W | 95 (2) |
| C9—C8—C7 | 121.8 (2) | H4W—O2W—H5W | 97 (7) |

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|---|---|---|---|
| C4—N1—N2—C7 | −176.00 (16) | N1A—N2A—C7A—C12A | 7.7 (3) |
| C4A—N1A—N2A—C7A | −177.76 (16) | C12—C7—C8—C9 | −1.6 (3) |
| O2—S1—C1—C2 | 56.35 (18) | N2—C7—C8—C9 | −179.29 (19) |
| O1—S1—C1—C2 | −63.79 (18) | C12A—C7A—C8A—C9A | −3.7 (3) |
| O3—S1—C1—C2 | 178.39 (16) | N2A—C7A—C8A—C9A | 177.34 (18) |
| O2—S1—C1—C6 | −130.20 (16) | C7—C8—C9—C10 | 1.5 (3) |
| O1—S1—C1—C6 | 109.65 (17) | C7A—C8A—C9A—C10A | 1.5 (3) |
| O3—S1—C1—C6 | −8.16 (19) | C13—N3—C10—C9 | 179.78 (19) |

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|---|---|---|---|
| O2A—S1A—C1A—C2A | −69.61 (18) | C15—N3—C10—C9 | −4.7 (3) |
| O3A—S1A—C1A—C2A | 169.97 (15) | C13—N3—C10—C11 | −0.8 (3) |
| O1A—S1A—C1A—C2A | 50.56 (18) | C15—N3—C10—C11 | 174.65 (18) |
| O2A—S1A—C1A—C6A | 106.12 (18) | C8—C9—C10—N3 | 178.38 (19) |
| O3A—S1A—C1A—C6A | −14.30 (19) | C8—C9—C10—C11 | −1.0 (3) |
| O1A—S1A—C1A—C6A | −133.71 (17) | C15A—N3A—C10A—C9A | −4.9 (3) |
| C6—C1—C2—C3 | −1.9 (3) | C13A—N3A—C10A—C9A | 173.20 (18) |
| S1—C1—C2—C3 | 171.55 (15) | C15A—N3A—C10A—C11A | 173.48 (18) |
| C6A—C1A—C2A—C3A | 0.0 (3) | C13A—N3A—C10A—C11A | −8.4 (3) |
| S1A—C1A—C2A—C3A | 175.81 (16) | C8A—C9A—C10A—N3A | 179.49 (18) |
| C1—C2—C3—C4 | −1.4 (3) | C8A—C9A—C10A—C11A | 1.1 (3) |
| C1A—C2A—C3A—C4A | 0.2 (3) | N3—C10—C11—C12 | −178.63 (19) |
| C2—C3—C4—C5 | 3.4 (3) | C9—C10—C11—C12 | 0.8 (3) |
| C2—C3—C4—N1 | −172.66 (18) | N3A—C10A—C11A—C12A | −179.79 (18) |
| N2—N1—C4—C5 | 4.0 (3) | C9A—C10A—C11A—C12A | −1.4 (3) |
| N2—N1—C4—C3 | 179.91 (18) | C10—C11—C12—C7 | −0.9 (3) |
| C2A—C3A—C4A—C5A | −0.3 (3) | C8—C7—C12—C11 | 1.3 (3) |
| C2A—C3A—C4A—N1A | −179.21 (18) | N2—C7—C12—C11 | 178.69 (19) |
| N2A—N1A—C4A—C3A | 174.69 (17) | C10A—C11A—C12A—C7A | −0.9 (3) |
| N2A—N1A—C4A—C5A | −4.2 (3) | C8A—C7A—C12A—C11A | 3.4 (3) |
| C3—C4—C5—C6 | −2.2 (3) | N2A—C7A—C12A—C11A | −177.73 (19) |
| N1—C4—C5—C6 | 173.63 (18) | C10—N3—C13—C14 | −81.0 (2) |
| C3A—C4A—C5A—C6A | 0.2 (3) | C15—N3—C13—C14 | 103.4 (2) |
| N1A—C4A—C5A—C6A | 179.01 (19) | C10A—N3A—C13A—C14A | −71.0 (2) |
| C4—C5—C6—C1 | −1.1 (3) | C15A—N3A—C13A—C14A | 107.2 (2) |
| C2—C1—C6—C5 | 3.2 (3) | N3—C13—C14—O4 | −179.84 (16) |
| S1—C1—C6—C5 | −170.31 (15) | N3A—C13A—C14A—O4A | −66.8 (2) |
| C4A—C5A—C6A—C1A | 0.0 (3) | C10—N3—C15—C16 | 84.3 (2) |
| C2A—C1A—C6A—C5A | −0.1 (3) | C13—N3—C15—C16 | −100.0 (2) |
| S1A—C1A—C6A—C5A | −175.79 (16) | C10A—N3A—C15A—C16A | 90.8 (2) |
| N1—N2—C7—C8 | −178.25 (18) | C13A—N3A—C15A—C16A | −87.4 (2) |
| N1—N2—C7—C12 | 4.3 (3) | N3—C15—C16—O5 | 66.0 (2) |
| N1A—N2A—C7A—C8A | −173.40 (18) | N3A—C15A—C16A—O5A | 85.7 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*W*···O1$^{i}$ | 0.88 (1) | 1.93 (1) | 2.802 (2) | 176 (3) |
| O1*W*—H2*W*···O5$^{ii}$ | 0.88 (1) | 1.84 (1) | 2.711 (2) | 168 (2) |
| O2*W*—H3*W*···O1*A*$^{ii}$ | 0.91 (1) | 2.05 (1) | 2.945 (2) | 172 (3) |
| O2*W*—H4*W*···O2*W*$^{iii}$ | 0.89 (1) | 2.29 (4) | 3.031 (5) | 141 (6) |
| O4—H1*H*···O3*A*$^{iv}$ | 0.84 (3) | 2.05 (3) | 2.885 (2) | 169 (3) |
| O5—H2*H*···O1*A* | 0.81 (3) | 1.90 (3) | 2.698 (2) | 169 (3) |
| O4*A*—H3*H*···O1$^{i}$ | 0.80 (3) | 1.96 (3) | 2.756 (2) | 177 (3) |
| O5*A*—H4*H*···O4*A* | 0.97 (3) | 1.79 (3) | 2.734 (2) | 165 (3) |
| N4—H1*N*···O3 | 0.87 (3) | 2.09 (3) | 2.957 (3) | 176 (2) |
| N4—H2*N*···O2$^{v}$ | 0.95 (3) | 1.92 (3) | 2.841 (3) | 163 (2) |
| N4—H3*N*···O3*A*$^{vi}$ | 0.89 (3) | 2.05 (3) | 2.923 (3) | 167 (2) |

| | | | | |
|---|---|---|---|---|
| N4—H4*N*···O1*W* | 1.13 (4) | 1.69 (4) | 2.818 (3) | 176 (3) |
| N5—H5*N*···O2*A* | 0.91 (3) | 1.89 (3) | 2.784 (3) | 164 (2) |
| N5—H6*N*···O5*A*$^{vii}$ | 0.89 (4) | 2.21 (4) | 2.935 (3) | 138 (3) |
| N5—H6*N*···O1*W*$^{viii}$ | 0.89 (4) | 2.51 (4) | 3.094 (3) | 123 (3) |
| N5—H7*N*···O3$^{ix}$ | 0.96 (4) | 2.03 (4) | 2.952 (3) | 159 (3) |
| N5—H8*N*···O4$^{x}$ | 0.96 (3) | 2.00 (3) | 2.943 (3) | 168 (3) |

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

### Ammonium 3-{2-(4-hydroxyphenyl)diazen-1-yl]benzenesulfonate (NH45)

*Crystal data*

$NH_4^+ \cdot C_{12}H_9N_2O_4S^-$
$M_r$ = 295.31
Orthorhombic, *Pccn*
*a* = 12.6592 (3) Å
*b* = 28.3597 (7) Å
*c* = 7.1268 (2) Å
*V* = 2558.60 (11) Å$^3$
*Z* = 8
*F*(000) = 1232
$D_x$ = 1.533 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3229 reflections
*θ* = 1.0–27.5°
*μ* = 0.27 mm$^{-1}$
*T* = 123 K
Cut needle, yellow-orange
0.30 × 0.10 × 0.05 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
phi and *ω* scans
5346 measured reflections
2923 independent reflections
1850 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.069
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.8°
*h* = −16→16
*k* = −36→36
*l* = −9→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.118
*S* = 1.03
2923 reflections
201 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 2.7325P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.45 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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.94027 (5) | 0.56239 (2) | 0.18127 (9) | 0.01749 (18) |
| O1 | 0.85668 (15) | 0.55115 (7) | 0.0463 (3) | 0.0287 (5) |
| O2 | 1.04360 (14) | 0.56367 (7) | 0.0953 (3) | 0.0278 (5) |
| O3 | 0.93547 (16) | 0.53174 (7) | 0.3437 (3) | 0.0301 (5) |

| | | | | |
|---|---|---|---|---|
| O4 | 1.24530 (16) | 0.93197 (7) | 0.0369 (3) | 0.0255 (5) |
| N1 | 1.02393 (17) | 0.73916 (8) | 0.2062 (3) | 0.0203 (5) |
| N2 | 0.99241 (18) | 0.78005 (9) | 0.2434 (3) | 0.0203 (5) |
| N3 | 0.6639 (2) | 0.50958 (11) | 0.1885 (4) | 0.0229 (6) |
| C1 | 0.9119 (2) | 0.62035 (10) | 0.2584 (3) | 0.0168 (6) |
| C2 | 0.9772 (2) | 0.65733 (10) | 0.2101 (4) | 0.0180 (6) |
| H2 | 1.039750 | 0.651624 | 0.139888 | 0.022* |
| C3 | 0.9512 (2) | 0.70309 (10) | 0.2644 (4) | 0.0176 (6) |
| C4 | 0.8602 (2) | 0.71133 (10) | 0.3688 (4) | 0.0206 (6) |
| H4 | 0.842341 | 0.742487 | 0.406115 | 0.025* |
| C5 | 0.7955 (2) | 0.67381 (10) | 0.4181 (4) | 0.0222 (7) |
| H5 | 0.733423 | 0.679368 | 0.489583 | 0.027* |
| C6 | 0.8209 (2) | 0.62841 (10) | 0.3640 (4) | 0.0202 (6) |
| H6 | 0.776597 | 0.602809 | 0.398488 | 0.024* |
| C7 | 1.0619 (2) | 0.81667 (10) | 0.1844 (4) | 0.0181 (6) |
| C8 | 1.1588 (2) | 0.80929 (10) | 0.0944 (4) | 0.0220 (6) |
| H8 | 1.181883 | 0.778107 | 0.068094 | 0.026* |
| C9 | 1.2208 (2) | 0.84727 (10) | 0.0436 (4) | 0.0208 (6) |
| H9 | 1.286594 | 0.842325 | −0.017454 | 0.025* |
| C10 | 1.1862 (2) | 0.89298 (10) | 0.0825 (4) | 0.0182 (6) |
| C11 | 1.0895 (2) | 0.90081 (10) | 0.1682 (4) | 0.0200 (6) |
| H11 | 1.065732 | 0.932015 | 0.191929 | 0.024* |
| C12 | 1.0278 (2) | 0.86232 (10) | 0.2188 (4) | 0.0191 (6) |
| H12 | 0.961388 | 0.867354 | 0.277683 | 0.023* |
| H1N | 0.673 (3) | 0.4784 (13) | 0.204 (5) | 0.042 (10)* |
| H2N | 0.731 (3) | 0.5206 (11) | 0.146 (5) | 0.046 (10)* |
| H3N | 0.641 (2) | 0.5235 (11) | 0.292 (4) | 0.026 (8)* |
| H4N | 0.619 (3) | 0.5176 (12) | 0.096 (5) | 0.048 (11)* |
| H1H | 1.306 (3) | 0.9239 (14) | 0.020 (6) | 0.063 (13)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0153 (3) | 0.0193 (3) | 0.0179 (3) | 0.0001 (3) | 0.0004 (3) | 0.0010 (3) |
| O1 | 0.0239 (11) | 0.0337 (13) | 0.0287 (11) | 0.0064 (9) | −0.0101 (9) | −0.0126 (10) |
| O2 | 0.0172 (10) | 0.0255 (11) | 0.0407 (12) | 0.0015 (9) | 0.0095 (9) | 0.0013 (10) |
| O3 | 0.0488 (13) | 0.0207 (11) | 0.0208 (10) | 0.0014 (10) | 0.0054 (10) | 0.0055 (9) |
| O4 | 0.0188 (10) | 0.0256 (12) | 0.0319 (11) | −0.0016 (10) | 0.0045 (10) | 0.0032 (10) |
| N1 | 0.0191 (11) | 0.0208 (13) | 0.0211 (12) | −0.0016 (10) | −0.0006 (10) | −0.0001 (11) |
| N2 | 0.0211 (13) | 0.0203 (13) | 0.0194 (12) | −0.0017 (11) | −0.0025 (9) | 0.0018 (10) |
| N3 | 0.0259 (14) | 0.0243 (15) | 0.0185 (13) | 0.0008 (12) | 0.0015 (13) | −0.0021 (13) |
| C1 | 0.0166 (14) | 0.0197 (15) | 0.0141 (13) | 0.0011 (12) | −0.0008 (11) | 0.0025 (12) |
| C2 | 0.0138 (13) | 0.0220 (15) | 0.0182 (14) | 0.0010 (12) | −0.0001 (11) | −0.0002 (12) |
| C3 | 0.0135 (13) | 0.0231 (15) | 0.0163 (13) | −0.0053 (12) | −0.0040 (11) | 0.0036 (12) |
| C4 | 0.0185 (13) | 0.0198 (16) | 0.0235 (14) | 0.0025 (12) | −0.0007 (12) | −0.0006 (13) |
| C5 | 0.0168 (14) | 0.0278 (18) | 0.0218 (15) | 0.0033 (12) | 0.0023 (12) | 0.0002 (13) |
| C6 | 0.0165 (13) | 0.0239 (16) | 0.0200 (14) | −0.0017 (12) | −0.0003 (12) | 0.0025 (13) |
| C7 | 0.0173 (12) | 0.0212 (14) | 0.0156 (13) | −0.0023 (12) | −0.0055 (12) | −0.0001 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8 | 0.0217 (14) | 0.0219 (16) | 0.0225 (15) | 0.0016 (12) | −0.0022 (12) | −0.0031 (13) |
| C9 | 0.0162 (14) | 0.0270 (17) | 0.0193 (14) | 0.0004 (12) | 0.0000 (11) | −0.0023 (13) |
| C10 | 0.0172 (14) | 0.0211 (16) | 0.0163 (14) | −0.0034 (12) | −0.0031 (11) | 0.0014 (12) |
| C11 | 0.0193 (13) | 0.0186 (15) | 0.0222 (14) | 0.0020 (11) | −0.0021 (12) | 0.0007 (13) |
| C12 | 0.0158 (13) | 0.0241 (16) | 0.0174 (13) | −0.0008 (12) | −0.0015 (11) | −0.0007 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.4449 (19) | C3—C4 | 1.392 (4) |
| S1—O3 | 1.449 (2) | C4—C5 | 1.387 (4) |
| S1—O1 | 1.4648 (19) | C4—H4 | 0.9500 |
| S1—C1 | 1.770 (3) | C5—C6 | 1.382 (4) |
| O4—C10 | 1.374 (3) | C5—H5 | 0.9500 |
| O4—H1H | 0.82 (4) | C6—H6 | 0.9500 |
| N1—N2 | 1.254 (3) | C7—C12 | 1.387 (4) |
| N1—C3 | 1.437 (3) | C7—C8 | 1.400 (4) |
| N2—C7 | 1.425 (3) | C8—C9 | 1.381 (4) |
| N3—H1N | 0.90 (4) | C8—H8 | 0.9500 |
| N3—H2N | 0.96 (4) | C9—C10 | 1.396 (4) |
| N3—H3N | 0.89 (3) | C9—H9 | 0.9500 |
| N3—H4N | 0.90 (4) | C10—C11 | 1.386 (4) |
| C1—C2 | 1.379 (4) | C11—C12 | 1.390 (4) |
| C1—C6 | 1.395 (4) | C11—H11 | 0.9500 |
| C2—C3 | 1.394 (4) | C12—H12 | 0.9500 |
| C2—H2 | 0.9500 | | |
| | | | |
| O2—S1—O3 | 113.06 (12) | C3—C4—H4 | 120.2 |
| O2—S1—O1 | 112.41 (12) | C6—C5—C4 | 120.4 (3) |
| O3—S1—O1 | 111.33 (12) | C6—C5—H5 | 119.8 |
| O2—S1—C1 | 106.97 (12) | C4—C5—H5 | 119.8 |
| O3—S1—C1 | 107.48 (12) | C5—C6—C1 | 119.7 (3) |
| O1—S1—C1 | 105.04 (12) | C5—C6—H6 | 120.2 |
| C10—O4—H1H | 109 (3) | C1—C6—H6 | 120.2 |
| N2—N1—C3 | 113.2 (2) | C12—C7—C8 | 119.6 (3) |
| N1—N2—C7 | 114.5 (2) | C12—C7—N2 | 115.9 (2) |
| H1N—N3—H2N | 105 (3) | C8—C7—N2 | 124.6 (2) |
| H1N—N3—H3N | 112 (3) | C9—C8—C7 | 120.1 (3) |
| H2N—N3—H3N | 114 (3) | C9—C8—H8 | 120.0 |
| H1N—N3—H4N | 114 (3) | C7—C8—H8 | 120.0 |
| H2N—N3—H4N | 105 (3) | C8—C9—C10 | 119.6 (3) |
| H3N—N3—H4N | 107 (3) | C8—C9—H9 | 120.2 |
| C2—C1—C6 | 120.4 (3) | C10—C9—H9 | 120.2 |
| C2—C1—S1 | 120.5 (2) | O4—C10—C11 | 117.1 (2) |
| C6—C1—S1 | 119.2 (2) | O4—C10—C9 | 122.0 (2) |
| C1—C2—C3 | 119.8 (2) | C11—C10—C9 | 120.9 (3) |
| C1—C2—H2 | 120.1 | C10—C11—C12 | 119.0 (3) |
| C3—C2—H2 | 120.1 | C10—C11—H11 | 120.5 |
| C4—C3—C2 | 120.0 (2) | C12—C11—H11 | 120.5 |

| | | | |
|---|---|---|---|
| C4—C3—N1 | 124.4 (3) | C7—C12—C11 | 120.8 (3) |
| C2—C3—N1 | 115.6 (2) | C7—C12—H12 | 119.6 |
| C5—C4—C3 | 119.7 (3) | C11—C12—H12 | 119.6 |
| C5—C4—H4 | 120.2 | | |

| | | | |
|---|---|---|---|
| C3—N1—N2—C7 | −178.9 (2) | C4—C5—C6—C1 | −0.3 (4) |
| O2—S1—C1—C2 | 8.3 (3) | C2—C1—C6—C5 | 0.8 (4) |
| O3—S1—C1—C2 | 130.0 (2) | S1—C1—C6—C5 | −177.2 (2) |
| O1—S1—C1—C2 | −111.3 (2) | N1—N2—C7—C12 | 178.2 (2) |
| O2—S1—C1—C6 | −173.6 (2) | N1—N2—C7—C8 | −0.8 (4) |
| O3—S1—C1—C6 | −51.9 (2) | C12—C7—C8—C9 | 1.3 (4) |
| O1—S1—C1—C6 | 66.7 (2) | N2—C7—C8—C9 | −179.7 (2) |
| C6—C1—C2—C3 | −1.0 (4) | C7—C8—C9—C10 | −0.1 (4) |
| S1—C1—C2—C3 | 177.0 (2) | C8—C9—C10—O4 | 179.3 (2) |
| C1—C2—C3—C4 | 0.6 (4) | C8—C9—C10—C11 | −1.3 (4) |
| C1—C2—C3—N1 | −179.6 (2) | O4—C10—C11—C12 | −179.2 (2) |
| N2—N1—C3—C4 | −6.4 (4) | C9—C10—C11—C12 | 1.3 (4) |
| N2—N1—C3—C2 | 173.9 (2) | C8—C7—C12—C11 | −1.3 (4) |
| C2—C3—C4—C5 | −0.1 (4) | N2—C7—C12—C11 | 179.6 (2) |
| N1—C3—C4—C5 | −179.8 (2) | C10—C11—C12—C7 | −0.1 (4) |
| C3—C4—C5—C6 | −0.1 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H1*N*···O2$^{i}$ | 0.90 (4) | 2.48 (3) | 3.002 (4) | 118 (3) |
| N3—H1*N*···O4$^{ii}$ | 0.90 (4) | 2.35 (3) | 2.913 (4) | 121 (3) |
| N3—H1*N*···O4$^{iii}$ | 0.90 (4) | 2.50 (4) | 3.162 (4) | 131 (3) |
| N3—H2*N*···O1 | 0.96 (4) | 1.94 (4) | 2.893 (3) | 171 (3) |
| N3—H3*N*···O1$^{iv}$ | 0.89 (3) | 1.97 (3) | 2.822 (3) | 159 (3) |
| N3—H4*N*···O3$^{v}$ | 0.90 (4) | 1.97 (4) | 2.831 (3) | 160 (3) |
| O4—H1*H*···O2$^{vi}$ | 0.82 (4) | 2.00 (4) | 2.707 (3) | 144 (4) |

Symmetry codes: (i) *x*−1/2, −*y*+1, −*z*+1/2; (ii) *x*−1/2, *y*−1/2, −*z*; (iii) −*x*+2, *y*−1/2, −*z*+1/2; (iv) −*x*+3/2, *y*, *z*+1/2; (v) −*x*+3/2, *y*, *z*−1/2; (vi) −*x*+5/2, −*y*+3/2, *z*.

## Poly[aqua{4-[2-(4-hydroxyphenyl)diazen-1-yl]benzenesulfonato}potassium] (K1)

*Crystal data*

[K($C_{12}H_9N_2O_4S$)($H_2O$)]
$M_r$ = 334.39
Triclinic, $P\overline{1}$
*a* = 5.9620 (7) Å
*b* = 7.2033 (11) Å
*c* = 31.929 (5) Å
*α* = 83.852 (14)°
*β* = 86.361 (15)°
*γ* = 88.868 (15)°
*V* = 1360.5 (3) Å$^3$

*Z* = 4
*F*(000) = 688
$D_x$ = 1.633 Mg m$^{-3}$
Synchrotron radiation, *λ* = 0.689 Å
Cell parameters from 10143 reflections
*θ* = 2.5–26.0°
*μ* = 0.51 mm$^{-1}$
*T* = 150 K
Plate, yellow
0.20 × 0.14 × 0.03 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: Daresbury SRS Station 9.8
$\omega$ scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min}$ = 0.751, $T_{max}$ = 1.000
10143 measured reflections
5691 independent reflections
4405 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 2.5°
$h$ = −7→7
$k$ = −9→9
$l$ = −40→39

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.180
$S$ = 1.02
5691 reflections
403 parameters
6 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1366P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.59 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.67 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.

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| K1 | 0.36696 (11) | −0.01827 (9) | 0.75009 (2) | 0.02505 (19) |
| K2 | 1.04941 (11) | −0.53233 (10) | 0.77938 (2) | 0.02465 (19) |
| S1 | −0.11322 (12) | −0.24184 (10) | 0.69389 (2) | 0.01926 (19) |
| S2 | 0.54537 (12) | −0.32684 (11) | 0.83209 (2) | 0.0213 (2) |
| O1 | 0.0904 (4) | −0.2355 (3) | 0.71678 (6) | 0.0268 (5) |
| O2 | −0.2642 (4) | −0.0810 (3) | 0.69821 (6) | 0.0277 (5) |
| O3 | −0.2304 (4) | −0.4194 (3) | 0.70500 (7) | 0.0281 (5) |
| O4 | 0.5925 (4) | −0.2727 (3) | 0.32948 (6) | 0.0285 (5) |
| O5 | 0.6597 (4) | −0.4995 (3) | 0.82126 (7) | 0.0306 (5) |
| O6 | 0.6621 (4) | −0.1604 (4) | 0.81212 (7) | 0.0371 (6) |
| O7 | 0.3080 (4) | −0.3207 (3) | 0.82324 (6) | 0.0254 (5) |
| O8 | 0.9857 (4) | −0.0787 (3) | 1.18624 (6) | 0.0246 (5) |
| O1W | 0.9915 (4) | −0.7663 (3) | 0.70673 (7) | 0.0294 (5) |
| O2W | 1.4663 (4) | −0.6612 (3) | 0.75731 (7) | 0.0267 (5) |
| N1 | 0.1526 (4) | −0.2478 (4) | 0.51080 (8) | 0.0241 (6) |
| N2 | 0.3607 (4) | −0.2444 (4) | 0.50057 (8) | 0.0244 (6) |
| N3 | 0.6433 (4) | −0.2553 (3) | 1.01425 (7) | 0.0211 (5) |
| N4 | 0.8446 (4) | −0.2370 (3) | 1.02266 (8) | 0.0219 (5) |
| C1 | −0.0256 (5) | −0.2344 (4) | 0.63988 (8) | 0.0188 (6) |
| C2 | 0.1890 (5) | −0.3011 (4) | 0.62818 (9) | 0.0224 (6) |
| H2 | 0.289784 | −0.343168 | 0.649169 | 0.027* |
| C3 | 0.2539 (5) | −0.3054 (4) | 0.58561 (9) | 0.0229 (6) |

