Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide

Mustafa Bookwala, Saloni Patel, Patrick T. Flaherty and Peter L. D. Wildfong*

Graduate School of Pharmaceutical Sciences, Duquesne University, 600 Forbes Avenue, Pittsburgh, PA 15282, USA. *Correspondence e-mail: wildfongp@duq.edu

Received 28 March 2022
Accepted 4 April 2022

Edited by J. T. Mague, Tulane University, USA

Keywords: bromopropamide; crystal structure; single-crystal XRD; structural analogue.

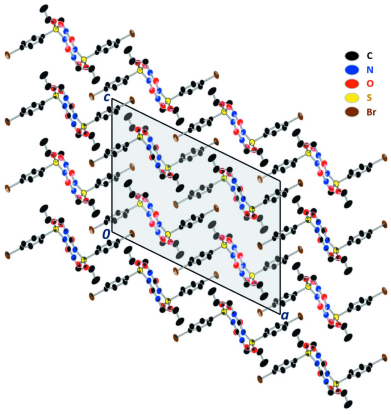
CCDC reference: 2164397

Supporting information: this article has supporting information at journals.iucr.org/e

The title compound, $C_{10}H_{13}BrN_2O_3S$, **1**, contains a sulfonyl urea moiety, which possesses potential therapeutic functions (*e.g.*, anti-diabetic and herbicidal). The geometry of **1** is similar to its closely related analogues, chlorpropamide and tolbutamide. This compound crystallizes in the monoclinic space group *C*2/*c*, having one molecule in its asymmetric unit. The crystal structure of **1**, recorded at 296 K, shows intermolecular N—H···O and C—H···O-type infinite hydrogen-bonded chains involving the sulfonyl urea moiety. Hirshfeld surface analysis and the two-dimensional fingerprint plots confirmed hydrogen bonding as the dominant feature in the crystal packing.

1. Chemical context

The title compound, **1**, also known as bromopropamide, is a sulfonyl urea structural analogue, whose chemical structure is shown in the scheme. Compounds containing sulfonyl urea as the structural core have been used extensively for the treatment of Type II diabetes (McLamore *et al.*, 1959), by stimulating insulin secretion from pancreatic β-cells by binding to the ATP-sensitive potassium channel (Proks *et al.*, 2002). Additionally, sulfonyl urea structural analogues have shown therapeutic action as herbicides and diuretic agents (Tanwar *et al.*, 2017). Thus, the title compound was synthesized in order to perform biological characterization. The crystal structures of several sulfonyl urea compounds have been reported, especially molecules closely related to **1** that contain the *N*-carbamoylbenzenesulfonamide substructure, all of which have multiple polymorphic forms (Kimura *et al.*, 1999; Drebushchak *et al.*, 2006; Fedorov *et al.*, 2017). Subtle changes to the molecule have shown drastic effects on its biological activity and also the arrangement of molecules in the crystal structure (Bieszczad *et al.*, 2020). Thus, it is of interest to not only confirm the molecular structure of bromopropamide, but to also identify its crystal packing relative to other structural analogues.



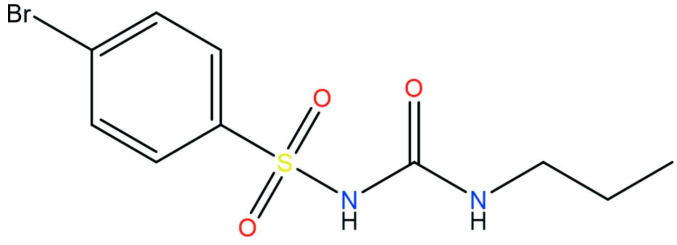


Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	1.94	2.791 (3)	172
N2—H2···O2^{i}	0.86	2.24	2.998 (3)	147
N2—H2···O3^{i}	0.86	2.64	3.351 (3)	141

