In the title compound, C_{32}H_{29}BrN_{2}O_{10}S_{3}, the benzothiazole and thiophene ring systems subtend an interplanar angle of 7.43 (12)^\circ. The NH_{2} group forms intramolecular hydrogen bonds to N_{thiazole} and O_{carbonyl}. The S_{galactose}−C_{thiophene} bond is short [1.759 (2) Å]. The molecules are connected to form ribbons parallel to the b axis by two ‘weak’ hydrogen bonds and a short N_{amino}−S_{galactose} contact.

Structure description

Benzothiazoles are the most widely applied class of heterocyclic compounds in medicinal chemistry, and benzothiazole derivatives have been employed in many pharmaceutical preparations (Bonde et al., 2015), because they offer a variety of pharmacological properties useful in treating many diseases (Wang et al., 2009). As clinical drugs, they often act with high therapeutic efficacy (Huang et al., 2009). The broad pharmacological activities of benzothiazoles suggest that they are also important for developing future drugs (Rana et al., 2008). Recently we have explored various novel synthetic methods to obtain benzothiazole derivatives (Azzam et al. 2017a,b, 2020a,b,c, 2021; Elgemeie et al., 2009, 2018). We found that our reported dihydro-pyridine S-glycosides have a strong anti-P-glycoprotein effect against human tumor cells.
(Scala et al., 1997). Consistent with these outcomes and our past research (Elgemeie et al., 2015, 2016, 2019, 2020b), the purpose of the current study was to design and synthesize benzothiazole-based thiophene thioglycosides. The synthesis of our target benzothiazole-2-thiophene thioglycoside was carried out by the reaction of benzothiazole 2-thiophenethiol derivative 1 with 2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl bromide 2 in the presence of potassium hydroxide to give the corresponding benzothiazole-2-thiophene S-glycoside 3 in good yield (Fig. 1). It has been suggested that the cis-(α) sugars react via a simple SN2 reaction to give the β-glycoside products such as 3 (Masoud et al., 2017; Hammad et al., 2018). The structure of 3 was confirmed based on the spectroscopic data (13C NMR, 1H NMR, and IR). The 1H NMR spectrum of compound 3 showed the anomeric proton as a doublet at δ = 5.39 p.p.m. with a spin–spin coupling constant (J1,2 = 8.8 Hz) confirming the β-configuration. The other six protons of galactose resonated at δ 4.00–5.30 p.p.m. In order to establish the structure of the product unambiguously, its crystal structure was determined and is reported here. To the best of our knowledge, this is the first reported X-ray structure of the new compound type benzothiazole-2-thiophene thioglycoside.

The structure of 3 is shown in Fig. 2. The dimensions of the benzothiazole moiety are as expected (a selection of molecular dimensions is presented in Table 1). The benzothiazole and thiophene ring systems are approximately coplanar (interplanar angle 7.43 (12)°), a geometry that is reinforced by the two intramolecular hydrogen bonds from the NH2 group to the thiazole nitrogen atom and the CO group (Table 2), whereas the bromophenyl and thiophene rings subtend an angle of 58.23 (6)°. The intramolecular S2–S3 contact is 3.1416 (8) Å.

The β configuration (equatorial position of the sulfur atom) at the anomeric carbon of the sugar (here C31) is confirmed, as is the axial configuration of the substituent at C34, characteristic of galactose. The galactose ring displays a slightly

**Table 1**
Selected geometric parameters (Å, °).

| Bond/Angle | Value   |
|------------|---------|
| S1–C2      | 1.703 (2) |
| S1–C5      | 1.731 (2) |
| S2–C6      | 1.762 (2) |
| C2–S1–C5   | 92.15 (12) |
| C12–S2–C6  | 88.99 (12) |
| C2–S3–C31  | 98.36 (11) |
| C6–N1–C7   | 111.1 (2)  |
| O1–C31–C32–C33 | 53.1 (2) |
| C31–O1–C35–C34 | 69.4 (2) |
| C31–C32–C33–C34 | 45.9 (3) |

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

| D–H · · · A | D–H | H · · · A | D · · · A | D–H · · · A |
|------------|-----|---------|---------|-----------|
| N2–H01···N1 | 0.86 (3) | 2.12 (3) | 2.746 (3) | 129 (3) |
| N2–H02···O10 | 0.86 (3) | 2.14 (3) | 2.795 (3) | 133 (3) |
| C31–H31···O6 | 1.00 | 2.34 | 3.290 (3) | 158 |
| C36–H36B···O9 | 0.99 | 2.33 | 3.294 (4) | 164 |

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

Figure 1
Reaction scheme.

Figure 2
The molecule of 3 in the crystal. Ellipsoids represent 50% probability levels. The dashed lines indicate intramolecular hydrogen bonds.
flattened chair conformation, with absolute torsion angles < 50° about C32—C33 and C33—C34. The configurations at C31—C35 are S, R, S, R, R, respectively. The S3—C31 bond is as expected longer than S3—C2, with values of 1.819 (2) and 1.759 (2) Å, respectively; the latter is significantly shorter than the values found for similar compounds in search of the ipso carbon atom. Dashed lines indicate hydrogen bonds or N···S contacts.

