Single-crystal structure analysis of non-deuterated triglycine sulfate by neutron diffraction at 20 and 298 K: a new disorder model for the 298 K structure

Yukana Terasawa,a Takashi Ohhara,b Sota Sato,c Satoshi Yoshida,d and Toru Asahi,e,*

*aSchool of Advanced Science and Engineering, Waseda University, 2-2 Wakamatsu-cho, Shinjuku-ku, Tokyo, 162-8480, Japan, bJ-PARC Center, Japan Atomic Energy Agency, Shirakata 2-4, Tokai, Ibaraki, 319-1195, Japan, cIntegrated Molecular Structure Analysis Laboratory, Department of Applied Chemistry, School of Engineering, The University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo, 113-8656, Japan, dDepartment of Applied Chemistry, School of Engineering, The University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo, 113-8656, Japan, eFaculty of Science and Engineering, Waseda University, 2-2 Wakamatsu-cho, Shinjuku-ku, Tokyo, 162-8480, Japan, and fResearch Organization for Nano & Life Innovation, Waseda University, 513 Wasedatsurumaki-cho, Shinjuku-ku, Tokyo, 162-0041, Japan. *Correspondence e-mail: tasahi@waseda.jp

Precise single-crystal structure analyses of the title compound, bis(glycinium) sulfate–glycine (1/1), 2C2H6NO2+·C2H5NO2–·SO42– (or C6H17N3O10S), non-deuterated triglycine sulfate (HTGS) at 20 K and 298 K were undertaken using time-of-flight neutron diffraction data. At 20 K for the O–H bond between the glycinium cation and the zwitterionic, unprotonated glycine molecule that is associated with the ferroelectric behaviour of HTGS, O–H = 1.070 (3), H/C1/C1/C1 O = 1.408 (3) [C14 = 0.338 (4)], O/C1/C1/C1 O = 2.4777 (15) Å and O–H/C1/C1/C1 O = 179.0 (4)°, which is in good agreement with previous studies. Two reasonable structures for the same three atoms were refined for the 298 K dataset. One is a single-minimum potential-energy model, with O–H = 1.090 (12), H/C1/C1/C1 O = 1.361 (12) [C14 = 0.271 (17)], O/C1/C1/C1 O = 2.450 (7) Å and O–H/C1/C1/C1 O = 179.2 (11)°, having the H atom with a large ellipticity along the bond path between the O atoms. The other is a double-minimum potential-energy model having two H atom sites with occupancies of 0.876 (8) and 0.124 (8): for the major occupancy component, O–H = 1.065 (12), H/C1/C1/C1 O = 1.387 (12), O/C1/C1/C1 O = 2.451 (7) Å and O–H/C1/C1/C1 O = 178.2 (11)° and for the minor component, O–H = 1.06 (4), H/C1/C1/C1 O = 1.41 (4), O/C1/C1/C1 O = 2.451 (7) Å and O–H/C1/C1/C1 O = 166 (2)°. These models did not show any significant differences in R factors. In addition, the unit-cell parameters and other structural parameters of HTGS did not show any major differences compared to those of partially deuterated TGS and fully deuterated TGS for both 20 K and 298 K.

1. Chemical context

Triglycine sulfate, 2(C2H4NO2)+·(C2H5NO2)·(SO4)2– (TGS), is a hydrogen-bond ferroelectric material (Matthias et al. 1956) exhibiting a second-order and order–disorder-type ferroelectric phase transition at a Curie temperature (Tc) of 322 K (Triebwasser, 1958). The TGS structure belongs to the point group C2h and the space group P21/m in the paraelectric phase and C2 and P21 in the ferroelectric phase, respectively (Wood & Holden, 1957). Because of its high pyroelectricity, TGS has long been used as a material for pyroelectric sensors. Therefore, determining the crystal structure of TGS is essential for understanding such physical properties.

The atomic coordinates, except for those of the hydrogen atoms, of TGS at room temperature were first determined using single-crystal X-ray diffraction (Hoshino et al., 1959).
The study assumed the presence of one neutral glycine molecule ($\text{C}_2\text{H}_5\text{NO}_2$) exhibiting a zwitterionic configuration, and two monoprotonated glycinium ions ($\text{C}_2\text{H}_6\text{NO}_2^+$), from the detailed analysis of the bond lengths and angles of the glycine molecules. The authors also proposed a hydrogen-bonding scheme and pointed out that the hydrogen atom that lies between the oxygen atom of the carboxyl group in the glycine III cation (GIII) and the O atom in the glycine II molecule (GII) plays a crucial role in the dipole reversal. Many structural studies on TGS have subsequently been conducted (see Database survey): most of them were X-ray diffraction studies, but some of them were neutron diffraction studies. The atomic coordinates of non-deuterated TGS (hereinafter, designated as HTGS in place of TGS), including those of the hydrogen atoms at room temperature, were first revealed using single-crystal neutron diffraction (Padmanabhan & Yadav, 1971) and the atomic arrangements including hydrogen atoms of the zwitterion and glycinium ions were directly observed. The neutron diffraction experiment revealed that the hydrogen atom forming the $\text{O}\cdots\text{H}^\cdot\cdot\cdot\text{O}$ hydrogen bond between the GIII and GII species was closer to the GIII O atom compared to that in GII. This result agreed with that obtained by Hoshino et al. (1959). The structure refinement of HTGS with an applied external electric field at 298 K revealed the placement of all the hydrogen atoms and the unambiguous definition of the hydrogen-bonding scheme in an ordered domain structure (Kay & Kleinberg, 1973).

Crystal-structure refinements of partially deuterated TGS (DTGS), where deuterium replaced the H atoms except for the hydrogen atoms of the methylene ($\text{CH}_2$) group in each glycine molecule and those in sulfuric acid molecules at 40 K and 180 K (Protas et al., 1997) showed that the refined structures were consistent with those of the HTGS reported by Kay & Kleinberg (1973). Protas et al. (1997) also observed that HTGS and DTGS in the ferroelectric phase had a consistent structure from 40 K to 298 K. The deuterium atom lying between GIII and GII was $\sim0.40$ Å closer to the O atom of the carboxyl group of GIII than that of GII at both temperatures. In contrast, the crystal-structure refinement of HTGS at room temperature showed positional disorder over two adjacent sites of the amino group in glycinium cation I (GI) (Choudhury & Chitra, 2008). However, this is not in agreement with the refined structure of HTGS reported by Padmanabhan & Yadav (1971) where the GI species was analysed as an ordered structure.

In the crystal structure of fully deuterated TGS (FDTGS), all the hydrogen atoms in the glycine molecules and sulfuric acid molecules are substituted by deuterium atoms; the crystal structures did not show major changes between 20 K and 295 K (Hudspheth et al., 2013). The unit-cell parameters of these FDTGS structures were consistent with those of HTGS (Kay & Kleinberg, 1973; Choudhury & Chitra, 2008) and DTGS (Protas et al., 1997).

Structural analysis of DTGS at 40 K and FDTGS at 20 K have been undertaken by Protas et al. (1997) and Hudspheth et al. (2013), respectively, as mentioned above. However, a precise structural analysis of HTGS including hydrogen atoms at low temperatures has not been reported. Furthermore, two different structures of HTGS at $\sim298$ K were reported: one is an ordered structure by Padmanabhan & Yadav (1971) and the other is a disordered structure by Choudhury & Chitra (2008).

With this motivation, in this study, single-crystal neutron diffraction of HTGS has been conducted at 20 K and 298 K in the ferroelectric phase. The single crystal neutron diffractometer SENJU (Ohhara et al. 2016) at the J-PARC facility, which enables us to measure multiple Bragg reflections with high efficiency at low temperatures by combining high-power neutron sources and a time-of-flight Laue diffraction method, has firstly determined the precise crystal structure of HTGS at 20 K under suppression of thermal vibrations, including the atomic coordinates of the hydrogen atoms. Furthermore, a new structural model of HTGS at 298 K is proposed in addition to the structural model reported previously.

2. Structural commentary

2.1. Structural Refinement of HTGS at 20 K

The refined structures at 20 K are shown in Figs. 1 and 2. It was confirmed that the GI $\text{C}_2\text{H}_6\text{NO}_2$ glycine molecule containing C15 exhibits the neutral zwitterion configuration,
Table 1
Lattice constants, interatomic distances and angles for HTGS, DTGS, and FDTGS at low temperatures.

|                      | This study | Protas et al. (1997) | Hudspeth et al. (2013) |
|----------------------|------------|----------------------|------------------------|
| Temperature (K)      | HTGS       | DTGS                 | FDTGS                 |
|                      | 20 (2)     | 40                   | 20 (2)                 |
| a (Å)                | 9.3946 (8) | 9.406 (5)            | 9.409 (2)              |
| b (Å)                | 12.5338 (11)| 12.614 (5)          | 12.558 (3)             |
| c (Å)                | 5.6630 (4) | 5.654 (5)            | 5.673 (1)              |
| β (Å)                | 110.500 (7)| 110.49 (2)           | 110.44 (2)             |
| V (Å³)               | 624.59 (9) | 628.4 (7)            | 628.2 (2)              |
| O15—O3 (Å)           | 2.4777 (15)| 2.486 (5)            | –                      |
| O15—H(D)15 (Å)       | 1.070 (3)  | 1.041 (5)            | –                      |
| H(D)15—O3 (Å)        | 1.408 (3)  | 1.445 (6)            | –                      |
| O15—H(D)15—O3 (°)    | 179.0 (4)  | 178.4 (6)            | –                      |

Symmetry code for HTGS in this study: (i) 3 – x, –1/2 + y, 2 – z.

...and the other two GI and GIII glycine moieties (C17 and C20, respectively) exist as monoprotonated C₂H₆NO₂⁺ glycinium ions. The most significant feature of these glycine/glycinium species are the N—C—C—O(H) torsion angles (Terasawa et al. 2021), viz.: 21.1 (1)° for N11—C17—C19—O7, 1.5 (1)° for N14—C15—C18—O10 and 1.4 (1)° for N21—C20—C16—O2. The sulfate ion shows its expected tetrahedral shape with bond distances of 1.480 (2) Å (S1—O4), 1.470 (2) Å (S1—O5), 1.477 (2) Å (S1—O6) and 1.472 (2) Å (S1—O8) and bond angles of 110.3 (1)° (O4—S1—O5), 107.9 (1)° (O4—S1—O6), 108.7 (1)° (O4—S1—O8), 109.7 (1)° (O5—S1—O6), 110.6 (1)° (O5—S1—O8) and 109.7 (1)° (O6—S1—O8). The slight differences among these distances and angles may arise because of the different hydrogen bonds accepted by these O atoms. Numerous N—H—O and O—H—O hydrogen bonds (see supporting information) are formed between the glycine or glycinium species and the sulfate ions; four N—H—O hydrogen bonds and one O—H—O hydrogen bond are formed by GI, five N—H—O hydrogen bonds are formed by GII and five N—H—O hydrogen bonds with the sulfate ion and one O—H—O hydrogen bond to the glycine molecule is formed by GIII.