| | | | | |
|---|---|---|---|---|
| H3 | 0.399776 | −0.348997 | 0.577331 | 0.027* |
| C4 | 0.1017 (5) | −0.2448 (4) | 0.55507 (9) | 0.0210 (6) |
| C5 | −0.1142 (5) | −0.1801 (5) | 0.56692 (9) | 0.0261 (7) |
| H5 | −0.216726 | −0.140838 | 0.546051 | 0.031* |
| C6 | −0.1768 (5) | −0.1740 (4) | 0.60948 (9) | 0.0221 (6) |
| H6 | −0.321876 | −0.128863 | 0.617822 | 0.027* |
| C7 | 0.4134 (5) | −0.2564 (4) | 0.45656 (9) | 0.0223 (6) |
| C8 | 0.6279 (5) | −0.2012 (4) | 0.44089 (9) | 0.0240 (6) |
| H8 | 0.731988 | −0.162898 | 0.459482 | 0.029* |
| C9 | 0.6902 (5) | −0.2017 (4) | 0.39842 (9) | 0.0223 (6) |
| H9 | 0.834366 | −0.158982 | 0.387603 | 0.027* |
| C10 | 0.5398 (5) | −0.2654 (4) | 0.37162 (9) | 0.0214 (6) |
| C11 | 0.3261 (5) | −0.3279 (5) | 0.38736 (9) | 0.0250 (6) |
| H11 | 0.225448 | −0.374106 | 0.369166 | 0.030* |
| C12 | 0.2637 (5) | −0.3213 (5) | 0.42974 (9) | 0.0251 (7) |
| H12 | 0.118444 | −0.361226 | 0.440581 | 0.030* |
| C13 | 0.5661 (5) | −0.3265 (4) | 0.88708 (9) | 0.0201 (6) |
| C14 | 0.3888 (5) | −0.2573 (4) | 0.91189 (9) | 0.0239 (6) |
| H14 | 0.250402 | −0.221333 | 0.899981 | 0.029* |
| C15 | 0.4177 (5) | −0.2415 (4) | 0.95442 (9) | 0.0227 (6) |
| H15 | 0.297484 | −0.197564 | 0.971870 | 0.027* |
| C16 | 0.6237 (5) | −0.2906 (4) | 0.97118 (9) | 0.0192 (6) |
| C17 | 0.7970 (5) | −0.3667 (4) | 0.94658 (9) | 0.0219 (6) |
| H17 | 0.934209 | −0.405492 | 0.958599 | 0.026* |
| C18 | 0.7675 (5) | −0.3852 (4) | 0.90454 (9) | 0.0235 (6) |
| H18 | 0.884138 | −0.437672 | 0.887653 | 0.028* |
| C19 | 0.8724 (5) | −0.2009 (4) | 1.06517 (8) | 0.0199 (6) |
| C20 | 1.0813 (5) | −0.1333 (4) | 1.07323 (9) | 0.0216 (6) |
| H20 | 1.196039 | −0.116093 | 1.051188 | 0.026* |
| C21 | 1.1211 (5) | −0.0912 (4) | 1.11342 (9) | 0.0214 (6) |
| H21 | 1.263161 | −0.044719 | 1.118899 | 0.026* |
| C22 | 0.9530 (5) | −0.1169 (4) | 1.14585 (9) | 0.0209 (6) |
| C23 | 0.7465 (5) | −0.1886 (4) | 1.13802 (9) | 0.0222 (6) |
| H23 | 0.633336 | −0.207817 | 1.160321 | 0.027* |
| C24 | 0.7039 (5) | −0.2324 (4) | 1.09797 (9) | 0.0214 (6) |
| H24 | 0.563378 | −0.282682 | 1.092802 | 0.026* |
| H4H | 0.736 (2) | −0.251 (5) | 0.3235 (11) | 0.025 (9)* |
| H8H | 1.121 (4) | −0.034 (7) | 1.1861 (17) | 0.075 (17)* |
| H1W | 0.909 (6) | −0.666 (3) | 0.7025 (12) | 0.040 (11)* |
| H2W | 0.908 (7) | −0.862 (5) | 0.7037 (16) | 0.060 (15)* |
| H3W | 1.510 (7) | −0.619 (6) | 0.7314 (6) | 0.043 (11)* |
| H4W | 1.541 (7) | −0.598 (6) | 0.7737 (12) | 0.051 (13)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| K1 | 0.0278 (4) | 0.0274 (4) | 0.0203 (3) | 0.0015 (3) | −0.0027 (3) | −0.0038 (3) |
| K2 | 0.0222 (4) | 0.0303 (4) | 0.0206 (3) | 0.0020 (3) | −0.0018 (2) | 0.0010 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| S1 | 0.0225 (4) | 0.0210 (4) | 0.0138 (3) | 0.0025 (3) | 0.0000 (3) | −0.0006 (3) |
| S2 | 0.0225 (4) | 0.0284 (4) | 0.0133 (3) | −0.0015 (3) | −0.0034 (3) | −0.0025 (3) |
| O1 | 0.0307 (12) | 0.0315 (12) | 0.0184 (10) | 0.0001 (9) | −0.0038 (8) | −0.0022 (9) |
| O2 | 0.0298 (12) | 0.0324 (12) | 0.0202 (11) | 0.0116 (9) | 0.0018 (8) | −0.0032 (9) |
| O3 | 0.0328 (12) | 0.0253 (12) | 0.0248 (11) | −0.0031 (9) | 0.0047 (9) | 0.0007 (9) |
| O4 | 0.0298 (12) | 0.0408 (14) | 0.0146 (10) | 0.0061 (10) | −0.0013 (8) | −0.0032 (9) |
| O5 | 0.0295 (12) | 0.0425 (14) | 0.0223 (11) | 0.0108 (10) | −0.0040 (9) | −0.0148 (10) |
| O6 | 0.0503 (15) | 0.0400 (15) | 0.0209 (11) | −0.0217 (12) | −0.0086 (10) | 0.0043 (10) |
| O7 | 0.0260 (11) | 0.0308 (12) | 0.0206 (10) | 0.0020 (9) | −0.0076 (8) | −0.0054 (9) |
| O8 | 0.0288 (12) | 0.0326 (12) | 0.0127 (9) | −0.0007 (9) | −0.0031 (8) | −0.0034 (8) |
| O1W | 0.0303 (13) | 0.0298 (13) | 0.0277 (12) | 0.0033 (10) | −0.0002 (9) | −0.0034 (10) |
| O2W | 0.0299 (12) | 0.0301 (12) | 0.0201 (11) | −0.0065 (9) | 0.0039 (9) | −0.0046 (9) |
| N1 | 0.0303 (14) | 0.0253 (14) | 0.0169 (12) | 0.0039 (10) | −0.0013 (10) | −0.0038 (10) |
| N2 | 0.0268 (13) | 0.0285 (14) | 0.0173 (12) | 0.0021 (10) | 0.0004 (10) | −0.0011 (10) |
| N3 | 0.0247 (13) | 0.0244 (13) | 0.0145 (11) | 0.0036 (10) | −0.0051 (9) | −0.0027 (9) |
| N4 | 0.0292 (14) | 0.0206 (13) | 0.0162 (11) | 0.0042 (10) | −0.0030 (9) | −0.0037 (9) |
| C1 | 0.0262 (15) | 0.0155 (13) | 0.0146 (13) | −0.0017 (11) | 0.0016 (10) | −0.0020 (10) |
| C2 | 0.0247 (15) | 0.0246 (15) | 0.0174 (13) | 0.0030 (11) | −0.0010 (11) | −0.0007 (11) |
| C3 | 0.0207 (14) | 0.0276 (16) | 0.0195 (14) | 0.0047 (11) | −0.0004 (11) | −0.0007 (11) |
| C4 | 0.0218 (14) | 0.0246 (15) | 0.0163 (13) | 0.0020 (11) | −0.0007 (10) | −0.0014 (11) |
| C5 | 0.0242 (15) | 0.0342 (18) | 0.0187 (14) | 0.0072 (12) | −0.0029 (11) | 0.0018 (12) |
| C6 | 0.0190 (14) | 0.0270 (16) | 0.0200 (14) | 0.0043 (11) | −0.0036 (11) | −0.0004 (11) |
| C7 | 0.0264 (15) | 0.0251 (15) | 0.0147 (13) | 0.0057 (11) | −0.0021 (11) | 0.0010 (11) |
| C8 | 0.0267 (15) | 0.0255 (16) | 0.0210 (15) | 0.0001 (12) | −0.0020 (11) | −0.0080 (12) |
| C9 | 0.0228 (14) | 0.0226 (15) | 0.0213 (14) | −0.0006 (11) | −0.0003 (11) | −0.0017 (11) |
| C10 | 0.0247 (15) | 0.0240 (15) | 0.0149 (13) | 0.0057 (11) | −0.0025 (10) | 0.0005 (11) |
| C11 | 0.0235 (15) | 0.0318 (17) | 0.0201 (14) | 0.0042 (12) | −0.0055 (11) | −0.0037 (12) |
| C12 | 0.0216 (15) | 0.0320 (17) | 0.0207 (14) | 0.0034 (12) | −0.0008 (11) | −0.0001 (12) |
| C13 | 0.0255 (15) | 0.0218 (14) | 0.0131 (12) | 0.0022 (11) | −0.0056 (10) | −0.0007 (10) |
| C14 | 0.0230 (15) | 0.0292 (16) | 0.0189 (14) | 0.0035 (11) | −0.0007 (11) | −0.0005 (11) |
| C15 | 0.0250 (15) | 0.0252 (16) | 0.0182 (13) | 0.0028 (11) | −0.0003 (11) | −0.0039 (11) |
| C16 | 0.0233 (14) | 0.0170 (14) | 0.0172 (13) | −0.0010 (10) | −0.0024 (10) | 0.0004 (10) |
| C17 | 0.0227 (14) | 0.0252 (15) | 0.0187 (14) | 0.0033 (11) | −0.0071 (11) | −0.0040 (11) |
| C18 | 0.0240 (15) | 0.0307 (16) | 0.0161 (13) | 0.0067 (12) | −0.0029 (11) | −0.0040 (11) |
| C19 | 0.0282 (15) | 0.0212 (14) | 0.0110 (12) | 0.0085 (11) | −0.0057 (10) | −0.0042 (10) |
| C20 | 0.0164 (13) | 0.0290 (16) | 0.0192 (14) | −0.0004 (11) | −0.0012 (10) | −0.0019 (11) |
| C21 | 0.0222 (14) | 0.0228 (15) | 0.0193 (14) | −0.0012 (11) | −0.0039 (11) | −0.0012 (11) |
| C22 | 0.0274 (15) | 0.0201 (14) | 0.0148 (13) | 0.0041 (11) | −0.0040 (11) | 0.0012 (10) |
| C23 | 0.0263 (15) | 0.0248 (15) | 0.0147 (13) | 0.0000 (11) | 0.0000 (11) | 0.0002 (11) |
| C24 | 0.0201 (14) | 0.0225 (15) | 0.0213 (14) | 0.0001 (11) | −0.0044 (11) | 0.0009 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| K1—O1 | 2.642 (2) | N3—N4 | 1.258 (4) |
| K1—O2W$^{i}$ | 2.688 (3) | N3—C16 | 1.437 (4) |
| K1—O2$^{ii}$ | 2.728 (2) | N4—C19 | 1.429 (4) |
| K1—O6 | 2.829 (3) | C1—C6 | 1.396 (4) |
| K1—O8$^{iii}$ | 2.957 (2) | C1—C2 | 1.400 (4) |

| | | | |
|---|---|---|---|
| K1—O7 | 3.027 (2) | C2—C3 | 1.393 (4) |
| K1—O4$^{iv}$ | 3.116 (2) | C2—H2 | 0.9500 |
| K1—O1W$^{i}$ | 3.149 (2) | C3—C4 | 1.404 (4) |
| K1—S2 | 3.4555 (11) | C3—H3 | 0.9500 |
| K1—K2$^{i}$ | 4.1142 (12) | C4—C5 | 1.403 (4) |
| K1—K2$^{v}$ | 4.1810 (12) | C5—C6 | 1.391 (4) |
| K2—O5 | 2.625 (2) | C5—H5 | 0.9500 |
| K2—O2W | 2.713 (2) | C6—H6 | 0.9500 |
| K2—O7$^{ii}$ | 2.736 (2) | C7—C8 | 1.393 (4) |
| K2—O1$^{ii}$ | 2.769 (2) | C7—C12 | 1.396 (4) |
| K2—O8$^{vi}$ | 2.904 (2) | C8—C9 | 1.383 (4) |
| K2—O3$^{ii}$ | 3.020 (2) | C8—H8 | 0.9500 |
| K2—O1W | 3.050 (3) | C9—C10 | 1.395 (4) |
| K2—C22$^{vi}$ | 3.285 (3) | C9—H9 | 0.9500 |
| K2—C23$^{vi}$ | 3.414 (3) | C10—C11 | 1.404 (4) |
| K2—S1$^{ii}$ | 3.4353 (11) | C11—C12 | 1.386 (4) |
| K2—S2 | 3.7108 (11) | C11—H11 | 0.9500 |
| K2—H1W | 2.91 (4) | C12—H12 | 0.9500 |
| K2—H4W | 2.96 (4) | C13—C18 | 1.395 (4) |
| S1—O1 | 1.460 (2) | C13—C14 | 1.397 (4) |
| S1—O2 | 1.466 (2) | C14—C15 | 1.396 (4) |
| S1—O3 | 1.469 (2) | C14—H14 | 0.9500 |
| S1—C1 | 1.765 (3) | C15—C16 | 1.395 (4) |
| S2—O7 | 1.460 (2) | C15—H15 | 0.9500 |
| S2—O6 | 1.464 (2) | C16—C17 | 1.397 (4) |
| S2—O5 | 1.470 (2) | C17—C18 | 1.385 (4) |
| S2—C13 | 1.768 (3) | C17—H17 | 0.9500 |
| O4—C10 | 1.368 (3) | C18—H18 | 0.9500 |
| O4—H4H | 0.880 (10) | C19—C20 | 1.395 (4) |
| O8—C22 | 1.374 (4) | C19—C24 | 1.408 (4) |
| O8—H8H | 0.876 (11) | C20—C21 | 1.386 (4) |
| O1W—H1W | 0.872 (10) | C20—H20 | 0.9500 |
| O1W—H2W | 0.877 (10) | C21—C22 | 1.395 (4) |
| O2W—H3W | 0.875 (10) | C21—H21 | 0.9500 |
| O2W—H4W | 0.878 (10) | C22—C23 | 1.392 (4) |
| N1—N2 | 1.262 (4) | C23—C24 | 1.389 (4) |
| N1—C4 | 1.429 (4) | C23—H23 | 0.9500 |
| N2—C7 | 1.432 (4) | C24—H24 | 0.9500 |

| | | | |
|---|---|---|---|
| O1—K1—O2W$^{i}$ | 143.99 (8) | S1$^{ii}$—K2—H4W | 110.8 (9) |
| O1—K1—O2$^{ii}$ | 96.82 (7) | S2—K2—H4W | 149.6 (4) |
| O2W$^{i}$—K1—O2$^{ii}$ | 94.96 (7) | K1$^{vii}$—K2—H4W | 54.5 (6) |
| O1—K1—O6 | 122.07 (7) | H1W—K2—H4W | 102.9 (8) |
| O2W$^{i}$—K1—O6 | 93.29 (8) | O1—S1—O2 | 113.45 (14) |
| O2$^{ii}$—K1—O6 | 81.29 (7) | O1—S1—O3 | 111.38 (13) |
| O1—K1—O8$^{iii}$ | 91.74 (7) | O2—S1—O3 | 111.79 (13) |
| O2W$^{i}$—K1—O8$^{iii}$ | 78.80 (7) | O1—S1—C1 | 106.56 (13) |
| O2$^{ii}$—K1—O8$^{iii}$ | 171.32 (7) | O2—S1—C1 | 106.74 (13) |

| | | | |
|---|---|---|---|
| O6—K1—O8$^{iii}$ | 92.97 (7) | O3—S1—C1 | 106.42 (13) |
| O1—K1—O7 | 81.20 (7) | O1—S1—K2$^{v}$ | 51.36 (9) |
| O2W$^{i}$—K1—O7 | 124.81 (7) | O2—S1—K2$^{v}$ | 122.43 (9) |
| O2$^{ii}$—K1—O7 | 112.15 (7) | O3—S1—K2$^{v}$ | 61.34 (10) |
| O6—K1—O7 | 48.56 (6) | C1—S1—K2$^{v}$ | 130.60 (10) |
| O8$^{iii}$—K1—O7 | 67.71 (6) | O7—S2—O6 | 111.40 (15) |
| O1—K1—O4$^{iv}$ | 94.29 (7) | O7—S2—O5 | 112.83 (14) |
| O2W$^{i}$—K1—O4$^{iv}$ | 59.54 (7) | O6—S2—O5 | 111.82 (15) |
| O2$^{ii}$—K1—O4$^{iv}$ | 67.50 (6) | O7—S2—C13 | 108.58 (13) |
| O6—K1—O4$^{iv}$ | 134.83 (7) | O6—S2—C13 | 106.38 (14) |
| O8$^{iii}$—K1—O4$^{iv}$ | 113.39 (6) | O5—S2—C13 | 105.38 (13) |
| O7—K1—O4$^{iv}$ | 175.43 (7) | O7—S2—K1 | 60.88 (9) |
| O1—K1—O1W$^{i}$ | 71.39 (7) | O6—S2—K1 | 53.06 (10) |
| O2W$^{i}$—K1—O1W$^{i}$ | 72.75 (7) | O5—S2—K1 | 117.59 (9) |
| O2$^{ii}$—K1—O1W$^{i}$ | 114.51 (7) | C13—S2—K1 | 136.61 (10) |
| O6—K1—O1W$^{i}$ | 159.23 (7) | O7—S2—K2 | 132.58 (9) |
| O8$^{iii}$—K1—O1W$^{i}$ | 69.63 (6) | O6—S2—K2 | 77.99 (12) |
| O7—K1—O1W$^{i}$ | 127.79 (6) | O5—S2—K2 | 34.05 (10) |
| O4$^{iv}$—K1—O1W$^{i}$ | 50.58 (6) | C13—S2—K2 | 112.84 (10) |
| O1—K1—S2 | 99.79 (5) | K1—S2—K2 | 100.28 (2) |
| O2W$^{i}$—K1—S2 | 113.31 (6) | S1—O1—K1 | 145.40 (14) |
| O2$^{ii}$—K1—S2 | 93.51 (5) | S1—O1—K2$^{v}$ | 104.32 (11) |
| O6—K1—S2 | 24.42 (5) | K1—O1—K2$^{v}$ | 101.16 (7) |
| O8$^{iii}$—K1—S2 | 83.59 (5) | S1—O2—K1$^{v}$ | 133.87 (12) |
| O7—K1—S2 | 24.92 (4) | S1—O3—K2$^{v}$ | 93.40 (11) |
| O4$^{iv}$—K1—S2 | 157.67 (5) | C10—O4—K1$^{iv}$ | 135.45 (18) |
| O1W$^{i}$—K1—S2 | 151.14 (5) | C10—O4—H4H | 110 (2) |
| O1—K1—K2$^{i}$ | 111.21 (5) | K1$^{iv}$—O4—H4H | 72 (2) |
| O2W$^{i}$—K1—K2$^{i}$ | 40.61 (5) | S2—O5—K2 | 127.68 (14) |
| O2$^{ii}$—K1—K2$^{i}$ | 131.67 (5) | S2—O6—K1 | 102.52 (12) |
| O6—K1—K2$^{i}$ | 112.19 (6) | S2—O7—K2$^{v}$ | 133.41 (13) |
| O8$^{iii}$—K1—K2$^{i}$ | 44.90 (5) | S2—O7—K1 | 94.20 (10) |
| O7—K1—K2$^{i}$ | 110.51 (5) | K2$^{v}$—O7—K1 | 92.89 (6) |
| O4$^{iv}$—K1—K2$^{i}$ | 71.74 (5) | C22—O8—K2$^{vi}$ | 93.35 (16) |
| O1W$^{i}$—K1—K2$^{i}$ | 47.40 (5) | C22—O8—K1$^{iii}$ | 126.30 (17) |
| S2—K1—K2$^{i}$ | 118.03 (2) | K2$^{vi}$—O8—K1$^{iii}$ | 89.15 (6) |
| O1—K1—K2$^{v}$ | 40.53 (5) | C22—O8—H8H | 107 (4) |
| O2W$^{i}$—K1—K2$^{v}$ | 157.68 (5) | K2$^{vi}$—O8—H8H | 114 (4) |
| O2$^{ii}$—K1—K2$^{v}$ | 106.51 (6) | K1$^{iii}$—O8—H8H | 120 (4) |
| O6—K1—K2$^{v}$ | 84.18 (5) | K2—O1W—K1$^{vii}$ | 83.14 (6) |
| O8$^{iii}$—K1—K2$^{v}$ | 79.19 (5) | K2—O1W—H1W | 72 (3) |
| O7—K1—K2$^{v}$ | 40.81 (4) | K1$^{vii}$—O1W—H1W | 155 (3) |
| O4$^{iv}$—K1—K2$^{v}$ | 134.64 (5) | K2—O1W—H2W | 134 (3) |
| O1W$^{i}$—K1—K2$^{v}$ | 102.80 (5) | K1$^{vii}$—O1W—H2W | 93 (3) |
| S2—K1—K2$^{v}$ | 60.30 (2) | H1W—O1W—H2W | 108 (4) |
| K2$^{i}$—K1—K2$^{v}$ | 120.54 (2) | K1$^{vii}$—O2W—K2 | 99.23 (7) |
| O5—K2—O2W | 160.56 (7) | K1$^{vii}$—O2W—H3W | 103 (3) |
| O5—K2—O7$^{ii}$ | 99.88 (7) | K2—O2W—H3W | 112 (3) |

| | | | |
|---|---|---|---|
| O2W—K2—O7$^{ii}$ | 79.33 (7) | K1$^{vii}$—O2W—H4W | 139 (3) |
| O5—K2—O1$^{ii}$ | 108.12 (7) | K2—O2W—H4W | 97 (3) |
| O2W—K2—O1$^{ii}$ | 91.20 (7) | H3W—O2W—H4W | 106 (4) |
| O7$^{ii}$—K2—O1$^{ii}$ | 84.47 (7) | N2—N1—C4 | 113.4 (3) |
| O5—K2—O8$^{vi}$ | 83.48 (7) | N1—N2—C7 | 113.7 (3) |
| O2W—K2—O8$^{vi}$ | 79.34 (7) | N4—N3—C16 | 112.2 (2) |
| O7$^{ii}$—K2—O8$^{vi}$ | 114.54 (7) | N3—N4—C19 | 114.2 (2) |
| O1$^{ii}$—K2—O8$^{vi}$ | 156.28 (7) | C6—C1—C2 | 121.0 (3) |
| O5—K2—O3$^{ii}$ | 81.94 (7) | C6—C1—S1 | 119.4 (2) |
| O2W—K2—O3$^{ii}$ | 113.62 (7) | C2—C1—S1 | 119.5 (2) |
| O7$^{ii}$—K2—O3$^{ii}$ | 130.17 (7) | C3—C2—C1 | 119.7 (3) |
| O1$^{ii}$—K2—O3$^{ii}$ | 49.17 (6) | C3—C2—H2 | 120.2 |
| O8$^{vi}$—K2—O3$^{ii}$ | 115.13 (7) | C1—C2—H2 | 120.2 |
| O5—K2—O1W | 109.02 (7) | C2—C3—C4 | 119.4 (3) |
| O2W—K2—O1W | 74.07 (7) | C2—C3—H3 | 120.3 |
| O7$^{ii}$—K2—O1W | 151.04 (7) | C4—C3—H3 | 120.3 |
| O1$^{ii}$—K2—O1W | 84.82 (7) | C5—C4—C3 | 120.8 (3) |
| O8$^{vi}$—K2—O1W | 71.71 (6) | C5—C4—N1 | 116.1 (3) |
| O3$^{ii}$—K2—O1W | 54.96 (6) | C3—C4—N1 | 123.1 (3) |
| O5—K2—C22$^{vi}$ | 75.97 (8) | C6—C5—C4 | 119.5 (3) |
| O2W—K2—C22$^{vi}$ | 84.63 (7) | C6—C5—H5 | 120.3 |
| O7$^{ii}$—K2—C22$^{vi}$ | 92.32 (7) | C4—C5—H5 | 120.3 |
| O1$^{ii}$—K2—C22$^{vi}$ | 175.16 (7) | C5—C6—C1 | 119.7 (3) |
| O8$^{vi}$—K2—C22$^{vi}$ | 24.68 (7) | C5—C6—H6 | 120.1 |
| O3$^{ii}$—K2—C22$^{vi}$ | 135.03 (7) | C1—C6—H6 | 120.1 |
| O1W—K2—C22$^{vi}$ | 96.36 (7) | C8—C7—C12 | 120.0 (3) |
| O5—K2—C23$^{vi}$ | 91.18 (7) | C8—C7—N2 | 116.5 (3) |
| O2W—K2—C23$^{vi}$ | 70.03 (7) | C12—C7—N2 | 123.5 (3) |
| O7$^{ii}$—K2—C23$^{vi}$ | 71.42 (7) | C9—C8—C7 | 120.4 (3) |
| O1$^{ii}$—K2—C23$^{vi}$ | 151.52 (7) | C9—C8—H8 | 119.8 |
| O8$^{vi}$—K2—C23$^{vi}$ | 43.13 (7) | C7—C8—H8 | 119.8 |
| O3$^{ii}$—K2—C23$^{vi}$ | 158.09 (7) | C8—C9—C10 | 119.5 (3) |
| O1W—K2—C23$^{vi}$ | 109.00 (7) | C8—C9—H9 | 120.3 |
| C22$^{vi}$—K2—C23$^{vi}$ | 23.89 (7) | C10—C9—H9 | 120.3 |
| O5—K2—S1$^{ii}$ | 92.88 (6) | O4—C10—C9 | 122.4 (3) |
| O2W—K2—S1$^{ii}$ | 106.08 (5) | O4—C10—C11 | 117.1 (3) |
| O7$^{ii}$—K2—S1$^{ii}$ | 105.92 (5) | C9—C10—C11 | 120.6 (3) |
| O1$^{ii}$—K2—S1$^{ii}$ | 24.32 (5) | C12—C11—C10 | 119.3 (3) |
| O8$^{vi}$—K2—S1$^{ii}$ | 139.42 (5) | C12—C11—H11 | 120.4 |
| O3$^{ii}$—K2—S1$^{ii}$ | 25.26 (4) | C10—C11—H11 | 120.4 |
| O1W—K2—S1$^{ii}$ | 71.45 (5) | C11—C12—C7 | 120.2 (3) |
| C22$^{vi}$—K2—S1$^{ii}$ | 160.14 (6) | C11—C12—H12 | 119.9 |
| C23$^{vi}$—K2—S1$^{ii}$ | 175.50 (5) | C7—C12—H12 | 119.9 |
| O5—K2—S2 | 18.27 (6) | C18—C13—C14 | 120.9 (3) |
| O2W—K2—S2 | 166.25 (6) | C18—C13—S2 | 118.7 (2) |
| O7$^{ii}$—K2—S2 | 88.14 (5) | C14—C13—S2 | 120.3 (2) |
| O1$^{ii}$—K2—S2 | 93.22 (5) | C15—C14—C13 | 119.1 (3) |
| O8$^{vi}$—K2—S2 | 101.09 (5) | C15—C14—H14 | 120.4 |

| | | | |
|---|---|---|---|
| O3$^{ii}$—K2—S2 | 78.85 (4) | C13—C14—H14 | 120.4 |
| O1W—K2—S2 | 119.29 (5) | C16—C15—C14 | 119.8 (3) |
| C22$^{vi}$—K2—S2 | 90.29 (5) | C16—C15—H15 | 120.1 |
| C23$^{vi}$—K2—S2 | 100.72 (5) | C14—C15—H15 | 120.1 |
| S1$^{ii}$—K2—S2 | 82.67 (2) | C15—C16—C17 | 120.7 (3) |
| O5—K2—K1$^{vii}$ | 127.22 (6) | C15—C16—N3 | 115.7 (2) |
| O2W—K2—K1$^{vii}$ | 40.16 (5) | C17—C16—N3 | 123.6 (3) |
| O7$^{ii}$—K2—K1$^{vii}$ | 112.89 (5) | C18—C17—C16 | 119.6 (3) |
| O1$^{ii}$—K2—K1$^{vii}$ | 114.97 (5) | C18—C17—H17 | 120.2 |
| O8$^{vi}$—K2—K1$^{vii}$ | 45.95 (4) | C16—C17—H17 | 120.2 |
| O3$^{ii}$—K2—K1$^{vii}$ | 104.05 (5) | C17—C18—C13 | 119.8 (3) |
| O1W—K2—K1$^{vii}$ | 49.46 (5) | C17—C18—H18 | 120.1 |
| C22$^{vi}$—K2—K1$^{vii}$ | 62.99 (5) | C13—C18—H18 | 120.1 |
| C23$^{vi}$—K2—K1$^{vii}$ | 63.93 (5) | C20—C19—C24 | 120.4 (3) |
| S1$^{ii}$—K2—K1$^{vii}$ | 114.76 (2) | C20—C19—N4 | 116.1 (2) |
| S2—K2—K1$^{vii}$ | 145.49 (3) | C24—C19—N4 | 123.5 (3) |
| O5—K2—H1W | 100.8 (7) | C21—C20—C19 | 119.9 (3) |
| O2W—K2—H1W | 86.6 (5) | C21—C20—H20 | 120.0 |
| O7$^{ii}$—K2—H1W | 153.4 (8) | C19—C20—H20 | 120.0 |
| O1$^{ii}$—K2—H1W | 73.3 (6) | C20—C21—C22 | 120.1 (3) |
| O8$^{vi}$—K2—H1W | 84.4 (5) | C20—C21—H21 | 119.9 |
| O3$^{ii}$—K2—H1W | 38.4 (2) | C22—C21—H21 | 119.9 |
| O1W—K2—H1W | 16.6 (2) | O8—C22—C23 | 117.9 (2) |
| C22$^{vi}$—K2—H1W | 108.8 (6) | O8—C22—C21 | 122.1 (3) |
| C23$^{vi}$—K2—H1W | 124.5 (4) | C23—C22—C21 | 119.9 (3) |
| S1$^{ii}$—K2—H1W | 56.5 (4) | O8—C22—K2$^{vi}$ | 61.97 (14) |
| S2—K2—H1W | 107.2 (5) | C23—C22—K2$^{vi}$ | 83.25 (17) |
| K1$^{vii}$—K2—H1W | 66.0 (2) | C21—C22—K2$^{vi}$ | 123.43 (19) |
| O5—K2—H4W | 153.1 (7) | C24—C23—C22 | 120.7 (3) |
| O2W—K2—H4W | 17.1 (4) | C24—C23—K2$^{vi}$ | 129.2 (2) |
| O7$^{ii}$—K2—H4W | 62.3 (4) | C22—C23—K2$^{vi}$ | 72.86 (16) |
| O1$^{ii}$—K2—H4W | 90.9 (9) | C24—C23—H23 | 119.6 |
| O8$^{vi}$—K2—H4W | 86.3 (8) | C22—C23—H23 | 119.6 |
| O3$^{ii}$—K2—H4W | 124.8 (7) | K2$^{vi}$—C23—H23 | 70.0 |
| O1W—K2—H4W | 91.1 (4) | C23—C24—C19 | 118.9 (3) |
| C22$^{vi}$—K2—H4W | 84.4 (9) | C23—C24—H24 | 120.6 |
| C23$^{vi}$—K2—H4W | 64.8 (9) | C19—C24—H24 | 120.6 |