The N—H donor groups do not participate in intermolecular hydrogen bonding, but two short and acceptably linear C—H···O contacts between the galactose moieties may be classed as ‘weak’ hydrogen bonds (Table 2). Additionally, a short contact N2···S3 of 3.249 (2) Å is observed (operator x, 1 + y, z). The net effect is to form ribbons of molecules parallel to the b axis (Fig. 3).

Synthesis and crystallization

Thiophene thiol derivative 1 (2.23 g, 5 mmol) was dissolved in acetone (10 ml) containing 0.5 ml of aq. KOH (0.25 g, 5 mmol). The mixture was warmed to 50°C for 15 min. After cooling, a solution of 2,3,4,6-tetra-O-acetyl-β-d-galacto-

| Table 3 Experimental details. |
|--------------------------------|
| Crystal data                  |
| Chemical formula              | C32H29BrN2O10S3                          |
| M                           | 777.66                                   |
| Crystal system, space group  | Monoclinic, P21                          |
| Temperature (K)              | 100                                      |
| a, b, c (Å)                  | 16.99261 (18), 6.02635 (7), 17.4076 (2)  |
| β (°)                        | 107.8307 (12)                            |
| V (Å³)                       | 1696.97 (3)                              |
| Z                            | 2                                        |
| Radiation type               | Cu Kα                                    |
| μ (mm⁻¹)                     | 3.89                                     |
| Crystal size (mm)            | 0.15 × 0.06 × 0.02                       |

| Data collection              |
| Diffractometer              | XtaLAB Synergy                           |
| Absorption correction       | Multi-scan (CrysAlis PRO; Rigaku OD, 2021) |
| Tmin, Tmax                  | 0.781, 1.000                             |
| No. of measured, independent and observed | 71096, 7129, 7052                         |
| No. of reflections          | 445                                      |
| No. of restraints           | 2                                        |
| H-atom treatment           | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³)        | 0.37, −0.72                              |
| Absolute structure parameter | −0.019 (7)                              |

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHELXT (Sheldrick, 2015a), SHELXL2018/5 (Sheldrick, 2015b), XP (Siemens, 1994) and OLEX2 (Dolomanov et al., 2009).

Yellow solid, yield 65%, m.p. 403–405 K (EtOH); IR (KBr, cm⁻¹): ν 3406–3281 (NH₂), 2923 (ArCH), 1748 (4Ac-CO), 1720 (CO); ¹H NMR (400 MHz, DMSO-d₆): δ 8.19, 1.91, 1.94, 2.01 (4 s, 12H, 4 × OAc), 4.00–4.02 (m, 2H, H-6'), 4.32 (t, J = 6.0 Hz, 1H, H-5'), 5.15 (t, J = 8.0 Hz, 1H, H-4'), 5.25–5.30 (m, 2H, H-3', H-2'), 5.39 (d, J = 8.8 Hz, 1H, H-1'), 7.52 (t, J = 7.4 Hz, 1H, benzothiazole-H), 7.61 (t, J = 7.4 Hz, 1H, benzo(thiazole-H), 7.77–7.79 (m, 4H, Ar—H), 8.14 (d, J = 7.6 Hz, 1H, benzothiazole-H), 8.21 (d, J = 8.0 Hz, 1H, benzothiazole-H), 8.93 (s, br, D₂O excl., 2H, NH₂); Analysis: calculated for C₃₂H₂₉BrN₂O₁₀S₃ (777.66): C, 49.42; H, 3.76; N, 3.60; S, 12.37%. Found: C, 49.39; H, 3.73; N, 3.67; S, 12.40%.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.
Acknowledgements
The authors acknowledge support by the Open Access Publication Funds of the Technical University of Braunschweig.