2.2. Structural Refinement of HTGS at 298 K

The refined structures at 298 K are shown in Figs. 3, 4 and 5. The crystallographic symmetry, the contents of the asymmetric...
unit, and the features of the molecular structures are consistent with those for the 20 K structure apart the disordered N11/N11B amino group [refined site occupancies = 0.874 (8):0.126 (8)] in the GI cation and the O—H/C1/C1/C1/O association for GIII and GII. Two models were refined considering the H atom between O15 in GIII and O3 in GII. For one model (298 K model 1), the H15 atom was refined with a large ellipticity along the bond path between O15 and O3 as a single minimum potential energy structure [Fig. 5(a)]. A double-minimum potential-energy structure could be deduced because the distance between O15 and O3 did not increase with an increase in the temperature; thus for the other model (298 K model 2), a pair of hydrogen atoms were refined along the bond path between O15 and O3i, the double-minimum potential structure [Fig. 5(b)].

The key parameters for the O15—H15/C1/C1/C1O3i hydrogen bond at 298 K are summarized in Table 2. The residuals for models 1 and 2 (Table 3) are almost identical: model 2 has one more variable parameter than model 1 (358 compared to 357). For model 1, H15 is 0.271 (17) Å closer to O15 in GIII than O3i in GII. On the other hand, the distance between O15 and H15 [1.090 (12) Å] is almost the same as that at 20 K despite there being no distance restraint for the H15···O3i separation. Therefore, the mixed structure (model 2) of the major ferro-
electric phase and minor paraelectric phase is strongly suggested, because the occupancies of N11 and N11B and H15 and H3 are related by symmetry.

The unit-cell parameters and bond lengths for HTGS, DTGS, and FDTGS at 298 K are listed in Table 2. The lattice parameters did not show any major differences and this result shows good agreement with that previously reported for DTGS (Protas et al., 1997). We may conclude that the intermolecular distances and angles do not change significantly upon deuteration.

In the previous studies using single-crystal neutron diffraction, Kay & Kleinberg (1973) proposed an ordered structure of HTGS because the domains were oriented by applying an external electric field. Hudspeth & Goossens (2012) proposed an ordered structure for FDTGS because \( T_C \) for FDTGS increased by approximately 12 K compared to HTGS. Choudhury & Chitra (2008) proposed a disordered structure for the GI amino group with unequal occupancies of N11 (88%) and N11B (12%); this occupancy ratio is in excellent agreement with the results in this study. For the hydrogen atom between the oxygen atom of the carboxyl group in GI11 and that in the GI11, the O—O distance was 2.470 (9) Å, and the H atom was approximately 0.241 Å closer to the GI11 O atom than that in GI11. They concluded that the structure of HTGS at room temperature has a single minimum potential energy in the O—H···O hydrogen-bond path between GI111 and GI1. In this study, two reasonable structures were refined as a single-minimum potential-energy model and a double-minimum model without any significant differences. Therefore, we conclude that there is a significant possibility of a double-minimum potential-energy model for HTGS at 298 K.

3. Supramolecular features

Hydrogen bonds in the refined structures were consistent with those reported previously (see supporting information) and no additional intermolecular interactions were found. Therefore, the 20 K and 298 K structures form essentially the structural motif of a three-dimensional network of N—H···O and O—H···O hydrogen bonds between glycinium cations, glycine molecules and sulfate ions.

4. Database survey

The Cambridge Structural Database (Version 5.42, update of November 2020; Groom et al. 2016) was searched for structures of triglycine sulfate and it returned no fewer than 29 hits: six of these records are structures obtained using neutron diffraction. The lattice constants of these structures are consistent with those of this study. The ionic states of glycine and the sulfate ion for five structures obtained using single-crystal neutron data are consistent with those for this study in which one neutral, zwitterionic glycine molecule and two monoprotonated glycinium ions occur [CSD refcodes TGLYSU01 (Protas et al., 1997); TGLYSU02 (Padmanabhan & Yadav, 1971); TGLYSU03 (Protas et al., 1997); TGLYSU11 (Kay & Kleinberg, 1973); and TGLYS25 (Cheng et al., 1986)]. In contrast, hydrogen atoms were not assigned in some of the structures obtained using X-ray diffraction: refcodes TGLYSU (Hoshino et al., 1959); TGLYSU13 (Itoh & Mitsui, 1973); TGLYSU28 (Choudhury & Chitra, 2008); TGLYSU29 (Kawasaki et al., 2021) and TGLYSU30 (Kawasaki et al., 2021). Furthermore, in several structures, some hydrogen atoms are missing: refcodes TGLYSU04 (Fletcher et al., 1976); TGLYSU07 (Solans et al., 1985); TGLYSU15 (Itoh & Mitsui, 1973); TGLYSU21, TGLYSU22, TGLYSU23 (Kolontsova et al., 1990). In one structure, HSO₃⁻ ions were proposed to be present: refcode TGLYSU04 (Fletcher et al., 1976).

5. Synthesis and crystallization

The HTGS crystals were grown in an aqueous solution by the slow evaporation method at \( ~293 \) K. Glycine (13.06 g; FUJIFILM Wako Pure Chemical Corporation; purity \( \geq 99.0\% \)) and sulfuric acid (3.1 ml; FUJI FILM; molar ratio 3:1) was added to 50 ml of water in a 100 ml beaker. They were dissolved by heating at \( ~313 \) K with a 300 r.p.m. magnetic stirrer. After completely dissolving them, plastic films were double-wrapped around the beaker, and some holes were knocked in the films to evaporate the water slowly. The beaker was left to stand at \( ~293 \) K. HTGS was crystallized after approximately a month, and then the solution was filtered. The collected crystals were dried in a desiccator at \( ~293 \) K.

6. Refinement

Crystal data, data collection, and structural refinement details are summarized in Table 3. All data were collected using the single-crystal neutron diffractometer SENJU (Ohhara et al., 2016) at beamline BL18 of the Materials and Life Science Facility, Japan Proton Accelerator Research Complex. The crystal (colourless cube, \( ~2.8 \) mm edge length) mounted on an aluminum pin was cooled to 20 K in a closed-cycle helium cryostat. The crystal was surrounded by 41 two-dimensional scintillation area detectors during the data collection. The same crystal was used for the measurement at 298 K after warming to room temperature. Three-dimensional data of \( (x, y, \lambda) \) were measured in 16 different orientations for each dataset. The measurement time was 1.5 h for one orientation; the raw data were processed using STARGazer (Ohhara et al. 2009) to generate HKLF files and visualize \( (x, y) \) slice maps and merged TOF profiles.

\textit{SHELXL2018} (Sheldrick 2015b) was used for least-squares refinements with neutron scattering lengths (fm) of 2.847 (S), 5.805 (O), –3.741 (H), 9.360 (N) and 6.648 (C). A reported structure determined by single-crystal X-ray diffraction (Hoshino et al., 1959) was used as the initial structural model. All atoms, including hydrogen atoms, were refined with \( U_{ij} \) values. For the 298 K data, the refinement was initially performed without the hydrogen atom(s) between O15 and O3 to minimize the model dependence. A nuclear density distribution (Fig. 6) with a large ellipticity along the bond path between O15 and O3 was observed. One hydrogen atom was
assigned to this position and refined as a single-minimum potential-energy model (298 K model 1). In 298 K model 2, two hydrogen atoms (H15 and H3) with the restrictions listed below were included: (i) H15 and H3 were refined anisotropically and constrained to have the same displacement factors; (ii) O15 and H15 and O3 and H3 were restrained to have the same distances; (iii) the occupancies of H15 for H3 were linked to those of N11 and N11B.

Acknowledgements

The neutron diffraction measurements were conducted under the J-PARC MLF Fast Track Proposal No.2020BF1801.

Funding information

Funding for this research was provided by: Mitsubishi Materials Corporation (grant No. AXA30Z001000); Waseda University Grants for Special Research Projects (grant No. BARD01107201).

References

Cheng, Z., Cheng, Y., Zhang, P. & Yan, Q. (1986). Acta Phys. Sin. 35, 643–52.
Choudhury, R. R. & Chitra, R. (2008). Pramana – J. Phys. 71, 911–915.
Fletcher, S. R., Keve, E. T. & Skapski, A. C. (1976). Ferroelectrics, 14, 789–799.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). Acta Cryst. B72, 171–179.
Hoshino, S., Okaya, Y. & Pepinsky, R. (1959). Phys. Rev. 115, 323–330.
Hudspeth, J. M. & Goossens, D. J. (2012). J. Cryst. Growth, 338, 177–180.
Hudspeth, J. M., Goossens, D. J., Gutmann, M. J. & Studer, A. J. (2013). *Cryst. Res. Technol.* **48**, 169–180.

Itoh, K. & Mitsui, T. (1973). *Ferroelectrics* **5**, 235–251.

Kawasaki, T., Kaimori, Y., Shimada, S., Hara, N., Sato, S., Suzuki, K., Asahi, T., Matsumoto, A. & Soai, S. (2021). *Chem. Commun.* **57**, 5999–6002.

Kay, M. I. & Kleinberg, R. (1973). *Ferroelectrics* **5**, 45–52.

Kolontsova, E. V., Red’ko, S. V., Struchkov, Yu. T. & Yanovskii, A. I. (1990). *Sov. Phys. Crystallogr.* **35**, 126–129.

Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platting, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.

Matthias, B. T., Miller, C. E. & Remeika, J. P. (1956). *Phys. Rev.* **104**, 849–850.

Ohhara, T., Kiyotani, T., Noda, Y. & Arai, M. (2016). *J. Appl. Cryst.* **49**, 120–127.

Ohhara, T., Kusaka, K., Hosoya, T., Kurihara, K., Tomoyori, K., Niimura, N., Tanaka, I., Suzuki, J., Nakatani, T., Otomo, T., Matsuoka, S., Tomita, K., Nishimaki, Y., Ajima, T. & Ryufuku, S. (2009). *Nucl. Instrum. Methods Phys. Res. A* **600**, 195–197.