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

2. Structural commentary

Bromopropamide crystallizes in the centrosymmetric and achiral monoclinic space group *C*2/*c*, having one molecule in the asymmetric unit (Fig. 1). The Br1—C1 bond length [1.887 (2) Å] is in good agreement with other structures containing a bromophenyl moiety (Khamees *et al.*, 2019; Arif Tawfeeq *et al.*, 2019). The bond length between C1—C2 [1.363 (4) Å], is the shortest among all the bond lengths in the phenyl group, possibly due to the inductive effect of bromine. The brominated phenyl ring is almost perpendicular [C4—S1—N1 = 105.65 (11)°] to the sulfonyl urea *n*-propyl group, resulting in an L-shaped molecular structure. This is similar to chlorpropamide, a structural analogue of **1** [Cambridge Structural Database (CSD; Groom *et al.*, 2016) refcode: BEDMIG10; 105.87°; Drebushchak *et al.*, 2009). The sum of the bond angles around N1 and N2 is 360°, indicating sp^2 hybridization, caused by the delocalization of the lone electron pair of N1 and N2 into the π bond of the carbonyl group. This is also supported by the trigonal-planar molecular geometry of C7—N1—S1 [123.94 (17)°], C7—N1—H1 (118°), S1—N1—H1 (118°), C7—N2—C8 [123.5 (2)°], C7—N2—H2 (118.3°), and C8—N2—H2 (118.3°). The C7—N2 bond length is 1.319 (3) Å, which is lower than the typical range; however, the values are similar to those in the crystal structures of bromopropamide analogues, chlorpropamide (1.315 Å; CSD refcode: BEDMIG14; Drebushchak *et al.*, 2009) and tolbutamide (1.319 Å; CSD refcode: ZZZPUS13; Drebushchak *et al.*, 2011). The propyl chain takes the stable *trans* conformation so as to have a maximum distance of 3.794 Å between N2 and C10, while C10 exhibits rotational disorder, possibly due to the X-ray diffraction experiments being conducted at 296 K. Overall, the crystal structure of **1** showcases bond lengths (Allen *et al.*, 1987) and angles typical of the expected ranges.

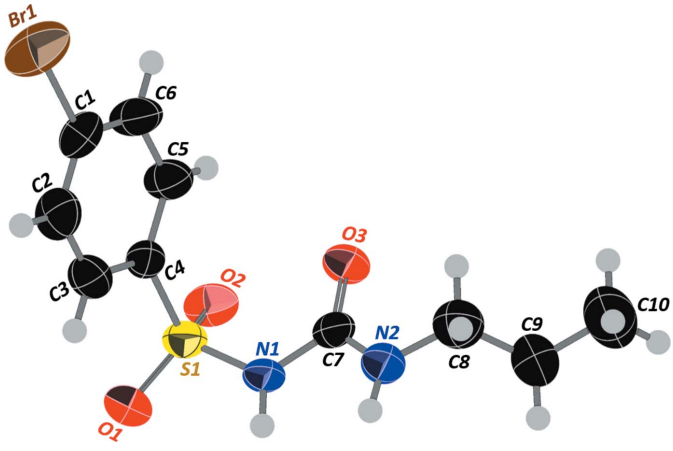


Figure 1
The molecular structure of 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide with atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

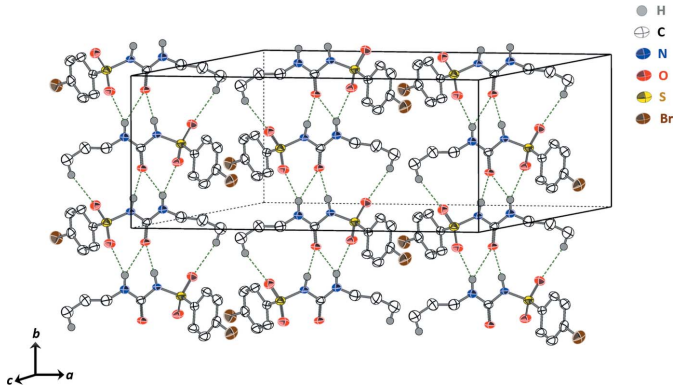


Figure 2
Infinite hydrogen-bonding involving the sulfonyl urea moiety in 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide. Hydrogen bonding between N1—H1···O2, N2—H2···O2, N2—H2···O3, and C10—H10*C*···O1 is shown as green dotted lines. Displacement ellipsoids are drawn at the 30% probability level. Only H atoms involved in hydrogen bonding are shown.