References
Abu-Zaied, M. A., Elgemeie, G. H. & Jones, P. G. (2019a). Acta Cryst. E75, 1820–1823.
Abu-Zaied, M. A., El-Telbani, E. M., Elgemeie, G. H. & Nawwar, G. A. (2019b). Eur. J. Med. Chem. 46, 229–235.
Abu-Zaied, M. A., Loutfy, S. A., Hassan, A. E. & Elgemeie, G. H. (2019c). Drug. Des. Dev. Ther. 13, 2437–2457.
Abu-Zaied, M. A., Mahmoud, N. M. & Elgemeie, G. H. (2020). Am. Chem. Soc. (Omega), 5, 20042–20050.
Elgemeie, G. H., Abu-Zaied, M. A. & Loutfy, S. A. (2017). Eur. J. Med. Chem., 139–149.
Abu-Zaied, M. A., Mahmoud, N. M. & Elgemeie, G. H. (2020). Acta Cryst. E77, 171–179.
Hammad, S. F., Masoud, D. M., Elgemeie, G. H. & Jones, P. G. (2018). Acta Cryst. E74, 853–856.
Huang, Q., Mao, J., Wan, B., Wang, Y., Brun, R., Franzblau, S. G. & Kozikowski, A. P. (2009). J. Med. Chem. 52, 6757–6767.
Khiar, N., Alonso, I., Rodriguez, N., Fernandez-Mayoralas, A., Jimenez-Barbero, J., Nieto, O., Cano, F., Foces-Foces, C. & Martin-Lomas, M. (1997). Tetrahedron Lett. 38, 8267–8270.
Masoud, D. M., Hammad, S. F., Elgemeie, G. H. & Jones, P. G. (2017). Acta Cryst. E73, 1751–1754.
Parsons, S., Flack, H. D. & Wagner, T. (2013). Acta Cryst. B69, 249–259.
Rana, A., Siddiqui, N., Khan, S. A., Haque, E. S. & Bhat, M. A. (2008). Eur. J. Med. Chem. 43, 1114–1122.
Rigaku D (2021). CrysAlis PRO. Rigaku Oxford Diffraction, Yarnton, England.
Scala, S., Akhmed, K., Rao, U. S., Paull, K., Lin, L., Dickstein, B., Lee, J., Elgemeie, G. H., Stein, W. D. & Bates, S. E. P. (1997). Mol. Pharmacol. 51, 1024–1033.
Sheldrick, G. M. (2015a). Acta Cryst. C71, 3–8.
Sheldrick, G. M. (2015b). Acta Cryst. A71, 3–8.
Siemens (1994). XP. Siemens Analytical X-Ray Instruments, Madison, Wisconsin, USA.
Smith, R., Zeng, X., Müller-Bunz, H. & Zhu, X. (2013). Tetrahedron Lett. 54, 5348–5350.
Wang, X., Sarris, K., Kage, K., Zhang, D., Brown, S. P., Kolasa, T., Surowy, C., El Kouhen, O. F., Muchmore, S. W., Brioni, J. D. & Stewart, A. O. (2009). J. Med. Chem. 52, 170–180.
full crystallographic data

IUCrData (2022). 7, x220412  [https://doi.org/10.1107/S2414314622004126]

4-Amino-5-(4-bromobenzoyl)-3-(benzo[d]thiazol-2-yl)-2-[(2',3',4',6'-tetra-O-acetyl-\(\beta\)-D-galactopyranosyl)sulfanyl]thiophene

Rasha A. Azzam, Galal H. Elgemeie, Nagwa M. Gad and Peter G. Jones

4-Amino-5-(4-bromobenzoyl)-3-(benzo[d]thiazol-2-yl)-2-[(2',3',4',6'-tetra-O-acetyl-\(\beta\)-D-galactopyranosyl)sulfanyl]thiophene

Crystal data

\(\text{C}_{32}\text{H}_{29}\text{BrN}_{2}\text{O}_{10}\text{S}_{3}\)

\(M_r = 777.66\)

Monoclinic, \(P2_1\)

\(a = 16.99261\) (18) \(\text{Å}\)

\(b = 6.02635\) (7) \(\text{Å}\)

\(c = 17.4076\) (2) \(\text{Å}\)

\(\beta = 107.8307\) (12)°

\(V = 1696.97\) (3) \(\text{Å}^3\)

\(Z = 2\)

Data collection

XtaLAB Synergy diffractometer

Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels \(\text{mm}^{-1}\)

\(\omega\) scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2021)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.023\)

\(wR(F^2) = 0.062\)

\(S = 1.04\)

7129 reflections

445 parameters

2 restraints

Primary atom site location: dual

Hydrogen site location: mixed

\(F(000) = 796\)

\(D_x = 1.522\) \(\text{Mg m}^{-3}\)

\(\text{Cu K\(\alpha\) radiation, } \lambda = 1.54184\) \(\text{Å}\)

Cell parameters from 91570 reflections

\(\theta = 2.7–77.5°\)

\(\mu = 3.89\) \(\text{mm}^{-1}\)

\(T = 100\) K

Lath, pale yellow

0.15 \(\times\) 0.06 \(\times\) 0.02 mm

\(T_{\text{min}} = 0.781, T_{\text{max}} = 1.000\)

107906 measured reflections

7129 independent reflections

7052 reflections with \(I > 2\sigma(I)\)

\(R_{\text{int}} = 0.032\)

\(\theta_{\text{max}} = 77.8°, \theta_{\text{min}} = 2.7°\)

\(h = -21\rightarrow21\)

\(k = -7\rightarrow7\)

\(l = -22\rightarrow22\)

H atoms treated by a mixture of independent and constrained refinement

\(w = 1/[(\sigma(F^2)^2 + (0.0397P)^2 + 0.6757P)]\)

where \(P = (F^2 + 2F_c^2)/3\)

\((\Delta/\sigma)_{\text{max}} = 0.003\)

\(\Delta\rho_{\text{max}} = 0.37\) \(\text{e} \text{ Å}^{-3}\)

\(\Delta\rho_{\text{min}} = -0.72\) \(\text{e} \text{ Å}^{-3}\)

Absolute structure: Flack \(x\) determined using 3095 quotients \([(I^+)−(I^−)]/[(I^+)+(I^−)]\) (Parsons et al., 2013)

Absolute structure parameter: −0.019 (7)
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.