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|---|---|---|---|
| O2—S1—O1—K1 | −22.5 (2) | N2—C7—C8—C9 | −177.6 (3) |
| O3—S1—O1—K1 | −149.71 (19) | C7—C8—C9—C10 | −2.7 (5) |
| C1—S1—O1—K1 | 94.6 (2) | K1$^{iv}$—O4—C10—C9 | −75.6 (4) |
| K2$^{v}$—S1—O1—K1 | −136.2 (2) | K1$^{iv}$—O4—C10—C11 | 105.2 (3) |
| O2—S1—O1—K2$^{v}$ | 113.68 (12) | C8—C9—C10—O4 | −178.8 (3) |
| O3—S1—O1—K2$^{v}$ | −13.50 (14) | C8—C9—C10—C11 | 0.3 (4) |
| C1—S1—O1—K2$^{v}$ | −129.16 (11) | O4—C10—C11—C12 | −179.3 (3) |
| O1—S1—O2—K1$^{v}$ | −95.7 (2) | C9—C10—C11—C12 | 1.6 (4) |
| O3—S1—O2—K1$^{v}$ | 31.2 (2) | C10—C11—C12—C7 | −1.1 (5) |
| C1—S1—O2—K1$^{v}$ | 147.23 (17) | C8—C7—C12—C11 | −1.2 (5) |

| | | | |
|---|---|---|---|
| K2$^{v}$—S1—O2—K1$^{v}$ | −37.8 (2) | N2—C7—C12—C11 | 179.6 (3) |
| O1—S1—O3—K2$^{v}$ | 11.99 (12) | O7—S2—C13—C18 | 161.7 (2) |
| O2—S1—O3—K2$^{v}$ | −116.08 (11) | O6—S2—C13—C18 | −78.3 (3) |
| C1—S1—O3—K2$^{v}$ | 127.74 (11) | O5—S2—C13—C18 | 40.6 (3) |
| O7—S2—O5—K2 | 133.50 (15) | K1—S2—C13—C18 | −131.3 (2) |
| O6—S2—O5—K2 | 7.0 (2) | K2—S2—C13—C18 | 5.3 (3) |
| C13—S2—O5—K2 | −108.19 (16) | O7—S2—C13—C14 | −22.4 (3) |
| K1—S2—O5—K2 | 65.57 (17) | O6—S2—C13—C14 | 97.6 (3) |
| O7—S2—O6—K1 | −18.44 (16) | O5—S2—C13—C14 | −143.5 (3) |
| O5—S2—O6—K1 | 108.85 (13) | K1—S2—C13—C14 | 44.6 (3) |
| C13—S2—O6—K1 | −136.60 (12) | K2—S2—C13—C14 | −178.8 (2) |
| K2—S2—O6—K1 | 112.84 (9) | C18—C13—C14—C15 | 2.0 (5) |
| O6—S2—O7—K2$^{v}$ | 114.81 (18) | S2—C13—C14—C15 | −173.8 (2) |
| O5—S2—O7—K2$^{v}$ | −11.9 (2) | C13—C14—C15—C16 | 1.6 (4) |
| C13—S2—O7—K2$^{v}$ | −128.36 (16) | C14—C15—C16—C17 | −4.2 (4) |
| K1—S2—O7—K2$^{v}$ | 97.99 (15) | C14—C15—C16—N3 | 175.1 (3) |
| K2—S2—O7—K2$^{v}$ | 21.6 (2) | N4—N3—C16—C15 | −155.9 (3) |
| O6—S2—O7—K1 | 16.82 (14) | N4—N3—C16—C17 | 23.3 (4) |
| O5—S2—O7—K1 | −109.92 (12) | C15—C16—C17—C18 | 3.1 (4) |
| C13—S2—O7—K1 | 133.65 (11) | N3—C16—C17—C18 | −176.1 (3) |
| K2—S2—O7—K1 | −76.44 (11) | C16—C17—C18—C13 | 0.5 (5) |
| C4—N1—N2—C7 | 177.2 (2) | C14—C13—C18—C17 | −3.1 (5) |
| C16—N3—N4—C19 | 179.8 (2) | S2—C13—C18—C17 | 172.8 (2) |
| O1—S1—C1—C6 | −157.8 (2) | N3—N4—C19—C20 | −164.3 (3) |
| O2—S1—C1—C6 | −36.3 (3) | N3—N4—C19—C24 | 16.5 (4) |
| O3—S1—C1—C6 | 83.2 (3) | C24—C19—C20—C21 | −2.0 (4) |
| K2$^{v}$—S1—C1—C6 | 149.3 (2) | N4—C19—C20—C21 | 178.8 (3) |
| O1—S1—C1—C2 | 26.5 (3) | C19—C20—C21—C22 | 0.2 (4) |
| O2—S1—C1—C2 | 148.1 (2) | K2$^{vi}$—O8—C22—C23 | 64.3 (3) |
| O3—S1—C1—C2 | −92.4 (3) | K1$^{iii}$—O8—C22—C23 | −27.1 (4) |
| K2$^{v}$—S1—C1—C2 | −26.4 (3) | K2$^{vi}$—O8—C22—C21 | −113.8 (3) |
| C6—C1—C2—C3 | 0.7 (5) | K1$^{iii}$—O8—C22—C21 | 154.8 (2) |
| S1—C1—C2—C3 | 176.3 (2) | K1$^{iii}$—O8—C22—K2$^{vi}$ | −91.41 (16) |
| C1—C2—C3—C4 | −0.8 (5) | C20—C21—C22—O8 | 179.4 (3) |
| C2—C3—C4—C5 | 0.0 (5) | C20—C21—C22—C23 | 1.3 (4) |
| C2—C3—C4—N1 | −178.8 (3) | C20—C21—C22—K2$^{vi}$ | 103.9 (3) |
| N2—N1—C4—C5 | 157.3 (3) | O8—C22—C23—C24 | −179.2 (3) |
| N2—N1—C4—C3 | −23.8 (4) | C21—C22—C23—C24 | −1.0 (4) |
| C3—C4—C5—C6 | 0.8 (5) | K2$^{vi}$—C22—C23—C24 | −125.9 (3) |
| N1—C4—C5—C6 | 179.7 (3) | O8—C22—C23—K2$^{vi}$ | −53.2 (2) |
| C4—C5—C6—C1 | −0.8 (5) | C21—C22—C23—K2$^{vi}$ | 124.9 (3) |
| C2—C1—C6—C5 | 0.1 (5) | C22—C23—C24—C19 | −0.7 (4) |
| S1—C1—C6—C5 | −175.5 (2) | K2$^{vi}$—C23—C24—C19 | −93.2 (3) |
| N1—N2—C7—C8 | 162.4 (3) | C20—C19—C24—C23 | 2.3 (4) |
| N1—N2—C7—C12 | −18.4 (4) | N4—C19—C24—C23 | −178.5 (3) |
| C12—C7—C8—C9 | 3.1 (5) | | |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4*H*···O1*W*$^{viii}$ | 0.88 (1) | 1.83 (2) | 2.676 (3) | 160 (3) |
| O8—H8*H*···O6$^{ix}$ | 0.88 (1) | 1.93 (3) | 2.748 (3) | 155 (5) |
| O1*W*—H1*W*···O3$^{ii}$ | 0.87 (1) | 1.95 (2) | 2.801 (3) | 165 (4) |
| O1*W*—H2*W*···O2$^{vii}$ | 0.88 (1) | 1.93 (1) | 2.803 (4) | 177 (5) |
| O2*W*—H3*W*···O3$^{x}$ | 0.88 (1) | 2.20 (3) | 2.866 (3) | 133 (4) |
| O2*W*—H3*W*···O4$^{viii}$ | 0.88 (1) | 2.29 (3) | 2.905 (3) | 127 (3) |
| O2*W*—H4*W*···O5$^{ii}$ | 0.88 (1) | 1.92 (2) | 2.778 (3) | 165 (4) |

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

## Poly[diaqua{4-[2-(4-aminophenyl)diazen-1-yl]benzenesulfonato}potassium] (K3)

*Crystal data*

$[K(C_{12}H_{10}N_3O_3S)(H_2O)_2]$
$M_r$ = 351.42
Triclinic, $P\overline{1}$
*a* = 13.3058 (2) Å
*b* = 13.6247 (2) Å
*c* = 18.4664 (3) Å
*α* = 88.373 (1)°
*β* = 73.971 (1)°
*γ* = 66.313 (1)°
*V* = 2933.52 (8) Å$^3$
*Z* = 8
*F*(000) = 1456
$D_x$ = 1.591 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 13626 reflections
*θ* = 0.4–27.5°
*μ* = 0.53 mm$^{-1}$
*T* = 123 K
Sheet, yellow
0.7 × 0.3 × 0.02 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
*ω* and phi scans
26000 measured reflections
13392 independent reflections
10848 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.6°
*h* = −17→17
*k* = −17→17
*l* = −23→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
*S* = 1.03
13392 reflections
895 parameters
24 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 2.5026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.80 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.79 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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| K1 | 0.36432 (3) | 0.16149 (3) | 0.04727 (2) | 0.01821 (9) | |
| K2 | 0.56585 (4) | 0.34009 (3) | 0.00078 (2) | 0.02089 (10) | |
| K3 | 0.91040 (4) | 0.14309 (4) | 0.03328 (3) | 0.03042 (11) | |
| K4 | 1.11003 (4) | 0.34080 (3) | −0.05349 (2) | 0.02172 (10) | |
| S1 | 0.60885 (4) | 0.15844 (4) | 0.13423 (2) | 0.01617 (10) | |
| S1A | 0.58680 (4) | 0.12938 (4) | −0.11362 (2) | 0.01558 (10) | |
| S1B | 0.90352 (4) | 0.37392 (4) | 0.11946 (2) | 0.01655 (10) | |
| S1C | 0.88336 (4) | 0.33155 (4) | −0.12057 (2) | 0.01540 (10) | |
| O1 | 0.66126 (12) | 0.04249 (11) | 0.14108 (8) | 0.0230 (3) | |
| O1A | 0.56604 (12) | 0.04564 (11) | −0.07064 (7) | 0.0224 (3) | |
| O1B | 0.99145 (14) | 0.26429 (11) | 0.09571 (8) | 0.0291 (3) | |
| O2 | 0.50128 (12) | 0.19267 (11) | 0.11594 (8) | 0.0235 (3) | |
| O1W | 0.17161 (13) | 0.17402 (12) | 0.16143 (8) | 0.0256 (3) | |
| O2A | 0.49160 (12) | 0.23543 (11) | −0.08722 (8) | 0.0234 (3) | |
| O2B | 0.93443 (13) | 0.45183 (11) | 0.07491 (8) | 0.0268 (3) | |
| O3 | 0.68945 (12) | 0.19462 (11) | 0.08320 (7) | 0.0225 (3) | |
| O2W | 0.38778 (13) | 0.44516 (13) | 0.13668 (9) | 0.0281 (3) | |
| O3A | 0.69498 (12) | 0.13426 (13) | −0.11593 (8) | 0.0279 (3) | |
| O3B | 0.79182 (13) | 0.37860 (15) | 0.12144 (9) | 0.0365 (4) | |
| O3W | 0.60988 (13) | 0.50992 (12) | 0.05734 (8) | 0.0257 (3) | |
| O4W | 0.90005 (13) | −0.02527 (12) | 0.13023 (9) | 0.0282 (3) | |
| O5W | 1.12711 (13) | −0.00124 (12) | 0.05792 (9) | 0.0293 (3) | |
| H9W | 1.1620 (18) | −0.0209 (19) | 0.0100 (6) | 0.035* | |
| H10W | 1.1896 (14) | −0.0422 (17) | 0.0707 (12) | 0.035* | |
| O6W | 1.22994 (13) | 0.13309 (11) | −0.03169 (8) | 0.0233 (3) | |
| O7W | 1.24230 (13) | 0.37602 (11) | 0.03183 (8) | 0.0255 (3) | |
| O8W | 1.31574 (13) | 0.33491 (12) | −0.15548 (9) | 0.0275 (3) | |
| H15W | 1.3758 (15) | 0.3093 (17) | −0.1379 (13) | 0.033* | |
| H16W | 1.324 (2) | 0.3912 (13) | −0.1745 (13) | 0.033* | |
| O1C | 0.76263 (16) | 0.35833 (19) | −0.08326 (10) | 0.0220 (4) | 0.878 (4) |
| O2C | 0.91077 (19) | 0.42551 (14) | −0.12630 (9) | 0.0228 (4) | 0.878 (4) |
| O3C | 0.95984 (17) | 0.24694 (16) | −0.08543 (11) | 0.0239 (4) | 0.878 (4) |
| O4C | 0.7885 (13) | 0.3113 (13) | −0.0773 (8) | 0.0220 (4) | 0.122 (4) |
| O5C | 0.8522 (14) | 0.4454 (11) | −0.1293 (7) | 0.0228 (4) | 0.122 (4) |
| O6C | 0.9823 (14) | 0.2762 (13) | −0.1002 (9) | 0.0239 (4) | 0.122 (4) |
| N1 | 0.49955 (13) | 0.33938 (12) | 0.44977 (9) | 0.0172 (3) | |
| N1A | 0.62709 (13) | 0.03685 (12) | −0.43692 (9) | 0.0173 (3) | |
| N1B | 0.87099 (13) | 0.46335 (12) | 0.44147 (9) | 0.0176 (3) | |
| N1C | 1.00015 (13) | 0.16488 (12) | −0.44137 (9) | 0.0168 (3) | |
| N2 | 0.58070 (13) | 0.34190 (12) | 0.47114 (9) | 0.0171 (3) | |
| N2A | 0.63343 (13) | −0.05449 (12) | −0.45571 (9) | 0.0176 (3) | |
| N2B | 0.86504 (13) | 0.55398 (12) | 0.46122 (9) | 0.0173 (3) | |
| N2C | 0.92154 (13) | 0.15932 (12) | −0.46417 (9) | 0.0174 (3) | |
| N3 | 0.46770 (17) | 0.50272 (14) | 0.77320 (9) | 0.0212 (3) | |
| N3A | 0.69006 (15) | −0.15131 (16) | −0.76079 (10) | 0.0226 (4) | |

| | | | | |
|---|---|---|---|---|
| N3B | 0.81564 (15) | 0.63159 (16) | 0.76852 (10) | 0.0238 (4) |
| N3C | 1.04330 (17) | 0.00391 (14) | −0.76838 (9) | 0.0221 (4) |
| C1 | 0.57751 (15) | 0.21914 (14) | 0.22572 (10) | 0.0147 (3) |
| C1A | 0.59373 (15) | 0.10016 (14) | −0.20806 (10) | 0.0146 (3) |
| C1B | 0.89745 (15) | 0.40302 (14) | 0.21380 (10) | 0.0152 (3) |
| C1C | 0.91372 (15) | 0.27882 (14) | −0.21423 (10) | 0.0149 (3) |
| C2 | 0.46687 (16) | 0.25495 (14) | 0.27370 (10) | 0.0174 (4) |
| H2 | 0.406898 | 0.252756 | 0.256284 | 0.021* |
| C2A | 0.59565 (16) | 0.00262 (15) | −0.23074 (10) | 0.0173 (4) |
| H2A | 0.589860 | −0.047690 | −0.194892 | 0.021* |
| C2B | 0.89368 (16) | 0.50142 (15) | 0.23675 (10) | 0.0174 (4) |
| H2B | 0.897118 | 0.552580 | 0.201182 | 0.021* |
| C2C | 0.82832 (15) | 0.27106 (14) | −0.24037 (10) | 0.0166 (4) |
| H2C | 0.753033 | 0.291570 | −0.207218 | 0.020* |
| C3 | 0.44475 (16) | 0.29419 (15) | 0.34773 (11) | 0.0184 (4) |
| H3 | 0.369302 | 0.318596 | 0.381298 | 0.022* |
| C3A | 0.60606 (15) | −0.02100 (15) | −0.30590 (10) | 0.0169 (4) |
| H3A | 0.607199 | −0.087354 | −0.321551 | 0.020* |
| C3B | 0.88492 (15) | 0.52458 (15) | 0.31145 (10) | 0.0169 (4) |
| H3B | 0.882456 | 0.591479 | 0.327216 | 0.020* |
| C3C | 0.85370 (16) | 0.23313 (14) | −0.31523 (10) | 0.0166 (4) |
| H3C | 0.796056 | 0.226894 | −0.333317 | 0.020* |
| C4 | 0.53297 (16) | 0.29782 (14) | 0.37283 (10) | 0.0156 (4) |
| C4A | 0.61481 (15) | 0.05321 (14) | −0.35826 (10) | 0.0158 (4) |
| C4B | 0.87976 (15) | 0.44910 (15) | 0.36324 (10) | 0.0162 (4) |
| C4C | 0.96408 (15) | 0.20436 (14) | −0.36353 (10) | 0.0154 (3) |
| C5 | 0.64367 (15) | 0.26369 (14) | 0.32360 (10) | 0.0168 (4) |
| H5 | 0.703310 | 0.267634 | 0.340529 | 0.020* |
| C5A | 0.61020 (16) | 0.15177 (15) | −0.33499 (10) | 0.0179 (4) |
| H5A | 0.614125 | 0.202845 | −0.370557 | 0.022* |
| C5B | 0.88510 (16) | 0.35028 (15) | 0.34002 (10) | 0.0179 (4) |
| H5B | 0.882686 | 0.298779 | 0.375479 | 0.022* |
| C5C | 1.04950 (16) | 0.21144 (15) | −0.33698 (10) | 0.0179 (4) |
| H5C | 1.124708 | 0.191197 | −0.370185 | 0.022* |
| C6 | 0.66621 (16) | 0.22407 (15) | 0.24999 (11) | 0.0180 (4) |
| H6 | 0.741426 | 0.200389 | 0.216181 | 0.022* |
| C6A | 0.59988 (16) | 0.17544 (14) | −0.25996 (10) | 0.0167 (4) |
| H6A | 0.597033 | 0.242455 | −0.244075 | 0.020* |
| C6B | 0.89394 (16) | 0.32676 (15) | 0.26520 (10) | 0.0176 (4) |
| H6B | 0.897544 | 0.259428 | 0.249272 | 0.021* |
| C6C | 1.02495 (16) | 0.24797 (15) | −0.26216 (10) | 0.0173 (4) |
| H6C | 1.083268 | 0.251967 | −0.243697 | 0.021* |
| C7 | 0.54673 (16) | 0.38451 (14) | 0.54765 (10) | 0.0157 (4) |
| C7A | 0.64460 (15) | −0.07259 (14) | −0.53316 (10) | 0.0158 (4) |
| C7B | 0.85779 (15) | 0.56769 (15) | 0.53871 (10) | 0.0162 (4) |
| C7C | 0.95806 (16) | 0.11810 (14) | −0.54119 (10) | 0.0155 (4) |
| C8 | 0.43404 (16) | 0.42187 (15) | 0.59522 (10) | 0.0173 (4) |
| H8 | 0.374791 | 0.420291 | 0.576679 | 0.021* |

| | | | | |
|---|---|---|---|---|
| C8A | 0.65069 (15) | −0.17206 (15) | −0.55619 (11) | 0.0178 (4) |
| H8A | 0.646107 | −0.222375 | −0.520123 | 0.021* |
| C8B | 0.84642 (16) | 0.66756 (15) | 0.56518 (11) | 0.0182 (4) |
| H8B | 0.846841 | 0.721222 | 0.531250 | 0.022* |
| C8C | 0.87466 (16) | 0.11037 (15) | −0.56976 (10) | 0.0177 (4) |
| H8C | 0.798442 | 0.131704 | −0.537895 | 0.021* |
| C9 | 0.40933 (16) | 0.46096 (15) | 0.66906 (11) | 0.0186 (4) |
| H9 | 0.332944 | 0.485441 | 0.701215 | 0.022* |
| C9A | 0.66334 (16) | −0.19812 (15) | −0.63115 (11) | 0.0180 (4) |
| H9A | 0.667436 | −0.266082 | −0.646125 | 0.022* |
| C9B | 0.83449 (16) | 0.68896 (15) | 0.64097 (11) | 0.0190 (4) |
| H9B | 0.826049 | 0.757445 | 0.658631 | 0.023* |
| C9C | 0.90248 (16) | 0.07160 (15) | −0.64467 (11) | 0.0185 (4) |
| H9C | 0.845055 | 0.066993 | −0.663857 | 0.022* |
| C10 | 0.49517 (16) | 0.46511 (14) | 0.69734 (10) | 0.0168 (4) |
| C10A | 0.67011 (15) | −0.12459 (15) | −0.68493 (10) | 0.0175 (4) |
| C10B | 0.83482 (15) | 0.61027 (15) | 0.69145 (10) | 0.0177 (4) |
| C10C | 1.01397 (16) | 0.03949 (14) | −0.69172 (10) | 0.0167 (4) |
| C11 | 0.60659 (16) | 0.42965 (15) | 0.64948 (11) | 0.0181 (4) |
| H11 | 0.665465 | 0.432619 | 0.667803 | 0.022* |
| C11A | 0.66057 (16) | −0.02318 (15) | −0.66082 (11) | 0.0197 (4) |
| H11A | 0.663013 | 0.028064 | −0.696478 | 0.024* |
| C11B | 0.85061 (16) | 0.50880 (15) | 0.66346 (11) | 0.0192 (4) |
| H11B | 0.853477 | 0.453906 | 0.696747 | 0.023* |
| C11C | 1.09807 (16) | 0.04553 (15) | −0.66239 (11) | 0.0187 (4) |
| H11C | 1.174597 | 0.022944 | −0.694008 | 0.022* |
| C12 | 0.63237 (16) | 0.38987 (15) | 0.57499 (11) | 0.0186 (4) |
| H12 | 0.708552 | 0.366292 | 0.542639 | 0.022* |
| C12A | 0.64776 (16) | 0.00269 (15) | −0.58628 (11) | 0.0179 (4) |
| H12A | 0.641058 | 0.071506 | −0.570813 | 0.021* |
| C12B | 0.86200 (16) | 0.48764 (15) | 0.58844 (11) | 0.0181 (4) |
| H12B | 0.872754 | 0.418489 | 0.570389 | 0.022* |
| C12C | 1.07081 (16) | 0.08399 (15) | −0.58800 (10) | 0.0175 (4) |
| H12C | 1.128572 | 0.087303 | −0.568568 | 0.021* |
| H1N | 0.395 (2) | 0.554 (2) | 0.7950 (14) | 0.031 (6)* |
| H2N | 0.516 (2) | 0.518 (2) | 0.7822 (15) | 0.033 (7)* |
| H3N | 0.673 (2) | −0.200 (2) | −0.7689 (15) | 0.039 (8)* |
| H4N | 0.666 (2) | −0.097 (2) | −0.7876 (15) | 0.036 (7)* |
| H5N | 0.852 (2) | 0.576 (2) | 0.7920 (15) | 0.037 (7)* |
| H6N | 0.831 (2) | 0.685 (2) | 0.7808 (15) | 0.035 (7)* |
| H7N | 0.991 (2) | −0.009 (2) | −0.7789 (16) | 0.043 (8)* |
| H8N | 1.111 (2) | −0.047 (2) | −0.7869 (14) | 0.031 (7)* |
| H1W | 0.155 (2) | 0.1254 (19) | 0.1873 (15) | 0.064 (10)* |
| H2W | 0.1166 (15) | 0.1971 (18) | 0.1402 (12) | 0.031 (7)* |
| H3W | 0.3304 (17) | 0.4996 (15) | 0.1291 (17) | 0.056 (9)* |
| H4W | 0.358 (2) | 0.3993 (17) | 0.1527 (17) | 0.063 (10)* |
| H5W | 0.581 (2) | 0.531 (3) | 0.1053 (7) | 0.082 (12)* |
| H6W | 0.6785 (12) | 0.4633 (18) | 0.0554 (15) | 0.049 (8)* |