3. Supramolecular features

The crystal packing of the title compound is dominated by hydrogen bonding, which is shown in Fig. 2. Geometric details of the hydrogen bonds are listed in Table 1. Intermolecular

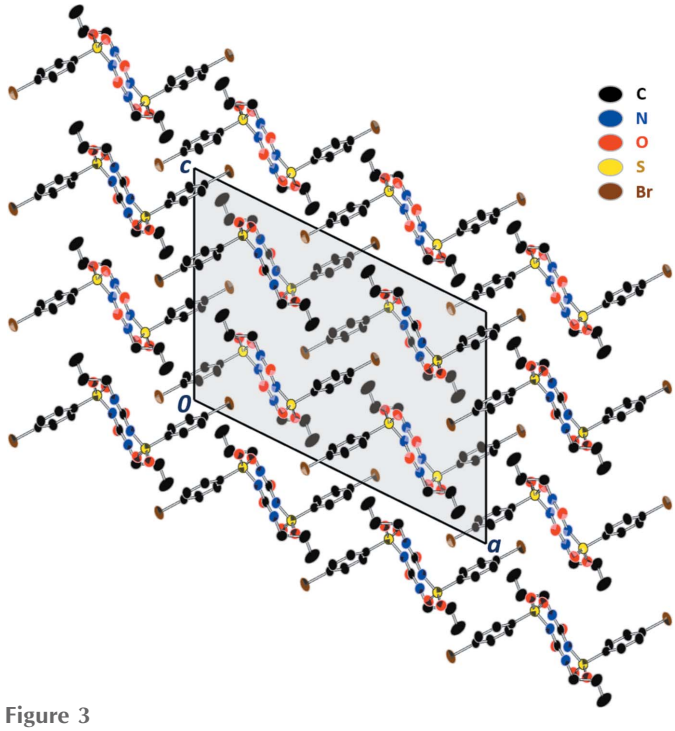


Figure 3
The crystal packing in 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide viewed along the *b* axis. Antiparallel stacking of the bromophenyl has a centroid-to-centroid distance of 4.213 Å. Displacement ellipsoids are drawn at the 30% probability level. H atoms are not shown for clarity.

N—H···O-type hydrogen bonds link the molecules into infinite chains, which stretch along the *b*-axis direction (Fig. 2). Hydrogen bonding between the H1 and O3 atoms of neighboring molecules have distances of H1···O3 = 1.94 Å, N1···O3 = 2.791 (3) Å. The strongest of these is N1—H1···O3, with an angle of 171.9°, followed in rank-order of strength by the hydrogen-bonds between H2···O2 = 2.24 Å, N2···O2 = 2.998 (3) Å (angle of 146.8° between N2—H2···O2) and H2···O3 = 2.642 Å, N2···O3 = 3.351 (3) Å (angle of 140.6° between N2—H2···O3). Additionally, weak C—H···O type hydrogen bonds also help, to some extent, with the molecular packing. The intermolecular distance between H10*C*···O1 is 2.61 Å; C10···O1 is 4.028 Å with an angle of 173.7° between C10—H10*C*···O1. The distances and angles of the C—H···O-type hydrogen bond observed in the present structure are within the reported ranges (Desiraju, 1991; Gumireddy *et al.*, 2021). Overall, the atoms involved in hydrogen bonding for bromopropamide are identical to those in the crystal structure for its analogue chlorpropamide (CSD refcode: BEDMIG10; Drebushchak *et al.*, 2009). Fig. 3 shows the unit cell of the title compound along the *b*-axis. It appears that the anti-parallel flanked phenyl rings are stacked. However, a centroid-to-centroid distance of 4.213 (2) Å, which is outside the range of π–π stacking interactions (Chulvi *et al.*, 2015; Ahmed *et al.*, 2019), supports its absence.