Padmanabhan, V. M. & Yadav, V. S. (1971). *Current Science* (Bangalore) **40**, 60–61.

Protas, J., Gerbaux, X., Hadni, A. & Schweiss, P. (1997). *Ferroelectrics*, **193**, 51–62.

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

Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.

Terasawa, Y., Kikuta, T., Ichiki, M., Sato, S., Ishikawa, K. & Asahi, T. (2021). *J. Phys. Chem. Solids*, **151**, 109890.

Triebwasser, S. (1958). *IBM J. Res. & Dev.* **2**, 212–217.

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

Wood, E. A. & Holden, A. N. (1957). *Acta Cryst.* **10**, 145–146.
supporting information

*Acta Cryst.* (2022). E78, 306-312  [https://doi.org/10.1107/S2056989022000858]

Single-crystal structure analysis of non-deuterated triglycine sulfate by neutron diffraction at 20 and 298 K: a new disorder model for the 298 K structure

**Yukana Terasawa, Takashi Ohhara, Sota Sato, Satoshi Yoshida and Toru Asahi**

Computing details

For all structures, data collection: *STARGazer* (Ohhara et al., 2009); cell refinement: *STARGazer* (Ohhara et al., 2009); data reduction: *STARGazer* (Ohhara et al., 2009); program(s) used to solve structure: A reported structure determined by single-crystal X-ray diffraction (Hoshino et al., 1959) was used as the initial structure model. Program(s) used to refine structure: *SHELXT2018/3* (Sheldrick, 2015) for (20K); *SHELXL2018/3* (Sheldrick, 2015) for 298KModel1, 298KModel2. For all structures, molecular graphics: *Mercury* (Macrae et al., 2020); software used to prepare material for publication: *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

**Bis(glycinium) sulfate–glycine (1/1) (20K)**

**Crystal data**

2C2H6NO2⁺·SO4²⁻·C2H5NO2

| Parameter | Value |
|-----------|-------|
| Mr        | 323.28 |
| Monoclinic, P2₁ |
| Hall symbol: P2yb |
| a         | 9.3946 (8) Å |
| b         | 12.5338 (11) Å |
| c         | 5.6630 (4) Å |
| β         | 110.500 (7)° |
| V         | 624.59 (9) Å³ |
| Z         | 2 |

**Data collection**

Time-of-flight Laue-type single crystal neutron diffractometer

| Parameter | Value |
|-----------|-------|
| Radiation source: spallation neutron |
| Detector resolution: 4 pixels mm⁻¹ |
| time–of–flight Laue method scans |
| 40510 measured reflections |
| 10169 independent reflections |

**Refinement**

Refinement on $F^2$

| Parameter | Value |
|-----------|-------|
| Least-squares matrix: full |
| $R[F^2 > 2σ(F^2)] = 0.073$ |
| $wR(F^2) = 0.193$ |
| $S = 1.04$ |
| 40510 reflections |
| 350 parameters |

1 restraint

All H-atom parameters refined

$w = 1/[σ(F_c)^2 + (0.1332P)^2 + 0.072P]$ where $P = (F_c^2 + 2F_s^2)/3$

$(Δ/σ)_{max} = 0.001$

$Δρ_{max} = 3.87 e Å^{-3}$

$Δρ_{min} = -6.28 e Å^{-3}$

$F(000) = 130.536$

$D_x = 1.719$ Mg m⁻³

Neutrons radiation, $λ = 1$ Å

Cell parameters from 8174 reflections

$θ = 6.6–83.3°$

$μ = 0.49$ mm⁻¹

$T = 20$ K

Block, colorless

$2.80 \times 2.80 \times 2.80$ mm

$R_{int} = N/A$

$θ_{max} = 86.1°$, $θ_{min} = 7.5°$

$h = -23→23$

$k = -31→31$

$l = -13→14$

33150 reflections with $I > 2σ(I)$

$Δρ_{max} = 3.87 e Å^{-3}$

$Δρ_{min} = -6.28 e Å^{-3}$
Extinction correction: SHELXL2018/3
(Sheldrick 2015)
Extinction coefficient: 0.119 (3)

Absolute structure: Indeterminate for a neutron structure

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. reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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

|    | x           | y           | z           | U(eq)       |
|----|-------------|-------------|-------------|-------------|
| S1 | 0.9987 (2)  | 0.25000 (15)| 0.2280 (4)  | 0.00149 (18)|
| O3 | 1.46271 (12)| 0.96537 (9) | 0.8104 (2)  | 0.00493 (13)|
| O2 | 1.20940 (13)| 0.49653 (10)| 0.7552 (2)  | 0.00564 (13)|
| O4 | 0.85445 (12)| 0.24777 (9) | 0.0101 (2)  | 0.00407 (11)|
| O5 | 0.96806 (13)| 0.25228 (9) | 0.4652 (2)  | 0.00438 (12)|
| O6 | 1.08235 (12)| 0.34725 (8) | 0.2072 (2)  | 0.00397 (12)|
| O7 | 1.39163 (12)| 0.73620 (10)| −0.0953 (2) | 0.00519 (13)|
| H7 | 1.2917 (3)  | 0.7379 (3)  | −0.0588 (6) | 0.0160 (4)  |
| O8 | 1.08828 (12)| 0.15498 (8) | 0.2172 (2)  | 0.00403 (12)|
| O9 | 1.50274 (14)| 0.72358 (11)| 0.3230 (2)  | 0.00628 (14)|
| O10| 1.22125 (13)| 0.98973 (10)| 0.7827 (2)  | 0.00528 (13)|
| O15| 1.44257 (13)| 0.50951 (11)| 0.7343 (2)  | 0.00643 (14)|
| H15| 1.4843 (4)  | 0.4899 (3)  | 0.9306 (6)  | 0.0162 (4)  |
| N11| 1.64288 (8) | 0.78825 (6) | −0.17333 (14)| 0.00468 (8)|
| H11A| 1.5930 (5) | 0.7383 (3)  | −0.3227 (7) | 0.0206 (5)  |
| H11B| 1.7487 (3) | 0.8093 (3)  | −0.1799 (7) | 0.0167 (4)  |
| H11C| 1.5753 (4) | 0.8567 (3)  | −0.2021 (6) | 0.0165 (4)  |
| N14| 1.10601 (8) | 0.92901 (5) | 0.29966 (13)| 0.00407 (8)|
| H14A| 1.0741 (4) | 0.9005 (3)  | 0.1183 (6)  | 0.0189 (5)  |
| H14B| 1.0637 (4) | 1.0044 (3)  | 0.2961 (8)  | 0.0202 (6)  |
| H14C| 1.0593 (4) | 0.8781 (3)  | 0.3977 (7)  | 0.0186 (5)  |
| N21| 1.07375 (7) | 0.57354 (5) | 0.28504 (13)| 0.00395 (8)|
| H21A| 1.0403 (4) | 0.6295 (3)  | 0.3907 (7)  | 0.0184 (5)  |
| H21B| 1.0241 (4) | 0.5017 (3)  | 0.2905 (7)  | 0.0194 (5)  |
| H21C| 1.0335 (4) | 0.6013 (3)  | 0.1022 (6)  | 0.0165 (4)  |
| C15| 1.27299 (11)| 0.92965 (8) | 0.42088 (19)| 0.00464 (11)|
| H15A| 1.3210 (5) | 0.9838 (4)  | 0.3185 (7)  | 0.0231 (7)  |
| H15B| 1.3161 (5) | 0.8502 (3)  | 0.4096 (8)  | 0.0227 (7)  |
| C16| 1.29570 (10)| 0.51955 (7) | 0.64503 (18)| 0.00353 (10)|
| C17| 1.65950 (10)| 0.73969 (8) | 0.07314 (19)| 0.00453 (11)|
| H17A| 1.7376 (4)| 0.7882 (3)  | 0.2221 (7)  | 0.0200 (6)  |
| H17B| 1.7072 (5) | 0.6594 (3)  | 0.0824 (9)  | 0.0217 (6)  |
| C18| 1.31907 (10)| 0.96459 (7) | 0.69381 (17)| 0.00311 (10)|
| C19| 1.50928 (10)| 0.73247 (7) | 0.11344 (18)| 0.00345 (10)|
### Atomic displacement parameters (Å²)