| | | | | |
|---|---|---|---|---|
| H7W | 0.8265 (9) | −0.0080 (19) | 0.1414 (16) | 0.047 (8)* |
| H8W | 0.9269 (19) | −0.0950 (8) | 0.1270 (16) | 0.045 (8)* |
| H11W | 1.187 (2) | 0.110 (2) | 0.0020 (12) | 0.050 (9)* |
| H12W | 1.275 (2) | 0.0765 (15) | −0.0625 (13) | 0.057 (9)* |
| H13W | 1.1940 (18) | 0.4350 (14) | 0.0606 (13) | 0.052 (9)* |
| H14W | 1.2900 (18) | 0.400 (2) | 0.0029 (13) | 0.049 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| K1 | 0.01762 (19) | 0.0189 (2) | 0.01943 (19) | −0.00785 (16) | −0.00678 (15) | 0.00224 (15) |
| K2 | 0.0220 (2) | 0.0183 (2) | 0.0234 (2) | −0.00772 (16) | −0.00867 (17) | 0.00016 (16) |
| K3 | 0.0264 (2) | 0.0278 (2) | 0.0359 (3) | −0.0133 (2) | −0.0032 (2) | −0.00333 (19) |
| K4 | 0.0225 (2) | 0.0200 (2) | 0.0208 (2) | −0.00755 (17) | −0.00514 (17) | 0.00190 (16) |
| S1 | 0.0161 (2) | 0.0179 (2) | 0.0145 (2) | −0.00651 (17) | −0.00479 (17) | −0.00092 (16) |
| S1A | 0.0172 (2) | 0.0162 (2) | 0.0146 (2) | −0.00747 (17) | −0.00571 (17) | 0.00230 (16) |
| S1B | 0.0171 (2) | 0.0197 (2) | 0.0143 (2) | −0.00834 (18) | −0.00570 (17) | 0.00287 (17) |
| S1C | 0.0157 (2) | 0.0162 (2) | 0.0139 (2) | −0.00658 (17) | −0.00328 (17) | −0.00098 (16) |
| O1 | 0.0270 (7) | 0.0178 (7) | 0.0206 (7) | −0.0065 (6) | −0.0051 (6) | −0.0022 (5) |
| O1A | 0.0317 (8) | 0.0200 (7) | 0.0152 (6) | −0.0124 (6) | −0.0034 (6) | 0.0026 (5) |
| O1B | 0.0400 (9) | 0.0211 (7) | 0.0234 (7) | −0.0050 (6) | −0.0159 (7) | −0.0036 (6) |
| O2 | 0.0198 (7) | 0.0300 (8) | 0.0218 (7) | −0.0090 (6) | −0.0092 (6) | −0.0023 (6) |
| O1W | 0.0237 (7) | 0.0269 (8) | 0.0246 (7) | −0.0088 (6) | −0.0068 (6) | 0.0037 (6) |
| O2A | 0.0293 (8) | 0.0171 (7) | 0.0213 (7) | −0.0045 (6) | −0.0112 (6) | −0.0021 (5) |
| O2B | 0.0412 (9) | 0.0249 (7) | 0.0153 (7) | −0.0181 (7) | −0.0031 (6) | 0.0030 (6) |
| O3 | 0.0229 (7) | 0.0296 (8) | 0.0167 (7) | −0.0136 (6) | −0.0045 (6) | 0.0044 (6) |
| O2W | 0.0238 (8) | 0.0273 (8) | 0.0330 (8) | −0.0092 (7) | −0.0102 (7) | 0.0027 (7) |
| O3A | 0.0242 (7) | 0.0443 (9) | 0.0244 (7) | −0.0199 (7) | −0.0127 (6) | 0.0090 (7) |
| O3B | 0.0242 (8) | 0.0722 (12) | 0.0224 (8) | −0.0264 (8) | −0.0114 (6) | 0.0106 (8) |
| O3W | 0.0290 (8) | 0.0219 (7) | 0.0251 (8) | −0.0075 (6) | −0.0105 (6) | 0.0018 (6) |
| O4W | 0.0220 (8) | 0.0272 (8) | 0.0359 (9) | −0.0093 (6) | −0.0101 (7) | 0.0025 (7) |
| O5W | 0.0285 (8) | 0.0292 (8) | 0.0333 (8) | −0.0107 (7) | −0.0159 (7) | 0.0064 (7) |
| O6W | 0.0253 (8) | 0.0214 (7) | 0.0206 (7) | −0.0071 (6) | −0.0060 (6) | −0.0036 (6) |
| O7W | 0.0297 (8) | 0.0208 (7) | 0.0230 (7) | −0.0084 (6) | −0.0059 (6) | 0.0009 (6) |
| O8W | 0.0272 (8) | 0.0265 (8) | 0.0303 (8) | −0.0103 (6) | −0.0122 (7) | 0.0040 (6) |
| O1C | 0.0169 (9) | 0.0284 (12) | 0.0186 (7) | −0.0099 (9) | −0.0004 (6) | −0.0047 (8) |
| O2C | 0.0276 (12) | 0.0219 (8) | 0.0209 (8) | −0.0156 (8) | −0.0011 (8) | −0.0036 (6) |
| O3C | 0.0296 (10) | 0.0208 (10) | 0.0174 (9) | −0.0041 (7) | −0.0102 (8) | 0.0018 (7) |
| O4C | 0.0169 (9) | 0.0284 (12) | 0.0186 (7) | −0.0099 (9) | −0.0004 (6) | −0.0047 (8) |
| O5C | 0.0276 (12) | 0.0219 (8) | 0.0209 (8) | −0.0156 (8) | −0.0011 (8) | −0.0036 (6) |
| O6C | 0.0296 (10) | 0.0208 (10) | 0.0174 (9) | −0.0041 (7) | −0.0102 (8) | 0.0018 (7) |
| N1 | 0.0179 (8) | 0.0182 (8) | 0.0163 (7) | −0.0079 (6) | −0.0054 (6) | 0.0003 (6) |
| N1A | 0.0177 (8) | 0.0191 (8) | 0.0155 (7) | −0.0076 (6) | −0.0052 (6) | 0.0009 (6) |
| N1B | 0.0176 (8) | 0.0194 (8) | 0.0169 (8) | −0.0079 (6) | −0.0059 (6) | 0.0013 (6) |
| N1C | 0.0181 (8) | 0.0173 (7) | 0.0156 (7) | −0.0076 (6) | −0.0049 (6) | 0.0001 (6) |
| N2 | 0.0182 (8) | 0.0168 (8) | 0.0168 (7) | −0.0074 (6) | −0.0052 (6) | 0.0003 (6) |
| N2A | 0.0163 (8) | 0.0198 (8) | 0.0160 (8) | −0.0066 (6) | −0.0046 (6) | 0.0010 (6) |
| N2B | 0.0152 (7) | 0.0193 (8) | 0.0163 (8) | −0.0060 (6) | −0.0043 (6) | 0.0008 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N2C | 0.0182 (8) | 0.0175 (8) | 0.0163 (7) | −0.0067 (6) | −0.0054 (6) | 0.0005 (6) |
| N3 | 0.0243 (9) | 0.0227 (9) | 0.0179 (8) | −0.0096 (8) | −0.0079 (7) | 0.0004 (7) |
| N3A | 0.0252 (9) | 0.0265 (9) | 0.0183 (8) | −0.0116 (8) | −0.0081 (7) | 0.0016 (7) |
| N3B | 0.0217 (9) | 0.0293 (10) | 0.0182 (8) | −0.0073 (8) | −0.0069 (7) | −0.0027 (7) |
| N3C | 0.0244 (9) | 0.0233 (9) | 0.0165 (8) | −0.0070 (8) | −0.0064 (7) | −0.0016 (7) |
| C1 | 0.0159 (8) | 0.0132 (8) | 0.0147 (8) | −0.0058 (7) | −0.0045 (7) | 0.0018 (7) |
| C1A | 0.0121 (8) | 0.0167 (8) | 0.0137 (8) | −0.0052 (7) | −0.0030 (7) | 0.0009 (7) |
| C1B | 0.0128 (8) | 0.0182 (9) | 0.0138 (8) | −0.0059 (7) | −0.0035 (7) | 0.0010 (7) |
| C1C | 0.0173 (9) | 0.0136 (8) | 0.0138 (8) | −0.0064 (7) | −0.0044 (7) | 0.0006 (6) |
| C2 | 0.0152 (9) | 0.0183 (9) | 0.0195 (9) | −0.0070 (7) | −0.0061 (7) | 0.0013 (7) |
| C2A | 0.0177 (9) | 0.0176 (9) | 0.0181 (9) | −0.0084 (7) | −0.0058 (7) | 0.0039 (7) |
| C2B | 0.0180 (9) | 0.0178 (9) | 0.0172 (9) | −0.0081 (7) | −0.0055 (7) | 0.0045 (7) |
| C2C | 0.0146 (8) | 0.0173 (9) | 0.0172 (9) | −0.0065 (7) | −0.0034 (7) | 0.0011 (7) |
| C3 | 0.0142 (9) | 0.0198 (9) | 0.0199 (9) | −0.0067 (7) | −0.0034 (7) | 0.0000 (7) |
| C3A | 0.0177 (9) | 0.0157 (9) | 0.0182 (9) | −0.0077 (7) | −0.0051 (7) | 0.0009 (7) |
| C3B | 0.0168 (9) | 0.0157 (9) | 0.0190 (9) | −0.0078 (7) | −0.0046 (7) | 0.0018 (7) |
| C3C | 0.0173 (9) | 0.0166 (9) | 0.0174 (9) | −0.0080 (7) | −0.0057 (7) | 0.0012 (7) |
| C4 | 0.0187 (9) | 0.0127 (8) | 0.0158 (8) | −0.0064 (7) | −0.0053 (7) | 0.0011 (7) |
| C4A | 0.0130 (8) | 0.0186 (9) | 0.0161 (9) | −0.0064 (7) | −0.0048 (7) | 0.0021 (7) |
| C4B | 0.0128 (8) | 0.0202 (9) | 0.0147 (8) | −0.0057 (7) | −0.0044 (7) | 0.0020 (7) |
| C4C | 0.0171 (9) | 0.0126 (8) | 0.0153 (8) | −0.0057 (7) | −0.0036 (7) | 0.0010 (7) |
| C5 | 0.0151 (8) | 0.0179 (9) | 0.0191 (9) | −0.0075 (7) | −0.0066 (7) | 0.0009 (7) |
| C5A | 0.0193 (9) | 0.0178 (9) | 0.0178 (9) | −0.0083 (7) | −0.0064 (7) | 0.0051 (7) |
| C5B | 0.0203 (9) | 0.0172 (9) | 0.0172 (9) | −0.0084 (7) | −0.0059 (7) | 0.0045 (7) |
| C5C | 0.0150 (9) | 0.0200 (9) | 0.0174 (9) | −0.0067 (7) | −0.0030 (7) | −0.0004 (7) |
| C6 | 0.0153 (9) | 0.0193 (9) | 0.0187 (9) | −0.0069 (7) | −0.0039 (7) | −0.0007 (7) |
| C6A | 0.0187 (9) | 0.0151 (8) | 0.0173 (9) | −0.0077 (7) | −0.0058 (7) | 0.0019 (7) |
| C6B | 0.0183 (9) | 0.0165 (9) | 0.0181 (9) | −0.0079 (7) | −0.0043 (7) | 0.0009 (7) |
| C6C | 0.0156 (9) | 0.0198 (9) | 0.0182 (9) | −0.0078 (7) | −0.0066 (7) | 0.0012 (7) |
| C7 | 0.0181 (9) | 0.0137 (8) | 0.0161 (8) | −0.0072 (7) | −0.0054 (7) | 0.0018 (7) |
| C7A | 0.0145 (8) | 0.0179 (9) | 0.0152 (8) | −0.0068 (7) | −0.0045 (7) | 0.0006 (7) |
| C7B | 0.0131 (8) | 0.0193 (9) | 0.0150 (8) | −0.0061 (7) | −0.0029 (7) | 0.0005 (7) |
| C7C | 0.0185 (9) | 0.0138 (8) | 0.0137 (8) | −0.0059 (7) | −0.0052 (7) | 0.0020 (7) |
| C8 | 0.0168 (9) | 0.0184 (9) | 0.0179 (9) | −0.0082 (7) | −0.0055 (7) | 0.0027 (7) |
| C8A | 0.0156 (9) | 0.0189 (9) | 0.0197 (9) | −0.0071 (7) | −0.0065 (7) | 0.0038 (7) |
| C8B | 0.0168 (9) | 0.0182 (9) | 0.0205 (9) | −0.0073 (7) | −0.0067 (7) | 0.0023 (7) |
| C8C | 0.0169 (9) | 0.0175 (9) | 0.0184 (9) | −0.0069 (7) | −0.0050 (7) | 0.0021 (7) |
| C9 | 0.0169 (9) | 0.0202 (9) | 0.0176 (9) | −0.0075 (7) | −0.0033 (7) | 0.0021 (7) |
| C9A | 0.0166 (9) | 0.0170 (9) | 0.0205 (9) | −0.0067 (7) | −0.0055 (7) | −0.0003 (7) |
| C9B | 0.0163 (9) | 0.0189 (9) | 0.0218 (9) | −0.0067 (7) | −0.0060 (7) | −0.0029 (7) |
| C9C | 0.0190 (9) | 0.0185 (9) | 0.0207 (9) | −0.0081 (7) | −0.0095 (8) | 0.0029 (7) |
| C10 | 0.0229 (9) | 0.0134 (8) | 0.0157 (8) | −0.0081 (7) | −0.0073 (7) | 0.0039 (7) |
| C10A | 0.0118 (8) | 0.0216 (9) | 0.0179 (9) | −0.0054 (7) | −0.0045 (7) | −0.0009 (7) |
| C10B | 0.0113 (8) | 0.0235 (9) | 0.0171 (9) | −0.0057 (7) | −0.0041 (7) | −0.0012 (7) |
| C10C | 0.0223 (9) | 0.0123 (8) | 0.0150 (8) | −0.0057 (7) | −0.0067 (7) | 0.0023 (7) |
| C11 | 0.0175 (9) | 0.0176 (9) | 0.0226 (9) | −0.0079 (7) | −0.0101 (8) | 0.0019 (7) |
| C11A | 0.0213 (9) | 0.0214 (9) | 0.0192 (9) | −0.0109 (8) | −0.0073 (8) | 0.0060 (7) |
| C11B | 0.0196 (9) | 0.0219 (9) | 0.0182 (9) | −0.0096 (8) | −0.0072 (7) | 0.0051 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11C | 0.0182 (9) | 0.0197 (9) | 0.0181 (9) | −0.0083 (7) | −0.0042 (7) | 0.0023 (7) |
| C12 | 0.0169 (9) | 0.0172 (9) | 0.0213 (9) | −0.0069 (7) | −0.0048 (7) | 0.0010 (7) |
| C12A | 0.0191 (9) | 0.0174 (9) | 0.0195 (9) | −0.0089 (7) | −0.0072 (7) | 0.0023 (7) |
| C12B | 0.0175 (9) | 0.0186 (9) | 0.0190 (9) | −0.0081 (7) | −0.0053 (7) | 0.0005 (7) |
| C12C | 0.0156 (9) | 0.0196 (9) | 0.0185 (9) | −0.0073 (7) | −0.0066 (7) | 0.0018 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| K1—O2 | 2.6531 (14) | N2C—C7C | 1.421 (2) |
| K1—O1A$^{i}$ | 2.6570 (14) | N3—C10 | 1.402 (2) |
| K1—O6W$^{ii}$ | 2.7388 (15) | N3—H1N | 0.92 (3) |
| K1—O7W$^{ii}$ | 2.7669 (15) | N3—H2N | 0.81 (3) |
| K1—O1W | 2.7806 (15) | N3A—C10A | 1.382 (2) |
| K1—O1A | 2.8284 (14) | N3A—H3N | 0.82 (3) |
| K1—O2A | 2.9928 (15) | N3A—H4N | 0.88 (3) |
| K1—S1A | 3.4580 (6) | N3B—C10B | 1.392 (2) |
| K1—K4$^{ii}$ | 4.1781 (6) | N3B—H5N | 0.90 (3) |
| K1—K2 | 4.2073 (6) | N3B—H6N | 0.88 (3) |
| K1—K1$^{i}$ | 4.4315 (8) | N3C—C10C | 1.405 (2) |
| K2—O3 | 2.7462 (14) | N3C—H7N | 0.86 (3) |
| K2—O1C | 2.7536 (17) | N3C—H8N | 0.87 (3) |
| K2—O4C | 2.784 (14) | C1—C2 | 1.385 (2) |
| K2—O2A | 2.7929 (14) | C1—C6 | 1.399 (3) |
| K2—O2W | 2.8559 (16) | C1A—C2A | 1.393 (3) |
| K2—O3W$^{iii}$ | 2.8666 (15) | C1A—C6A | 1.395 (2) |
| K2—O3W | 2.8901 (16) | C1B—C6B | 1.393 (3) |
| K2—O2 | 3.0708 (15) | C1B—C2B | 1.395 (3) |
| K2—O3A | 3.1446 (16) | C1C—C2C | 1.392 (3) |
| K2—S1 | 3.4381 (6) | C1C—C6C | 1.396 (3) |
| K2—S1A | 3.4869 (6) | C2—C3 | 1.391 (3) |
| K2—K2$^{iii}$ | 3.9929 (8) | C2—H2 | 0.9500 |
| K2—H6W | 3.00 (3) | C2A—C3A | 1.390 (3) |
| K3—O3 | 2.6206 (14) | C2A—H2A | 0.9500 |
| K3—O3C | 2.649 (2) | C2B—C3B | 1.386 (3) |
| K3—O1B | 2.7285 (16) | C2B—H2B | 0.9500 |
| K3—O5W$^{iv}$ | 2.8713 (16) | C2C—C3C | 1.391 (2) |
| K3—O4W | 2.8932 (16) | C2C—H2C | 0.9500 |
| K3—O5W | 2.9166 (15) | C3—C4 | 1.394 (3) |
| K3—O6C | 3.181 (17) | C3—H3 | 0.9500 |
| K3—O3B | 3.2009 (19) | C3A—C4A | 1.396 (3) |
| K3—O4C | 3.261 (16) | C3A—H3A | 0.9500 |
| K3—O4W$^{iv}$ | 3.2971 (17) | C3B—C4B | 1.393 (3) |
| K3—S1B | 3.5219 (7) | C3B—H3B | 0.9500 |
| K3—K3$^{iv}$ | 3.6855 (9) | C3C—C4C | 1.393 (3) |
| K3—H9W$^{iv}$ | 2.48 (2) | C3C—H3C | 0.9500 |
| K4—O6C | 2.542 (17) | C4—C5 | 1.395 (3) |
| K4—O2B$^{v}$ | 2.6854 (15) | C4A—C5A | 1.394 (3) |
| K4—O6W | 2.7164 (15) | C4B—C5B | 1.393 (3) |

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|---|---|---|---|
| K4—O2B | 2.7701 (15) | C4C—C5C | 1.393 (3) |
| K4—O8W | 2.8338 (16) | C5—C6 | 1.384 (3) |
| K4—O7W | 2.8395 (16) | C5—H5 | 0.9500 |
| K4—O3C | 2.954 (2) | C5A—C6A | 1.388 (3) |
| K4—O2C | 3.097 (2) | C5A—H5A | 0.9500 |
| K4—O1B | 3.1503 (16) | C5B—C6B | 1.389 (3) |
| K4—S1B | 3.4918 (6) | C5B—H5B | 0.9500 |
| K4—S1C | 3.6089 (6) | C5C—C6C | 1.387 (2) |
| S1—O2 | 1.4497 (14) | C5C—H5C | 0.9500 |
| S1—O3 | 1.4565 (14) | C6—H6 | 0.9500 |
| S1—O1 | 1.4664 (14) | C6A—H6A | 0.9500 |
| S1—C1 | 1.7680 (18) | C6B—H6B | 0.9500 |
| S1A—O1A | 1.4452 (14) | C6C—H6C | 0.9500 |
| S1A—O3A | 1.4564 (14) | C7—C12 | 1.395 (3) |
| S1A—O2A | 1.4691 (14) | C7—C8 | 1.401 (3) |
| S1A—C1A | 1.7690 (18) | C7A—C8A | 1.396 (3) |
| S1B—O2B | 1.4447 (14) | C7A—C12A | 1.404 (3) |
| S1B—O3B | 1.4519 (15) | C7B—C8B | 1.395 (3) |
| S1B—O1B | 1.4635 (15) | C7B—C12B | 1.399 (3) |
| S1B—C1B | 1.7706 (18) | C7C—C8C | 1.393 (3) |
| S1C—O6C | 1.380 (16) | C7C—C12C | 1.400 (3) |
| S1C—O4C | 1.421 (15) | C8—C9 | 1.380 (3) |
| S1C—O5C | 1.453 (13) | C8—H8 | 0.9500 |
| S1C—O1C | 1.4562 (18) | C8A—C9A | 1.386 (3) |
| S1C—O2C | 1.4592 (17) | C8A—H8A | 0.9500 |
| S1C—O3C | 1.4715 (18) | C8B—C9B | 1.390 (3) |
| S1C—C1C | 1.7670 (18) | C8B—H8B | 0.9500 |
| O1W—H1W | 0.871 (10) | C8C—C9C | 1.390 (3) |
| O1W—H2W | 0.869 (9) | C8C—H8C | 0.9500 |
| O2W—H3W | 0.866 (10) | C9—C10 | 1.401 (3) |
| O2W—H4W | 0.869 (10) | C9—H9 | 0.9500 |
| O3W—H5W | 0.867 (10) | C9A—C10A | 1.402 (3) |
| O3W—H6W | 0.869 (10) | C9A—H9A | 0.9500 |
| O4W—H7W | 0.873 (10) | C9B—C10B | 1.400 (3) |
| O4W—H8W | 0.867 (10) | C9B—H9B | 0.9500 |
| O5W—H9W | 0.870 (9) | C9C—C10C | 1.391 (3) |
| O5W—H10W | 0.888 (9) | C9C—H9C | 0.9500 |
| O6W—H11W | 0.866 (10) | C10—C11 | 1.392 (3) |
| O6W—H12W | 0.862 (10) | C10A—C11A | 1.409 (3) |
| O7W—H13W | 0.873 (10) | C10B—C11B | 1.404 (3) |
| O7W—H14W | 0.876 (10) | C10C—C11C | 1.401 (3) |
| O8W—H15W | 0.882 (9) | C11—C12 | 1.391 (3) |
| O8W—H16W | 0.870 (9) | C11—H11 | 0.9500 |
| N1—N2 | 1.260 (2) | C11A—C12A | 1.378 (3) |
| N1—C4 | 1.425 (2) | C11A—H11A | 0.9500 |
| N1A—N2A | 1.265 (2) | C11B—C12B | 1.377 (3) |
| N1A—C4A | 1.430 (2) | C11B—H11B | 0.9500 |
| N1B—N2B | 1.263 (2) | C11C—C12C | 1.381 (3) |

| | | | |
|---|---|---|---|
| N1B—C4B | 1.429 (2) | C11C—H11C | 0.9500 |
| N1C—N2C | 1.259 (2) | C12—H12 | 0.9500 |
| N1C—C4C | 1.429 (2) | C12A—H12A | 0.9500 |
| N2—C7 | 1.421 (2) | C12B—H12B | 0.9500 |
| N2A—C7A | 1.415 (2) | C12C—H12C | 0.9500 |
| N2B—C7B | 1.420 (2) | | |

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|---|---|---|---|
| O2—K1—O1A$^{i}$ | 93.46 (5) | O1A—S1A—K2 | 112.62 (6) |
| O2—K1—O6W$^{ii}$ | 176.42 (5) | O3A—S1A—K2 | 64.38 (6) |
| O1A$^{i}$—K1—O6W$^{ii}$ | 88.31 (4) | O2A—S1A—K2 | 50.50 (6) |
| O2—K1—O7W$^{ii}$ | 96.46 (5) | C1A—S1A—K2 | 139.34 (6) |
| O1A$^{i}$—K1—O7W$^{ii}$ | 166.72 (5) | K1—S1A—K2 | 74.573 (13) |
| O6W$^{ii}$—K1—O7W$^{ii}$ | 82.29 (4) | O2B—S1B—O3B | 114.07 (10) |
| O2—K1—O1W | 106.10 (5) | O2B—S1B—O1B | 111.81 (9) |
| O1A$^{i}$—K1—O1W | 81.52 (4) | O3B—S1B—O1B | 110.35 (10) |
| O6W$^{ii}$—K1—O1W | 77.24 (5) | O2B—S1B—C1B | 107.17 (9) |
| O7W$^{ii}$—K1—O1W | 87.25 (5) | O3B—S1B—C1B | 106.78 (9) |
| O2—K1—O1A | 86.00 (4) | O1B—S1B—C1B | 106.18 (8) |
| O1A$^{i}$—K1—O1A | 72.27 (5) | O2B—S1B—K4 | 49.10 (6) |
| O6W$^{ii}$—K1—O1A | 91.58 (4) | O3B—S1B—K4 | 120.29 (6) |
| O7W$^{ii}$—K1—O1A | 117.16 (4) | O1B—S1B—K4 | 64.44 (6) |
| O1W—K1—O1A | 151.82 (4) | C1B—S1B—K4 | 132.51 (6) |
| O2—K1—O2A | 84.65 (4) | O2B—S1B—K3 | 121.36 (6) |
| O1A$^{i}$—K1—O2A | 121.15 (4) | O3B—S1B—K3 | 65.35 (8) |
| O6W$^{ii}$—K1—O2A | 91.77 (4) | O1B—S1B—K3 | 46.46 (6) |
| O7W$^{ii}$—K1—O2A | 68.76 (4) | C1B—S1B—K3 | 129.95 (6) |
| O1W—K1—O2A | 154.89 (4) | K4—S1B—K3 | 79.306 (14) |
| O1A—K1—O2A | 48.89 (4) | O6C—S1C—O4C | 113.4 (8) |
| O2—K1—S1A | 83.01 (3) | O6C—S1C—O5C | 117.1 (8) |
| O1A$^{i}$—K1—S1A | 96.24 (3) | O4C—S1C—O5C | 112.0 (8) |
| O6W$^{ii}$—K1—S1A | 93.70 (3) | O1C—S1C—O2C | 112.83 (10) |
| O7W$^{ii}$—K1—S1A | 93.72 (3) | O1C—S1C—O3C | 112.65 (11) |
| O1W—K1—S1A | 170.69 (4) | O2C—S1C—O3C | 110.62 (11) |
| O1A—K1—S1A | 24.01 (3) | O6C—S1C—C1C | 105.1 (6) |
| O2A—K1—S1A | 25.02 (3) | O4C—S1C—C1C | 105.8 (6) |
| O2—K1—K4$^{ii}$ | 138.74 (3) | O5C—S1C—C1C | 101.8 (5) |
| O1A$^{i}$—K1—K4$^{ii}$ | 127.69 (3) | O1C—S1C—C1C | 107.36 (9) |
| O6W$^{ii}$—K1—K4$^{ii}$ | 39.82 (3) | O2C—S1C—C1C | 106.53 (9) |
| O7W$^{ii}$—K1—K4$^{ii}$ | 42.49 (3) | O3C—S1C—C1C | 106.40 (9) |
| O1W—K1—K4$^{ii}$ | 80.55 (3) | O6C—S1C—K4 | 31.8 (7) |
| O1A—K1—K4$^{ii}$ | 107.32 (3) | O4C—S1C—K4 | 126.2 (6) |
| O2A—K1—K4$^{ii}$ | 76.73 (3) | O5C—S1C—K4 | 85.8 (7) |
| S1A—K1—K4$^{ii}$ | 93.909 (13) | O1C—S1C—K4 | 132.42 (7) |
| O2—K1—K2 | 46.70 (3) | O2C—S1C—K4 | 58.12 (9) |
| O1A$^{i}$—K1—K2 | 126.75 (3) | O3C—S1C—K4 | 52.50 (9) |
| O6W$^{ii}$—K1—K2 | 129.93 (3) | C1C—S1C—K4 | 120.10 (6) |
| O7W$^{ii}$—K1—K2 | 66.47 (3) | O6C—S1C—K3 | 54.9 (7) |
| O1W—K1—K2 | 135.07 (3) | O4C—S1C—K3 | 58.5 (7) |

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|---|---|---|---|
| O1A—K1—K2 | 71.44 (3) | O5C—S1C—K3 | 139.2 (5) |
| O2A—K1—K2 | 41.51 (3) | O1C—S1C—K3 | 80.14 (9) |
| S1A—K1—K2 | 53.027 (11) | O2C—S1C—K3 | 126.37 (8) |
| K4$^{ii}$—K1—K2 | 99.729 (12) | O3C—S1C—K3 | 32.52 (9) |
| O2—K1—K1$^{i}$ | 89.53 (3) | C1C—S1C—K3 | 119.03 (6) |
| O1A$^{i}$—K1—K1$^{i}$ | 37.44 (3) | K4—S1C—K3 | 74.665 (13) |
| O6W$^{ii}$—K1—K1$^{i}$ | 89.99 (3) | S1A—O1A—K1$^{i}$ | 148.88 (8) |
| O7W$^{ii}$—K1—K1$^{i}$ | 151.03 (4) | S1A—O1A—K1 | 103.22 (7) |
| O1W—K1—K1$^{i}$ | 118.31 (3) | K1$^{i}$—O1A—K1 | 107.73 (5) |
| O1A—K1—K1$^{i}$ | 34.83 (3) | S1B—O1B—K3 | 110.66 (8) |
| O2A—K1—K1$^{i}$ | 83.71 (3) | S1B—O1B—K4 | 90.78 (7) |
| S1A—K1—K1$^{i}$ | 58.808 (12) | K3—O1B—K4 | 98.91 (4) |
| K4$^{ii}$—K1—K1$^{i}$ | 123.824 (16) | S1—O2—K1 | 152.11 (8) |
| K2—K1—K1$^{i}$ | 98.949 (14) | S1—O2—K2 | 91.86 (7) |
| O3—K2—O1C | 89.81 (6) | K1—O2—K2 | 94.34 (4) |
| O3—K2—O4C | 78.8 (3) | K1—O1W—H1W | 132 (2) |
| O3—K2—O2A | 109.42 (4) | K1—O1W—H2W | 104.3 (16) |
| O1C—K2—O2A | 110.70 (5) | H1W—O1W—H2W | 99.6 (18) |
| O4C—K2—O2A | 108.2 (3) | S1A—O2A—K2 | 105.55 (7) |
| O3—K2—O2W | 90.13 (4) | S1A—O2A—K1 | 95.50 (7) |
| O1C—K2—O2W | 136.34 (5) | K2—O2A—K1 | 93.24 (4) |
| O4C—K2—O2W | 141.4 (3) | S1B—O2B—K4$^{v}$ | 148.49 (9) |
| O2A—K2—O2W | 110.39 (5) | S1B—O2B—K4 | 107.69 (7) |
| O3—K2—O3W$^{iii}$ | 166.01 (5) | K4$^{v}$—O2B—K4 | 103.59 (5) |
| O1C—K2—O3W$^{iii}$ | 103.96 (6) | S1—O3—K3 | 139.98 (8) |
| O2A—K2—O3W$^{iii}$ | 68.31 (4) | S1—O3—K2 | 105.64 (7) |
| O2W—K2—O3W$^{iii}$ | 78.26 (4) | K3—O3—K2 | 113.39 (5) |
| O3—K2—O3W | 91.51 (4) | K2—O2W—H3W | 113 (2) |
| O1C—K2—O3W | 64.79 (5) | K2—O2W—H4W | 105 (2) |
| O4C—K2—O3W | 71.9 (3) | H3W—O2W—H4W | 102.9 (19) |
| O2A—K2—O3W | 158.82 (4) | S1A—O3A—K2 | 90.93 (7) |
| O2W—K2—O3W | 71.57 (5) | S1B—O3B—K3 | 90.31 (8) |
| O3W$^{iii}$—K2—O3W | 92.17 (4) | K2$^{iii}$—O3W—K2 | 87.83 (4) |
| O3—K2—O2 | 48.75 (4) | K2$^{iii}$—O3W—H5W | 101 (2) |
| O1C—K2—O2 | 137.53 (6) | K2—O3W—H5W | 118 (2) |
| O4C—K2—O2 | 125.3 (3) | K2$^{iii}$—O3W—H6W | 156.1 (18) |
| O2A—K2—O2 | 80.89 (4) | K2—O3W—H6W | 89.1 (19) |
| O2W—K2—O2 | 63.80 (4) | H5W—O3W—H6W | 101.7 (19) |
| O3W$^{iii}$—K2—O2 | 117.99 (4) | K3—O4W—K3$^{iv}$ | 72.75 (4) |
| O3W—K2—O2 | 116.97 (4) | K3—O4W—H7W | 98.2 (17) |
| O3—K2—O3A | 79.15 (4) | K3$^{iv}$—O4W—H7W | 125.5 (19) |
| O1C—K2—O3A | 75.40 (5) | K3—O4W—H8W | 138.6 (18) |
| O4C—K2—O3A | 67.3 (3) | K3$^{iv}$—O4W—H8W | 66.2 (18) |
| O2A—K2—O3A | 47.21 (4) | H7W—O4W—H8W | 101.3 (18) |
| O2W—K2—O3A | 146.90 (4) | K3$^{iv}$—O5W—K3 | 79.10 (4) |
| O3W$^{iii}$—K2—O3A | 106.42 (4) | K3$^{iv}$—O5W—H9W | 54.9 (17) |
| O3W—K2—O3A | 139.15 (4) | K3—O5W—H9W | 94.2 (15) |
| O2—K2—O3A | 86.27 (4) | K3$^{iv}$—O5W—H10W | 105.4 (16) |

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| O3—K2—S1 | 24.08 (3) | K3—O5W—H10W | 173.6 (15) |
| O1C—K2—S1 | 113.82 (5) | H9W—O5W—H10W | 92.1 (15) |
| O4C—K2—S1 | 102.4 (3) | K4—O6W—K1$^{vi}$ | 99.97 (5) |
| O2A—K2—S1 | 97.78 (3) | K4—O6W—H11W | 110.5 (18) |
| O2W—K2—S1 | 74.17 (3) | K1$^{vi}$—O6W—H11W | 102 (2) |
| O3W$^{iii}$—K2—S1 | 142.21 (4) | K4—O6W—H12W | 132.8 (19) |
| O3W—K2—S1 | 102.89 (3) | K1$^{vi}$—O6W—H12W | 104 (2) |
| O2—K2—S1 | 24.93 (3) | H11W—O6W—H12W | 103.3 (18) |
| O3A—K2—S1 | 84.46 (3) | K1$^{vi}$—O7W—K4 | 96.35 (5) |
| O3—K2—S1A | 90.20 (3) | K1$^{vi}$—O7W—H13W | 138.7 (18) |
| O1C—K2—S1A | 97.37 (4) | K4—O7W—H13W | 103.0 (19) |
| O4C—K2—S1A | 90.9 (3) | K1$^{vi}$—O7W—H14W | 108.2 (17) |
| O2A—K2—S1A | 23.95 (3) | K4—O7W—H14W | 109.1 (19) |
| O2W—K2—S1A | 126.29 (4) | H13W—O7W—H14W | 99.4 (17) |
| O3W$^{iii}$—K2—S1A | 90.46 (3) | K4—O8W—H15W | 114.6 (16) |
| O3W—K2—S1A | 162.07 (4) | K4—O8W—H16W | 124.1 (16) |
| O2—K2—S1A | 76.98 (3) | H15W—O8W—H16W | 98.9 (16) |
| O3A—K2—S1A | 24.68 (3) | S1C—O1C—K2 | 160.77 (14) |
| S1—K2—S1A | 85.573 (14) | S1C—O2C—K4 | 98.29 (10) |
| O3—K2—K2$^{iii}$ | 135.69 (3) | S1C—O3C—K3 | 130.10 (12) |
| O1C—K2—K2$^{iii}$ | 82.24 (5) | S1C—O3C—K4 | 104.22 (11) |
| O2A—K2—K2$^{iii}$ | 114.18 (3) | K3—O3C—K4 | 105.93 (6) |
| O2W—K2—K2$^{iii}$ | 67.98 (3) | S1C—O4C—K2 | 161.7 (10) |
| O3W$^{iii}$—K2—K2$^{iii}$ | 46.33 (3) | S1C—O4C—K3 | 99.6 (7) |
| O3W—K2—K2$^{iii}$ | 45.84 (3) | K2—O4C—K3 | 95.5 (4) |
| O2—K2—K2$^{iii}$ | 131.69 (3) | S1C—O6C—K4 | 131.6 (9) |
| O3A—K2—K2$^{iii}$ | 138.62 (3) | S1C—O6C—K3 | 104.4 (9) |
| S1—K2—K2$^{iii}$ | 136.779 (19) | K4—O6C—K3 | 102.3 (5) |
| S1A—K2—K2$^{iii}$ | 133.976 (19) | N2—N1—C4 | 113.70 (15) |
| O3—K2—H6W | 78.2 (4) | N2A—N1A—C4A | 113.11 (15) |
| O1C—K2—H6W | 55.3 (4) | N2B—N1B—C4B | 113.55 (15) |
| O4C—K2—H6W | 59.6 (5) | N2C—N1C—C4C | 113.74 (15) |
| O2A—K2—H6W | 164.9 (5) | N1—N2—C7 | 113.51 (15) |
| O2W—K2—H6W | 82.1 (4) | N1A—N2A—C7A | 114.38 (15) |
| O3W$^{iii}$—K2—H6W | 107.5 (3) | N1B—N2B—C7B | 113.58 (15) |
| O3W—K2—H6W | 16.8 (2) | N1C—N2C—C7C | 113.67 (15) |
| O2—K2—H6W | 113.1 (5) | C10—N3—H1N | 117.5 (15) |
| O3A—K2—H6W | 125.1 (3) | C10—N3—H2N | 113.3 (19) |
| S1—K2—H6W | 93.8 (5) | H1N—N3—H2N | 111 (2) |
| S1A—K2—H6W | 149.7 (3) | C10A—N3A—H3N | 114.1 (19) |
| K2$^{iii}$—K2—H6W | 61.5 (3) | C10A—N3A—H4N | 115.9 (17) |
| O3—K3—O3C | 111.17 (5) | H3N—N3A—H4N | 112 (3) |
| O3—K3—O1B | 116.49 (5) | C10B—N3B—H5N | 115.7 (17) |
| O3C—K3—O1B | 81.50 (6) | C10B—N3B—H6N | 115.6 (17) |
| O3—K3—O5W$^{iv}$ | 75.72 (5) | H5N—N3B—H6N | 106 (2) |
| O3C—K3—O5W$^{iv}$ | 90.88 (6) | C10C—N3C—H7N | 112.5 (19) |
| O1B—K3—O5W$^{iv}$ | 167.29 (5) | C10C—N3C—H8N | 115.8 (16) |
| O3—K3—O4W | 77.80 (4) | H7N—N3C—H8N | 113 (2) |