4. Hirshfeld surface analysis

Hirshfeld surface analysis was carried out using *CrystalExplorer17.5* (Turner *et al.*, 2017; Spackman *et al.*, 2021) mapped over d_{norm}, which was estimated by the calculations of the external and internal distances to the nearest nucleus and built over a volume of 322.24 Å^3 having an area of 304.35 Å^2, with scaled color of −0.6347 a.u. (red) to 1.2043 a.u. (blue). The Hirshfeld surface of **1**, shown in Fig. 4, displays close contacts between N1—H1···O3, N2—H2···O2, N2—H2···O3, and C10—H10*C*···O1, supporting the conclusions about hydrogen-bonding interactions. Hirshfeld surfaces and their associated two-dimensional fingerprint plots were used to quantify the various intermolecular interactions. The overall two-dimensional fingerprint plot for bromopropamide (Fig. 5*a*) and those delineated into major contacts: H···H, O···H/H···O, Br···H/H···Br, and C···H/H···C are shown in Fig. 5*b*–*e*. The other contacts have lower contributions, with individual contributions <4.3% and a sum <12.8%. The H···H interatomic contacts, which appear as a single spike in the center at $d_e = d_i$ = 1.1 Å (Fig. 5*b*), generated 39.4% of the Hirshfeld surface, denoting these contacts have a significant effect on the molecular packing. The O···H/H···O interatomic contacts, which appear as a pair of spikes with tips at $d_e + d_i \sim$1.75 Å (Fig. 5*c*), represent 25.8% of the total surface and confirms the prominent role of multiple hydrogen bonds in the molecular arrangement within the crystal structure. Br···H/H···Br and C···H/H···C contribute 12.2% and 9.8%, respectively, to the Hirshfeld surface. The placement of molecules in the crystal structure of the title compound results in efficient packing, as seen in the Hirshfeld surface analysis, which is further supported by the crystallographic density of

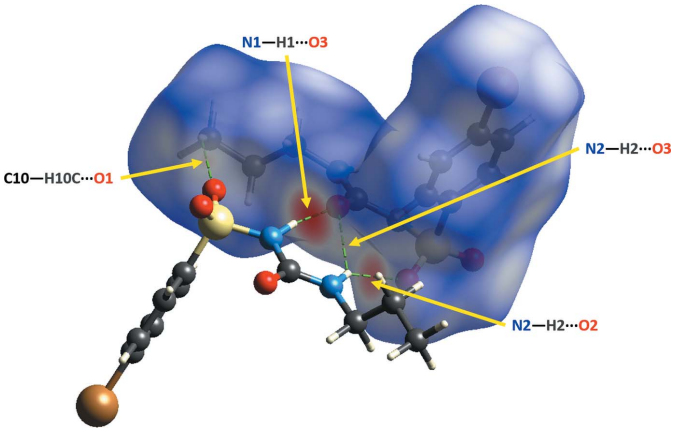


Figure 4
Hirshfeld surface of 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide mapped over d_{norm}, displays close contacts in the crystal. The non-covalent interactions indicated by the red spots are labeled.