| Atom | U¹¹  | U²²  | U³³  | U¹²  | U¹³  | U²³  |
|------|------|------|------|------|------|------|
| S1   | 0.0013 (5) | 0.0009 (4) | 0.0022 (4) | 0.0000 (3) | 0.0005 (4) | 0.0000 (4) |
| O3   | 0.0027 (3) | 0.0063 (3) | 0.0046 (3) | 0.0002 (2) | −0.0001 (2) | −0.0012 (2) |
| O2   | 0.0042 (3) | 0.0077 (3) | 0.0049 (3) | −0.0005 (2) | 0.0015 (2) | 0.0020 (3) |
| O4   | 0.0021 (3) | 0.0053 (3) | 0.0038 (3) | 0.0001 (2) | −0.0003 (2) | 0.0001 (2) |
| O5   | 0.0062 (3) | 0.0039 (3) | 0.0037 (3) | −0.0001 (2) | 0.0026 (2) | 0.0001 (2) |
| O6   | 0.0038 (3) | 0.0021 (2) | 0.0063 (3) | −0.0010 (2) | 0.0022 (2) | 0.0001 (2) |
| O7   | 0.0028 (3) | 0.0081 (3) | 0.0042 (3) | −0.0004 (2) | 0.0007 (2) | 0.0004 (2) |
| H7   | 0.0104 (8) | 0.0207 (11) | 0.0168 (10) | 0.0000 (8) | 0.0047 (7) | −0.0004 (9) |
| O8   | 0.0040 (3) | 0.0022 (2) | 0.0058 (3) | 0.0013 (2) | 0.0015 (2) | −0.0002 (2) |
| O9   | 0.0054 (3) | 0.0094 (4) | 0.0039 (3) | −0.0011 (3) | 0.0016 (2) | 0.0003 (3) |
| O10  | 0.0035 (3) | 0.0072 (3) | 0.0050 (3) | 0.0005 (2) | 0.0014 (2) | −0.0020 (3) |
| O15  | 0.0036 (3) | 0.0101 (4) | 0.0052 (3) | 0.0019 (3) | 0.0012 (2) | 0.0022 (3) |
| H15  | 0.0140 (9) | 0.0174 (10) | 0.0166 (10) | 0.0008 (8) | 0.0044 (8) | 0.0015 (9) |
| N11  | 0.00353 (17) | 0.00565 (19) | 0.00521 (18) | 0.00037 (14) | 0.00198 (15) | 0.00040 (15) |
| H11A | 0.0232 (14) | 0.0202 (12) | 0.0149 (10) | −0.0037 (11) | 0.0025 (10) | −0.0057 (9) |
| H11B | 0.0118 (9) | 0.0202 (11) | 0.0201 (11) | −0.0011 (8) | 0.0082 (8) | 0.0021 (10) |
| H11C | 0.0179 (11) | 0.0148 (10) | 0.0179 (10) | 0.0056 (8) | 0.0076 (9) | 0.0044 (8) |
| N14  | 0.00371 (17) | 0.00394 (16) | 0.00353 (17) | −0.00007 (13) | −0.00001 (13) | −0.00041 (13) |
| H14A | 0.0205 (12) | 0.0239 (13) | 0.0097 (8) | −0.0023 (10) | 0.0022 (8) | −0.0053 (8) |
| H14B | 0.0198 (12) | 0.0125 (9) | 0.0243 (14) | 0.0061 (9) | 0.0027 (11) | −0.0024 (9) |
| H14C | 0.0163 (11) | 0.0227 (13) | 0.0179 (11) | −0.0053 (9) | 0.0072 (9) | 0.0042 (10) |
| N21  | 0.00371 (17) | 0.00376 (17) | 0.00370 (17) | −0.00009 (13) | 0.00047 (14) | 0.00026 (13) |
| H21A | 0.0174 (11) | 0.0204 (12) | 0.0178 (11) | 0.0057 (9) | 0.0068 (9) | −0.0048 (9) |
| H21B | 0.0191 (12) | 0.0146 (10) | 0.0218 (13) | −0.0075 (9) | 0.0038 (10) | 0.0036 (9) |
| H21C | 0.0176 (11) | 0.0198 (11) | 0.0103 (8) | 0.0022 (8) | 0.0028 (8) | 0.0044 (8) |
| C15  | 0.0034 (2) | 0.0064 (3) | 0.0038 (2) | −0.0001 (2) | 0.00083 (19) | −0.0014 (2) |
| H15A | 0.0211 (13) | 0.0320 (17) | 0.0173 (11) | −0.0101 (12) | 0.0081 (11) | 0.0032 (12) |
| H15B | 0.0194 (12) | 0.0198 (12) | 0.0240 (14) | 0.0089 (10) | 0.0014 (11) | −0.0090 (11) |
| C16  | 0.0029 (2) | 0.0038 (2) | 0.0037 (2) | 0.00049 (18) | 0.00095 (19) | 0.00052 (19) |
| C17  | 0.0026 (2) | 0.0055 (3) | 0.0053 (3) | 0.0030 (19) | 0.0011 (2) | 0.0011 (2) |
| H17A | 0.0146 (10) | 0.0279 (15) | 0.0150 (10) | −0.0087 (10) | 0.0019 (8) | −0.0037 (10) |
| H17B | 0.0212 (13) | 0.0136 (10) | 0.0333 (18) | 0.0086 (9) | 0.0131 (13) | 0.0081 (11) |
| C18  | 0.0022 (2) | 0.0031 (2) | 0.0033 (2) | 0.00014 (17) | 0.00010 (18) | −0.00052 (18) |
| C19  | 0.0028 (2) | 0.0036 (2) | 0.0038 (2) | −0.00029 (17) | 0.00102 (19) | 0.00007 (18) |
| C20  | 0.0038 (2) | 0.0052 (3) | 0.0040 (2) | 0.00017 (19) | 0.0014 (2) | 0.00104 (19) |
| H20A | 0.0187 (12) | 0.0167 (11) | 0.0230 (14) | −0.0069 (9) | 0.0028 (10) | 0.0060 (10) |
| H20B | 0.0221 (13) | 0.0288 (16) | 0.0150 (10) | 0.0096 (12) | 0.0081 (10) | −0.0033 (11) |
### Geometric parameters (Å, °)

| Bond/Centered Angle | Distance/Angle (Å/°) |
|---------------------|----------------------|
| S1—O5               | 1.470 (2)            |
| S1—O8               | 1.472 (2)            |
| S1—O6               | 1.477 (2)            |
| S1—O4               | 1.480 (2)            |
| O3—C18              | 1.2778 (14)          |
| O3—H15i             | 1.408 (4)            |
| O2—C16              | 1.2181 (15)          |
| O7—H7               | 1.030 (3)            |
| O7—C19              | 1.3063 (14)          |
| O9—C19              | 1.2149 (15)          |
| O10—C18             | 1.2333 (15)          |
| O15—H15             | 1.070 (4)            |
| O5—S1—O8            | 110.60 (14)          |
| O5—S1—O6            | 109.68 (14)          |
| O8—S1—O6            | 109.68 (13)          |
| O5—S1—O4            | 110.31 (13)          |
| O8—S1—O4            | 108.66 (14)          |
| O6—S1—O4            | 107.86 (13)          |
| C18—O3—H15i         | 117.66 (16)          |
| H7—O7—C19           | 111.1 (2)            |
| H15—O15—C16         | 112.3 (2)            |
| H11A—N11—H11B       | 107.0 (3)            |
| H11A—N11—H11C       | 107.2 (3)            |
| H11B—N11—H11C       | 108.7 (3)            |
| H11A—N11—C17        | 113.2 (2)            |
| H11B—N11—C17        | 110.6 (2)            |
| H11C—N11—C17        | 110.0 (2)            |
| H14B—N14—H14A       | 109.0 (3)            |
| H14B—N14—H14C       | 110.1 (3)            |
| H14A—N14—H14C       | 106.9 (3)            |
| H14B—N14—C15        | 110.3 (2)            |
| H14A—N14—C15        | 111.0 (2)            |
| H14C—N14—C15        | 109.5 (2)            |
| H21B—N21—H21C       | 108.6 (3)            |
| H21B—N21—H21A       | 110.1 (3)            |
| H21C—N21—H21A       | 105.8 (3)            |
| H21B—N21—C20        | 111.4 (2)            |
| H21C—N21—C20        | 111.5 (2)            |
| H21A—N21—C20        | 109.4 (2)            |

**Supporting Information**

*Acta Cryst. (2022). E78, 306-312*
Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|---------|
| O7—H7···O4ii | 1.030 (3) | 1.497 (3) | 2.5258 (17) | 176.3 (4) |
| O15—H15···O3iii | 1.070 (3) | 1.408 (3) | 2.4777 (15) | 179.0 (4) |
| N11—H11A···O9iv | 1.024 (4) | 1.894 (4) | 2.8099 (13) | 147.1 (3) |
| N11—H11B···O6v | 1.042 (3) | 1.715 (3) | 2.7557 (14) | 176.7 (3) |
| N11—H11B···O8v | 1.042 (3) | 2.522 (4) | 3.1097 (14) | 115.1 (3) |
| N11—H11C···O3iv | 1.046 (4) | 1.741 (4) | 2.7736 (14) | 168.5 (3) |
| N14—H14A···O4ii | 1.028 (3) | 2.233 (4) | 2.9718 (13) | 127.4 (3) |
| N14—H14A···O6ii | 1.028 (3) | 2.026 (4) | 2.9771 (13) | 152.7 (3) |
| N14—H14B···O8vi | 1.023 (4) | 1.972 (4) | 2.8658 (12) | 144.4 (3) |
| N14—H14B···O2vii | 1.023 (4) | 2.481 (4) | 2.9914 (15) | 110.2 (3) |
| N14—H14C···O5vii | 1.039 (4) | 1.815 (4) | 2.7952 (13) | 155.8 (3) |
| N21—H21A···O5vii | 1.039 (4) | 1.757 (4) | 2.7501 (13) | 158.5 (3) |
| N21—H21B···O6 | 1.020 (4) | 2.110 (4) | 2.8758 (12) | 130.3 (3) |
| N21—H21B···O10viii | 1.020 (4) | 2.200 (4) | 2.8612 (15) | 121.0 (3) |
| N21—H21C···O4ii | 1.031 (3) | 2.313 (4) | 2.9677 (13) | 120.1 (3) |
| N21—H21C···O8ii | 1.031 (3) | 1.893 (3) | 2.9010 (13) | 165.1 (3) |

Symmetry code: (i) −x+3, y+1/2, −z+2.
C15—H15...O15<sub>a</sub> 1.088 (5) 2.365 (5) 3.2496 (17) 137.4 (3)
C15—H15...O9 1.085 (4) 2.537 (5) 3.5320 (18) 152.0 (3)
C17—H17...O10<sub>b</sub> 1.095 (4) 2.280 (4) 3.3311 (16) 160.2 (4)
C20—H20...O9 1.087 (4) 2.369 (4) 3.2703 (18) 139.2 (3)
C20—H20...O4<sub>ii</sub> 1.087 (4) 2.532 (4) 3.0948 (15) 111.2 (3)

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

Bis(glycinium) sulfate–glycine (1/1) (298KModel1)

Crystal data
2C<sub>2</sub>H<sub>6</sub>NO<sub>2</sub>·SO<sub>4</sub>·C<sub>2</sub>H<sub>5</sub>NO<sub>2</sub>  F(000) = 130.536
Mr = 323.28
Monoclinic, P<sub>2</sub>1
Hall symbol: P2yb
a = 9.3910 (14) Å
b = 12.6021 (18) Å
c = 5.7125 (7) Å
β = 110.306 (13)°
V = 634.04 (16) Å<sup>3</sup>
Z = 2

Data collection
Time-of-flight Laue-type single crystal neutron
diffractometer
Radiation source: spallation neutron
Detector resolution: 4 pixels mm<sup>-1</sup>
time–of–flight Laue method scans
14190 measured reflections
3132 independent reflections
10685 reflections with I > 2σ(I)

Refinement
Refinement on F<sup>2</sup>
Least-squares matrix: full
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.080
wR(F<sup>2</sup>) = 0.209
S = 1.05
14190 reflections
357 parameters
1 restraint
All H-atom parameters refined
w = 1/[σ<sup>2</sup>(F<sup>2</sup>) + (0.1405P)<sup>2</sup>]
where P = (F<sup>2</sup> + 2F<sup>c</sup><sup>2</sup>)<sup>1/2</sup>

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.