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| O3C—K3—O4W | 162.84 (5) | C2—C1—C6 | 121.15 (17) |
| O1B—K3—O4W | 108.15 (5) | C2—C1—S1 | 119.32 (14) |
| O5W$^{iv}$—K3—O4W | 76.90 (5) | C6—C1—S1 | 119.41 (14) |
| O3—K3—O5W | 141.48 (5) | C2A—C1A—C6A | 120.45 (16) |
| O3C—K3—O5W | 107.21 (5) | C2A—C1A—S1A | 120.64 (14) |
| O1B—K3—O5W | 72.00 (4) | C6A—C1A—S1A | 118.89 (14) |
| O5W$^{iv}$—K3—O5W | 100.90 (4) | C6B—C1B—C2B | 120.54 (17) |
| O4W—K3—O5W | 64.31 (4) | C6B—C1B—S1B | 118.77 (14) |
| O3—K3—O6C | 114.4 (3) | C2B—C1B—S1B | 120.67 (14) |
| O1B—K3—O6C | 75.9 (3) | C2C—C1C—C6C | 120.71 (17) |
| O5W$^{iv}$—K3—O6C | 96.3 (3) | C2C—C1C—S1C | 120.99 (14) |
| O4W—K3—O6C | 164.5 (3) | C6C—C1C—S1C | 118.28 (14) |
| O5W—K3—O6C | 104.1 (3) | C1—C2—C3 | 119.08 (17) |
| O3—K3—O3B | 72.30 (4) | C1—C2—H2 | 120.5 |
| O3C—K3—O3B | 83.22 (5) | C3—C2—H2 | 120.5 |
| O1B—K3—O3B | 46.78 (4) | C3A—C2A—C1A | 119.91 (17) |
| O5W$^{iv}$—K3—O3B | 142.66 (4) | C3A—C2A—H2A | 120.0 |
| O4W—K3—O3B | 113.80 (5) | C1A—C2A—H2A | 120.0 |
| O5W—K3—O3B | 116.10 (4) | C3B—C2B—C1B | 120.03 (17) |
| O6C—K3—O3B | 80.1 (3) | C3B—C2B—H2B | 120.0 |
| O3—K3—O4C | 72.3 (3) | C1B—C2B—H2B | 120.0 |
| O1B—K3—O4C | 100.0 (3) | C3C—C2C—C1C | 119.71 (17) |
| O5W$^{iv}$—K3—O4C | 79.8 (3) | C3C—C2C—H2C | 120.1 |
| O4W—K3—O4C | 145.7 (3) | C1C—C2C—H2C | 120.1 |
| O5W—K3—O4C | 145.8 (3) | C2—C3—C4 | 120.20 (17) |
| O6C—K3—O4C | 42.6 (4) | C2—C3—H3 | 119.9 |
| O3B—K3—O4C | 72.6 (3) | C4—C3—H3 | 119.9 |
| O3—K3—O4W$^{iv}$ | 131.51 (4) | C2A—C3A—C4A | 119.70 (17) |
| O3C—K3—O4W$^{iv}$ | 55.72 (5) | C2A—C3A—H3A | 120.1 |
| O1B—K3—O4W$^{iv}$ | 107.63 (4) | C4A—C3A—H3A | 120.1 |
| O5W$^{iv}$—K3—O4W$^{iv}$ | 59.69 (4) | C2B—C3B—C4B | 119.58 (17) |
| O4W—K3—O4W$^{iv}$ | 107.25 (4) | C2B—C3B—H3B | 120.2 |
| O5W—K3—O4W$^{iv}$ | 70.16 (4) | C4B—C3B—H3B | 120.2 |
| O3B—K3—O4W$^{iv}$ | 136.67 (4) | C2C—C3C—C4C | 119.64 (17) |
| O3—K3—S1B | 96.20 (3) | C2C—C3C—H3C | 120.2 |
| O3C—K3—S1B | 78.29 (4) | C4C—C3C—H3C | 120.2 |
| O1B—K3—S1B | 22.88 (3) | C3—C4—C5 | 120.29 (17) |
| O5W$^{iv}$—K3—S1B | 163.31 (4) | C3—C4—N1 | 114.87 (16) |
| O4W—K3—S1B | 116.15 (4) | C5—C4—N1 | 124.83 (16) |
| O5W—K3—S1B | 94.47 (3) | C5A—C4A—C3A | 120.20 (17) |
| O6C—K3—S1B | 73.5 (3) | C5A—C4A—N1A | 115.36 (16) |
| O3B—K3—S1B | 24.35 (3) | C3A—C4A—N1A | 124.43 (17) |
| O4C—K3—S1B | 83.8 (3) | C5B—C4B—C3B | 120.33 (17) |
| O4W$^{iv}$—K3—S1B | 121.09 (3) | C5B—C4B—N1B | 114.82 (16) |
| O3—K3—K3$^{iv}$ | 115.28 (4) | C3B—C4B—N1B | 124.84 (17) |
| O3C—K3—K3$^{iv}$ | 104.25 (4) | C5C—C4C—C3C | 120.44 (17) |
| O1B—K3—K3$^{iv}$ | 121.03 (4) | C5C—C4C—N1C | 114.83 (16) |
| O5W$^{iv}$—K3—K3$^{iv}$ | 50.99 (3) | C3C—C4C—N1C | 124.72 (16) |

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| O4W—K3—K3$^{iv}$ | 58.69 (3) | C6—C5—C4 | 119.71 (17) |
| O5W—K3—K3$^{iv}$ | 49.91 (3) | C6—C5—H5 | 120.1 |
| O3B—K3—K3$^{iv}$ | 165.36 (4) | C4—C5—H5 | 120.1 |
| O4W$^{iv}$—K3—K3$^{iv}$ | 48.56 (3) | C6A—C5A—C4A | 120.13 (17) |
| S1B—K3—K3$^{iv}$ | 143.90 (2) | C6A—C5A—H5A | 119.9 |
| O3—K3—H9W$^{iv}$ | 62.8 (4) | C4A—C5A—H5A | 119.9 |
| O3C—K3—H9W$^{iv}$ | 105.2 (4) | C6B—C5B—C4B | 120.25 (17) |
| O1B—K3—H9W$^{iv}$ | 173.2 (4) | C6B—C5B—H5B | 119.9 |
| O5W$^{iv}$—K3—H9W$^{iv}$ | 16.7 (3) | C4B—C5B—H5B | 119.9 |
| O4W—K3—H9W$^{iv}$ | 65.0 (4) | C6C—C5C—C4C | 120.11 (17) |
| O5W—K3—H9W$^{iv}$ | 104.2 (5) | C6C—C5C—H5C | 119.9 |
| O3B—K3—H9W$^{iv}$ | 134.4 (5) | C4C—C5C—H5C | 119.9 |
| O4W$^{iv}$—K3—H9W$^{iv}$ | 75.7 (3) | C5—C6—C1 | 119.55 (17) |
| S1B—K3—H9W$^{iv}$ | 158.7 (4) | C5—C6—H6 | 120.2 |
| K3$^{iv}$—K3—H9W$^{iv}$ | 56.5 (5) | C1—C6—H6 | 120.2 |
| O6C—K4—O6W | 87.6 (3) | C5A—C6A—C1A | 119.56 (17) |
| O2B$^{v}$—K4—O6W | 158.58 (5) | C5A—C6A—H6A | 120.2 |
| O6C—K4—O2B | 92.2 (4) | C1A—C6A—H6A | 120.2 |
| O2B$^{v}$—K4—O2B | 76.41 (5) | C5B—C6B—C1B | 119.26 (17) |
| O6W—K4—O2B | 111.99 (4) | C5B—C6B—H6B | 120.4 |
| O6C—K4—O8W | 119.9 (4) | C1B—C6B—H6B | 120.4 |
| O2B$^{v}$—K4—O8W | 75.67 (5) | C5C—C6C—C1C | 119.36 (17) |
| O6W—K4—O8W | 87.78 (5) | C5C—C6C—H6C | 120.3 |
| O2B—K4—O8W | 143.65 (5) | C1C—C6C—H6C | 120.3 |
| O6C—K4—O7W | 163.8 (4) | C12—C7—C8 | 119.49 (17) |
| O2B$^{v}$—K4—O7W | 80.59 (5) | C12—C7—N2 | 116.54 (16) |
| O6W—K4—O7W | 81.35 (4) | C8—C7—N2 | 123.96 (16) |
| O2B—K4—O7W | 81.20 (5) | C8A—C7A—C12A | 119.35 (17) |
| O8W—K4—O7W | 71.68 (4) | C8A—C7A—N2A | 116.26 (16) |
| O2B$^{v}$—K4—O3C | 119.29 (5) | C12A—C7A—N2A | 124.38 (16) |
| O6W—K4—O3C | 81.43 (5) | C8B—C7B—C12B | 119.37 (17) |
| O2B—K4—O3C | 88.02 (5) | C8B—C7B—N2B | 116.68 (16) |
| O8W—K4—O3C | 126.13 (5) | C12B—C7B—N2B | 123.95 (17) |
| O7W—K4—O3C | 154.52 (5) | C8C—C7C—C12C | 119.49 (17) |
| O2B$^{v}$—K4—O2C | 72.75 (5) | C8C—C7C—N2C | 116.32 (16) |
| O6W—K4—O2C | 126.89 (4) | C12C—C7C—N2C | 124.18 (16) |
| O2B—K4—O2C | 81.89 (5) | C9—C8—C7 | 119.86 (17) |
| O8W—K4—O2C | 111.10 (5) | C9—C8—H8 | 120.1 |
| O7W—K4—O2C | 151.11 (4) | C7—C8—H8 | 120.1 |
| O3C—K4—O2C | 46.87 (5) | C9A—C8A—C7A | 120.66 (17) |
| O6C—K4—O1B | 79.0 (4) | C9A—C8A—H8A | 119.7 |
| O2B$^{v}$—K4—O1B | 123.67 (4) | C7A—C8A—H8A | 119.7 |
| O6W—K4—O1B | 66.06 (4) | C9B—C8B—C7B | 120.32 (17) |
| O2B—K4—O1B | 47.47 (4) | C9B—C8B—H8B | 119.8 |
| O8W—K4—O1B | 147.95 (4) | C7B—C8B—H8B | 119.8 |
| O7W—K4—O1B | 85.73 (4) | C9C—C8C—C7C | 120.29 (17) |
| O3C—K4—O1B | 70.13 (5) | C9C—C8C—H8C | 119.9 |
| O2C—K4—O1B | 99.72 (4) | C7C—C8C—H8C | 119.9 |

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| O6C—K4—S1B | 81.9 (4) | C8—C9—C10 | 121.02 (17) |
| O2B$^{v}$—K4—S1B | 99.57 (3) | C8—C9—H9 | 119.5 |
| O6W—K4—S1B | 90.68 (3) | C10—C9—H9 | 119.5 |
| O2B—K4—S1B | 23.22 (3) | C8A—C9A—C10A | 120.26 (17) |
| O8W—K4—S1B | 158.02 (4) | C8A—C9A—H9A | 119.9 |
| O7W—K4—S1B | 86.41 (3) | C10A—C9A—H9A | 119.9 |
| O3C—K4—S1B | 75.16 (4) | C8B—C9B—C10B | 120.45 (17) |
| O2C—K4—S1B | 87.21 (3) | C8B—C9B—H9B | 119.8 |
| O1B—K4—S1B | 24.78 (3) | C10B—C9B—H9B | 119.8 |
| O6C—K4—S1C | 16.6 (3) | C8C—C9C—C10C | 120.35 (17) |
| O2B$^{v}$—K4—S1C | 96.17 (4) | C8C—C9C—H9C | 119.8 |
| O6W—K4—S1C | 104.10 (3) | C10C—C9C—H9C | 119.8 |
| O2B—K4—S1C | 84.45 (3) | C11—C10—C9 | 118.90 (17) |
| O8W—K4—S1C | 121.29 (3) | C11—C10—N3 | 121.55 (17) |
| O7W—K4—S1C | 165.64 (3) | C9—C10—N3 | 119.54 (18) |
| O3C—K4—S1C | 23.28 (4) | N3A—C10A—C9A | 121.31 (18) |
| O2C—K4—S1C | 23.58 (3) | N3A—C10A—C11A | 119.94 (18) |
| O1B—K4—S1C | 84.49 (3) | C9A—C10A—C11A | 118.71 (17) |
| S1B—K4—S1C | 80.317 (14) | N3B—C10B—C9B | 121.73 (18) |
| O2B$^{v}$—K4—K1$^{vi}$ | 120.62 (4) | N3B—C10B—C11B | 119.62 (18) |
| O6W—K4—K1$^{vi}$ | 40.21 (3) | C9B—C10B—C11B | 118.61 (17) |
| O2B—K4—K1$^{vi}$ | 99.50 (3) | C9C—C10C—C11C | 119.19 (17) |
| O8W—K4—K1$^{vi}$ | 75.67 (3) | C9C—C10C—N3C | 121.44 (18) |
| O7W—K4—K1$^{vi}$ | 41.16 (3) | C11C—C10C—N3C | 119.35 (18) |
| O3C—K4—K1$^{vi}$ | 119.71 (4) | C12—C11—C10 | 120.48 (17) |
| O2C—K4—K1$^{vi}$ | 166.57 (3) | C12—C11—H11 | 119.8 |
| O1B—K4—K1$^{vi}$ | 72.38 (3) | C10—C11—H11 | 119.8 |
| S1B—K4—K1$^{vi}$ | 89.108 (13) | C12A—C11A—C10A | 120.94 (17) |
| S1C—K4—K1$^{vi}$ | 142.993 (15) | C12A—C11A—H11A | 119.5 |
| O2—S1—O3 | 112.68 (8) | C10A—C11A—H11A | 119.5 |
| O2—S1—O1 | 113.18 (9) | C12B—C11B—C10B | 120.96 (17) |
| O3—S1—O1 | 111.78 (8) | C12B—C11B—H11B | 119.5 |
| O2—S1—C1 | 107.16 (8) | C10B—C11B—H11B | 119.5 |
| O3—S1—C1 | 106.88 (8) | C12C—C11C—C10C | 120.65 (17) |
| O1—S1—C1 | 104.50 (8) | C12C—C11C—H11C | 119.7 |
| O2—S1—K2 | 63.22 (6) | C10C—C11C—H11C | 119.7 |
| O3—S1—K2 | 50.28 (6) | C11—C12—C7 | 120.23 (17) |
| O1—S1—K2 | 141.39 (6) | C11—C12—H12 | 119.9 |
| C1—S1—K2 | 113.31 (6) | C7—C12—H12 | 119.9 |
| O1A—S1A—O3A | 113.91 (9) | C11A—C12A—C7A | 120.02 (17) |
| O1A—S1A—O2A | 111.82 (8) | C11A—C12A—H12A | 120.0 |
| O3A—S1A—O2A | 110.19 (9) | C7A—C12A—H12A | 120.0 |
| O1A—S1A—C1A | 107.12 (8) | C11B—C12B—C7B | 120.22 (17) |
| O3A—S1A—C1A | 106.79 (8) | C11B—C12B—H12B | 119.9 |
| O2A—S1A—C1A | 106.56 (8) | C7B—C12B—H12B | 119.9 |
| O1A—S1A—K1 | 52.78 (6) | C11C—C12C—C7C | 120.01 (17) |
| O3A—S1A—K1 | 126.06 (6) | C11C—C12C—H12C | 120.0 |
| O2A—S1A—K1 | 59.48 (6) | C7C—C12C—H12C | 120.0 |

| | | | |
|---|---|---|---|
| C1A—S1A—K1 | 127.14 (6) | | |
| | | | |
| O3A—S1A—O1A—K1$^{i}$ | −55.79 (18) | O2B—S1B—C1B—C2B | 15.16 (17) |
| O2A—S1A—O1A—K1$^{i}$ | 178.46 (14) | O3B—S1B—C1B—C2B | −107.45 (16) |
| C1A—S1A—O1A—K1$^{i}$ | 62.06 (18) | O1B—S1B—C1B—C2B | 134.79 (15) |
| K1—S1A—O1A—K1$^{i}$ | −173.9 (2) | K4—S1B—C1B—C2B | 64.89 (17) |
| K2—S1A—O1A—K1$^{i}$ | −126.67 (14) | K3—S1B—C1B—C2B | −179.04 (11) |
| O3A—S1A—O1A—K1 | 118.15 (8) | O6C—S1C—C1C—C2C | 147.2 (8) |
| O2A—S1A—O1A—K1 | −7.61 (9) | O4C—S1C—C1C—C2C | 26.9 (7) |
| C1A—S1A—O1A—K1 | −124.01 (7) | O5C—S1C—C1C—C2C | −90.2 (7) |
| K2—S1A—O1A—K1 | 47.27 (7) | O1C—S1C—C1C—C2C | 1.15 (19) |
| O2B—S1B—O1B—K3 | −113.15 (9) | O2C—S1C—C1C—C2C | −119.97 (17) |
| O3B—S1B—O1B—K3 | 14.94 (11) | O3C—S1C—C1C—C2C | 121.98 (17) |
| C1B—S1B—O1B—K3 | 130.29 (8) | K4—S1C—C1C—C2C | 177.73 (12) |
| K4—S1B—O1B—K3 | −99.83 (7) | K3—S1C—C1C—C2C | 89.32 (15) |
| O2B—S1B—O1B—K4 | −13.32 (9) | O6C—S1C—C1C—C6C | −34.6 (8) |
| O3B—S1B—O1B—K4 | 114.77 (7) | O4C—S1C—C1C—C6C | −154.9 (7) |
| C1B—S1B—O1B—K4 | −129.88 (7) | O5C—S1C—C1C—C6C | 87.9 (7) |
| K3—S1B—O1B—K4 | 99.83 (7) | O1C—S1C—C1C—C6C | 179.31 (16) |
| O3—S1—O2—K1 | 93.56 (19) | O2C—S1C—C1C—C6C | 58.20 (18) |
| O1—S1—O2—K1 | −34.5 (2) | O3C—S1C—C1C—C6C | −59.85 (18) |
| C1—S1—O2—K1 | −149.16 (17) | K4—S1C—C1C—C6C | −4.11 (17) |
| K2—S1—O2—K1 | 102.90 (19) | K3—S1C—C1C—C6C | −92.51 (15) |
| O3—S1—O2—K2 | −9.33 (8) | C6—C1—C2—C3 | −1.4 (3) |
| O1—S1—O2—K2 | −137.39 (7) | S1—C1—C2—C3 | 174.55 (14) |
| C1—S1—O2—K2 | 107.94 (7) | C6A—C1A—C2A—C3A | 1.3 (3) |
| O1A—S1A—O2A—K2 | 101.94 (8) | S1A—C1A—C2A—C3A | −177.25 (14) |
| O3A—S1A—O2A—K2 | −25.82 (9) | C6B—C1B—C2B—C3B | −0.8 (3) |
| C1A—S1A—O2A—K2 | −141.32 (7) | S1B—C1B—C2B—C3B | 177.75 (14) |
| K1—S1A—O2A—K2 | 94.92 (6) | C6C—C1C—C2C—C3C | −0.6 (3) |
| O1A—S1A—O2A—K1 | 7.03 (9) | S1C—C1C—C2C—C3C | 177.50 (13) |
| O3A—S1A—O2A—K1 | −120.74 (7) | C1—C2—C3—C4 | 0.4 (3) |
| C1A—S1A—O2A—K1 | 123.77 (7) | C1A—C2A—C3A—C4A | 0.2 (3) |
| K2—S1A—O2A—K1 | −94.92 (6) | C1B—C2B—C3B—C4B | −0.1 (3) |
| O3B—S1B—O2B—K4$^{v}$ | 62.58 (19) | C1C—C2C—C3C—C4C | −0.7 (3) |
| O1B—S1B—O2B—K4$^{v}$ | −171.33 (15) | C2—C3—C4—C5 | 1.0 (3) |
| C1B—S1B—O2B—K4$^{v}$ | −55.38 (19) | C2—C3—C4—N1 | −179.85 (16) |
| K4—S1B—O2B—K4$^{v}$ | 172.7 (2) | N2—N1—C4—C3 | −179.97 (16) |
| K3—S1B—O2B—K4$^{v}$ | 137.35 (14) | N2—N1—C4—C5 | −0.9 (3) |
| O3B—S1B—O2B—K4 | −110.12 (10) | C2A—C3A—C4A—C5A | −1.6 (3) |
| O1B—S1B—O2B—K4 | 15.96 (10) | C2A—C3A—C4A—N1A | 179.28 (17) |
| C1B—S1B—O2B—K4 | 131.92 (7) | N2A—N1A—C4A—C5A | 178.53 (16) |
| K3—S1B—O2B—K4 | −35.35 (9) | N2A—N1A—C4A—C3A | −2.4 (3) |
| O2—S1—O3—K3 | −156.23 (11) | C2B—C3B—C4B—C5B | 1.0 (3) |
| O1—S1—O3—K3 | −27.45 (15) | C2B—C3B—C4B—N1B | 179.82 (17) |
| C1—S1—O3—K3 | 86.33 (13) | N2B—N1B—C4B—C5B | −179.28 (16) |
| K2—S1—O3—K3 | −167.08 (16) | N2B—N1B—C4B—C3B | 1.8 (3) |
| O2—S1—O3—K2 | 10.85 (9) | C2C—C3C—C4C—C5C | 1.2 (3) |

| | | | |
|---|---|---|---|
| O1—S1—O3—K2 | 139.64 (7) | C2C—C3C—C4C—N1C | −179.76 (16) |
| C1—S1—O3—K2 | −106.59 (8) | N2C—N1C—C4C—C5C | −179.89 (16) |
| O1A—S1A—O3A—K2 | −104.72 (7) | N2C—N1C—C4C—C3C | 1.0 (3) |
| O2A—S1A—O3A—K2 | 21.89 (8) | C3—C4—C5—C6 | −1.3 (3) |
| C1A—S1A—O3A—K2 | 137.24 (7) | N1—C4—C5—C6 | 179.62 (17) |
| K1—S1A—O3A—K2 | −44.44 (7) | C3A—C4A—C5A—C6A | 1.7 (3) |
| O2B—S1B—O3B—K3 | 114.97 (8) | N1A—C4A—C5A—C6A | −179.15 (16) |
| O1B—S1B—O3B—K3 | −11.87 (8) | C3B—C4B—C5B—C6B | −0.9 (3) |
| C1B—S1B—O3B—K3 | −126.84 (7) | N1B—C4B—C5B—C6B | −179.90 (16) |
| K4—S1B—O3B—K3 | 59.70 (7) | C3C—C4C—C5C—C6C | −0.4 (3) |
| O2C—S1C—O1C—K2 | −150.2 (3) | N1C—C4C—C5C—C6C | −179.56 (16) |
| O3C—S1C—O1C—K2 | −24.1 (3) | C4—C5—C6—C1 | 0.3 (3) |
| C1C—S1C—O1C—K2 | 92.7 (3) | C2—C1—C6—C5 | 1.1 (3) |
| K4—S1C—O1C—K2 | −83.3 (3) | S1—C1—C6—C5 | −174.87 (14) |
| K3—S1C—O1C—K2 | −24.8 (3) | C4A—C5A—C6A—C1A | −0.2 (3) |
| O1C—S1C—O2C—K4 | 126.88 (9) | C2A—C1A—C6A—C5A | −1.2 (3) |
| O3C—S1C—O2C—K4 | −0.33 (10) | S1A—C1A—C6A—C5A | 177.32 (14) |
| C1C—S1C—O2C—K4 | −115.57 (7) | C4B—C5B—C6B—C1B | 0.0 (3) |
| K3—S1C—O2C—K4 | 32.34 (10) | C2B—C1B—C6B—C5B | 0.8 (3) |
| O1C—S1C—O3C—K3 | −1.26 (17) | S1B—C1B—C6B—C5B | −177.75 (14) |
| O2C—S1C—O3C—K3 | 126.05 (13) | C4C—C5C—C6C—C1C | −0.9 (3) |
| C1C—S1C—O3C—K3 | −118.63 (12) | C2C—C1C—C6C—C5C | 1.4 (3) |
| K4—S1C—O3C—K3 | 125.70 (15) | S1C—C1C—C6C—C5C | −176.79 (14) |
| O1C—S1C—O3C—K4 | −126.95 (9) | N1—N2—C7—C12 | −179.85 (16) |
| O2C—S1C—O3C—K4 | 0.35 (11) | N1—N2—C7—C8 | −0.9 (2) |
| C1C—S1C—O3C—K4 | 115.67 (8) | N1A—N2A—C7A—C8A | −179.35 (16) |
| K3—S1C—O3C—K4 | −125.70 (15) | N1A—N2A—C7A—C12A | −0.5 (3) |
| O6C—S1C—O4C—K2 | 145 (3) | N1B—N2B—C7B—C8B | −177.56 (16) |
| O5C—S1C—O4C—K2 | 10 (3) | N1B—N2B—C7B—C12B | 2.8 (3) |
| C1C—S1C—O4C—K2 | −100 (3) | N1C—N2C—C7C—C8C | −179.80 (16) |
| K4—S1C—O4C—K2 | 112 (3) | N1C—N2C—C7C—C12C | 1.4 (3) |
| K3—S1C—O4C—K2 | 145 (3) | C12—C7—C8—C9 | −1.7 (3) |
| O6C—S1C—O4C—K3 | 0.0 (9) | N2—C7—C8—C9 | 179.36 (16) |
| O5C—S1C—O4C—K3 | −135.2 (6) | C12A—C7A—C8A—C9A | 2.0 (3) |
| C1C—S1C—O4C—K3 | 114.7 (3) | N2A—C7A—C8A—C9A | −179.08 (16) |
| K4—S1C—O4C—K3 | −33.8 (8) | C12B—C7B—C8B—C9B | −2.7 (3) |
| O4C—S1C—O6C—K4 | −121.5 (11) | N2B—C7B—C8B—C9B | 177.61 (16) |
| O5C—S1C—O6C—K4 | 11.3 (15) | C12C—C7C—C8C—C9C | −1.6 (3) |
| C1C—S1C—O6C—K4 | 123.4 (10) | N2C—C7C—C8C—C9C | 179.58 (16) |
| K3—S1C—O6C—K4 | −121.5 (13) | C7—C8—C9—C10 | 0.7 (3) |
| O4C—S1C—O6C—K3 | 0.0 (10) | C7A—C8A—C9A—C10A | −0.1 (3) |
| O5C—S1C—O6C—K3 | 132.8 (6) | C7B—C8B—C9B—C10B | 0.6 (3) |
| C1C—S1C—O6C—K3 | −115.1 (4) | C7C—C8C—C9C—C10C | 0.4 (3) |
| K4—S1C—O6C—K3 | 121.5 (13) | C8—C9—C10—C11 | 0.4 (3) |
| C4—N1—N2—C7 | 179.35 (14) | C8—C9—C10—N3 | −178.37 (17) |
| C4A—N1A—N2A—C7A | 179.46 (15) | C8A—C9A—C10A—N3A | 175.81 (17) |
| C4B—N1B—N2B—C7B | −179.25 (14) | C8A—C9A—C10A—C11A | −1.8 (3) |
| C4C—N1C—N2C—C7C | −178.85 (14) | C8B—C9B—C10B—N3B | −175.77 (17) |