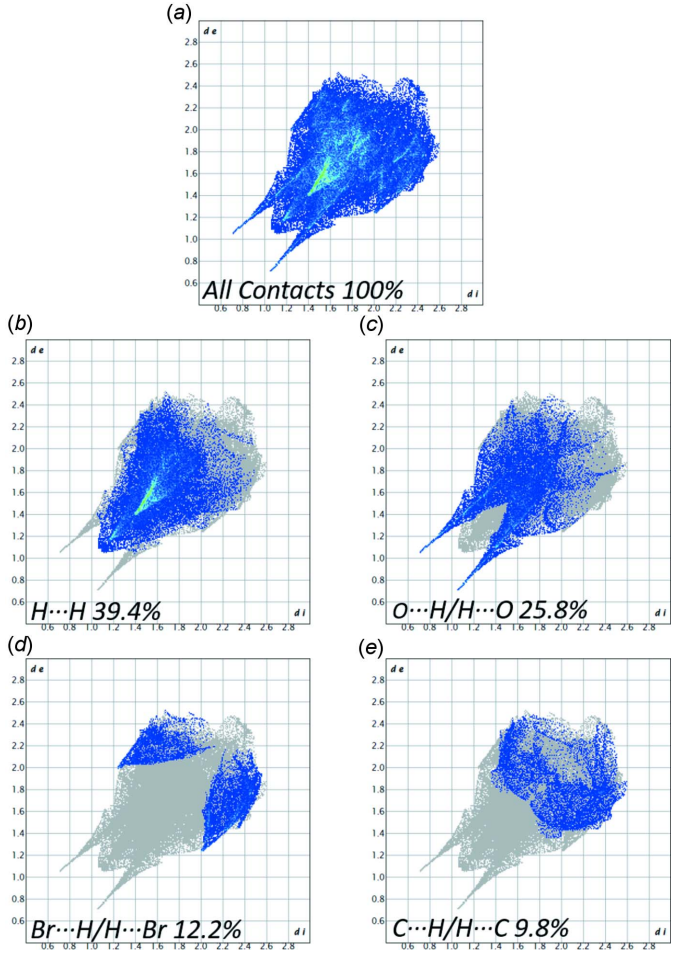


Figure 5
The two-dimensional fingerprint plots of 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide with their relative contribution to the Hirshfeld surface. The units of d_i and d_e are Å.

1.626 g cm^{-3}, which is relatively higher than other small molecule organic compounds (Bookwala *et al.*, 2020, 2022).

5. Database survey

A search in the Cambridge Structural Database (Version 5.41, update of March 2020; Groom *et al.*, 2016) for compounds possessing the sulfonyl urea substructure resulted in 178 hits, reinforcing the importance of this scaffold as having potential as an anti-diabetic or diuretic drug, and a herbicide. Of the 178 hits, 82 were distributed among chlorpropamide (deposited structures: 20), tolazamide (deposited structures: 40), and tolbutamide (deposited structures: 22), all of which share a close structural relationship to bromopropamide. The search was then narrowed to identify compounds containing *N*-(propylcarbamoyl)benzenesulfonamide, which resulted in identification of only chlorpropamide polymorphs, confirming the absence of reported crystal structures for analogues having different halogen substitutions. An exact search for the title compound resulted in zero hits, further supporting the previous claim. Thus, X–ray studies were important to identify, if any, changes in the crystal structure by replacing the peripheral Cl with a Br atom.

6. Synthesis and crystallization

The synthesis of 4-bromo-*N*-(*n*-propylcarbamoyl)benzenesulfonamide used *in situ* formation of *n*-propylisocyanate from *n*-propylcarbamic chloride with direct capture by 4-bromobenzenesulfonamide in the presence of excess potassium carbonate in refluxing toluene (Fig. 6). This is a new methodology to generate sulfonyl ureas in an atom-efficient manner with identical chemical characterization to prior methods proceeding *via* carbamate (Marshall & Sigal, 1958) or carbonate (Tanwar *et al.*, 2017) intermediates. A manuscript describing the optimization of this synthetic strategy is in preparation.

n-Propylcarbamic chloride (labeled **2** in Fig. 6): A solution of triphosgene (2.24 g, 22.62 mMol as phosgene) in 25 mL of dichloromethane (DCM) was cooled in a 100 mL round-bottom flask. A solution of triethylamine (TEA) (5.6 mL, 40 mMol), *n*-propylamine (labeled **1** in Fig. 6) (1.7 mL, 20.1 mMol) and 10 mL of DCM was added to the triphosgene solution with slow dropwise addition over 15 min maintaining an internal temperature between 278 and 283 K. The cooling bath was removed following addition and the reaction was permitted to stir for an additional 2 h at 296 K. The reaction mixture was cooled in an ice/water bath and then transferred to a 125 mL separatory funnel previously cooled in ice–water. The mixture was then washed with 3 × 5 mL portions of ice-cold water, 2 × 5 mL of ice-cold 0.5 *N* HCl, 2 × 5 mL portions of ice-cold brine, dried Na_2SO_4, decanted, and the solvent was carefully removed under reduced pressure without heating to theoretical mass. The conversion to *n*-propylcarbamic chloride was confirmed with IR absorbance of 1734 cm^{-1} and afforded 2.5 g (98% of a light yellow oil) and stored at 253 K until use.

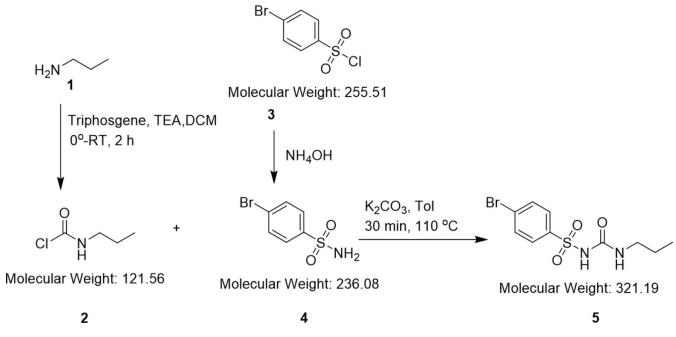


Figure 6
Reaction scheme for the synthesis of 4-bromo-*N*-(propylcarbamoyl)-benzenesulfonamide.