Refrinement. reflns Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.
### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|   | x     | y     | z     | U\(_{iso}\)/U\(_{eq}\) | Occ. (<1) |
|---|-------|-------|-------|-------------------------|-----------|
| S1| 0.9997 (5) | 0.7500 (5) | 0.7732 (8) | 0.0132 (7) |           |
| O2| 0.9154 (4)  | 0.8459 (2)  | 0.7922 (8) | 0.0239 (7) |           |
| O3| 0.5395 (5)  | 1.4692 (4)  | 0.1995 (9) | 0.0336 (9) |           |
| O4| 0.7789 (5)  | 1.4949 (3)  | 0.2233 (8) | 0.0308 (8) |           |
| O5| 1.1424 (3)  | 0.7495 (4)  | 0.9937 (5) | 0.0239 (5) |           |
| O6| 1.0347 (4)  | 0.7517 (3)  | 0.5439 (5) | 0.0252 (5) |           |
| O7| 0.7864 (5)  | 0.9977 (4)  | 0.2398 (8) | 0.0313 (8) |           |
| O8| 0.6071 (4)  | 1.2431 (4)  | 1.0767 (7) | 0.0318 (7) |           |
| O9| 0.7083 (6)  | 1.2436 (8)  | 1.0440 (12)| 0.0370 (12)|           |
| O10| 0.4960 (5) | 1.2334 (5)  | 0.6667 (8) | 0.0435 (12)|           |
| O12| 0.9126 (5) | 0.6560 (3)  | 0.7868 (8) | 0.0260 (7) |           |
| O15| 0.5516 (5) | 1.0201 (5)  | 0.2419 (10)| 0.0419 (11)|           |
| H15| 0.5111 (11) | 0.9968 (7)  | 0.046 (2)  | 0.874 (8)  | 0.050 (2) |
| N11| 0.3571 (3)  | 1.2889 (3)  | 1.1627 (6) | 0.0318 (6) | 0.874 (8) |
| H11A| 0.4246 (11) | 1.3547 (10)| 1.200 (2)  | 0.051 (2)  | 0.874 (8) |
| H11B| 0.4071 (13) | 1.2382 (13)| 1.305 (2)  | 0.063 (3)  | 0.874 (8) |
| H11C| 0.2518 (10)| 1.3094 (9)  | 1.176 (2)  | 0.048 (2)  | 0.874 (8) |
| N11B| 0.364 (2)   | 1.224 (2)   | 1.167 (4)  | 0.0318 (6) | 0.126 (8) |
| H11D| 0.427371    | 1.284076    | 1.276505   | 0.051 (2)  | 0.126 (8) |
| H11E| 0.421468    | 1.153071    | 1.217566   | 0.063 (3)  | 0.126 (8) |
| H11F| 0.260990    | 1.218760    | 1.192923   | 0.048 (2)  | 0.126 (8) |
| N16| 0.9163 (3)  | 1.07320 (14)| 0.7057 (4) | 0.0229 (4) |           |
| H16A| 0.9555 (11) | 1.1246 (8)  | 0.6000 (17)| 0.0451 (18)|           |
| H16B| 0.9613 (13) | 0.9994 (7)  | 0.7083 (17)| 0.053 (2)  |           |
| H16C| 0.9530 (12) | 1.1040 (6)  | 0.8831 (14)| 0.0414 (17)|           |
| N21| 0.8957 (3)  | 1.42963 (15)| 0.6984 (4) | 0.0221 (4) |           |
| H21A| 0.9418 (11) | 1.3815 (9)  | 0.6004 (18)| 0.049 (2)  |           |
| H21B| 0.9277 (12) | 1.3988 (7)  | 0.8734 (14)| 0.046 (2)  |           |
| H21C| 0.9367 (12) | 1.5046 (7)  | 0.7098 (16)| 0.052 (2)  |           |
| C13| 0.6830 (3)  | 1.4683 (2)  | 0.3127 (6) | 0.0204 (5) |           |
| C14| 0.7510 (4)  | 1.0688 (3)  | 0.6007 (6) | 0.0257 (6) |           |
| H14A| 0.7068 (12) | 1.1483 (8)  | 0.597 (2)  | 0.059 (3)  |           |
| H14B| 0.7088 (15) | 1.0190 (11)| 0.719 (2)  | 0.064 (3)  |           |
| C17| 0.3401 (3)  | 1.2452 (3)  | 0.9149 (5) | 0.0256 (5) |           |
| H17A| 0.2645 (11) | 1.2958 (10)| 0.7802 (18)| 0.061 (3)  |           |
| H17B| 0.2911 (14) | 1.1675 (8)  | 0.895 (3)  | 0.073 (4)  |           |
| C18| 0.6979 (4)  | 1.0246 (2)  | 0.3395 (6) | 0.0230 (6) |           |
| C19| 0.4893 (3)  | 1.2402 (2)  | 0.8730 (5) | 0.0221 (5) |           |
| C20| 0.7303 (4)  | 1.4306 (3)  | 0.5794 (6) | 0.0258 (6) |           |
| H20A| 0.6812 (15) | 1.4810 (13)| 0.682 (2)  | 0.074 (4)  |           |
| H20B| 0.6890 (13) | 1.3519 (10)| 0.584 (2)  | 0.065 (3)  |           |
Atomic displacement parameters (Å²)

|   | U¹¹ | U¹² | U¹³ | U²² | U²³ | U³³ |
|---|-----|-----|-----|-----|-----|-----|
| S1| 0.0134 (18) | 0.0106 (13) | 0.0159 (17) | 0.0002 (19) | 0.0057 (14) | −0.0022 (19) |
| O2| 0.0244 (15) | 0.0152 (12) | 0.0353 (19) | 0.0016 (11) | 0.0145 (14) | 0.0063 (10) |
| O3| 0.0181 (15) | 0.0438 (19) | 0.0325 (19) | 0.0056 (14) | 0.0099 (13) | 0.0029 (15) |
| O4| 0.0239 (17) | 0.0405 (18) | 0.0244 (16) | 0.0118 (14) | 0.0039 (13) | −0.0039 (14) |
| O5| 0.0147 (11) | 0.0363 (12) | 0.0188 (10) | 0.0010 (15) | 0.0035 (9) | 0.0018 (14) |
| O6| 0.0337 (15) | 0.0271 (11) | 0.0178 (10) | −0.0003 (13) | 0.0127 (10) | −0.0006 (15) |
| O7| 0.0252 (17) | 0.0418 (18) | 0.0255 (16) | −0.0112 (15) | 0.0069 (14) | 0.0043 (14) |
| O8| 0.0142 (12) | 0.0518 (19) | 0.0286 (14) | −0.0027 (18) | 0.0065 (11) | 0.0003 (16) |
| O9| 0.021 (2) | 0.050 (3) | 0.039 (3) | −0.004 (3) | 0.010 (2) | 0.002 (3) |
| O10| 0.0310 (19) | 0.071 (3) | 0.0268 (16) | −0.0066 (19) | 0.0084 (14) | 0.006 (2) |
| O11| 0.0274 (17) | 0.0159 (12) | 0.035 (2) | 0.0001 (11) | 0.0111 (14) | −0.0072 (11) |
| H15| 0.031 (3) | 0.044 (3) | 0.071 (7) | −0.002 (4) | 0.015 (4) | −0.003 (3) |
| N11| 0.0174 (10) | 0.0492 (17) | 0.0316 (11) | −0.0032 (12) | 0.0119 (9) | −0.0065 (11) |
| H11A| 0.037 (4) | 0.074 (6) | 0.048 (5) | −0.025 (5) | 0.020 (4) | −0.017 (4) |
| H11B| 0.047 (5) | 0.088 (9) | 0.051 (5) | 0.025 (6) | 0.014 (4) | −0.009 (6) |
| H11C| 0.028 (4) | 0.060 (5) | 0.061 (6) | −0.010 (4) | 0.022 (4) | −0.005 (3) |
| H11D| 0.0174 (10) | 0.0492 (17) | 0.0316 (11) | −0.0032 (12) | 0.0119 (9) | −0.0065 (11) |
| H11E| 0.037 (4) | 0.074 (6) | 0.048 (5) | −0.025 (5) | 0.020 (4) | −0.017 (4) |
| H11F| 0.047 (5) | 0.088 (9) | 0.051 (5) | 0.025 (6) | 0.014 (4) | −0.009 (6) |
| N16| 0.0227 (10) | 0.0200 (8) | 0.0179 (8) | −0.0019 (6) | 0.0033 (7) | 0.0037 (7) |
| H16A| 0.039 (4) | 0.062 (5) | 0.033 (4) | 0.002 (3) | 0.012 (3) | −0.012 (3) |
| H16B| 0.057 (5) | 0.044 (4) | 0.042 (4) | −0.010 (3) | −0.003 (4) | 0.026 (4) |
| H16C| 0.056 (5) | 0.035 (3) | 0.028 (3) | −0.010 (2) | 0.008 (3) | −0.004 (3) |
| N21| 0.0244 (9) | 0.0216 (8) | 0.0161 (8) | 0.0036 (6) | 0.0017 (7) | −0.0029 (7) |
| H21A| 0.040 (4) | 0.070 (5) | 0.037 (4) | −0.004 (3) | 0.011 (3) | 0.015 (4) |
| H21B| 0.063 (6) | 0.042 (3) | 0.027 (3) | 0.010 (3) | 0.007 (3) | 0.003 (3) |
| C13| 0.0177 (11) | 0.0186 (10) | 0.0218 (12) | 0.0033 (8) | 0.0032 (9) | 0.0015 (9) |
| C14| 0.0268 (14) | 0.0270 (12) | 0.0252 (13) | −0.0068 (10) | 0.0113 (11) | −0.0012 (10) |
| H14A| 0.045 (5) | 0.052 (4) | 0.069 (7) | −0.027 (4) | 0.005 (4) | 0.017 (4) |
| H14B| 0.071 (7) | 0.088 (7) | 0.043 (5) | −0.003 (4) | 0.034 (5) | −0.028 (6) |
| C17| 0.0144 (10) | 0.0276 (10) | 0.0322 (12) | −0.0032 (12) | 0.0048 (9) | −0.0006 (11) |
| H17A| 0.039 (4) | 0.095 (7) | 0.047 (4) | 0.010 (4) | 0.010 (4) | 0.030 (5) |
| H17B| 0.058 (6) | 0.045 (4) | 0.127 (11) | −0.032 (6) | 0.046 (7) | −0.025 (4) |
| C18| 0.0217 (12) | 0.0226 (12) | 0.0244 (13) | −0.0035 (9) | 0.0076 (10) | −0.0026 (9) |
| C19| 0.0170 (10) | 0.0243 (11) | 0.0246 (10) | −0.0007 (9) | 0.0068 (8) | 0.0022 (9) |
| C20| 0.0215 (13) | 0.0313 (13) | 0.0246 (14) | 0.0060 (10) | 0.0079 (11) | −0.0011 (11) |
| H20A| 0.059 (6) | 0.120 (10) | 0.049 (6) | −0.001 (6) | 0.027 (5) | 0.035 (7) |
| H20B| 0.055 (6) | 0.064 (5) | 0.059 (6) | 0.029 (4) | −0.003 (4) | −0.031 (5) |
**Geometric parameters (Å, °)**