| | | | |
|---|---|---|---|
| O2—S1—C1—C2 | 20.81 (17) | C8B—C9B—C10B—C11B | 1.7 (3) |
| O3—S1—C1—C2 | 141.83 (15) | C8C—C9C—C10C—C11C | 0.8 (3) |
| O1—S1—C1—C2 | −99.54 (15) | C8C—C9C—C10C—N3C | −177.53 (17) |
| K2—S1—C1—C2 | 88.45 (15) | C9—C10—C11—C12 | −0.6 (3) |
| O2—S1—C1—C6 | −163.12 (14) | N3—C10—C11—C12 | 178.20 (17) |
| O3—S1—C1—C6 | −42.11 (17) | N3A—C10A—C11A—C12A | −175.97 (17) |
| O1—S1—C1—C6 | 76.52 (16) | C9A—C10A—C11A—C12A | 1.6 (3) |
| K2—S1—C1—C6 | −95.49 (14) | N3B—C10B—C11B—C12B | 175.56 (17) |
| O1A—S1A—C1A—C2A | −8.81 (17) | C9B—C10B—C11B—C12B | −2.0 (3) |
| O3A—S1A—C1A—C2A | 113.60 (16) | C9C—C10C—C11C—C12C | −0.7 (3) |
| O2A—S1A—C1A—C2A | −128.64 (15) | N3C—C10C—C11C—C12C | 177.57 (17) |
| K1—S1A—C1A—C2A | −64.70 (16) | C10—C11—C12—C7 | −0.4 (3) |
| K2—S1A—C1A—C2A | −176.40 (10) | C8—C7—C12—C11 | 1.5 (3) |
| O1A—S1A—C1A—C6A | 172.63 (14) | N2—C7—C12—C11 | −179.43 (16) |
| O3A—S1A—C1A—C6A | −64.96 (16) | C10A—C11A—C12A—C7A | 0.3 (3) |
| O2A—S1A—C1A—C6A | 52.80 (16) | C8A—C7A—C12A—C11A | −2.1 (3) |
| K1—S1A—C1A—C6A | 116.74 (14) | N2A—C7A—C12A—C11A | 179.05 (17) |
| K2—S1A—C1A—C6A | 5.0 (2) | C10B—C11B—C12B—C7B | −0.1 (3) |
| O2B—S1B—C1B—C6B | −166.25 (15) | C8B—C7B—C12B—C11B | 2.5 (3) |
| O3B—S1B—C1B—C6B | 71.14 (17) | N2B—C7B—C12B—C11B | −177.89 (17) |
| O1B—S1B—C1B—C6B | −46.61 (17) | C10C—C11C—C12C—C7C | −0.4 (3) |
| K4—S1B—C1B—C6B | −116.52 (14) | C8C—C7C—C12C—C11C | 1.6 (3) |
| K3—S1B—C1B—C6B | −0.45 (18) | N2C—C7C—C12C—C11C | −179.67 (17) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H1*N*···O3*B*$^{vii}$ | 0.92 (3) | 2.35 (3) | 3.210 (2) | 155 (2) |
| N3—H2*N*···O2*W*$^{vii}$ | 0.81 (3) | 2.40 (3) | 3.147 (2) | 154 (2) |
| N3*A*—H3*N*···O8*W*$^{viii}$ | 0.82 (3) | 2.27 (3) | 3.016 (2) | 153 (3) |
| N3*A*—H4*N*···O1$^{ix}$ | 0.88 (3) | 2.27 (3) | 3.115 (2) | 161 (2) |
| N3*B*—H5*N*···O2*C*$^{x}$ | 0.90 (3) | 2.50 (3) | 3.384 (3) | 167 (2) |
| N3*B*—H5*N*···O5*C*$^{x}$ | 0.90 (3) | 2.26 (3) | 3.078 (14) | 151 (2) |
| N3*B*—H6*N*···O1*W*$^{vii}$ | 0.88 (3) | 2.21 (3) | 3.065 (2) | 164 (2) |
| N3*C*—H7*N*···O4*W*$^{ix}$ | 0.86 (3) | 2.38 (3) | 3.149 (2) | 149 (2) |
| N3*C*—H8*N*···O3*A*$^{viii}$ | 0.87 (3) | 2.53 (3) | 3.330 (2) | 154 (2) |
| O1*W*—H1*W*···N3*C*$^{xi}$ | 0.87 (1) | 2.62 (2) | 3.428 (2) | 156 (2) |
| O1*W*—H2*W*···O1*B*$^{ii}$ | 0.87 (1) | 1.94 (1) | 2.808 (2) | 174 (2) |
| O2*W*—H3*W*···O1*C*$^{iii}$ | 0.87 (1) | 2.13 (1) | 2.963 (3) | 160 (3) |
| O2*W*—H3*W*···O5*C*$^{iii}$ | 0.87 (1) | 2.24 (3) | 2.973 (18) | 143 (2) |
| O2*W*—H4*W*···N3*B*$^{vii}$ | 0.87 (1) | 2.56 (2) | 3.322 (2) | 148 (2) |
| O3*W*—H5*W*···N3$^{vii}$ | 0.87 (1) | 2.25 (2) | 3.036 (2) | 151 (3) |
| O3*W*—H6*W*···O3*B* | 0.87 (1) | 2.15 (2) | 2.906 (2) | 145 (2) |
| O4*W*—H7*W*···O1 | 0.87 (1) | 2.03 (1) | 2.879 (2) | 166 (3) |
| O4*W*—H8*W*···O3*C*$^{iv}$ | 0.87 (1) | 2.02 (1) | 2.837 (2) | 156 (2) |
| O4*W*—H8*W*···O6*C*$^{iv}$ | 0.87 (1) | 2.27 (2) | 3.126 (16) | 170 (3) |

| | | | | |
|---|---|---|---|---|
| O5*W*—H9*W*···O3$^{iv}$ | 0.87 (1) | 2.66 (2) | 3.376 (2) | 141 (2) |
| O5*W*—H10*W*···O3*A*$^{iv}$ | 0.89 (1) | 1.93 (1) | 2.807 (2) | 169 (2) |
| O6*W*—H11*W*···O5*W* | 0.87 (1) | 2.11 (1) | 2.924 (2) | 158 (2) |
| O6*W*—H12*W*···O1$^{iv}$ | 0.86 (1) | 1.95 (1) | 2.7885 (19) | 164 (3) |
| O7*W*—H13*W*···O2*C*$^{v}$ | 0.87 (1) | 2.02 (1) | 2.896 (2) | 177 (3) |
| O7*W*—H13*W*···O5*C*$^{v}$ | 0.87 (1) | 1.88 (2) | 2.698 (12) | 156 (3) |
| O7*W*—H14*W*···O3*W*$^{v}$ | 0.88 (1) | 2.22 (1) | 3.085 (2) | 169 (2) |
| O8*W*—H15*W*···O2*A*$^{vi}$ | 0.88 (1) | 1.94 (1) | 2.808 (2) | 168 (2) |

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

## Poly[diaqua[4-(2-{4-[bis(2-hydroxyethyl)amino]phenyl}diazen-1-yl)benzenesulfonato]potassium] (K4)

*Crystal data*

$[K(C_{16}H_{18}N_3O_5S)(H_2O)_2]$
$M_r$ = 439.52
Monoclinic, $P2_1/n$
$a$ = 9.4006 (2) Å
$b$ = 12.1583 (3) Å
$c$ = 34.4743 (9) Å
$\beta$ = 95.496 (1)°
$V$ = 3922.14 (16) Å$^3$
$Z$ = 8
$F(000)$ = 1840
$D_x$ = 1.489 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4144 reflections
$\theta$ = 1.0–27.5°
$\mu$ = 0.42 mm$^{-1}$
$T$ = 123 K
Fragment, yellow-orange
0.20 × 0.18 × 0.08 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
$\omega$ and phi scans
14501 measured reflections
7971 independent reflections
4387 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.084
$\theta_{max}$ = 26.5°, $\theta_{min}$ = 1.8°
$h$ = −11→11
$k$ = −15→14
$l$ = −43→43

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.102
$S$ = 1.01
7971 reflections
560 parameters
112 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 3.3444P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| K1 | 0.62092 (8) | 0.71226 (6) | 0.01530 (2) | 0.0258 (2) | |

| | | | | | |
|---|---|---|---|---|---|
| K2 | 0.77029 (8) | 0.37647 (7) | 0.01526 (2) | 0.0271 (2) | |
| S1 | 1.00862 (10) | 0.25029 (7) | 0.08760 (3) | 0.0245 (2) | |
| S1A | 0.52799 (9) | 0.49509 (7) | 0.06827 (3) | 0.0199 (2) | |
| O1 | 0.8908 (2) | 0.20077 (19) | 0.06291 (7) | 0.0278 (6) | |
| O1A | 0.5147 (2) | 0.38622 (18) | 0.05067 (7) | 0.0266 (6) | |
| O2 | 1.0283 (3) | 0.36445 (19) | 0.07699 (7) | 0.0353 (7) | |
| O2A | 0.4295 (2) | 0.57474 (18) | 0.04875 (7) | 0.0249 (6) | |
| O3 | 1.1377 (2) | 0.1841 (2) | 0.08914 (7) | 0.0318 (7) | |
| O3A | 0.6749 (2) | 0.53511 (18) | 0.07197 (7) | 0.0249 (6) | |
| O4 | 0.3408 (3) | 0.3821 (2) | 0.43887 (8) | 0.0303 (6) | |
| H1H | 0.419 (2) | 0.421 (2) | 0.4435 (10) | 0.036* | |
| O4A | −0.1792 (2) | 0.37386 (19) | 0.42667 (7) | 0.0280 (6) | |
| H3H | −0.109 (2) | 0.420 (2) | 0.4311 (10) | 0.034* | |
| O5 | 0.4632 (3) | 0.1879 (2) | 0.46276 (8) | 0.0331 (7) | |
| H2H | 0.429 (4) | 0.2534 (15) | 0.4564 (10) | 0.040* | |
| O5A | −0.0092 (2) | 0.5506 (2) | 0.44089 (7) | 0.0282 (6) | |
| H4H | 0.066 (2) | 0.554 (3) | 0.4579 (7) | 0.034* | |
| O1W | 0.3676 (3) | 0.8304 (2) | 0.00680 (7) | 0.0318 (7) | |
| H1W | 0.373 (4) | 0.876 (2) | −0.0126 (7) | 0.038* | |
| H2W | 0.305 (3) | 0.863 (2) | 0.0197 (8) | 0.038* | |
| O2W | 1.1728 (3) | 0.4172 (2) | 0.01231 (8) | 0.0382 (7) | |
| H3W | 1.109 (3) | 0.378 (3) | −0.0014 (8) | 0.046* | |
| H4W | 1.156 (4) | 0.403 (3) | 0.0361 (4) | 0.046* | |
| O3W | 0.2480 (2) | 0.6137 (2) | 0.47825 (7) | 0.0295 (6) | |
| H5W | 0.296 (3) | 0.630 (3) | 0.4587 (6) | 0.035* | |
| H6W | 0.316 (2) | 0.586 (3) | 0.4947 (7) | 0.035* | |
| O4W | 0.5948 (3) | 0.4831 (2) | 0.45676 (8) | 0.0341 (7) | |
| H7W | 0.659 (3) | 0.455 (2) | 0.4428 (9) | 0.041* | |
| H8W | 0.599 (3) | 0.5528 (10) | 0.4505 (10) | 0.041* | |
| N1 | 0.7690 (3) | 0.2169 (3) | 0.24068 (8) | 0.0308 (8) | |
| N2 | 0.6618 (3) | 0.2771 (3) | 0.24382 (8) | 0.0308 (8) | |
| N1A | 0.3029 (6) | 0.5236 (5) | 0.22279 (16) | 0.0262 (14) | 0.661 (7) |
| N2A | 0.2019 (7) | 0.4551 (5) | 0.22954 (17) | 0.0274 (13) | 0.661 (7) |
| N2B | 0.2462 (13) | 0.5338 (11) | 0.2346 (3) | 0.0274 (13) | 0.339 (7) |
| N1B | 0.2353 (13) | 0.4540 (12) | 0.2145 (4) | 0.0262 (14) | 0.339 (7) |
| N3 | 0.3547 (3) | 0.2138 (2) | 0.37386 (8) | 0.0259 (7) | |
| N3A | −0.1168 (3) | 0.5177 (2) | 0.35650 (8) | 0.0265 (7) | |
| C1 | 0.9529 (3) | 0.2518 (3) | 0.13522 (10) | 0.0211 (8) | |
| C1A | 0.4756 (19) | 0.4898 (12) | 0.1160 (4) | 0.0164 (17) | 0.661 (7) |
| C2 | 0.8560 (4) | 0.3317 (3) | 0.14473 (10) | 0.0263 (9) | |
| H1 | 0.831089 | 0.390539 | 0.127249 | 0.032* | |
| C3 | 0.7963 (4) | 0.3250 (3) | 0.17975 (11) | 0.0290 (10) | |
| H2 | 0.731465 | 0.379860 | 0.186724 | 0.035* | |
| C4 | 0.8321 (4) | 0.2369 (3) | 0.20482 (10) | 0.0255 (9) | |
| C5 | 0.9339 (4) | 0.1616 (3) | 0.19610 (11) | 0.0303 (10) | |
| H3 | 0.963122 | 0.105188 | 0.214192 | 0.036* | |
| C6 | 0.9934 (4) | 0.1681 (3) | 0.16106 (10) | 0.0264 (9) | |
| H4 | 1.061896 | 0.115256 | 0.154753 | 0.032* | |

| | | | | | |
|---|---|---|---|---|---|
| C7 | 0.5910 (4) | 0.2558 (3) | 0.27774 (10) | 0.0268 (9) | |
| C8 | 0.4826 (4) | 0.3286 (3) | 0.28460 (11) | 0.0325 (10) | |
| H5 | 0.460671 | 0.387794 | 0.267050 | 0.039* | |
| C9 | 0.4060 (4) | 0.3164 (3) | 0.31657 (10) | 0.0298 (10) | |
| H6 | 0.334356 | 0.368631 | 0.321110 | 0.036* | |
| C10 | 0.4322 (4) | 0.2281 (3) | 0.34243 (10) | 0.0237 (9) | |
| C10A | −0.0325 (18) | 0.5018 (10) | 0.3265 (4) | 0.0260 (16) | 0.661 (7) |
| C11 | 0.5412 (4) | 0.1540 (3) | 0.33454 (11) | 0.0292 (10) | |
| H7 | 0.561914 | 0.093513 | 0.351601 | 0.035* | |
| C12 | 0.6185 (4) | 0.1666 (3) | 0.30281 (11) | 0.0292 (10) | |
| H8 | 0.690395 | 0.114777 | 0.298035 | 0.035* | |
| C2A | 0.3749 (17) | 0.4139 (10) | 0.1248 (3) | 0.0236 (15) | 0.661 (7) |
| H2A | 0.343592 | 0.358075 | 0.106681 | 0.028* | 0.661 (7) |
| C3A | 0.3191 (10) | 0.4205 (7) | 0.1612 (3) | 0.0233 (16) | 0.661 (7) |
| H3A | 0.250484 | 0.368325 | 0.167921 | 0.028* | 0.661 (7) |
| C4A | 0.3642 (8) | 0.5029 (6) | 0.1871 (2) | 0.0196 (17) | 0.661 (7) |
| C5A | 0.4737 (9) | 0.5739 (6) | 0.1789 (3) | 0.0230 (16) | 0.661 (7) |
| H5A | 0.509392 | 0.627066 | 0.197539 | 0.028* | 0.661 (7) |
| C6A | 0.5301 (15) | 0.5663 (10) | 0.1434 (4) | 0.0250 (14) | 0.661 (7) |
| H6A | 0.606084 | 0.613376 | 0.137698 | 0.030* | 0.661 (7) |
| C7A | 0.1305 (8) | 0.4756 (6) | 0.2625 (2) | 0.0213 (16) | 0.661 (7) |
| C8A | 0.0395 (9) | 0.3926 (6) | 0.2724 (3) | 0.0262 (16) | 0.661 (7) |
| H8A | 0.031325 | 0.327123 | 0.257344 | 0.031* | 0.661 (7) |
| C9A | −0.0391 (15) | 0.4038 (9) | 0.3039 (4) | 0.0292 (16) | 0.661 (7) |
| H9A | −0.098664 | 0.345127 | 0.310670 | 0.035* | 0.661 (7) |
| C11A | 0.0654 (15) | 0.5837 (7) | 0.3162 (3) | 0.0263 (16) | 0.661 (7) |
| H11A | 0.077171 | 0.648604 | 0.331527 | 0.032* | 0.661 (7) |
| C12A | 0.1434 (9) | 0.5712 (6) | 0.2847 (2) | 0.0244 (16) | 0.661 (7) |
| H12A | 0.206264 | 0.628024 | 0.278124 | 0.029* | 0.661 (7) |
| C1B | 0.470 (4) | 0.470 (3) | 0.1158 (7) | 0.0164 (17) | 0.339 (7) |
| C2B | 0.367 (3) | 0.391 (2) | 0.1201 (6) | 0.0236 (15) | 0.339 (7) |
| H2B | 0.344024 | 0.338534 | 0.099962 | 0.028* | 0.339 (7) |
| C3B | 0.297 (2) | 0.3878 (13) | 0.1538 (6) | 0.0233 (16) | 0.339 (7) |
| H3B | 0.226149 | 0.333454 | 0.156719 | 0.028* | 0.339 (7) |
| C4B | 0.3303 (16) | 0.4644 (11) | 0.1832 (5) | 0.0196 (17) | 0.339 (7) |
| C5B | 0.4338 (19) | 0.5440 (12) | 0.1789 (5) | 0.0230 (16) | 0.339 (7) |
| H5B | 0.456688 | 0.596294 | 0.198977 | 0.028* | 0.339 (7) |
| C6B | 0.504 (3) | 0.5470 (19) | 0.1452 (7) | 0.0250 (14) | 0.339 (7) |
| H6B | 0.574565 | 0.601377 | 0.142220 | 0.030* | 0.339 (7) |
| C7B | 0.1512 (15) | 0.5240 (11) | 0.2660 (4) | 0.0213 (16) | 0.339 (7) |
| C8B | 0.1485 (19) | 0.6142 (9) | 0.2907 (5) | 0.0244 (16) | 0.339 (7) |
| H8B | 0.207002 | 0.676301 | 0.286941 | 0.029* | 0.339 (7) |
| C9B | 0.060 (3) | 0.6136 (15) | 0.3208 (7) | 0.0263 (16) | 0.339 (7) |
| H9B | 0.058397 | 0.675243 | 0.337650 | 0.032* | 0.339 (7) |
| C10B | −0.025 (4) | 0.523 (2) | 0.3262 (8) | 0.0260 (16) | 0.339 (7) |
| C11B | −0.023 (3) | 0.4325 (16) | 0.3016 (8) | 0.0292 (16) | 0.339 (7) |
| H11B | −0.081131 | 0.370347 | 0.305289 | 0.035* | 0.339 (7) |
| C12B | 0.066 (2) | 0.4331 (10) | 0.2714 (5) | 0.0262 (16) | 0.339 (7) |

| | | | | | |
|---|---|---|---|---|---|
| H12B | 0.067473 | 0.371403 | 0.254579 | 0.031* | 0.339 (7) |
| C13 | 0.2378 (4) | 0.2887 (3) | 0.38086 (11) | 0.0293 (9) | |
| H10 | 0.174460 | 0.251538 | 0.398096 | 0.035* | |
| H9 | 0.180883 | 0.303336 | 0.355723 | 0.035* | |
| C13A | −0.2335 (4) | 0.4395 (3) | 0.35967 (11) | 0.0340 (10) | |
| H10Z | −0.265121 | 0.410732 | 0.333383 | 0.041* | |
| H9Z | −0.315305 | 0.478847 | 0.369322 | 0.041* | |
| C14 | 0.2845 (4) | 0.3974 (3) | 0.39906 (11) | 0.0337 (10) | |
| H11 | 0.358573 | 0.430807 | 0.384207 | 0.040* | |
| H12 | 0.201974 | 0.448291 | 0.397926 | 0.040* | |
| C14A | −0.1938 (4) | 0.3438 (3) | 0.38641 (11) | 0.0325 (10) | |
| H12Z | −0.268054 | 0.286201 | 0.382158 | 0.039* | |
| H11Z | −0.102392 | 0.312115 | 0.379608 | 0.039* | |
| C15 | 0.3763 (4) | 0.1161 (3) | 0.39830 (11) | 0.0321 (10) | |
| H14 | 0.394140 | 0.052405 | 0.381567 | 0.038* | |
| H13 | 0.287528 | 0.101129 | 0.410683 | 0.038* | |
| C15A | −0.1184 (4) | 0.6188 (3) | 0.37928 (11) | 0.0299 (10) | |
| H13A | −0.209267 | 0.621970 | 0.391553 | 0.036* | |
| H14A | −0.117071 | 0.682160 | 0.361280 | 0.036* | |
| C16 | 0.4991 (4) | 0.1263 (3) | 0.42982 (11) | 0.0306 (10) | |
| H15 | 0.530215 | 0.051774 | 0.438510 | 0.037* | |
| H16 | 0.580613 | 0.162388 | 0.418761 | 0.037* | |
| C16A | 0.0029 (4) | 0.6315 (3) | 0.41055 (10) | 0.0282 (9) | |
| H16A | 0.094903 | 0.621735 | 0.399200 | 0.034* | |
| H15A | 0.001225 | 0.706329 | 0.421790 | 0.034* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| K1 | 0.0291 (5) | 0.0211 (4) | 0.0282 (5) | −0.0024 (4) | 0.0077 (4) | 0.0004 (4) |
| K2 | 0.0232 (5) | 0.0295 (5) | 0.0295 (5) | 0.0007 (4) | 0.0064 (4) | −0.0018 (4) |
| S1 | 0.0244 (5) | 0.0240 (5) | 0.0260 (5) | −0.0008 (5) | 0.0076 (4) | −0.0009 (5) |
| S1A | 0.0197 (5) | 0.0203 (5) | 0.0203 (5) | −0.0010 (4) | 0.0056 (4) | 0.0007 (4) |
| O1 | 0.0256 (14) | 0.0323 (15) | 0.0250 (14) | 0.0007 (12) | 0.0007 (12) | −0.0023 (12) |
| O1A | 0.0300 (15) | 0.0207 (14) | 0.0306 (15) | −0.0009 (12) | 0.0101 (12) | −0.0049 (12) |
| O2 | 0.0450 (17) | 0.0240 (15) | 0.0401 (17) | −0.0071 (13) | 0.0209 (14) | 0.0016 (13) |
| O2A | 0.0234 (14) | 0.0267 (14) | 0.0251 (14) | 0.0049 (12) | 0.0052 (12) | 0.0081 (12) |
| O3 | 0.0207 (14) | 0.0421 (16) | 0.0336 (16) | 0.0089 (13) | 0.0081 (12) | −0.0010 (13) |
| O3A | 0.0154 (13) | 0.0298 (14) | 0.0300 (15) | −0.0050 (11) | 0.0046 (12) | 0.0003 (12) |
| O4 | 0.0344 (16) | 0.0262 (15) | 0.0310 (16) | −0.0023 (13) | 0.0069 (14) | −0.0022 (13) |
| O4A | 0.0284 (16) | 0.0260 (15) | 0.0295 (15) | −0.0067 (12) | 0.0025 (13) | 0.0020 (13) |
| O5 | 0.0378 (17) | 0.0300 (16) | 0.0320 (16) | 0.0080 (14) | 0.0055 (13) | 0.0038 (14) |
| O5A | 0.0282 (16) | 0.0307 (15) | 0.0251 (16) | −0.0026 (13) | −0.0010 (12) | 0.0011 (13) |
| O1W | 0.0376 (17) | 0.0276 (15) | 0.0312 (17) | 0.0063 (13) | 0.0085 (13) | 0.0033 (13) |
| O2W | 0.0408 (19) | 0.0425 (18) | 0.0330 (17) | 0.0056 (14) | 0.0128 (15) | 0.0074 (15) |
| O3W | 0.0272 (16) | 0.0347 (16) | 0.0272 (16) | 0.0041 (13) | 0.0055 (13) | 0.0020 (14) |
| O4W | 0.0358 (17) | 0.0297 (15) | 0.0384 (17) | 0.0063 (14) | 0.0115 (13) | 0.0041 (14) |
| N1 | 0.033 (2) | 0.034 (2) | 0.0251 (18) | 0.0018 (17) | 0.0030 (16) | −0.0051 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N2 | 0.0289 (19) | 0.037 (2) | 0.0264 (19) | 0.0022 (17) | 0.0031 (16) | −0.0059 (16) |
| N1A | 0.020 (3) | 0.039 (3) | 0.021 (3) | 0.003 (2) | 0.012 (2) | 0.006 (3) |
| N2A | 0.027 (3) | 0.035 (3) | 0.021 (3) | 0.002 (3) | 0.011 (2) | 0.005 (3) |
| N2B | 0.027 (3) | 0.035 (3) | 0.021 (3) | 0.002 (3) | 0.011 (2) | 0.005 (3) |
| N1B | 0.020 (3) | 0.039 (3) | 0.021 (3) | 0.003 (2) | 0.012 (2) | 0.006 (3) |
| N3 | 0.0232 (17) | 0.0275 (18) | 0.0279 (18) | 0.0015 (15) | 0.0073 (15) | −0.0004 (16) |
| N3A | 0.0232 (17) | 0.0360 (19) | 0.0214 (17) | 0.0012 (16) | 0.0074 (14) | −0.0003 (16) |
| C1 | 0.016 (2) | 0.025 (2) | 0.023 (2) | −0.0074 (17) | 0.0021 (17) | −0.0016 (18) |
| C1A | 0.021 (2) | 0.011 (5) | 0.0182 (19) | 0.006 (3) | 0.0058 (17) | 0.005 (3) |
| C2 | 0.026 (2) | 0.024 (2) | 0.028 (2) | −0.0015 (18) | −0.0011 (19) | 0.0027 (18) |
| C3 | 0.029 (2) | 0.027 (2) | 0.031 (2) | 0.0012 (19) | 0.010 (2) | −0.007 (2) |
| C4 | 0.027 (2) | 0.031 (2) | 0.019 (2) | −0.0059 (19) | 0.0023 (18) | −0.0030 (18) |
| C5 | 0.036 (2) | 0.031 (2) | 0.024 (2) | 0.002 (2) | 0.003 (2) | 0.0025 (19) |
| C6 | 0.022 (2) | 0.031 (2) | 0.027 (2) | 0.0009 (18) | 0.0048 (18) | −0.0038 (19) |
| C7 | 0.024 (2) | 0.035 (2) | 0.021 (2) | −0.0002 (19) | 0.0003 (18) | −0.0058 (19) |
| C8 | 0.030 (2) | 0.038 (2) | 0.029 (2) | 0.004 (2) | 0.000 (2) | 0.002 (2) |
| C9 | 0.023 (2) | 0.038 (2) | 0.028 (2) | 0.0078 (19) | 0.0002 (19) | 0.002 (2) |
| C10 | 0.019 (2) | 0.027 (2) | 0.026 (2) | −0.0025 (18) | 0.0041 (17) | −0.0030 (18) |
| C10A | 0.018 (3) | 0.040 (5) | 0.020 (2) | 0.002 (3) | 0.0011 (18) | 0.004 (3) |
| C11 | 0.031 (2) | 0.027 (2) | 0.030 (2) | 0.0007 (19) | 0.0064 (19) | −0.0032 (18) |
| C12 | 0.027 (2) | 0.030 (2) | 0.031 (2) | 0.0053 (19) | 0.0056 (19) | −0.010 (2) |
| C2A | 0.023 (3) | 0.021 (5) | 0.027 (3) | −0.001 (4) | 0.008 (3) | 0.000 (2) |
| C3A | 0.010 (4) | 0.034 (5) | 0.026 (5) | −0.014 (3) | −0.002 (3) | 0.007 (4) |
| C4A | 0.019 (4) | 0.016 (5) | 0.024 (3) | −0.004 (3) | 0.003 (3) | 0.004 (3) |
| C5A | 0.033 (5) | 0.014 (4) | 0.022 (2) | −0.010 (3) | 0.003 (3) | 0.000 (3) |
| C6A | 0.037 (5) | 0.015 (4) | 0.024 (2) | −0.009 (3) | 0.008 (3) | 0.003 (3) |
| C7A | 0.019 (3) | 0.026 (5) | 0.019 (3) | −0.005 (4) | 0.001 (2) | −0.001 (3) |
| C8A | 0.024 (4) | 0.031 (5) | 0.023 (2) | −0.013 (3) | 0.003 (3) | −0.009 (4) |
| C9A | 0.026 (4) | 0.042 (5) | 0.021 (3) | −0.008 (4) | 0.006 (2) | −0.002 (4) |
| C11A | 0.029 (3) | 0.025 (5) | 0.026 (3) | 0.004 (4) | 0.007 (2) | 0.000 (3) |
| C12A | 0.023 (2) | 0.023 (5) | 0.028 (4) | −0.003 (4) | 0.004 (2) | 0.004 (4) |
| C1B | 0.021 (2) | 0.011 (5) | 0.0182 (19) | 0.006 (3) | 0.0058 (17) | 0.005 (3) |
| C2B | 0.023 (3) | 0.021 (5) | 0.027 (3) | −0.001 (4) | 0.008 (3) | 0.000 (2) |
| C3B | 0.010 (4) | 0.034 (5) | 0.026 (5) | −0.014 (3) | −0.002 (3) | 0.007 (4) |
| C4B | 0.019 (4) | 0.016 (5) | 0.024 (3) | −0.004 (3) | 0.003 (3) | 0.004 (3) |
| C5B | 0.033 (5) | 0.014 (4) | 0.022 (2) | −0.010 (3) | 0.003 (3) | 0.000 (3) |
| C6B | 0.037 (5) | 0.015 (4) | 0.024 (2) | −0.009 (3) | 0.008 (3) | 0.003 (3) |
| C7B | 0.019 (3) | 0.026 (5) | 0.019 (3) | −0.005 (4) | 0.001 (2) | −0.001 (3) |
| C8B | 0.023 (2) | 0.023 (5) | 0.028 (4) | −0.003 (4) | 0.004 (2) | 0.004 (4) |
| C9B | 0.029 (3) | 0.025 (5) | 0.026 (3) | 0.004 (4) | 0.007 (2) | 0.000 (3) |
| C10B | 0.018 (3) | 0.040 (5) | 0.020 (2) | 0.002 (3) | 0.0011 (18) | 0.004 (3) |
| C11B | 0.026 (4) | 0.042 (5) | 0.021 (3) | −0.008 (4) | 0.006 (2) | −0.002 (4) |
| C12B | 0.024 (4) | 0.031 (5) | 0.023 (2) | −0.013 (3) | 0.003 (3) | −0.009 (4) |
| C13 | 0.018 (2) | 0.042 (2) | 0.027 (2) | 0.0008 (19) | 0.0033 (18) | 0.000 (2) |
| C13A | 0.025 (2) | 0.048 (3) | 0.030 (2) | 0.000 (2) | 0.007 (2) | −0.009 (2) |
| C14 | 0.037 (2) | 0.030 (2) | 0.035 (2) | 0.011 (2) | 0.009 (2) | 0.004 (2) |
| C14A | 0.027 (2) | 0.033 (2) | 0.038 (3) | −0.0094 (19) | 0.005 (2) | −0.010 (2) |
| C15 | 0.026 (2) | 0.027 (2) | 0.044 (3) | −0.0039 (19) | 0.011 (2) | −0.002 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15A | 0.026 (2) | 0.033 (2) | 0.032 (2) | 0.0114 (19) | 0.0073 (19) | 0.010 (2) |
| C16 | 0.032 (2) | 0.025 (2) | 0.038 (2) | 0.0038 (19) | 0.016 (2) | 0.004 (2) |
| C16A | 0.034 (2) | 0.020 (2) | 0.031 (2) | −0.0014 (18) | 0.0087 (19) | 0.0006 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| K1—O2W$^{i}$ | 2.738 (3) | C5—H3 | 0.9500 |
| K1—O1A$^{ii}$ | 2.771 (3) | C6—H4 | 0.9500 |
| K1—O1W | 2.772 (3) | C7—C8 | 1.388 (5) |
| K1—O2A | 2.786 (2) | C7—C12 | 1.395 (5) |
| K1—O3W$^{iii}$ | 2.798 (3) | C8—C9 | 1.381 (5) |
| K1—O4A$^{iv}$ | 2.820 (3) | C8—H5 | 0.9500 |
| K1—O3A | 2.919 (2) | C9—C10 | 1.401 (5) |
| K1—S1A | 3.3728 (11) | C9—H6 | 0.9500 |
| K1—K2$^{ii}$ | 3.8802 (11) | C10—C11 | 1.411 (5) |
| K1—K2 | 4.3173 (11) | C10A—C9A | 1.420 (8) |
| K2—O2W$^{i}$ | 2.753 (3) | C10A—C11A | 1.423 (7) |
| K2—O5$^{v}$ | 2.794 (3) | C11—C12 | 1.379 (5) |
| K2—O1A | 2.800 (2) | C11—H7 | 0.9500 |
| K2—O2A$^{ii}$ | 2.821 (2) | C12—H8 | 0.9500 |
| K2—O1 | 2.862 (2) | C2A—C3A | 1.406 (7) |
| K2—O1W$^{ii}$ | 2.898 (3) | C2A—H2A | 0.9500 |
| K2—O3A | 2.947 (2) | C3A—C4A | 1.381 (8) |
| K2—O2 | 3.073 (3) | C3A—H3A | 0.9500 |
| K2—S1A | 3.3773 (11) | C4A—C5A | 1.393 (8) |
| K2—S1 | 3.5388 (13) | C5A—C6A | 1.384 (8) |
| S1—O2 | 1.452 (2) | C5A—H5A | 0.9500 |
| S1—O3 | 1.453 (2) | C6A—H6A | 0.9500 |
| S1—O1 | 1.461 (2) | C7A—C8A | 1.387 (8) |
| S1—C1 | 1.770 (3) | C7A—C12A | 1.391 (8) |
| S1A—O1A | 1.457 (2) | C8A—C9A | 1.377 (8) |
| S1A—O3A | 1.458 (2) | C8A—H8A | 0.9500 |
| S1A—O2A | 1.459 (2) | C9A—H9A | 0.9500 |
| S1A—C1A | 1.764 (9) | C11A—C12A | 1.376 (7) |
| S1A—C1B | 1.798 (17) | C11A—H11A | 0.9500 |
| O4—C14 | 1.435 (4) | C12A—H12A | 0.9500 |
| O4—H1H | 0.873 (10) | C1B—C2B | 1.3900 |
| O4A—C14A | 1.429 (4) | C1B—C6B | 1.3900 |
| O4A—H3H | 0.870 (10) | C2B—C3B | 1.3900 |
| O5—C16 | 1.428 (4) | C2B—H2B | 0.9500 |
| O5—H2H | 0.878 (10) | C3B—C4B | 1.3900 |
| O5A—C16A | 1.448 (4) | C3B—H3B | 0.9500 |
| O5A—H4H | 0.876 (10) | C4B—C5B | 1.3900 |
| O1W—H1W | 0.874 (10) | C5B—C6B | 1.3900 |
| O1W—H2W | 0.869 (10) | C5B—H5B | 0.9500 |
| O2W—H3W | 0.871 (10) | C6B—H6B | 0.9500 |
| O2W—H4W | 0.869 (10) | C7B—C8B | 1.3900 |
| O3W—H5W | 0.871 (10) | C7B—C12B | 1.3900 |