4-Bromobenzenesulfonamide (labeled **4** in Fig. 6): Synthesized using a variation of the published procedure (Anana *et al.*, 2006). Concentrated NH_4OH (150 mL, 1.10 mol) was charged into a 500 mL three-neck round-bottom flask equipped with an overhead stirrer, thermowell, and condenser. The reaction was then cooled in an ice/water bath to an internal temperature of 283 K. Solid 4-bromobenzenesulfonyl chloride (49.9954 g, 0.1957 mol) was added in portions over 5 min. The ice/water bath was removed and the mixture was stirred at room temperature for 15 min and then brought to 308 K for 30 min. After this, the reaction was warmed to reflux for an additional 30 min. The reaction was followed by thin-layer chromatography (TLC) [R_f = 0.69 (labeled **3** in Fig. 6), R_f = 0.54 (labeled **4** in Fig. 6) 1/1 hexane/ethyl acetate (H/EA), short wavelength ultra-violet (SWUV)]. The reaction was cooled to room temperature upon consumption of the starting material and then poured into 200 mL of ice-cold water. This heterogeneous mixture was brought to pH = 1 (pHydrion paper) with 6 *N* HCl. The precipitated white solid was collected on a #1 Whatman filter paper, pressed dry with a rubber dam, and dried 12 h in a drying pistol (P_2O_5, 150 mTorr, 383 K) to afford 43.03 g (93.5%) of a white solid. Proton identical with literature (Richardson *et al.*, 2007), m.p. 434–438 K (m.p. lit: 435 K).

4-Bromo-*N*-(*n*-propylcarbamoyl)benzenesulfonamide (labeled **5** in Fig. 6): *n*-Propylcarbamic chloride (labeled **2** in Fig. 6), (2.0 g, 15.9 mMol), toluene (15 mL), K_2CO_3, (2.019 g, 14.6 mMol), and 4-bromobenzenesulfonamide (labeled **4** in Fig. 6), (1.4306 g, 6.06 mMol) were added to a dry 100 mL round-bottom flask fitted with a straight condenser and brought to reflux for 30 min. Upon loss of the sulfonamide (TLC: R_f = 0.86, 1/1: H/EA SiO_2, SWUV, I_2), the heating was stopped, the oil bath was removed, and the reaction was permitted to cool to room temperature. The resulting white suspension was cooled in an ice/water bath and brought to a pH = 1 (pHydrion paper: red) with 6 *N* HCl. This mixture was extracted with 3 × 10 mL portions of EA, washed [3 × 5 mL 1 *N* HCl, then 2 × 5 mL NaCl (sat, aq.)], dried Mg_2SO_4, filtered under vacuum through #1 Whatman filter paper, and then the solvent was removed under reduced pressure to give 2.2 g of a white solid. This material was purified on a SiO_2 column (1/1: H/EA SiO_2, R_f = 0.66) then recrystallized from toluene to yield, after drying in a drying pistol at 383 K (P_2O_5,

150 µTorr), 0.87 g (41%) of fine white crystals. ^{1}H NMR was identical to prior synthesis (Tanwar *et al.*, 2017), m.p. 411 K (m.p. lit: 406–408 K).

Crystals obtained from toluene were very small; therefore, they were dissolved in methanol to obtain a supersaturated solution (37.5 mg mL^{-1}). This was placed in a 20 mL scintillation vial, which was covered with Parafilm® and punched with 5 pin holes to allow slow evaporation of methanol at room temperature over several days, until larger single crystals appeared.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically (aromatic C—H = 0.93 Å, amide N—H = 0.86 Å methylene C—H = 0.98 Å, and methyl C—H = 0.96 Å) and treated as riding atoms during refinement, with U_{iso} (H) = 1.2U_{eq}(aromatic C, amide N, and methylene C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate about their local threefold axes.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}BrN_2O_3S$
M_r	321.19
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	21.0939 (12), 9.2520 (6), 15.0283 (10)
β (°)	116.211 (4)
V (Å^3)	2631.4 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.28
Crystal size (mm)	0.25 × 0.12 × 0.05
Data collection	
Diffractometer	Bruker SMART APEXII
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.522, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15567, 2918, 1776
R_{int}	0.047
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.643
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.095, 1.01
No. of reflections	2918
No. of parameters	155
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.26

Computer programs: *SMART* and *SAINT* (Bruker, 1998), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), and *CrystalMaker* (Palmer, 2014).