| Bond/Distance   | Length/Angle       |
|----------------|--------------------|
| S1—O6          | 1.456 (5)          |
| S1—O12         | 1.457 (6)          |
| S1—O2          | 1.470 (6)          |
| S1—O5          | 1.487 (5)          |
| O3—C13         | 1.276 (5)          |
| O3—H15        | 1.361 (12)         |
| O4—C13         | 1.227 (5)          |
| O7—C18         | 1.208 (5)          |
| O9—H9          | 1.030 (7)          |
| O9—C19         | 1.298 (4)          |
| O10—C19        | 1.205 (5)          |
| O15—H15        | 1.089 (12)         |
| O15—C18        | 1.292 (5)          |
| N11—H11B       | 1.010 (12)         |
| N11—H11A       | 1.021 (10)         |
| N11—H11C       | 1.049 (9)          |
| N11—C17        | 1.475 (4)          |
| N11B—H11E      | 1.170 (5)          |
| N11B—H11F      | 1.130 (5)          |
| N11B—H11I      | 113.3 (6)          |
| H9—O9—C19      | 108.0 (4)          |
| H15—O15—C18    | 106.9 (4)          |
| H11B—N11—H11A  | 105.2 (11)         |
| H11B—N11—H11C  | 106.6 (10)         |
| H11A—N11—H11C  | 108.5 (8)          |
| H11B—N11—C17   | 113.6 (10)         |
| H11A—N11—C17   | 110.9 (6)          |
| H11C—N11—C17   | 111.6 (6)          |
| H11E—N11B—H11F | 109.5              |
| H11E—N11B—H11D | 109.5              |
| H11F—N11B—H11D | 109.5              |
| H11E—N11B—C17  | 109.5              |
| H11F—N11B—C17  | 109.5              |
| H11F—N11B—C17  | 109.5              |
| H11D—N11B—C17  | 109.5              |
| H16B—N16—H16C  | 109.6 (7)          |
| H16B—N16—H16A  | 110.5 (10)         |
| H16C—N16—H16A  | 105.9 (7)          |
| H16B—N16—C14   | 110.4 (7)          |
| H16C—N16—C14   | 111.4 (6)          |

---

**Notes:**
- C13—O3—H15i: 117.0 (5)  H21A—N21—C20: 109.6 (6)
- H9—O9—C19: 113.0 (5)  H14A—C14—C18: 109.4 (7)
- H15—O15—C18: 113.3 (6)  H14B—C14—C18: 109.5 (7)
- H11B—N11—H11A: 110.1 (3)  H14—C14—N16: 108.6 (7)
- H11B—N11—H11C: 108.0 (4)  H14A—C14—N16: 108.9 (12)
- H11A—N11—H11C: 113.6 (10)  H17A—C17—N11B: 133.5 (12)
- H11B—N11—C17: 110.9 (6)  H17A—C17—N11B: 81.1 (13)
- H11A—N11—C17: 111.6 (6)  H17A—C17—N11B: 107.1 (7)
- H11C—N11—C17: 111.6 (6)  H17B—C17—N11: 110.1 (9)
- H11E—N11B—H11F: 109.5  H17A—C17—N11: 109.6 (7)
- H11E—N11B—H11D: 109.5  H17B—C17—N11B: 109.6 (7)
- H11F—N11B—H11D: 109.5  H17B—C17—N11B: 109.2 (8)
- H11E—N11B—C17: 109.5  H11C—N11—C17: 111.8 (2)
- H11F—N11B—C17: 109.5  O7—C18—O15: 126.0 (4)
- H11D—N11B—C17: 109.5  O7—C18—C14: 121.7 (3)
- H16B—N16—H16C: 112.4 (4)
- H16B—N16—H16A: 124.1 (3)
- H16C—N16—H16A: 121.8 (3)
- H16B—N16—C14: 114.1 (3)
- H16C—N16—C14: 108.0 (13)
Hydrogen-bond geometry (Å, °)

| D—H···A       | D—H | H···A  | D···A  | D—H···A |
|---------------|-----|-------|--------|---------|
| O9—H9···O5ii  | 1.030 (7) | 1.492 (7) | 2.522 (5) | 176.8 (8) |
| O15—H15···O3iii | 1.090 (12) | 1.361 (12) | 2.450 (7) | 179.2 (10) |
| N11—H11A···O3iv | 1.020 (13) | 1.802 (13) | 2.809 (6) | 168.3 (10) |
| N11—H11B···O10iv | 1.012 (14) | 1.942 (12) | 2.806 (6) | 141.5 (13) |
### Supporting Information

| Bond          | D (Å) | E (Å) | F (Å) | ψ (°) |
|---------------|-------|-------|-------|-------|
| N11—H11C···O2v | 1.050 (11) | 1.705 (11) | 2.755 (5) | 177.7 (8) |
| N11—H11C···O12v | 1.050 (11) | 2.529 (12) | 3.132 (6) | 115.8 (8) |
| N16—H16A···O6vi | 1.036 (10) | 1.817 (11) | 2.786 (4) | 154.0 (9) |
| N16—H16B···O2 | 1.020 (10) | 2.074 (10) | 2.907 (3) | 137.4 (10) |
| N16—H16C···O5ii | 1.027 (8) | 2.258 (10) | 2.974 (5) | 125.5 (7) |
| N16—H16D···O12ii | 1.027 (8) | 1.982 (9) | 2.974 (5) | 161.7 (10) |
| N21—H21A···O6vi | 1.018 (11) | 1.879 (12) | 2.828 (4) | 153.8 (10) |
| N21—H21B···O2ii | 1.016 (8) | 2.076 (9) | 3.024 (5) | 153.4 (10) |
| N21—H21C···O5ii | 1.016 (8) | 2.213 (10) | 2.968 (5) | 129.9 (7) |
| C14—H14A···O10 | 1.082 (11) | 2.403 (13) | 3.287 (7) | 138.0 (9) |
| C17—H17B···O4x | 1.071 (11) | 2.305 (12) | 3.348 (5) | 164.2 (12) |
| C20—H20A···O15ix | 1.071 (15) | 2.423 (15) | 3.346 (7) | 143.7 (11) |
| C20—H20B···O10 | 1.069 (13) | 2.517 (14) | 3.467 (7) | 147.9 (10) |

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

---

**Bis(glycinium) sulfate–glycine (1/1) (298KModel2)**

**Crystal data**

| Compound | Formula | Mr | Crystal class | Space group | a (Å) | b (Å) | c (Å) | β (°) | V (Å³) | Z |
|----------|---------|----|---------------|-------------|-------|-------|-------|-------|-------|---|
| 2C2H6NO2·SO42−·C2H5NO2 | F(000) = 130.536 | 323.28 | Monoclinic, P21 | P21 | 9.3910 (14) | 12.6021 (18) | 5.7125 (7) | 110.306 (13) | 634.04 (16) | 2 |

**Data collection**

| Method | Time-of-flight Laue-type single crystal neutron diffractometer | 10685 reflections with I > 2σ(I) |
|--------|-----------------------------------------------------|----------------------------------|
| Radiation source: | Spallation neutron | Rint = N/A |
| Detector resolution: | 4 pixels mm⁻¹ | θmax = 83.3°, θmin = 7.9° |
| Time-of-flight Laue method scans | 14190 measured reflections | h = −15→15 |
| 3132 independent reflections | l = −9→9 |

**Refinement**

| Refinement on F² | All H-atom parameters refined |
|------------------|--------------------------------|
| Least-squares matrix: full | w = 1/[σ²(F²) + (0.1405P)²] |
| R[F² > 2σ(F²)] = 0.080 | where P = (F² + 2Fc²)/3 |
| wR(F²) = 0.209 | (Δσ)max < 0.001 |
| S = 1.05 | Δρmax = 1.50 e Å⁻³ |
| 14190 reflections | Δρmin = −1.70 e Å⁻³ |
| 358 parameters | Extinction correction: SHELXL-2018/3 (Sheldrick 2015) |

**Acta Cryst. (2022), E78, 306-312**

sup-11
Extinction coefficient: 0.106 (5)  
Absolute structure: Indeterminate for a neutron structure

**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.** reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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