| | | | |
|---|---|---|---|
| O3W—H6W | 0.879 (10) | C8B—C9B | 1.3900 |
| O4W—H7W | 0.875 (10) | C8B—H8B | 0.9500 |
| O4W—H8W | 0.876 (10) | C9B—C10B | 1.3900 |
| N1—N2 | 1.259 (4) | C9B—H9B | 0.9500 |
| N1—C4 | 1.442 (4) | C10B—C11B | 1.3900 |
| N2—C7 | 1.423 (4) | C11B—C12B | 1.3900 |
| N1A—N2A | 1.301 (11) | C11B—H11B | 0.9500 |
| N1A—C4A | 1.431 (9) | C12B—H12B | 0.9500 |
| N2A—C7A | 1.396 (9) | C13—C14 | 1.510 (5) |
| N2B—N1B | 1.19 (2) | C13—H10 | 0.9900 |
| N2B—C7B | 1.472 (17) | C13—H9 | 0.9900 |
| N1B—C4B | 1.471 (18) | C13A—C14A | 1.509 (5) |
| N3—C10 | 1.373 (4) | C13A—H10Z | 0.9900 |
| N3—C15 | 1.459 (4) | C13A—H9Z | 0.9900 |
| N3—C13 | 1.465 (4) | C14—H11 | 0.9900 |
| N3A—C10A | 1.376 (9) | C14—H12 | 0.9900 |
| N3A—C10B | 1.415 (13) | C14A—H12Z | 0.9900 |
| N3A—C15A | 1.459 (4) | C14A—H11Z | 0.9900 |
| N3A—C13A | 1.465 (4) | C15—C16 | 1.513 (5) |
| C1—C6 | 1.382 (5) | C15—H14 | 0.9900 |
| C1—C2 | 1.392 (5) | C15—H13 | 0.9900 |
| C1A—C2A | 1.377 (7) | C15A—C16A | 1.500 (5) |
| C1A—C6A | 1.387 (7) | C15A—H13A | 0.9900 |
| C2—C3 | 1.381 (5) | C15A—H14A | 0.9900 |
| C2—H1 | 0.9500 | C16—H15 | 0.9900 |
| C3—C4 | 1.397 (5) | C16—H16 | 0.9900 |
| C3—H2 | 0.9500 | C16A—H16A | 0.9900 |
| C4—C5 | 1.378 (5) | C16A—H15A | 0.9900 |
| C5—C6 | 1.381 (5) | | |

| | | | |
|---|---|---|---|
| O2W$^{i}$—K1—O1A$^{ii}$ | 75.62 (8) | K1$^{viii}$—O3W—H6W | 108 (2) |
| O2W$^{i}$—K1—O1W | 152.98 (9) | H5W—O3W—H6W | 101.2 (19) |
| O1A$^{ii}$—K1—O1W | 79.29 (8) | H7W—O4W—H8W | 101.1 (19) |
| O2W$^{i}$—K1—O2A | 107.81 (7) | N2—N1—C4 | 112.0 (3) |
| O1A$^{ii}$—K1—O2A | 79.38 (7) | N1—N2—C7 | 113.9 (3) |
| O1W—K1—O2A | 76.85 (7) | N2A—N1A—C4A | 113.7 (7) |
| O2W$^{i}$—K1—O3W$^{iii}$ | 85.91 (7) | N1A—N2A—C7A | 116.5 (7) |
| O1A$^{ii}$—K1—O3W$^{iii}$ | 97.77 (7) | N1B—N2B—C7B | 109.7 (13) |
| O1W—K1—O3W$^{iii}$ | 87.69 (7) | N2B—N1B—C4B | 109.5 (14) |
| O2A—K1—O3W$^{iii}$ | 164.54 (7) | C10—N3—C15 | 120.3 (3) |
| O2W$^{i}$—K1—O4A$^{iv}$ | 123.44 (8) | C10—N3—C13 | 120.9 (3) |
| O1A$^{ii}$—K1—O4A$^{iv}$ | 157.87 (7) | C15—N3—C13 | 118.4 (3) |
| O1W—K1—O4A$^{iv}$ | 79.66 (8) | C10A—N3A—C15A | 124.0 (5) |
| O2A—K1—O4A$^{iv}$ | 102.35 (7) | C10B—N3A—C15A | 113.5 (10) |
| O3W$^{iii}$—K1—O4A$^{iv}$ | 74.59 (7) | C10A—N3A—C13A | 117.0 (6) |
| O2W$^{i}$—K1—O3A | 74.04 (7) | C10B—N3A—C13A | 126.5 (11) |
| O1A$^{ii}$—K1—O3A | 105.50 (7) | C15A—N3A—C13A | 117.4 (3) |
| O1W—K1—O3A | 123.06 (7) | C6—C1—C2 | 120.5 (3) |

| | | | |
|---|---|---|---|
| O2A—K1—O3A | 50.05 (6) | C6—C1—S1 | 120.2 (3) |
| O3W$^{iii}$—K1—O3A | 144.00 (7) | C2—C1—S1 | 119.0 (3) |
| O4A$^{iv}$—K1—O3A | 91.71 (7) | C2A—C1A—C6A | 121.1 (5) |
| O2W$^{i}$—K1—S1A | 88.10 (6) | C2A—C1A—S1A | 119.3 (8) |
| O1A$^{ii}$—K1—S1A | 89.05 (5) | C6A—C1A—S1A | 119.4 (7) |
| O1W—K1—S1A | 101.45 (6) | C3—C2—C1 | 119.6 (3) |
| O2A—K1—S1A | 25.17 (5) | C3—C2—H1 | 120.2 |
| O3W$^{iii}$—K1—S1A | 169.52 (6) | C1—C2—H1 | 120.2 |
| O4A$^{iv}$—K1—S1A | 101.78 (6) | C2—C3—C4 | 119.5 (3) |
| O3A—K1—S1A | 25.51 (4) | C2—C3—H2 | 120.3 |
| O2W$^{i}$—K1—K2$^{ii}$ | 115.47 (6) | C4—C3—H2 | 120.3 |
| O1A$^{ii}$—K1—K2$^{ii}$ | 46.16 (5) | C5—C4—C3 | 120.3 (3) |
| O1W—K1—K2$^{ii}$ | 48.19 (6) | C5—C4—N1 | 115.2 (3) |
| O2A—K1—K2$^{ii}$ | 46.57 (5) | C3—C4—N1 | 124.4 (3) |
| O3W$^{iii}$—K1—K2$^{ii}$ | 121.42 (6) | C4—C5—C6 | 120.0 (4) |
| O4A$^{iv}$—K1—K2$^{ii}$ | 120.07 (5) | C4—C5—H3 | 120.0 |
| O3A—K1—K2$^{ii}$ | 94.36 (5) | C6—C5—H3 | 120.0 |
| S1A—K1—K2$^{ii}$ | 68.99 (2) | C5—C6—C1 | 119.8 (3) |
| O2W$^{i}$—K1—K2 | 38.29 (5) | C5—C6—H4 | 120.1 |
| O1A$^{ii}$—K1—K2 | 73.45 (5) | C1—C6—H4 | 120.1 |
| O1W—K1—K2 | 140.00 (6) | C8—C7—C12 | 119.1 (3) |
| O2A—K1—K2 | 69.85 (5) | C8—C7—N2 | 115.6 (3) |
| O3W$^{iii}$—K1—K2 | 124.19 (5) | C12—C7—N2 | 125.3 (3) |
| O4A$^{iv}$—K1—K2 | 128.17 (6) | C9—C8—C7 | 121.0 (4) |
| O3A—K1—K2 | 42.86 (5) | C9—C8—H5 | 119.5 |
| S1A—K1—K2 | 50.28 (2) | C7—C8—H5 | 119.5 |
| K2$^{ii}$—K1—K2 | 92.11 (2) | C8—C9—C10 | 121.2 (3) |
| O2W$^{i}$—K2—O5$^{v}$ | 82.66 (8) | C8—C9—H6 | 119.4 |
| O2W$^{i}$—K2—O1A | 108.37 (7) | C10—C9—H6 | 119.4 |
| O5$^{v}$—K2—O1A | 158.69 (8) | N3—C10—C9 | 121.7 (3) |
| O2W$^{i}$—K2—O2A$^{ii}$ | 71.04 (8) | N3—C10—C11 | 121.4 (3) |
| O5$^{v}$—K2—O2A$^{ii}$ | 88.68 (7) | C9—C10—C11 | 116.9 (3) |
| O1A—K2—O2A$^{ii}$ | 78.30 (7) | N3A—C10A—C9A | 122.1 (8) |
| O2W$^{i}$—K2—O1 | 143.04 (8) | N3A—C10A—C11A | 121.4 (8) |
| O5$^{v}$—K2—O1 | 85.24 (7) | C9A—C10A—C11A | 116.5 (5) |
| O1A—K2—O1 | 95.18 (7) | C12—C11—C10 | 122.0 (4) |
| O2A$^{ii}$—K2—O1 | 143.46 (7) | C12—C11—H7 | 119.0 |
| O2W$^{i}$—K2—O1W$^{ii}$ | 142.86 (9) | C10—C11—H7 | 119.0 |
| O5$^{v}$—K2—O1W$^{ii}$ | 83.60 (7) | C11—C12—C7 | 119.9 (3) |
| O1A—K2—O1W$^{ii}$ | 76.71 (7) | C11—C12—H8 | 120.1 |
| O2A$^{ii}$—K2—O1W$^{ii}$ | 74.31 (7) | C7—C12—H8 | 120.1 |
| O1—K2—O1W$^{ii}$ | 69.22 (7) | C1A—C2A—C3A | 118.7 (5) |
| O2W$^{i}$—K2—O3A | 73.39 (7) | C1A—C2A—H2A | 120.6 |
| O5$^{v}$—K2—O3A | 150.57 (7) | C3A—C2A—H2A | 120.6 |
| O1A—K2—O3A | 49.80 (6) | C4A—C3A—C2A | 120.1 (5) |
| O2A$^{ii}$—K2—O3A | 99.16 (7) | C4A—C3A—H3A | 120.0 |
| O1—K2—O3A | 103.69 (7) | C2A—C3A—H3A | 120.0 |
| O1W$^{ii}$—K2—O3A | 125.83 (7) | C3A—C4A—C5A | 120.3 (5) |

| | | | |
|---|---|---|---|
| O2W$^{i}$—K2—O2 | 96.65 (8) | C3A—C4A—N1A | 124.1 (6) |
| O5$^{v}$—K2—O2 | 85.25 (7) | C5A—C4A—N1A | 115.6 (6) |
| O1A—K2—O2 | 110.70 (7) | C6A—C5A—C4A | 119.4 (5) |
| O2A$^{ii}$—K2—O2 | 166.92 (7) | C6A—C5A—H5A | 120.3 |
| O1—K2—O2 | 47.48 (6) | C4A—C5A—H5A | 120.3 |
| O1W$^{ii}$—K2—O2 | 116.36 (7) | C5A—C6A—C1A | 119.9 (5) |
| O3A—K2—O2 | 80.90 (6) | C5A—C6A—H6A | 120.1 |
| O2W$^{i}$—K2—S1A | 87.77 (6) | C1A—C6A—H6A | 120.1 |
| O5$^{v}$—K2—S1A | 169.57 (6) | C8A—C7A—C12A | 119.7 (5) |
| O1A—K2—S1A | 25.12 (5) | C8A—C7A—N2A | 115.2 (6) |
| O2A$^{ii}$—K2—S1A | 84.29 (5) | C12A—C7A—N2A | 125.0 (6) |
| O1—K2—S1A | 104.98 (5) | C9A—C8A—C7A | 120.8 (5) |
| O1W$^{ii}$—K2—S1A | 101.81 (5) | C9A—C8A—H8A | 119.6 |
| O3A—K2—S1A | 25.51 (4) | C7A—C8A—H8A | 119.6 |
| O2—K2—S1A | 100.02 (5) | C8A—C9A—C10A | 121.1 (6) |
| O2W$^{i}$—K2—S1 | 120.50 (7) | C8A—C9A—H9A | 119.5 |
| O5$^{v}$—K2—S1 | 85.91 (6) | C10A—C9A—H9A | 119.5 |
| O1A—K2—S1 | 102.88 (6) | C12A—C11A—C10A | 121.7 (6) |
| O2A$^{ii}$—K2—S1 | 166.36 (6) | C12A—C11A—H11A | 119.1 |
| O1—K2—S1 | 23.47 (5) | C10A—C11A—H11A | 119.1 |
| O1W$^{ii}$—K2—S1 | 92.63 (6) | C11A—C12A—C7A | 120.1 (5) |
| O3A—K2—S1 | 91.63 (5) | C11A—C12A—H12A | 120.0 |
| O2—K2—S1 | 24.07 (4) | C7A—C12A—H12A | 120.0 |
| S1A—K2—S1 | 102.59 (3) | C2B—C1B—C6B | 120.0 |
| O2W$^{i}$—K2—K1$^{ii}$ | 111.72 (6) | C2B—C1B—S1A | 119.2 (15) |
| O5$^{v}$—K2—K1$^{ii}$ | 113.78 (6) | C6B—C1B—S1A | 119.0 (14) |
| O1A—K2—K1$^{ii}$ | 45.54 (5) | C1B—C2B—C3B | 120.0 |
| O2A$^{ii}$—K2—K1$^{ii}$ | 45.83 (5) | C1B—C2B—H2B | 120.0 |
| O1—K2—K1$^{ii}$ | 105.13 (5) | C3B—C2B—H2B | 120.0 |
| O1W$^{ii}$—K2—K1$^{ii}$ | 45.48 (5) | C4B—C3B—C2B | 120.0 |
| O3A—K2—K1$^{ii}$ | 91.22 (5) | C4B—C3B—H3B | 120.0 |
| O2—K2—K1$^{ii}$ | 147.08 (5) | C2B—C3B—H3B | 120.0 |
| S1A—K2—K1$^{ii}$ | 66.10 (2) | C5B—C4B—C3B | 120.0 |
| S1—K2—K1$^{ii}$ | 126.19 (3) | C5B—C4B—N1B | 128.2 (12) |
| O2W$^{i}$—K2—K1 | 38.04 (5) | C3B—C4B—N1B | 111.5 (12) |
| O5$^{v}$—K2—K1 | 119.77 (6) | C6B—C5B—C4B | 120.0 |
| O1A—K2—K1 | 70.56 (5) | C6B—C5B—H5B | 120.0 |
| O2A$^{ii}$—K2—K1 | 66.99 (5) | C4B—C5B—H5B | 120.0 |
| O1—K2—K1 | 144.74 (5) | C5B—C6B—C1B | 120.0 |
| O1W$^{ii}$—K2—K1 | 133.12 (6) | C5B—C6B—H6B | 120.0 |
| O3A—K2—K1 | 42.37 (5) | C1B—C6B—H6B | 120.0 |
| O2—K2—K1 | 106.25 (5) | C8B—C7B—C12B | 120.0 |
| S1A—K2—K1 | 50.19 (2) | C8B—C7B—N2B | 115.7 (11) |
| S1—K2—K1 | 126.42 (3) | C12B—C7B—N2B | 124.3 (11) |
| K1$^{ii}$—K2—K1 | 87.89 (2) | C7B—C8B—C9B | 120.0 |
| O2—S1—O3 | 114.37 (15) | C7B—C8B—H8B | 120.0 |
| O2—S1—O1 | 110.80 (15) | C9B—C8B—H8B | 120.0 |
| O3—S1—O1 | 112.16 (14) | C8B—C9B—C10B | 120.0 |

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|---|---|---|---|
| O2—S1—C1 | 106.26 (15) | C8B—C9B—H9B | 120.0 |
| O3—S1—C1 | 107.00 (16) | C10B—C9B—H9B | 120.0 |
| O1—S1—C1 | 105.62 (14) | C9B—C10B—C11B | 120.0 |
| O2—S1—K2 | 59.68 (11) | C9B—C10B—N3A | 122.3 (15) |
| O3—S1—K2 | 137.40 (11) | C11B—C10B—N3A | 117.7 (15) |
| O1—S1—K2 | 51.27 (10) | C10B—C11B—C12B | 120.0 |
| C1—S1—K2 | 115.17 (12) | C10B—C11B—H11B | 120.0 |
| O1A—S1A—O3A | 112.48 (14) | C12B—C11B—H11B | 120.0 |
| O1A—S1A—O2A | 112.69 (15) | C11B—C12B—C7B | 120.0 |
| O3A—S1A—O2A | 111.87 (14) | C11B—C12B—H12B | 120.0 |
| O1A—S1A—C1A | 109.6 (4) | C7B—C12B—H12B | 120.0 |
| O3A—S1A—C1A | 106.1 (6) | N3—C13—C14 | 114.8 (3) |
| O2A—S1A—C1A | 103.5 (7) | N3—C13—H10 | 108.6 |
| O1A—S1A—C1B | 101.9 (9) | C14—C13—H10 | 108.6 |
| O3A—S1A—C1B | 110.0 (11) | N3—C13—H9 | 108.6 |
| O2A—S1A—C1B | 107.3 (13) | C14—C13—H9 | 108.6 |
| O1A—S1A—K1 | 120.14 (10) | H10—C13—H9 | 107.5 |
| O3A—S1A—K1 | 59.59 (10) | N3A—C13A—C14A | 113.8 (3) |
| O2A—S1A—K1 | 54.29 (9) | N3A—C13A—H10Z | 108.8 |
| C1A—S1A—K1 | 130.1 (4) | C14A—C13A—H10Z | 108.8 |
| C1B—S1A—K1 | 137.7 (9) | N3A—C13A—H9Z | 108.8 |
| O1A—S1A—K2 | 54.68 (9) | C14A—C13A—H9Z | 108.8 |
| O3A—S1A—K2 | 60.50 (9) | H10Z—C13A—H9Z | 107.7 |
| O2A—S1A—K2 | 117.69 (10) | O4—C14—C13 | 110.5 (3) |
| C1A—S1A—K2 | 138.8 (7) | O4—C14—H11 | 109.6 |
| C1B—S1A—K2 | 134.4 (13) | C13—C14—H11 | 109.6 |
| K1—S1A—K2 | 79.52 (3) | O4—C14—H12 | 109.6 |
| S1—O1—K2 | 105.26 (12) | C13—C14—H12 | 109.6 |
| S1A—O1A—K1$^{ii}$ | 138.52 (14) | H11—C14—H12 | 108.1 |
| S1A—O1A—K2 | 100.21 (11) | O4A—C14A—C13A | 113.0 (3) |
| K1$^{ii}$—O1A—K2 | 88.30 (7) | O4A—C14A—H12Z | 109.0 |
| S1—O2—K2 | 96.25 (12) | C13A—C14A—H12Z | 109.0 |
| S1A—O2A—K1 | 100.55 (11) | O4A—C14A—H11Z | 109.0 |
| S1A—O2A—K2$^{ii}$ | 147.79 (14) | C13A—C14A—H11Z | 109.0 |
| K1—O2A—K2$^{ii}$ | 87.60 (7) | H12Z—C14A—H11Z | 107.8 |
| S1A—O3A—K1 | 94.89 (11) | N3—C15—C16 | 113.7 (3) |
| S1A—O3A—K2 | 93.98 (11) | N3—C15—H14 | 108.8 |
| K1—O3A—K2 | 94.78 (7) | C16—C15—H14 | 108.8 |
| C14—O4—H1H | 109 (2) | N3—C15—H13 | 108.8 |
| C14A—O4A—K1$^{vi}$ | 120.3 (2) | C16—C15—H13 | 108.8 |
| C14A—O4A—H3H | 110 (2) | H14—C15—H13 | 107.7 |
| K1$^{vi}$—O4A—H3H | 103 (2) | N3A—C15A—C16A | 115.3 (3) |
| C16—O5—K2$^{vii}$ | 125.9 (2) | N3A—C15A—H13A | 108.5 |
| C16—O5—H2H | 113 (2) | C16A—C15A—H13A | 108.5 |
| K2$^{vii}$—O5—H2H | 100 (2) | N3A—C15A—H14A | 108.5 |
| C16A—O5A—H4H | 110 (2) | C16A—C15A—H14A | 108.5 |
| K1—O1W—K2$^{ii}$ | 86.33 (7) | H13A—C15A—H14A | 107.5 |
| K1—O1W—H1W | 107 (2) | O5—C16—C15 | 113.0 (3) |

| | | | |
|---|---|---|---|
| K2$^{ii}$—O1W—H1W | 114 (2) | O5—C16—H15 | 109.0 |
| K1—O1W—H2W | 143 (2) | C15—C16—H15 | 109.0 |
| K2$^{ii}$—O1W—H2W | 103 (2) | O5—C16—H16 | 109.0 |
| H1W—O1W—H2W | 102 (2) | C15—C16—H16 | 109.0 |
| K1$^{i}$—O2W—K2$^{i}$ | 103.67 (8) | H15—C16—H16 | 107.8 |
| K1$^{i}$—O2W—H3W | 88 (3) | O5A—C16A—C15A | 110.0 (3) |
| K2$^{i}$—O2W—H3W | 117 (2) | O5A—C16A—H16A | 109.7 |
| K1$^{i}$—O2W—H4W | 114 (3) | C15A—C16A—H16A | 109.7 |
| K2$^{i}$—O2W—H4W | 125 (2) | O5A—C16A—H15A | 109.7 |
| H3W—O2W—H4W | 103 (2) | C15A—C16A—H15A | 109.7 |
| K1$^{viii}$—O3W—H5W | 117 (2) | H16A—C16A—H15A | 108.2 |

| | | | |
|---|---|---|---|
| O2—S1—O1—K2 | −4.66 (15) | C13A—N3A—C10A—C11A | 167.9 (7) |
| O3—S1—O1—K2 | −133.81 (12) | N3—C10—C11—C12 | 179.2 (3) |
| C1—S1—O1—K2 | 109.99 (14) | C9—C10—C11—C12 | −0.3 (5) |
| O3A—S1A—O1A—K1$^{ii}$ | −117.96 (19) | C10—C11—C12—C7 | 0.9 (6) |
| O2A—S1A—O1A—K1$^{ii}$ | 9.6 (2) | C8—C7—C12—C11 | −2.0 (5) |
| C1A—S1A—O1A—K1$^{ii}$ | 124.3 (7) | N2—C7—C12—C11 | −179.1 (3) |
| C1B—S1A—O1A—K1$^{ii}$ | 124.3 (13) | C6A—C1A—C2A—C3A | 5.0 (8) |
| K1—S1A—O1A—K1$^{ii}$ | −51.1 (2) | S1A—C1A—C2A—C3A | −171.1 (14) |
| K2—S1A—O1A—K1$^{ii}$ | −99.11 (19) | C1A—C2A—C3A—C4A | 0.8 (8) |
| O3A—S1A—O1A—K2 | −18.85 (16) | C2A—C3A—C4A—C5A | −5.6 (9) |
| O2A—S1A—O1A—K2 | 108.73 (12) | C2A—C3A—C4A—N1A | 173.0 (9) |
| C1A—S1A—O1A—K2 | −136.6 (7) | N2A—N1A—C4A—C3A | 1.9 (10) |
| C1B—S1A—O1A—K2 | −136.6 (13) | N2A—N1A—C4A—C5A | −179.4 (6) |
| K1—S1A—O1A—K2 | 48.00 (13) | C3A—C4A—C5A—C6A | 4.6 (9) |
| O3—S1—O2—K2 | 132.16 (13) | N1A—C4A—C5A—C6A | −174.1 (9) |
| O1—S1—O2—K2 | 4.21 (13) | C4A—C5A—C6A—C1A | 1.2 (8) |
| C1—S1—O2—K2 | −110.04 (13) | C2A—C1A—C6A—C5A | −6.1 (8) |
| O1A—S1A—O2A—K1 | −111.70 (12) | S1A—C1A—C6A—C5A | 170.0 (14) |
| O3A—S1A—O2A—K1 | 16.21 (15) | N1A—N2A—C7A—C8A | −170.3 (7) |
| C1A—S1A—O2A—K1 | 130.0 (4) | N1A—N2A—C7A—C12A | 10.5 (10) |
| C1B—S1A—O2A—K1 | 136.9 (9) | C12A—C7A—C8A—C9A | 0.5 (9) |
| K2—S1A—O2A—K1 | −50.93 (11) | N2A—C7A—C8A—C9A | −178.8 (9) |
| O1A—S1A—O2A—K2$^{ii}$ | −9.3 (3) | C7A—C8A—C9A—C10A | 1.8 (9) |
| O3A—S1A—O2A—K2$^{ii}$ | 118.7 (2) | N3A—C10A—C9A—C8A | 175.8 (17) |
| C1A—S1A—O2A—K2$^{ii}$ | −127.5 (5) | C11A—C10A—C9A—C8A | −3.7 (8) |
| C1B—S1A—O2A—K2$^{ii}$ | −120.7 (9) | N3A—C10A—C11A—C12A | −175.8 (16) |
| K1—S1A—O2A—K2$^{ii}$ | 102.4 (2) | C9A—C10A—C11A—C12A | 3.7 (8) |
| K2—S1A—O2A—K2$^{ii}$ | 51.5 (3) | C10A—C11A—C12A—C7A | −1.6 (9) |
| O1A—S1A—O3A—K1 | 112.78 (13) | C8A—C7A—C12A—C11A | −0.6 (9) |
| O2A—S1A—O3A—K1 | −15.23 (14) | N2A—C7A—C12A—C11A | 178.6 (9) |
| C1A—S1A—O3A—K1 | −127.4 (6) | O1A—S1A—C1B—C2B | −32.3 (17) |
| C1B—S1A—O3A—K1 | −134.4 (12) | O3A—S1A—C1B—C2B | −151.8 (11) |
| K2—S1A—O3A—K1 | 95.15 (8) | O2A—S1A—C1B—C2B | 86.3 (15) |
| O1A—S1A—O3A—K2 | 17.63 (15) | K1—S1A—C1B—C2B | 141.8 (10) |
| O2A—S1A—O3A—K2 | −110.38 (12) | K2—S1A—C1B—C2B | −84.0 (19) |
| C1A—S1A—O3A—K2 | 137.4 (6) | O1A—S1A—C1B—C6B | 162.7 (14) |