References

Ahmed, M. N., Arif, M., Jabeen, F., Khan, H. A., Yasin, K. A., Tahir, M. N., Franconetti, A. & Frontera, A. (2019). *New J. Chem.* **43**, 8122–8131.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.

Anana, R., Rao, P. P. N., Chen, Q. H. & Knaus, E. E. (2006). *Bioorg. Med. Chem.* **14**, 5259–5265.

Arif Tawfeeq, N., Kwong, H. C., Mohamed Tahir, M. I. & Ravoof, T. B. S. A. (2019). *Acta Cryst.* E**75**, 774–779.

Bieszczad, B., Siwek, A., Wilczek, M., Trzybiński, D., Woźniak, K., Satała, G., Bojarski, A. J. & Mieczkowski, A. (2020). *Bioorg. Med. Chem. Lett.* **30**, Article 127493.

Bookwala, M., DeBoyace, K., Buckner, I. S. & Wildfong, P. L. D. (2020). *AAPS PharmSciTech*, **21**, https://doi.org/ 10.1208/s12249-020-1632-4.

Bookwala, M., Gumireddy, A., Aitken, J. A. & Wildfong, P. L. D. (2022). *J. Chem. Crystallogr.* **52**, 81–88.

Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chulvi, K., Costero, A., Ochando, L. E. & Gaviña, P. (2015). *Acta Cryst.* E**71**, o1069–o1070.

Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.

Drebushchak, T. N., Chesalov, Y. A. & Boldyreva, E. V. (2009). *Acta Cryst.* B**65**, 770–781.

Drebushchak, T. N., Chukanov, N. V. & Boldyreva, E. V. (2006). *Acta Cryst.* E**62**, o4393–o4395.

Drebushchak, T. N., Pankrushina, N. A. & Boldyreva, E. V. (2011). *Dokl. Phys. Chem.* **437**, 61–64.

Fedorov, A. Y., Rychkov, D. A., Losev, E. A., Zakharov, B. A., Stare, J. & Boldyreva, E. V. (2017). *CrystEngComm*, **19**, 2243–2252.

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

Gumireddy, A., DeBoyace, K., Rupprecht, A., Gupta, M., Patel, S., Flaherty, P. T. & Wildfong, P. L. D. (2021). *Acta Cryst.* E**77**, 360–365.

Khamees, H. A., Chaluvaiah, K., El-khatatneh, N. A., Swamynayaka, A., Chong, K. H., Dasappa, J. P. & Madegowda, M. (2019). *Acta Cryst.* E**75**, 1620–1626.

Kimura, K., Hirayama, F. & Uekama, K. (1999). *J. Pharm. Sci.* **88**, 385–391.

Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.

Marshall, F. & Sigal, J. M. (1958). *J. Org. Chem.* **23**, 927–929.

McLamore, W. M., Fanelli, G. M., P'an, S. Y. & Laubach, G. D. (1959). *Ann. N. Y. Acad. Sci.* **74**, 443–448.

Palmer, D. C. (2014). *CrystalMaker.* CrystalMaker Software Ltd, Begbroke, England.

Proks, P., Reimann, F., Green, N., Gribble, F. & Ashcroft, F. (2002). *Diabetes*, **51**, S368–S376.

Richardson, C. M., Nunns, C. L., Williamson, D. S., Parratt, M. J., Dokurno, P., Howes, R., Borgognoni, J., Drysdale, M. J., Finch, H., Hubbard, R. E., Jackson, P. S., Kierstan, P., Lentzen, G., Moore, J. D., Murray, J. B., Simmonite, H., Surgenor, A. E. & Torrance, C. J. (2007). *Bioorg. Med. Chem. Lett.* **17**, 3880–3885.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

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

Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.