|     | x      | y      | z      | Uiso*/Ueq | Occ. (<1) |
|-----|--------|--------|--------|-----------|-----------|
| S1  | 0.9997 | 0.7502 | 0.7732 | 0.0131 (7)| 0.124 (8) |
| O2  | 0.9154 | 0.8462 | 0.7921 | 0.0238 (7)|           |
| O3  | 0.5395 | 1.4695 | 0.1996 | 0.0037 (9)|           |
| H3  | 0.5153 | 1.4925 | 0.0111 | 0.0046 (2)| 0.124 (8) |
| O4  | 0.7790 | 1.4951 | 0.2233 | 0.0307 (8)|           |
| O5  | 1.1424 | 0.7497 | 0.9937 | 0.0239 (5)|           |
| O6  | 1.0347 | 0.7519 | 0.5440 | 0.0252 (5)|           |
| O7  | 0.7864 | 0.9980 | 0.2398 | 0.0313 (8)|           |
| O9  | 0.6071 | 1.2434 | 1.0767 | 0.0317 (7)|           |
| H9  | 0.7082 | 1.2438 | 1.0440 | 0.0369 (12)|          |
| O10 | 0.4960 | 1.2336 | 0.6668 | 0.0434 (12)|          |
| O12 | 0.9126 | 0.6563 | 0.7868 | 0.0261 (7)|           |
| O15 | 0.5517 | 1.0205 | 0.2419 | 0.0419 (11)|          |
| H15 | 0.5131 | 0.9970 | 0.0517 | 0.0046 (2)| 0.876 (8) |
| N11 | 0.3571 | 1.2892 | 1.1626 | 0.0316 (7)| 0.876 (8) |
| H11A| 0.4246 | 1.3552 | 1.2000 | 0.0052 (2)| 0.876 (8) |
| H11B| 0.4077 | 1.2388 | 1.3050 | 0.0062 (3)| 0.876 (8) |
| H11C| 0.2518 | 1.3095 | 1.1760 | 0.0048 (2)| 0.876 (8) |
| N11B| 0.3650 | 1.2260 | 1.1670 | 0.0031 (7)| 0.124 (8) |
| H11D| 0.4260 | 1.1567 | 1.2210 | 0.0052 (2)| 0.124 (8) |
| H11E| 0.2624 | 1.2188 | 1.1933 | 0.0062 (3)| 0.124 (8) |
| H11F| 0.4253 | 1.2881 | 1.2731 | 0.0048 (2)| 0.124 (8) |
| N16 | 0.9164 | 1.0734 | 0.7057 | 0.0228 (4)|           |
| H16A| 0.9553 | 1.1250 | 0.5999 | 0.0454 (18)|          |
| H16B| 0.9613 | 0.9997 | 0.7084 | 0.053 (2) |           |
| H16C| 0.9530 | 1.1042 | 0.8830 | 0.0414 (17)|          |
| N21 | 0.8957 | 1.4298 | 0.6984 | 0.0220 (4)|           |
| H21A| 0.9420 | 1.3818 | 0.6005 | 0.049 (2) |           |
| H21B| 0.9277 | 1.3990 | 0.8735 | 0.046 (2) |           |
| H21C| 0.9368 | 1.5049 | 0.7098 | 0.053 (2) |           |
| C13 | 0.6830 | 1.4686 | 0.3128 | 0.0203 (5)|           |
| C14 | 0.7510 | 1.0691 | 0.6007 | 0.0257 (6)|           |
| H14A| 0.7067 | 1.1486 | 0.597 (2)| 0.060 (3) |           |
| H14B| 0.7088 | 1.0192 | 0.719 (2)| 0.064 (3) |           |
| C17 | 0.3401 | 1.2455 | 0.9149 | 0.0256 (5)|           |
supporting information

Atomic displacement parameters (Å²)

|   | U¹¹ | U¹² | U¹³ | U²² | U²³ | U³³ |
|---|-----|-----|-----|-----|-----|-----|
| S1 | 0.0133 (18) | 0.0105 (13) | 0.0159 (17) | −0.0021 (19) | 0.0057 (14) | 0.0003 (19) |
| O2 | 0.0243 (15) | 0.0152 (12) | 0.0352 (19) | 0.0063 (10) | 0.0144 (14) | 0.0017 (11) |
| O3 | 0.0179 (15) | 0.0440 (19) | 0.0327 (19) | 0.0028 (15) | 0.0008 (13) | 0.0059 (15) |
| H3 | 0.027 (3) | 0.044 (3) | 0.062 (7) | −0.005 (3) | 0.008 (4) | −0.004 (4) |
| O4 | 0.0238 (17) | 0.0403 (18) | 0.0245 (17) | −0.0039 (14) | 0.0041 (13) | 0.0119 (14) |
| O5 | 0.0147 (11) | 0.0363 (12) | 0.0188 (10) | 0.0019 (14) | 0.0036 (9) | 0.0011 (15) |
| O6 | 0.0337 (15) | 0.0270 (11) | 0.0179 (10) | −0.0006 (15) | 0.0128 (10) | −0.0003 (13) |
| O7 | 0.0252 (17) | 0.0418 (18) | 0.0253 (16) | 0.0043 (14) | 0.0067 (14) | −0.0111 (15) |
| O8 | 0.0142 (12) | 0.0517 (19) | 0.0287 (14) | 0.0003 (16) | 0.0067 (11) | −0.0028 (18) |
| O9 | 0.021 (2) | 0.050 (3) | 0.039 (3) | 0.002 (3) | 0.010 (2) | −0.004 (3) |
| O10 | 0.0309 (19) | 0.071 (3) | 0.0268 (16) | 0.005 (2) | 0.0083 (14) | −0.0069 (19) |
| O11 | 0.0276 (17) | 0.0158 (12) | 0.035 (2) | −0.0072 (11) | 0.0113 (15) | 0.0001 (11) |
| O15 | 0.0222 (18) | 0.064 (3) | 0.038 (2) | −0.0124 (19) | 0.0085 (17) | −0.014 (2) |
| H15 | 0.026 (3) | 0.044 (3) | 0.062 (7) | −0.005 (3) | 0.008 (4) | −0.004 (4) |
| N11 | 0.0176 (10) | 0.0485 (18) | 0.0314 (11) | −0.0062 (11) | 0.0119 (9) | −0.0033 (12) |
| H11A | 0.038 (4) | 0.075 (6) | 0.048 (5) | −0.017 (4) | 0.020 (4) | −0.024 (5) |
| H11B | 0.046 (5) | 0.088 (9) | 0.048 (5) | −0.009 (6) | 0.013 (4) | 0.023 (6) |
| H11C | 0.028 (4) | 0.059 (5) | 0.061 (6) | −0.006 (3) | 0.023 (4) | −0.011 (4) |
| N11B | 0.0176 (10) | 0.0485 (18) | 0.0314 (11) | −0.0062 (11) | 0.0119 (9) | −0.0033 (12) |
| H11D | 0.038 (4) | 0.075 (6) | 0.048 (5) | −0.017 (4) | 0.020 (4) | −0.024 (5) |
| H11E | 0.046 (5) | 0.088 (9) | 0.048 (5) | −0.009 (6) | 0.013 (4) | 0.023 (6) |
| H11F | 0.028 (4) | 0.059 (5) | 0.061 (6) | −0.006 (3) | 0.023 (4) | −0.011 (4) |
| N16 | 0.0269 (10) | 0.0201 (8) | 0.0179 (8) | 0.0037 (7) | 0.0033 (7) | −0.0019 (6) |
| H16A | 0.039 (4) | 0.063 (5) | 0.033 (4) | −0.012 (4) | 0.011 (3) | 0.002 (3) |
| H16B | 0.057 (5) | 0.044 (4) | 0.042 (4) | 0.025 (4) | −0.003 (4) | −0.010 (3) |
| H16C | 0.056 (5) | 0.035 (3) | 0.028 (3) | −0.004 (3) | 0.008 (3) | −0.010 (2) |
| N21 | 0.0244 (9) | 0.0214 (8) | 0.0160 (8) | −0.0029 (7) | 0.0016 (7) | 0.0036 (6) |
| H21A | 0.040 (4) | 0.069 (5) | 0.036 (4) | 0.015 (4) | 0.011 (3) | −0.003 (3) |
| H21B | 0.063 (6) | 0.042 (3) | 0.026 (3) | 0.003 (3) | 0.007 (3) | 0.010 (3) |
| H21C | 0.057 (5) | 0.045 (4) | 0.041 (4) | −0.026 (4) | −0.001 (4) | 0.009 (3) |
| C13 | 0.0177 (11) | 0.0186 (10) | 0.0218 (12) | 0.0014 (9) | 0.0032 (9) | 0.0032 (8) |
| C14 | 0.0267 (14) | 0.0270 (12) | 0.0251 (13) | −0.0012 (10) | 0.0113 (11) | −0.0067 (10) |
| H14A | 0.045 (5) | 0.053 (4) | 0.069 (7) | 0.017 (4) | 0.005 (4) | −0.027 (4) |
| H14B | 0.072 (7) | 0.089 (7) | 0.044 (5) | −0.028 (6) | 0.034 (5) | −0.003 (4) |
| C17 | 0.0143 (10) | 0.0276 (10) | 0.0324 (12) | −0.0007 (11) | 0.0048 (9) | −0.0031 (12) |
| H17A | 0.040 (4) | 0.095 (7) | 0.046 (4) | 0.031 (5) | 0.010 (4) | 0.010 (5) |
| H17B | 0.058 (6) | 0.045 (4) | 0.126 (11) | −0.024 (4) | 0.045 (7) | −0.032 (6) |
|   | 0.0217 (12) | 0.0226 (12) | 0.0244 (13) | −0.0026 (9) | 0.0077 (10) | −0.0035 (9) |
|---|-----------|-------------|-------------|------------|------------|-----------|
| C19 | 0.0170 (10) | 0.0243 (11) | 0.0246 (11) | 0.0021 (9) | 0.0068 (8) | −0.0007 (9) |
| C20 | 0.0215 (13) | 0.0312 (13) | 0.0245 (14) | −0.0011 (11) | 0.0078 (11) | 0.0060 (10) |
| H20A | 0.060 (6) | 0.118 (10) | 0.049 (6) | 0.035 (7) | 0.028 (5) | −0.001 (6) |
| H20B | 0.055 (6) | 0.064 (5) | 0.059 (6) | −0.031 (5) | −0.003 (4) | 0.029 (4) |

**Geometric parameters (Å, °)**

| Bond | Distance (Å) | Angle (°) |
|------|--------------|-----------|
| S1—O12 | 1.456 (6) | N11B—H11F | 1.0300 |
| S1—O6 | 1.457 (5) | N11B—H11D | 1.0300 |
| S1—O2 | 1.469 (6) | N11B—C17 | 1.40 (2) |
| S1—O5 | 1.487 (5) | N16—H16B | 1.019 (8) |
| O3—H3 | 1.06 (4) | N16—H16C | 1.026 (7) |
| O3—C13 | 1.277 (5) | N16—H16A | 1.036 (9) |
| O3—H15i | 1.386 (12) | N16—C14 | 1.459 (4) |
| H3—H15i | 0.37 (4) | N21—H21C | 1.014 (8) |
| O4—C13 | 1.226 (5) | N21—H21B | 1.016 (7) |
| O7—C18 | 1.208 (5) | N21—H21A | 1.019 (10) |
| O9—H9 | 1.029 (7) | N21—C20 | 1.464 (4) |
| O9—C19 | 1.298 (4) | C13—C20 | 1.508 (4) |
| O10—C19 | 1.204 (5) | C14—H14A | 1.082 (9) |
| O15—H15 | 1.066 (12) | C14—H14B | 1.093 (11) |
| O15—C18 | 1.291 (5) | C14—C18 | 1.507 (5) |
| N11—H11B | 1.010 (12) | C17—H17A | 1.061 (10) |
| N11—H11A | 1.022 (11) | C17—H17B | 1.070 (9) |
| N11—H11C | 1.050 (9) | C17—C19 | 1.503 (4) |
| N11—C17 | 1.474 (4) | C20—H20B | 1.068 (10) |
| N11B—H11E | 1.0300 | C20—H20A | 1.074 (12) |