| | |
|---|---|
| C1B—S1A—O3A—K2 | 130.5 (12) |
| K1—S1A—O3A—K2 | −95.15 (8) |
| C4—N1—N2—C7 | 176.5 (3) |
| C4A—N1A—N2A—C7A | −175.4 (6) |
| C7B—N2B—N1B—C4B | 179.9 (11) |
| O2—S1—C1—C6 | −146.5 (3) |
| O3—S1—C1—C6 | −23.9 (3) |
| O1—S1—C1—C6 | 95.8 (3) |
| K2—S1—C1—C6 | 149.9 (2) |
| O2—S1—C1—C2 | 40.2 (3) |
| O3—S1—C1—C2 | 162.8 (3) |
| O1—S1—C1—C2 | −77.5 (3) |
| K2—S1—C1—C2 | −23.4 (3) |
| O1A—S1A—C1A—C2A | −29.6 (10) |
| O3A—S1A—C1A—C2A | −151.2 (7) |
| O2A—S1A—C1A—C2A | 90.9 (8) |
| K1—S1A—C1A—C2A | 145.2 (6) |
| K2—S1A—C1A—C2A | −87.9 (10) |
| O1A—S1A—C1A—C6A | 154.3 (7) |
| O3A—S1A—C1A—C6A | 32.6 (10) |
| O2A—S1A—C1A—C6A | −85.3 (9) |
| K1—S1A—C1A—C6A | −30.9 (14) |
| K2—S1A—C1A—C6A | 96.0 (9) |
| C6—C1—C2—C3 | −2.2 (5) |
| S1—C1—C2—C3 | 171.0 (3) |
| C1—C2—C3—C4 | −1.2 (5) |
| C2—C3—C4—C5 | 4.7 (5) |
| C2—C3—C4—N1 | −175.3 (3) |
| N2—N1—C4—C5 | −169.2 (3) |
| N2—N1—C4—C3 | 10.8 (5) |
| C3—C4—C5—C6 | −4.9 (5) |
| N1—C4—C5—C6 | 175.1 (3) |
| C4—C5—C6—C1 | 1.5 (5) |
| C2—C1—C6—C5 | 2.1 (5) |
| S1—C1—C6—C5 | −171.1 (3) |
| N1—N2—C7—C8 | 172.8 (3) |
| N1—N2—C7—C12 | −9.9 (5) |
| C12—C7—C8—C9 | 2.5 (6) |
| N2—C7—C8—C9 | 179.9 (3) |
| C7—C8—C9—C10 | −2.0 (6) |
| C15—N3—C10—C9 | 174.8 (3) |
| C13—N3—C10—C9 | 2.0 (5) |
| C15—N3—C10—C11 | −4.7 (5) |
| C13—N3—C10—C11 | −177.5 (3) |
| C8—C9—C10—N3 | −178.6 (3) |
| C8—C9—C10—C11 | 0.9 (5) |
| C15A—N3A—C10A—C9A | −177.1 (6) |
| O3A—S1A—C1B—C6B | 43 (2) |
| O2A—S1A—C1B—C6B | −78.7 (17) |
| K1—S1A—C1B—C6B | −23 (3) |
| K2—S1A—C1B—C6B | 111.0 (14) |
| C6B—C1B—C2B—C3B | 0.0 |
| S1A—C1B—C2B—C3B | −165 (3) |
| C1B—C2B—C3B—C4B | 0.0 |
| C2B—C3B—C4B—C5B | 0.0 |
| C2B—C3B—C4B—N1B | 174.5 (17) |
| N2B—N1B—C4B—C5B | 9 (2) |
| N2B—N1B—C4B—C3B | −165.3 (14) |
| C3B—C4B—C5B—C6B | 0.0 |
| N1B—C4B—C5B—C6B | −174 (2) |
| C4B—C5B—C6B—C1B | 0.0 |
| C2B—C1B—C6B—C5B | 0.0 |
| S1A—C1B—C6B—C5B | 165 (3) |
| N1B—N2B—C7B—C8B | −177.2 (13) |
| N1B—N2B—C7B—C12B | 1.6 (19) |
| C12B—C7B—C8B—C9B | 0.0 |
| N2B—C7B—C8B—C9B | 178.9 (17) |
| C7B—C8B—C9B—C10B | 0.0 |
| C8B—C9B—C10B—C11B | 0.0 |
| C8B—C9B—C10B—N3A | 180 (3) |
| C15A—N3A—C10B—C9B | 4 (3) |
| C13A—N3A—C10B—C9B | 165.0 (9) |
| C15A—N3A—C10B—C11B | −176.5 (10) |
| C13A—N3A—C10B—C11B | −15 (3) |
| C9B—C10B—C11B—C12B | 0.0 |
| N3A—C10B—C11B—C12B | −180 (3) |
| C10B—C11B—C12B—C7B | 0.0 |
| C8B—C7B—C12B—C11B | 0.0 |
| N2B—C7B—C12B—C11B | −178.8 (19) |
| C10—N3—C13—C14 | −78.6 (4) |
| C15—N3—C13—C14 | 108.5 (4) |
| C10A—N3A—C13A—C14A | 92.9 (10) |
| C10B—N3A—C13A—C14A | 99 (2) |
| C15A—N3A—C13A—C14A | −100.6 (4) |
| N3—C13—C14—O4 | −70.5 (4) |
| K1$^{vi}$—O4A—C14A—C13A | 176.5 (2) |
| N3A—C13A—C14A—O4A | 72.4 (4) |
| C10—N3—C15—C16 | 84.7 (4) |
| C13—N3—C15—C16 | −102.3 (4) |
| C10A—N3A—C15A—C16A | −79.5 (11) |
| C10B—N3A—C15A—C16A | −81.8 (19) |
| C13A—N3A—C15A—C16A | 115.0 (3) |
| K2$^{vii}$—O5—C16—C15 | 67.5 (4) |
| N3—C15—C16—O5 | 79.4 (4) |

| | | | |
|---|---|---|---|
| C13A—N3A—C10A—C9A | −11.6 (14) | N3A—C15A—C16A—O5A | −67.6 (4) |
| C15A—N3A—C10A—C11A | 2.4 (16) | | |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H1*H*···O4*W* | 0.87 (1) | 1.84 (1) | 2.703 (4) | 171 (4) |
| O5—H2*H*···O4 | 0.88 (1) | 1.85 (1) | 2.719 (3) | 173 (4) |
| O4*A*—H3*H*···O5*A* | 0.87 (1) | 1.86 (2) | 2.695 (3) | 161 (3) |
| O5*A*—H4*H*···O3*W* | 0.88 (1) | 1.93 (2) | 2.738 (3) | 154 (3) |
| O1*W*—H1*W*···O5*A*$^{iii}$ | 0.87 (1) | 2.22 (2) | 3.017 (3) | 151 (3) |
| O1*W*—H2*W*···O4$^{iv}$ | 0.87 (1) | 2.08 (1) | 2.906 (3) | 158 (3) |
| O2*W*—H3*W*···O5$^{v}$ | 0.87 (1) | 1.93 (1) | 2.791 (4) | 170 (4) |
| O2*W*—H4*W*···O2 | 0.87 (1) | 1.99 (2) | 2.795 (3) | 153 (3) |
| O3*W*—H5*W*···O3$^{ix}$ | 0.87 (1) | 1.93 (1) | 2.786 (3) | 167 (3) |
| O3*W*—H6*W*···O4*W*$^{x}$ | 0.88 (1) | 1.99 (1) | 2.821 (4) | 158 (3) |
| O4*W*—H7*W*···O4*A*$^{xi}$ | 0.88 (1) | 1.94 (1) | 2.789 (3) | 163 (3) |
| O4*W*—H8*W*···O1$^{ix}$ | 0.88 (1) | 1.86 (1) | 2.738 (3) | 180 (4) |

Symmetry codes: (iii) *x*+1/2, −*y*+3/2, *z*−1/2; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) *x*+1/2, −*y*+1/2, *z*−1/2; (ix) −*x*+3/2, *y*+1/2, −*z*+1/2; (x) −*x*+1, −*y*+1, −*z*+1; (xi) *x*+1, *y*, *z*.

## Poly[(3-{2-(4-hydroxyphenyl)diazen-1-yl]benzenesulfonato)potassium] (K5)

*Crystal data*

[K($C_{12}H_9N_2O_4S$)]
$M_r$ = 316.37
Orthorhombic, *Pccn*
*a* = 12.5535 (2) Å
*b* = 27.9698 (5) Å
*c* = 6.9982 (1) Å
*V* = 2457.20 (7) Å$^3$
*Z* = 8
*F*(000) = 1296
$D_x$ = 1.710 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3222 reflections
*θ* = 1.0–27.5°
*μ* = 0.62 mm$^{-1}$
*T* = 123 K
Needle, yellow
0.70 × 0.08 × 0.04 mm

*Data collection*

Enraf–Nonius KappaCCD diffractometer
Radiation source: sealed tube
*ω* and phi scans
5174 measured reflections
2802 independent reflections
1971 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.056
$\theta_{max}$ = 27.4°, $\theta_{min}$ = 1.5°
*h* = −16→16
*k* = −35→36
*l* = −9→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.092
*S* = 1.05
2802 reflections
185 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2 + 2.2145P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| K1 | 0.65356 (5) | 0.50424 (2) | 0.18594 (8) | 0.01900 (15) |
| S1 | 0.42945 (5) | 0.44186 (2) | 0.33020 (9) | 0.01226 (15) |
| O1 | 0.53205 (14) | 0.44178 (6) | 0.4257 (3) | 0.0221 (4) |
| O2 | 0.42970 (16) | 0.47067 (6) | 0.1573 (2) | 0.0232 (5) |
| O3 | 0.34195 (15) | 0.45457 (6) | 0.4572 (3) | 0.0219 (4) |
| O4 | 0.75203 (15) | 0.06592 (6) | 0.4630 (3) | 0.0176 (4) |
| N1 | 0.52435 (16) | 0.26259 (7) | 0.3155 (3) | 0.0151 (5) |
| N2 | 0.49261 (17) | 0.22095 (7) | 0.2784 (3) | 0.0151 (5) |
| C1 | 0.3142 (2) | 0.37266 (9) | 0.1500 (4) | 0.0154 (6) |
| H1 | 0.268119 | 0.397996 | 0.113452 | 0.018* |
| C2 | 0.4042 (2) | 0.38193 (8) | 0.2578 (3) | 0.0119 (5) |
| C3 | 0.4732 (2) | 0.34535 (8) | 0.3090 (4) | 0.0138 (5) |
| H3 | 0.535957 | 0.352124 | 0.379793 | 0.017* |
| C4 | 0.4493 (2) | 0.29836 (8) | 0.2554 (4) | 0.0128 (5) |
| C5 | 0.3585 (2) | 0.28883 (9) | 0.1480 (4) | 0.0149 (6) |
| H5 | 0.342403 | 0.256992 | 0.110563 | 0.018* |
| C6 | 0.2919 (2) | 0.32598 (9) | 0.0959 (4) | 0.0169 (6) |
| H6 | 0.229995 | 0.319477 | 0.022130 | 0.020* |
| C7 | 0.5637 (2) | 0.18368 (8) | 0.3358 (4) | 0.0144 (5) |
| C8 | 0.5289 (2) | 0.13728 (9) | 0.2979 (4) | 0.0158 (6) |
| H8 | 0.461027 | 0.132318 | 0.241007 | 0.019* |
| C9 | 0.59238 (19) | 0.09843 (9) | 0.3426 (4) | 0.0152 (5) |
| H9 | 0.568711 | 0.066853 | 0.316210 | 0.018* |
| C10 | 0.6911 (2) | 0.10615 (8) | 0.4263 (4) | 0.0137 (5) |
| C11 | 0.7258 (2) | 0.15214 (9) | 0.4697 (4) | 0.0154 (6) |
| H11 | 0.792806 | 0.156931 | 0.529763 | 0.019* |
| C12 | 0.6618 (2) | 0.19088 (9) | 0.4247 (4) | 0.0158 (6) |
| H12 | 0.684753 | 0.222373 | 0.454440 | 0.019* |
| H1H | 0.8159 (13) | 0.0744 (12) | 0.495 (5) | 0.055 (12)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| K1 | 0.0221 (3) | 0.0181 (3) | 0.0168 (3) | −0.0013 (2) | 0.0001 (3) | 0.0007 (2) |
| S1 | 0.0124 (3) | 0.0102 (3) | 0.0142 (3) | −0.0002 (2) | −0.0006 (3) | 0.0008 (3) |
| O1 | 0.0169 (10) | 0.0165 (9) | 0.0330 (11) | −0.0001 (8) | −0.0122 (9) | −0.0002 (9) |
| O2 | 0.0394 (12) | 0.0155 (9) | 0.0146 (10) | −0.0021 (9) | −0.0034 (9) | 0.0049 (8) |
| O3 | 0.0197 (11) | 0.0184 (9) | 0.0278 (11) | −0.0036 (8) | 0.0121 (9) | −0.0083 (8) |
| O4 | 0.0144 (10) | 0.0147 (8) | 0.0238 (10) | 0.0036 (8) | −0.0036 (9) | 0.0008 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.0159 (11) | 0.0137 (10) | 0.0158 (11) | 0.0004 (8) | 0.0033 (10) | −0.0003 (9) |
| N2 | 0.0168 (12) | 0.0124 (10) | 0.0161 (12) | 0.0016 (9) | 0.0019 (9) | −0.0006 (9) |
| C1 | 0.0146 (14) | 0.0151 (12) | 0.0165 (14) | 0.0018 (10) | −0.0010 (11) | 0.0005 (11) |
| C2 | 0.0134 (13) | 0.0114 (11) | 0.0109 (12) | −0.0015 (10) | 0.0014 (10) | −0.0005 (10) |
| C3 | 0.0124 (13) | 0.0157 (12) | 0.0134 (13) | −0.0022 (10) | 0.0013 (11) | −0.0004 (11) |
| C4 | 0.0121 (13) | 0.0134 (12) | 0.0130 (12) | 0.0024 (10) | 0.0040 (10) | 0.0021 (10) |
| C5 | 0.0155 (14) | 0.0130 (11) | 0.0161 (14) | −0.0015 (10) | 0.0009 (11) | −0.0022 (10) |
| C6 | 0.0149 (14) | 0.0203 (13) | 0.0154 (14) | −0.0016 (11) | −0.0036 (12) | −0.0008 (11) |
| C7 | 0.0156 (13) | 0.0137 (12) | 0.0138 (13) | 0.0020 (10) | 0.0025 (11) | 0.0016 (11) |
| C8 | 0.0125 (13) | 0.0185 (13) | 0.0164 (14) | −0.0019 (10) | 0.0016 (11) | 0.0005 (11) |
| C9 | 0.0162 (14) | 0.0128 (12) | 0.0164 (13) | −0.0005 (10) | 0.0034 (11) | 0.0000 (11) |
| C10 | 0.0135 (13) | 0.0124 (12) | 0.0153 (13) | 0.0023 (10) | 0.0025 (11) | 0.0007 (10) |
| C11 | 0.0115 (14) | 0.0171 (12) | 0.0177 (14) | −0.0005 (10) | −0.0022 (11) | 0.0003 (11) |
| C12 | 0.0165 (14) | 0.0113 (11) | 0.0197 (14) | −0.0017 (10) | 0.0010 (12) | −0.0015 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| K1—O2$^{i}$ | 2.7117 (18) | C1—C2 | 1.383 (3) |
| K1—O3$^{ii}$ | 2.7511 (18) | C1—C6 | 1.388 (3) |
| K1—O4$^{iii}$ | 2.7954 (19) | C1—H1 | 0.9500 |
| K1—O3$^{iv}$ | 2.815 (2) | C2—C3 | 1.387 (3) |
| K1—O1 | 2.863 (2) | C3—C4 | 1.399 (3) |
| K1—O2 | 2.970 (2) | C3—H3 | 0.9500 |
| K1—O4$^{v}$ | 3.0023 (19) | C4—C5 | 1.391 (3) |
| K1—S1 | 3.4611 (8) | C5—C6 | 1.383 (3) |
| K1—S1$^{iv}$ | 3.7788 (9) | C5—H5 | 0.9500 |
| K1—K1$^{vi}$ | 4.2551 (7) | C6—H6 | 0.9500 |
| K1—K1$^{vii}$ | 4.2551 (7) | C7—C12 | 1.394 (3) |
| K1—K1$^{i}$ | 4.6577 (12) | C7—C8 | 1.395 (3) |
| S1—O1 | 1.4510 (18) | C8—C9 | 1.384 (3) |
| S1—O2 | 1.4539 (18) | C8—H8 | 0.9500 |
| S1—O3 | 1.4568 (18) | C9—C10 | 1.387 (3) |
| S1—C2 | 1.779 (2) | C9—H9 | 0.9500 |
| O4—C10 | 1.385 (3) | C10—C11 | 1.392 (3) |
| O4—H1H | 0.866 (10) | C11—C12 | 1.385 (3) |
| N1—N2 | 1.258 (3) | C11—H11 | 0.9500 |
| N1—C4 | 1.437 (3) | C12—H12 | 0.9500 |
| N2—C7 | 1.430 (3) | | |

| | | | |
|---|---|---|---|
| O2$^{i}$—K1—O3$^{ii}$ | 134.72 (6) | O1—S1—O2 | 112.48 (12) |
| O2$^{i}$—K1—O4$^{iii}$ | 81.82 (6) | O1—S1—O3 | 112.88 (11) |
| O3$^{ii}$—K1—O4$^{iii}$ | 142.35 (6) | O2—S1—O3 | 111.97 (11) |
| O2$^{i}$—K1—O3$^{iv}$ | 84.43 (6) | O1—S1—C2 | 106.71 (11) |
| O3$^{ii}$—K1—O3$^{iv}$ | 97.73 (5) | O2—S1—C2 | 106.57 (11) |
| O4$^{iii}$—K1—O3$^{iv}$ | 73.57 (5) | O3—S1—C2 | 105.63 (11) |
| O2$^{i}$—K1—O1 | 118.14 (6) | O1—S1—K1 | 54.10 (8) |
| O3$^{ii}$—K1—O1 | 74.59 (5) | O2—S1—K1 | 58.40 (8) |
| O4$^{iii}$—K1—O1 | 97.72 (5) | O3—S1—K1 | 131.90 (7) |

| | | | |
|---|---|---|---|
| O3$^{iv}$—K1—O1 | 154.96 (5) | C2—S1—K1 | 122.44 (8) |
| O2$^{i}$—K1—O2 | 69.95 (6) | O1—S1—K1$^{viii}$ | 145.89 (8) |
| O3$^{ii}$—K1—O2 | 102.30 (5) | O2—S1—K1$^{viii}$ | 75.88 (8) |
| O4$^{iii}$—K1—O2 | 99.13 (5) | O3—S1—K1$^{viii}$ | 39.63 (8) |
| O3$^{iv}$—K1—O2 | 154.22 (5) | C2—S1—K1$^{viii}$ | 101.78 (8) |
| O1—K1—O2 | 48.88 (5) | K1—S1—K1$^{viii}$ | 122.356 (16) |
| O2$^{i}$—K1—O4$^{v}$ | 153.28 (5) | S1—O1—K1 | 101.66 (9) |
| O3$^{ii}$—K1—O4$^{v}$ | 71.30 (5) | S1—O2—K1$^{i}$ | 151.62 (11) |
| O4$^{iii}$—K1—O4$^{v}$ | 74.16 (4) | S1—O2—K1 | 96.95 (9) |
| O3$^{iv}$—K1—O4$^{v}$ | 99.53 (5) | K1$^{i}$—O2—K1 | 110.05 (6) |
| O1—K1—O4$^{v}$ | 55.46 (5) | S1—O3—K1$^{ii}$ | 129.83 (11) |
| O2—K1—O4$^{v}$ | 102.15 (5) | S1—O3—K1$^{viii}$ | 121.09 (10) |
| O2$^{i}$—K1—S1 | 94.33 (4) | K1$^{ii}$—O3—K1$^{viii}$ | 99.72 (6) |
| O3$^{ii}$—K1—S1 | 87.87 (4) | C10—O4—K1$^{ix}$ | 115.43 (15) |
| O4$^{iii}$—K1—S1 | 99.69 (4) | C10—O4—K1$^{v}$ | 129.03 (15) |
| O3$^{iv}$—K1—S1 | 173.25 (4) | K1$^{ix}$—O4—K1$^{v}$ | 94.37 (5) |
| O1—K1—S1 | 24.24 (4) | C10—O4—H1H | 110 (2) |
| O2—K1—S1 | 24.64 (3) | K1$^{ix}$—O4—H1H | 117 (2) |
| O4$^{v}$—K1—S1 | 78.64 (4) | K1$^{v}$—O4—H1H | 89 (2) |
| O2$^{i}$—K1—S1$^{iv}$ | 102.92 (4) | N2—N1—C4 | 112.1 (2) |
| O3$^{ii}$—K1—S1$^{iv}$ | 80.87 (4) | N1—N2—C7 | 114.8 (2) |
| O4$^{iii}$—K1—S1$^{iv}$ | 81.84 (4) | C2—C1—C6 | 119.4 (2) |
| O3$^{iv}$—K1—S1$^{iv}$ | 19.28 (4) | C2—C1—H1 | 120.3 |
| O1—K1—S1$^{iv}$ | 138.53 (4) | C6—C1—H1 | 120.3 |
| O2—K1—S1$^{iv}$ | 172.48 (4) | C1—C2—C3 | 120.8 (2) |
| O4$^{v}$—K1—S1$^{iv}$ | 85.31 (4) | C1—C2—S1 | 118.51 (19) |
| S1—K1—S1$^{iv}$ | 162.711 (19) | C3—C2—S1 | 120.68 (19) |
| O2$^{i}$—K1—K1$^{vi}$ | 59.40 (4) | C2—C3—C4 | 119.3 (2) |
| O3$^{ii}$—K1—K1$^{vi}$ | 137.30 (4) | C2—C3—H3 | 120.3 |
| O4$^{iii}$—K1—K1$^{vi}$ | 44.71 (4) | C4—C3—H3 | 120.3 |
| O3$^{iv}$—K1—K1$^{vi}$ | 39.59 (4) | C5—C4—C3 | 120.0 (2) |
| O1—K1—K1$^{vi}$ | 141.74 (4) | C5—C4—N1 | 124.2 (2) |
| O2—K1—K1$^{vi}$ | 118.87 (4) | C3—C4—N1 | 115.8 (2) |
| O4$^{v}$—K1—K1$^{vi}$ | 107.84 (4) | C6—C5—C4 | 119.6 (2) |
| S1—K1—K1$^{vi}$ | 134.620 (17) | C6—C5—H5 | 120.2 |
| S1$^{iv}$—K1—K1$^{vi}$ | 56.903 (18) | C4—C5—H5 | 120.2 |
| O2$^{i}$—K1—K1$^{vii}$ | 161.38 (4) | C5—C6—C1 | 120.8 (2) |
| O3$^{ii}$—K1—K1$^{vii}$ | 40.69 (4) | C5—C6—H6 | 119.6 |
| O4$^{iii}$—K1—K1$^{vii}$ | 101.97 (4) | C1—C6—H6 | 119.6 |
| O3$^{iv}$—K1—K1$^{vii}$ | 79.31 (4) | C12—C7—C8 | 119.8 (2) |
| O1—K1—K1$^{vii}$ | 79.70 (4) | C12—C7—N2 | 124.9 (2) |
| O2—K1—K1$^{vii}$ | 126.44 (4) | C8—C7—N2 | 115.4 (2) |
| O4$^{v}$—K1—K1$^{vii}$ | 40.92 (4) | C9—C8—C7 | 120.4 (2) |
| S1—K1—K1$^{vii}$ | 102.865 (15) | C9—C8—H8 | 119.8 |
| S1$^{iv}$—K1—K1$^{vii}$ | 60.202 (18) | C7—C8—H8 | 119.8 |
| K1$^{vi}$—K1—K1$^{vii}$ | 110.64 (3) | C8—C9—C10 | 119.2 (2) |
| O2$^{i}$—K1—K1$^{i}$ | 36.80 (4) | C8—C9—H9 | 120.4 |
| O3$^{ii}$—K1—K1$^{i}$ | 123.06 (5) | C10—C9—H9 | 120.4 |

| | | | |
|---|---|---|---|
| O4$^{iii}$—K1—K1$^{i}$ | 91.05 (4) | O4—C10—C9 | 116.4 (2) |
| O3$^{iv}$—K1—K1$^{i}$ | 121.17 (4) | O4—C10—C11 | 122.5 (2) |
| O1—K1—K1$^{i}$ | 81.68 (4) | C9—C10—C11 | 121.0 (2) |
| O2—K1—K1$^{i}$ | 33.16 (3) | C12—C11—C10 | 119.5 (2) |
| O4$^{v}$—K1—K1$^{i}$ | 130.86 (4) | C12—C11—H11 | 120.3 |
| S1—K1—K1$^{i}$ | 57.616 (16) | C10—C11—H11 | 120.3 |
| S1$^{iv}$—K1—K1$^{i}$ | 139.67 (2) | C11—C12—C7 | 120.1 (2) |
| K1$^{vi}$—K1—K1$^{i}$ | 90.662 (13) | C11—C12—H12 | 120.0 |
| K1$^{vii}$—K1—K1$^{i}$ | 158.51 (2) | C7—C12—H12 | 120.0 |
| | | | |
| O2—S1—O1—K1 | 1.78 (12) | O2—S1—C2—C3 | 125.4 (2) |
| O3—S1—O1—K1 | −126.11 (9) | O3—S1—C2—C3 | −115.4 (2) |
| C2—S1—O1—K1 | 118.29 (10) | K1—S1—C2—C3 | 62.7 (2) |
| K1$^{viii}$—S1—O1—K1 | −96.28 (13) | K1$^{viii}$—S1—C2—C3 | −156.05 (19) |
| O1—S1—O2—K1$^{i}$ | 160.5 (2) | C1—C2—C3—C4 | −1.9 (4) |
| O3—S1—O2—K1$^{i}$ | −71.1 (2) | S1—C2—C3—C4 | 177.85 (19) |
| C2—S1—O2—K1$^{i}$ | 43.9 (3) | C2—C3—C4—C5 | 1.5 (4) |
| K1—S1—O2—K1$^{i}$ | 162.2 (3) | C2—C3—C4—N1 | −179.5 (2) |
| K1$^{viii}$—S1—O2—K1$^{i}$ | −54.4 (2) | N2—N1—C4—C5 | −7.4 (3) |
| O1—S1—O2—K1 | −1.69 (11) | N2—N1—C4—C3 | 173.6 (2) |
| O3—S1—O2—K1 | 126.67 (9) | C3—C4—C5—C6 | −0.5 (4) |
| C2—S1—O2—K1 | −118.29 (10) | N1—C4—C5—C6 | −179.4 (2) |
| K1$^{viii}$—S1—O2—K1 | 143.38 (6) | C4—C5—C6—C1 | −0.2 (4) |
| O1—S1—O3—K1$^{ii}$ | 14.64 (16) | C2—C1—C6—C5 | −0.1 (4) |
| O2—S1—O3—K1$^{ii}$ | −113.50 (13) | N1—N2—C7—C12 | −0.6 (4) |
| C2—S1—O3—K1$^{ii}$ | 130.89 (13) | N1—N2—C7—C8 | 179.1 (2) |
| K1—S1—O3—K1$^{ii}$ | −46.90 (17) | C12—C7—C8—C9 | −2.0 (4) |
| K1$^{viii}$—S1—O3—K1$^{ii}$ | −139.42 (18) | N2—C7—C8—C9 | 178.3 (2) |
| O1—S1—O3—K1$^{viii}$ | 154.06 (10) | C7—C8—C9—C10 | 0.3 (4) |
| O2—S1—O3—K1$^{viii}$ | 25.92 (14) | K1$^{ix}$—O4—C10—C9 | −55.4 (3) |
| C2—S1—O3—K1$^{viii}$ | −89.69 (12) | K1$^{v}$—O4—C10—C9 | 64.2 (3) |
| K1—S1—O3—K1$^{viii}$ | 92.52 (11) | K1$^{ix}$—O4—C10—C11 | 124.8 (2) |
| C4—N1—N2—C7 | −179.7 (2) | K1$^{v}$—O4—C10—C11 | −115.6 (2) |
| C6—C1—C2—C3 | 1.2 (4) | C8—C9—C10—O4 | −178.3 (2) |
| C6—C1—C2—S1 | −178.5 (2) | C8—C9—C10—C11 | 1.5 (4) |
| O1—S1—C2—C1 | −175.3 (2) | O4—C10—C11—C12 | 178.4 (2) |
| O2—S1—C2—C1 | −54.9 (2) | C9—C10—C11—C12 | −1.5 (4) |
| O3—S1—C2—C1 | 64.3 (2) | C10—C11—C12—C7 | −0.3 (4) |
| K1—S1—C2—C1 | −117.61 (19) | C8—C7—C12—C11 | 2.0 (4) |
| K1$^{viii}$—S1—C2—C1 | 23.7 (2) | N2—C7—C12—C11 | −178.3 (2) |
| O1—S1—C2—C3 | 5.0 (2) | | |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O4—H1*H*···O1$^{v}$ | 0.87 (1) | 2.02 (3) | 2.732 (3) | 139 (3) |

Symmetry code: (v) −*x*+3/2, −*y*+1/2, *z*.