Short contacts:
3.1416 (0.0008) S2 - S3 3.2493 (0.0021) N2 - S3$_2$
Operator $2: x,1+y,z$

==============================================================================
Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
- 1.7949 (0.0074) x + 2.7373 (0.0043) y + 15.2223 (0.0070) z = 2.9267 (0.0054)
  * 0.0402 (0.0013) C2 * -0.0135 (0.0017) N1 * -0.0086 (0.0019) C6 * -0.0308 (0.0020) C7 * -0.0016 (0.0018) C8 *
  0.0379 (0.0020) C9 * 0.0338 (0.0019) C10 * -0.0142 (0.0018) C11 * -0.0432 (0.0020) C12
Rms deviation of fitted atoms = 0.0288
- 0.0073 (0.0162) x + 3.1508 (0.0048) y + 14.1284 (0.0110) z = 4.2446 (0.0123)
Angle to previous plane (with approximate esd) = 7.434 ( 0.118 )
  * 0.0061 (0.0010) S1 * 0.0046 (0.0013) C2 * -0.0152 (0.0014) C3 * 0.0207 (0.0014) C4 * -0.0162 (0.0013) C5 0.0201
  (0.0033) S3 0.0387 (0.0033) N2 -0.1146 (0.0038) C13 0.0343 (0.0044) O10
Rms deviation of fitted atoms = 0.0140
5.5692 (0.0163) x + 2.3719 (0.0054) y - 15.9799 (0.0068) z = 5.2155 (0.0164)
Angle to previous plane (with approximate esd) = 58.230 ( 0.061 )
  * -0.0124 (0.0016) C21 * -0.0049 (0.0017) C22 * 0.0186 (0.0017) C23 * -0.0152 (0.0018) C24 * -0.0023 (0.0018) C25 *
  0.0162 (0.0017) C26 -0.0309 (0.0037) C13 0.8520 (0.0040) O10 -0.1298 (0.0035) Br1
Rms deviation of fitted atoms = 0.0130

Refinement. The hydrogen atoms of the NH$_2$ group were refined freely, but with N—H distances restrained to be approximately equal (SADI). The methyl groups were refined as idealized rigid groups allowed to rotate but not tip, with C—H 0.98 Å and H—C—H 109.5 °. Other hydrogens were included using a riding model starting from calculated positions (C—Haromatic 0.95, C—Hmethylene 0.99, C—Hmethine 1.00 Å). The $U$(H) values were fixed at 1.5 or 1.2 times the equivalent $U_{iso}$ value of the parent carbon atoms for methyl and non-methyl hydrogens respectively.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\AA^2$)