| Bond | Distance (Å) | Angle (°) |
|------|--------------|-----------|
| O12—S1—O6 | 111.5 (4) | H21B—N21—H21A | 106.0 (8) |
| O12—S1—O2 | 109.8 (3) | H21C—N21—C20 | 110.0 (7) |
| O6—S1—O2 | 110.4 (4) | H21B—N21—C20 | 111.3 (7) |
| O12—S1—O5 | 108.0 (4) | H21A—N21—C20 | 109.6 (6) |
| O6—S1—O5 | 110.1 (3) | O4—C13—O3 | 125.8 (4) |
| O2—S1—O5 | 107.0 (4) | O4—C13—C20 | 120.4 (3) |
| H3—O3—C13 | 109.5 | O3—C13—C20 | 113.7 (3) |
| H3—O3—H15i | 8.0 | H14A—C14—H14B | 108.9 (12) |
| C13—O3—H15i | 117.4 (6) | H14A—C14—N16 | 108.7 (7) |
| H15i—H3—O3 | 149 (4) | H14B—C14—N16 | 109.3 (8) |
| H9—O9—C19 | 113.0 (5) | H14A—C14—C18 | 109.3 (7) |
| H15—O15—C18 | 112.7 (7) | H14B—C14—C18 | 109.5 (7) |
| H15—O15—H3ii | 6 (3) | N16—C14—C18 | 111.1 (3) |
| C18—O15—H3ii | 118.9 (11) | H17A—C17—H17B | 108.4 (12) |
| H3ii—H15—O15 | 155.4 | H17A—C17—N11B | 133.1 (12) |
| H11B—N11—H11A | 105.0 (11) | H17B—C17—N11B | 82.1 (14) |
| H11B—N11—H11C | 106.7 (10) | H17A—C17—N11 | 107.1 (7) |
| H11A—N11—H11C | 108.5 (9) | H17B—C17—N11 | 110.1 (9) |
| H11B—N11—C17 | 113.7 (10) | H17A—C17—C19 | 109.7 (7) |
| Bond | Angle | Torsion | Bond | Angle | Torsion |
|------|-------|---------|------|-------|---------|
| H11A—N11—C17 | 110.9 (6) | 110.9A (6) | H11B—C17—C19 | 109.6 (7) |
| H11C—N11—C17 | 111.7 (6) | 111.7A (6) | N11B—C17—C19 | 108.9 (8) |
| H11E—N11B—H11F | 109.5 | 109.5 | N11—C17—C19 | 111.8 (2) |
| H11E—N11B—H11D | 109.5 | 109.5 | O7—C18—O15 | 126.0 (4) |
| H11F—N11B—H11D | 109.5 | 109.5 | O7—C18—C14 | 121.7 (3) |
| H11E—N11B—C17 | 109.5 | 109.5 | O15—C18—C14 | 112.3 (4) |
| H11F—N11B—C17 | 109.5 | 109.5 | O10—C19—O9 | 124.2 (3) |
| H11D—N11B—C17 | 109.5 | 109.5 | O10—C19—C17 | 121.7 (3) |
| H16B—N16—H16C | 109.5 (7) | 109.5 (7) | O9—C19—C17 | 114.1 (3) |
| H16B—N16—H16A | 110.6 (10) | 110.6 (10) | H20B—C20—H20A | 108.2 (13) |
| H16C—N16—H16A | 105.9 (7) | 105.9 (7) | H20B—C20—N21 | 108.3 (7) |
| H16B—N16—C14 | 110.3 (7) | 110.3 (7) | H20A—C20—N21 | 109.6 (8) |
| H16C—N16—C14 | 111.4 (6) | 111.4 (6) | H20B—C20—C13 | 109.7 (7) |
| H16A—N16—C14 | 109.0 (6) | 109.0 (6) | H20A—C20—C13 | 109.6 (8) |
| H21C—N21—H21B | 108.5 (7) | 108.5 (7) | N21—C20—C13 | 111.4 (3) |
| H21C—N21—H21A | 111.3 (10) | 111.3 (10) | | | |

| Bond | Angle | Torsion | Bond | Angle | Torsion |
|------|-------|---------|------|-------|---------|
| C13—O3—H3—H15 | 176.0 | 169.8 | H3—O15—C18—O7 | -6.9 |
| H3—O3—C13—O4 | 3.4 | H3—O15—C18—C14 | 173.9 (7) |
| H15—O3—C13—O4 | 4.0 (8) | H14A—C14—C18—O15 | 119.8 (9) |
| H3—O3—C13—C20 | -176.7 | H14B—C14—C18—O7 | -120.9 (10) |
| H15—O3—C13—C20 | -176.1 (6) | N16—C14—C18—O7 | -0.1 (5) |
| H16B—N16—C14—H14A | -178.9 (11) | H14A—C14—C18—O15 | -59.8 (9) |
| H16C—N16—C14—H14A | 59.2 (10) | H14B—C14—C18—O15 | 59.4 (9) |
| H16A—N16—C14—H14A | -57.3 (11) | N16—C14—C18—O15 | -179.8 (4) |
| H16B—N16—C14—H14B | 62.3 (11) | H9—O9—C19—O10 | 3.6 (9) |
| H16C—N16—C14—H14B | -59.6 (9) | H9—O9—C19—C17 | -177.0 (7) |
| H16A—N16—C14—H14B | -176.1 (10) | H17A—C17—C19—O10 | -42.0 (9) |
| H16B—N16—C14—C18 | -58.6 (8) | H17B—C17—C19—O10 | 76.9 (11) |
| H16C—N16—C14—C18 | 179.5 (5) | N11B—C17—C19—O10 | 165.1 (12) |
| H16A—N16—C14—C18 | 63.0 (6) | N11—C17—C19—O10 | -160.7 (4) |
| H11E—N11B—C17—H17A | 43.9 | H17A—C17—C19—O9 | 138.5 (8) |
| H11F—N11B—C17—H17A | -76.1 | H17B—C17—C19—O9 | -102.5 (10) |
| H11D—N11B—C17—H17A | 163.9 | N11B—C17—C19—O9 | -14.3 (12) |
| H11E—N11B—C17—H17B | -64.0 | N11—C17—C19—O9 | 19.9 (5) |
| H11F—N11B—C17—H17B | 176.0 | H21C—N21—C20—H20B | -175.1 (11) |
| H11D—N11B—C17—H17B | 56.0 | H21B—N21—C20—H20B | -54.8 (10) |
| H11E—N11B—C17—C19 | -172.1 | H21A—N21—C20—H20B | 62.2 (11) |
| H11F—N11B—C17—C19 | 67.9 | H21C—N21—C20—H20A | -57.3 (12) |
| H11D—N11B—C17—C19 | -52.1 | H21B—N21—C20—H20A | 63.0 (11) |
| H11B—N11—C17—H17A | 167.0 (11) | H21A—N21—C20—H20B | -180.0 (11) |
| H11A—N11—C17—H17A | -74.9 (11) | H21C—N21—C20—C13 | 64.1 (7) |
| H11C—N11—C17—H17A | 46.3 (11) | H21B—N21—C20—C13 | -175.6 (6) |
| H11B—N11—C17—H17B | 49.3 (11) | H21A—N21—C20—C13 | -58.6 (8) |
| H11A—N11—C17—H17B | 167.4 (11) | O4—C13—C20—H20B | -120.9 (10) |
| H11C—N11—C17—H17B | -71.4 (11) | O3—C13—C20—H20B | 59.2 (10) |
| H11B—N11—C17—C19 | -72.8 (8) | O4—C13—C20—H20A | 120.5 (10) |

Acta Cryst. (2022). E78, 306-312

sup-15
### Hydrogen-bond geometry (Å, °)

| D—H···A         | D—H  | H····A | D····A | D—H···A |
|-----------------|-------|--------|--------|---------|
| O3—H3···O15i    | 1.06 (4) | 1.41 (4) | 2.451 (7) | 166 (2) |
| O9—H9···O5iii  | 1.029 (7) | 1.493 (7) | 2.522 (5) | 176.7 (8) |
| O15—H15···O3ii | 1.07 (1) | 1.39 (1) | 2.451 (7) | 178 (1) |
| N11—H11A···O3iv| 1.022 (13) | 1.800 (13) | 2.809 (6) | 168.3 (10) |
| N11—H11B···O10v| 1.011 (14) | 1.943 (12) | 2.807 (6) | 141.7 (13) |
| N11—H11C···O2v | 1.050 (11) | 1.706 (11) | 2.755 (5) | 177.5 (8) |
| N11—H11C···O12v| 1.050 (11) | 2.527 (12) | 3.132 (6) | 116.0 (8) |
| N16—H16B···O6vi| 1.036 (10) | 1.815 (11) | 2.785 (4) | 154.2 (9) |
| N16—H16B···O2  | 1.019 (10) | 2.074 (10) | 2.907 (3) | 137.5 (10) |
| N16—H16B···O4vii| 1.019 (10) | 2.333 (14) | 2.919 (6) | 115.4 (8) |
| N16—H16C···O12v| 1.026 (8) | 2.258 (10) | 2.974 (5) | 125.5 (7) |
| N16—H16C···O12vi| 1.026 (8) | 1.982 (9) | 2.974 (5) | 161.6 (10) |
| N21—H21A···O6vi| 1.019 (11) | 1.880 (12) | 2.829 (4) | 153.6 (10) |
| N21—H21B···O2viii| 1.017 (8) | 2.076 (9) | 3.024 (5) | 154.3 (10) |
| N21—H21B···O5viii| 1.017 (8) | 2.213 (10) | 2.969 (5) | 129.9 (7) |
| N21—H21C···O12vii| 1.015 (10) | 1.989 (10) | 2.892 (4) | 146.9 (10) |
| N21—H21C···O7vii| 1.015 (10) | 2.516 (13) | 3.007 (6) | 109.3 (7) |
| C14—H14A···O10  | 1.082 (11) | 2.402 (13) | 3.287 (7) | 138.0 (9) |
| C17—H17A···O7ix | 1.062 (11) | 2.583 (13) | 3.404 (5) | 133.8 (8) |
| C17—H17B···O4vii| 1.070 (11) | 2.307 (12) | 3.350 (5) | 164.2 (12) |
| C20—H20A···O15vii| 1.076 (15) | 2.423 (15) | 3.348 (7) | 143.3 (11) |
| C20—H20B···O10   | 1.068 (13) | 2.518 (14) | 3.468 (7) | 147.7 (10) |

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