|     | x          | y          | z          | $U_{iso}$/$U_{eq}$ |
|-----|------------|------------|------------|--------------------|
| Br1 | 1.17709 (2)| 0.30260 (5)| 0.13689 (2)| 0.03408 (9)        |
| S1  | 0.81796 (3)| 0.50666 (10)| 0.18829 (3)| 0.01870 (11)       |
| S2  | 0.51931 (3)| 0.50194 (10)| 0.15908 (3)| 0.01913 (11)       |
| S3  | 0.69078 (3)| 0.27394 (9) | 0.24112 (3) | 0.01767 (11)       |
| O1  | 0.76755 (10)| 0.4911 (3) | 0.37404 (9) | 0.0193 (3)         |
| O2  | 0.56621 (10)| 0.2394 (3) | 0.34351 (10)| 0.0210 (3)         |
| O3  | 0.64615 (12)| 0.2015 (3) | 0.51551 (11)| 0.0263 (4)         |
| O4  | 0.80626 (10)| 0.3125 (3) | 0.53459 (9) | 0.0236 (3)         |
| O5  | 0.85216 (11)| 0.8993 (3) | 0.41271 (12)| 0.0287 (4)         |
| O6  | 0.57638 (13)| -0.1299 (4)| 0.33101 (15)| 0.0391 (5)         |
| O7  | 0.55812 (15)| 0.4238 (4) | 0.55315 (13)| 0.0413 (5)         |
| O8  | 0.86193 (15)| 0.5092 (5) | 0.64867 (13)| 0.0468 (6)         |
| O9  | 0.98997 (13)| 0.9040 (4) | 0.46564 (14)| 0.0394 (5)         |
| O10 | 0.85254 (11)| 1.0388 (3) | 0.07162 (11)| 0.0243 (4)         |
| N1  | 0.54299 (12)| 0.8799 (3) | 0.09717 (11)| 0.0180 (4)         |
| N2  | 0.69030 (12)| 1.0127 (4) | 0.07769 (12)| 0.0196 (4)         |
| H01 | 0.6390 (17) | 1.048 (6)  | 0.0665 (18) | 0.023 (8)          |
| H02 | 0.726 (2)   | 1.093 (6)  | 0.065 (2)  | 0.039 (10)         |
| C2   | 0.71837 (13) | 0.4982 (4) | 0.19001 (13) | 0.0177 (4) |
| C3   | 0.67073 (14) | 0.6741 (4) | 0.14936 (13) | 0.0167 (4) |
| C4   | 0.71933 (13) | 0.8253 (4) | 0.11822 (12) | 0.0171 (4) |
| C5   | 0.80082 (14) | 0.7495 (4) | 0.13255 (13) | 0.0188 (5) |
| C6   | 0.58211 (14) | 0.7040 (4) | 0.13374 (13) | 0.0166 (4) |
| C7   | 0.45902 (14) | 0.8671 (4) | 0.08844 (13) | 0.0174 (4) |
| C8   | 0.40068 (14) | 1.0310 (4) | 0.05402 (13) | 0.0204 (5) |
| H8   | 0.416201 | 1.161819 | 0.031863 | 0.025* |
| C9   | 0.31929 (14) | 0.9979 (5) | 0.05296 (14) | 0.0220 (5) |
| H9   | 0.278981 | 1.108347 | 0.030320 | 0.026* |
| C10  | 0.29589 (13) | 0.8040 (5) | 0.08480 (13) | 0.0226 (5) |
| H10  | 0.240008 | 0.785287 | 0.083586 | 0.027* |
| C11  | 0.35290 (15) | 0.6397 (4) | 0.11791 (14) | 0.0215 (5) |
| H11  | 0.336876 | 0.507638 | 0.138925 | 0.026* |
| C12  | 0.43461 (14) | 0.6732 (4) | 0.11962 (13) | 0.0186 (4) |
| C13  | 0.86263 (14) | 0.8523 (4) | 0.10269 (14) | 0.0196 (5) |
| C14  | 0.93984 (14) | 0.7229 (4) | 0.10924 (14) | 0.0192 (5) |
| C15  | 0.93411 (14) | 0.5116 (4) | 0.07541 (14) | 0.0214 (5) |
| H22  | 0.881300 | 0.450400 | 0.048610 | 0.026* |
| C23  | 1.00516 (15) | 0.3892 (4) | 0.08054 (15) | 0.0221 (5) |
| H23  | 1.001565 | 0.247432 | 0.055855 | 0.026* |
| C24  | 1.08128 (14) | 0.4797 (5) | 0.12261 (15) | 0.0241 (5) |
| C25  | 1.08860 (15) | 0.6910 (5) | 0.15573 (16) | 0.0248 (5) |
| H25  | 1.141475 | 0.750716 | 0.183250 | 0.030* |
| C26  | 1.01739 (14) | 0.8141 (5) | 0.14802 (14) | 0.0219 (5) |
| H26  | 1.021491 | 0.906444 | 0.169162 | 0.026* |
| C31  | 0.68674 (14) | 0.4215 (4) | 0.33099 (14) | 0.0177 (4) |
| H31  | 0.649650 | 0.553540 | 0.315148 | 0.021* |
| C32  | 0.65357 (13) | 0.2649 (4) | 0.38322 (13) | 0.0189 (5) |
| H32  | 0.682566 | 0.118429 | 0.390455 | 0.023* |
| C33  | 0.66127 (15) | 0.3731 (4) | 0.46429 (14) | 0.0211 (5) |
| H33  | 0.617097 | 0.488234 | 0.456146 | 0.025* |
| C34  | 0.74480 (15) | 0.4824 (4) | 0.50439 (14) | 0.0225 (5) |
| H34  | 0.740969 | 0.577656 | 0.550143 | 0.027* |
| C35  | 0.76617 (15) | 0.6258 (4) | 0.44166 (14) | 0.0221 (5) |
| H35  | 0.722698 | 0.742486 | 0.422673 | 0.026* |
| C36  | 0.84913 (16) | 0.7367 (5) | 0.47261 (16) | 0.0277 (5) |
| H36A | 0.855276 | 0.809402 | 0.525127 | 0.033* |
| H36B | 0.894073 | 0.626681 | 0.479826 | 0.033* |
| C37  | 0.53570 (15) | 0.0359 (4) | 0.31928 (14) | 0.0222 (5) |
| C38  | 0.44510 (15) | 0.0517 (5) | 0.27487 (15) | 0.0255 (5) |
| H38A | 0.417097 | 0.130629 | 0.308282 | 0.038* |
| H38B | 0.421897 | −0.097856 | 0.263079 | 0.038* |
| H38C | 0.437085 | 0.132674 | 0.224223 | 0.038* |
| C39  | 0.59276 (16) | 0.2497 (5) | 0.55765 (15) | 0.0279 (6) |
| C40  | 0.5876 (2) | 0.0572 (6) | 0.61001 (17) | 0.0354 (7) |
| H40A | 0.578982 | −0.079620 | 0.578079 | 0.053* |
| H40B | 0.541162 | 0.079141 | 0.631549 | 0.053* |
| Atomic displacement parameters (Å²) |
|------------------------------------|
|  | $U_{11}$  | $U_{22}$  | $U_{33}$  | $U_{12}$  | $U_{13}$  | $U_{23}$  |
| Br1  | 0.01892 (12) | 0.02848 (15) | 0.05928 (18) | 0.00424 (11) | 0.01851 (11) | 0.00576 (13) |
| S1  | 0.0151 (2) | 0.0187 (3) | 0.0240 (2) | 0.0015 (2) | 0.00866 (19) | 0.0033 (2) |
| S2  | 0.0149 (2) | 0.0184 (3) | 0.0248 (2) | -0.0013 (2) | 0.00720 (19) | 0.0027 (2) |
| S3  | 0.0188 (2) | 0.0162 (3) | 0.0201 (2) | 0.0004 (2) | 0.00916 (18) | 0.0012 (2) |
| O1  | 0.0162 (7) | 0.0215 (9) | 0.0208 (7) | -0.0012 (7) | 0.00664 (6) | -0.0001 (7) |
| O2  | 0.0178 (8) | 0.0197 (9) | 0.0268 (8) | -0.0013 (6) | 0.0088 (6) | -0.0008 (6) |
| O3  | 0.0289 (9) | 0.0290 (10) | 0.0255 (8) | -0.0005 (8) | 0.0151 (7) | 0.0046 (7) |
| O4  | 0.0246 (8) | 0.0248 (9) | 0.0191 (7) | 0.0013 (8) | 0.0035 (6) | -0.0013 (7) |
| O5  | 0.0223 (9) | 0.0254 (10) | 0.0370 (10) | -0.0040 (7) | 0.0071 (7) | 0.0052 (8) |
| O6  | 0.0286 (10) | 0.0248 (11) | 0.0613 (13) | 0.0015 (8) | 0.0100 (9) | -0.0130 (9) |
| O7  | 0.0529 (14) | 0.0450 (14) | 0.0372 (11) | 0.0105 (11) | 0.0305 (10) | 0.0039 (9) |
| O8  | 0.0534 (14) | 0.0464 (14) | 0.0312 (10) | 0.0093 (12) | -0.0011 (9) | -0.0112 (10) |
| O9  | 0.0244 (10) | 0.0413 (12) | 0.0470 (12) | -0.0057 (9) | 0.0030 (9) | 0.0085 (10) |
| O10 | 0.0228 (8) | 0.0182 (9) | 0.0351 (9) | -0.0009 (7) | 0.0136 (7) | 0.0034 (7) |
| N1  | 0.0163 (9) | 0.0204 (10) | 0.0189 (8) | 0.0004 (7) | 0.0079 (7) | -0.0008 (7) |
| N2  | 0.0170 (9) | 0.0187 (10) | 0.0245 (9) | 0.0019 (8) | 0.0088 (7) | 0.0051 (8) |
| C2  | 0.0165 (10) | 0.0193 (11) | 0.0194 (9) | -0.0007 (9) | 0.0086 (8) | -0.0017 (9) |
| C3  | 0.0164 (10) | 0.0167 (11) | 0.0181 (10) | -0.0011 (8) | 0.0071 (8) | -0.0014 (8) |
| C4  | 0.0177 (10) | 0.0183 (11) | 0.0166 (9) | -0.0018 (9) | 0.0071 (8) | -0.0014 (9) |
| C5  | 0.0172 (10) | 0.0188 (12) | 0.0209 (10) | 0.0000 (8) | 0.0068 (8) | -0.0005 (8) |
| C6  | 0.0179 (11) | 0.0180 (11) | 0.0157 (9) | -0.0009 (8) | 0.0079 (8) | -0.0008 (8) |
| C7  | 0.0157 (10) | 0.0207 (12) | 0.0167 (9) | -0.0014 (8) | 0.0061 (8) | -0.0022 (8) |
| C8  | 0.0187 (11) | 0.0239 (13) | 0.0195 (10) | 0.0021 (9) | 0.0071 (8) | 0.0024 (9) |
| C9  | 0.0180 (11) | 0.0281 (13) | 0.0201 (10) | 0.0054 (10) | 0.0063 (8) | 0.0005 (10) |
| C10 | 0.0158 (9) | 0.0303 (13) | 0.0226 (10) | -0.0015 (11) | 0.0073 (8) | -0.0014 (11) |
| C11 | 0.0173 (11) | 0.0246 (13) | 0.0240 (11) | -0.0039 (9) | 0.0083 (9) | 0.0001 (9) |
| C12 | 0.0160 (11) | 0.0207 (12) | 0.0191 (10) | -0.0002 (9) | 0.0053 (8) | -0.0010 (9) |
| C13 | 0.0187 (10) | 0.0193 (12) | 0.0219 (10) | -0.0029 (8) | 0.0076 (8) | -0.0023 (8) |
| C21 | 0.0190 (11) | 0.0195 (11) | 0.0227 (10) | -0.0026 (9) | 0.0116 (9) | 0.0016 (9) |
| C22 | 0.0182 (11) | 0.0215 (12) | 0.0267 (11) | -0.0021 (10) | 0.0099 (9) | 0.0004 (10) |
| C23 | 0.0211 (11) | 0.0192 (11) | 0.0299 (12) | -0.0015 (9) | 0.0136 (9) | 0.0003 (9) |
| C24 | 0.0159 (11) | 0.0260 (13) | 0.0346 (12) | 0.0032 (10) | 0.0138 (9) | 0.0053 (11) |
| C25 | 0.0172 (11) | 0.0260 (13) | 0.0326 (13) | -0.0031 (10) | 0.0097 (9) | -0.0001 (10) |
### Geometric parameters (Å, °)

|        | 1.898 (2) | 1.703 (2) | 1.731 (2) | 1.762 (2) | 1.733 (2) | 1.759 (2) | 1.819 (2) | 1.412 (3) | 1.436 (3) | 1.441 (3) | 1.348 (3) | 1.439 (3) | 1.361 (3) | 1.442 (3) | 1.364 (3) | 1.443 (3) | 1.338 (3) | 1.196 (3) | 1.194 (4) | 1.205 (4) | 1.210 (3) | 1.237 (3) | 1.308 (3) | 1.390 (3) | 1.343 (3) | 1.389 (3) | 1.442 (3) | 1.406 (3) | 1.446 (3) |
|--------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| Br1—C24 | C24—C25 | 1.388 (4) |
| S1—C2   | C25—C26 | 1.390 (4) |
| S1—C5   | C31—C32 | 1.532 (3) |
| S2—C6   | C32—C33 | 1.523 (3) |
| S2—C12  | C33—C34 | 1.526 (3) |
| S3—C2   | C34—C35 | 1.521 (3) |
| S3—C31  | C35—C36 | 1.503 (3) |
| O1—C31  | C37—C38 | 1.499 (3) |
| O1—C35  | C39—C40 | 1.494 (4) |
| O2—C32  | C41—C42 | 1.490 (4) |
| O2—C37  | C43—C44 | 1.492 (4) |
| O3—C33  | N2—H01  | 0.86 (3)  |
| O3—C39  | N2—H02  | 0.86 (3)  |
| O4—C34  | C8—H8   | 0.9500    |
| O4—C41  | C9—H9   | 0.9500    |
| O5—C36  | C10—H10 | 0.9500    |
| O5—C43  | C11—H11 | 0.9500    |
| O6—C37  | C22—H22 | 0.9500    |
| O7—C39  | C23—H23 | 0.9500    |
| O8—C41  | C25—H25 | 0.9500    |
| O9—C43  | C26—H26 | 0.9500    |
| O10—C13 | C31—H31 | 1.0000    |
| N1—C6   | C32—H32 | 1.0000    |
| N1—C7   | C33—H33 | 1.0000    |
| N2—C4   | C34—H34 | 1.0000    |
| C2—C3   | C35—H35 | 1.0000    |
| C3—C4   | C36—H36A| 0.9900    |
| C3—C6   | C36—H36B| 0.9900    |
| C4—C5   | C38—H38A| 0.9800    |
| C5—C13  | C38—H38B| 0.9800    |
The extracted data from the document is as follows:

| Bond          | Distance   | Bond          | Distance   |
|---------------|------------|---------------|------------|
| C7—C8         | 1.398 (3)  | C38—H38C      | 0.9800     |
| C7—C12        | 1.404 (3)  | C40—H40A      | 0.9800     |
| C8—C9         | 1.392 (3)  | C40—H40B      | 0.9800     |
| C9—C10        | 1.402 (4)  | C40—H40C      | 0.9800     |
| C10—C11       | 1.382 (4)  | C42—H42A      | 0.9800     |
| C11—C12       | 1.394 (3)  | C42—H42B      | 0.9800     |
| C13—C21       | 1.500 (3)  | C42—H42C      | 0.9800     |
| C21—C22       | 1.394 (4)  | C44—H44A      | 0.9800     |
| C21—C26       | 1.396 (3)  | C44—H44B      | 0.9800     |
| C22—C23       | 1.394 (3)  | C44—H44C      | 0.9800     |
| C23—C24       | 1.388 (3)  |               |            |

The bond distances are given in Angstroms (Å) with standard deviations in parentheses. The data is from the IUCrData (2022) publication.
| Bond/Angle/Distance | Value     | Bond/Angle/Distance | Value     |
|---------------------|-----------|---------------------|-----------|
| C5—C13—C21         | 117.7 (2) | C33—C34—H34        | 109.5     |
| C22—C21—C13        | 119.8 (2) | O1—C35—H35         | 108.8     |
| C22—C21—C26        | 119.8 (2) | C36—C35—H35        | 108.8     |
| C26—C21—C13        | 120.4 (2) | C34—C35—H35        | 108.8     |
| C21—C22—C23        | 120.6 (2) | O5—C36—H36A        | 110.5     |
| C24—C23—C22        | 118.4 (2) | C35—C36—H36A       | 110.5     |
| C23—C24—Br1        | 118.1 (2) | O5—C36—H36B        | 110.5     |
| C23—C24—C25        | 122.0 (2) | C35—C36—H36B       | 110.5     |
| C25—C24—Br1        | 119.82 (19) | H36A—C36—H36B     | 108.7     |
| C24—C25—C26        | 119.0 (2) | C37—C38—H38A       | 109.5     |
| C25—C26—C21        | 120.1 (2) | C37—C38—H38B       | 109.5     |
| O1—C31—S3          | 108.36 (15) | H38A—C38—H38B   | 109.5     |
| O1—C31—C32         | 110.05 (18) | C37—C38—H38C    | 109.5     |
| C32—C31—S3         | 109.08 (16) | H38A—C38—H38C   | 109.5     |
| O2—C32—C31         | 107.04 (18) | H38B—C38—H38C   | 109.5     |
| O2—C32—C33         | 105.88 (18) | C39—C40—H40A     | 109.5     |
| C33—C32—C31        | 110.08 (19) | C39—C40—H40B     | 109.5     |
| O3—C33—C32         | 106.6 (2) | H40A—C40—H40B     | 109.5     |
| O3—C33—C34         | 110.14 (19) | C39—C40—H40C     | 109.5     |
| C32—C33—C34        | 114.36 (19) | H40A—C40—H40C   | 109.5     |
| O4—C34—C33         | 109.2 (2) | H40B—C40—H40C     | 109.5     |
| O4—C34—C35         | 111.1 (2) | C41—C42—H42A      | 109.5     |
| C35—C34—C33        | 108.05 (19) | C41—C42—H42B     | 109.5     |
| O1—C35—C34         | 109.6 (2) | H42A—C42—H42B     | 109.5     |
| O1—C35—C36         | 107.2 (2) | C41—C42—H42C      | 109.5     |
| C36—C35—C34        | 113.5 (2) | H42A—C42—H42C     | 109.5     |
| O5—C36—C35         | 106.3 (2) | H42B—C42—H42C     | 109.5     |
| O2—C37—C38         | 109.8 (2) | C43—C44—H44A      | 109.5     |
| O6—C37—O2          | 124.0 (2) | C43—C44—H44B      | 109.5     |
| O6—C37—C38         | 126.1 (2) | H44A—C44—H44B     | 109.5     |
| O3—C39—C40         | 109.7 (2) | C43—C44—H44C      | 109.5     |
| O7—C39—O3          | 123.3 (2) | H44A—C44—H44C     | 109.5     |
| O7—C39—C40         | 127.0 (3) | H44B—C44—H44C     | 109.5     |

Br1—C24—C25—C26  -176.89 (19)  
S1—C2—C3—C4      -2.1 (2)      
S1—C2—C3—C6      174.13 (19)  
S1—C5—C13—O10    -171.04 (18) 
S1—C5—C13—C21    10.0 (3)     
S3—C2—C3—C4      178.18 (17)  
S3—C2—C3—C6      -5.6 (4)     
S3—C31—C32—O2    -73.6 (2)    
S3—C31—C32—C33   171.81 (16)  
O1—C31—C32—O2    167.69 (18)  
O1—C31—C32—C33   53.1 (2)     
O1—C35—C36—O5    70.6 (3)     
O2—C32—C33—O3    76.8 (2)     
O2—C32—C33—C34   -161.3 (2)   

C7—N1—C6—S2      1.4 (2)      
C7—N1—C6—C3      179.0 (2)    
C7—C8—C9—C10     -0.7 (3)     
C8—C7—C12—S2     -179.43 (18) 
C8—C7—C12—C11    -0.6 (3)     
C8—C9—C10—C11    -0.2 (4)     
C9—C10—C11—C12   0.7 (4)      
C10—C11—C12—S2   178.28 (19) 
C10—C11—C12—C7   -0.3 (4)     
C12—S2—C6—N1     -1.70 (18)   
C12—S2—C6—C3     -179.36 (19) 
C12—C7—C8—C9     1.1 (3)      
C13—C21—C22—C23  179.7 (2)   
C13—C21—C26—C25  178.3 (2)   

IUCrData (2022). 7, x220412
O3—C33—C34—O4 47.2 (2)  
O3—C33—C34—C35 168.1 (2)  
O4—C34—C35—O1 61.9 (2)  
O4—C34—C35—C36 −58.0 (3)  
O10—C13—C21—C22 −123.4 (3)  
O10—C13—C21—C26 55.7 (3)  
N1—C7—C8—C9 −177.1 (2)  
N1—C7—C8—C12 −1.1 (2)  
N1—C7—C12—S2 177.7 (2)  
N2—C4—C5—S1 178.69 (18)  
N2—C4—C5—C13 −3.7 (4)  
C2—S1—C5—C4 1.97 (18)  
C2—S1—C5—C13 −175.7 (2)  
C2—S3—C31—O1 −66.64 (17)  
C2—S3—C31—C32 173.56 (15)  
C2—C3—C4—N2 3.6 (3)  
C2—C3—C4—C5 −172.8 (2)  
C4—C5—C13—O10 11.6 (4)  
C4—C5—C13—C21 −167.4 (2)  
C5—S1—C7—N1 178.1 (2)  
C5—S1—C7—C8 4.9 (3)  
C6—S2—C12—C7 1.47 (17)  
C6—S2—C12—C11 −177.2 (2)  
C6—N1—C7—C8 178.1 (2)  
C6—N1—C7—C12 −0.2 (3)  
C6—C3—C4—N2 4.9 (3)  
C6—C3—C4—C5 −172.8 (2)  

Hydrogen-bond geometry (Å, º)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|------|---------|
| N2—H01···N1 | 0.86 (3) | 2.12 (3) | 2.746 (3) | 129 (3) |
| N2—H02···O10 | 0.86 (3) | 2.14 (3) | 2.795 (3) | 133 (3) |
| C11—H11···Br1 | 0.95 | 2.97 | 3.710 (2) | 135 |
| C31—H31···O6i | 1.00 | 2.34 | 3.290 (3) | 158 |
| C35—H35···O6ii | 1.00 | 2.62 | 3.532 (3) | 151 |
| C36—H36B···O9iii | 0.99 | 2.33 | 3.294 (4) | 164 |

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