Tanwar, D. K., Ratan, A. & Gill, M. S. (2017). *Org. Biomol. Chem.* **15**, 4992–4999.

Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *CrystalExplorer.* Version 17. University of Western Australia.

supporting information

Acta Cryst. (2022). E**78**, 485-489 [https://doi.org/10.1107/S2056989022003723]

Crystal structure of 4-bromo-*N*-(propylcarbamoyl)benzenesulfonamide

Mustafa Bookwala, Saloni Patel, Patrick T. Flaherty and Peter L. D. Wildfong

Computing details

Data collection: *SMART* and *SAINT* (Bruker, 1998); cell refinement: *SMART* and *SAINT* (Bruker, 1998); data reduction: *SMART* and *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *CrystalMaker* (Palmer, 2014); software used to prepare material for publication: *SHELXL2018/3* (Sheldrick, 2015).

4-Bromo-*N*-(propylcarbamoyl)benzenesulfonamide

Crystal data

$C_{10}H_{13}BrN_2O_3S$
M_r = 321.19
Monoclinic, *C*2/*c*
a = 21.0939 (12) Å
b = 9.2520 (6) Å
c = 15.0283 (10) Å
β = 116.211 (4)°
V = 2631.4 (3) Å^3
Z = 8
F(000) = 1296
D_x = 1.622 Mg m^{-3}
Melting point: 411 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2918 reflections
θ = 2.2–27.2°
μ = 3.28 mm^{-1}
T = 296 K
Rectangular Plate, colorless
0.25 × 0.12 × 0.05 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: Fine-focus Sealed Tube
φ and *ω* Scans scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.522, T_{max} = 0.746
15567 measured reflections
2918 independent reflections
1776 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.2°, θ_{min} = 2.2°
h = −26→27
k = −11→11
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.095
S = 1.01
2918 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.7765P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 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}
Br1	0.62045 (2)	0.74137 (4)	0.56073 (3)	0.0982 (2)
S1	0.33290 (3)	0.91269 (7)	0.18667 (5)	0.0519 (2)
O1	0.34641 (11)	1.0325 (2)	0.13838 (14)	0.0677 (5)
N1	0.28060 (11)	0.9785 (2)	0.23075 (16)	0.0525 (6)
H1	0.267634	1.067206	0.217972	0.063*
C1	0.53543 (14)	0.7926 (3)	0.4505 (2)	0.0576 (7)
O2	0.30317 (11)	0.7841 (2)	0.13234 (14)	0.0629 (5)
N2	0.22679 (11)	0.9777 (2)	0.33323 (17)	0.0579 (6)
H2	0.223806	1.069690	0.323849	0.069*
C7	0.25586 (12)	0.8997 (3)	0.2876 (2)	0.0486 (6)
C2	0.52538 (15)	0.9312 (3)	0.4162 (2)	0.0664 (8)
H2A	0.560352	1.000298	0.446974	0.080*
O3	0.26161 (11)	0.76750 (17)	0.29256 (16)	0.0612 (5)
C3	0.46344 (14)	0.9687 (3)	0.3360 (2)	0.0599 (7)
H3	0.456463	1.062947	0.312127	0.072*
C4	0.41168 (13)	0.8659 (3)	0.29115 (18)	0.0460 (6)
C5	0.42201 (15)	0.7269 (3)	0.3267 (2)	0.0608 (8)
H5	0.386886	0.657922	0.296706	0.073*
C6	0.48436 (15)	0.6894 (3)	0.4068 (2)	0.0658 (8)
H6	0.491670	0.595259	0.430921	0.079*
C8	0.19943 (17)	0.9163 (4)	0.3988 (2)	0.0726 (9)
H8A	0.215639	0.974924	0.458349	0.087*
H8B	0.218898	0.820113	0.418427	0.087*
C9	0.12066 (19)	0.9070 (4)	0.3534 (3)	0.0960 (12)
H9A	0.103998	0.849364	0.293370	0.115*
H9B	0.100819	1.003157	0.335194	0.115*
C10	0.0955 (3)	0.8407 (5)	0.4230 (4)	0.1239 (17)
H10A	0.044787	0.836946	0.391608	0.186*
H10B	0.111388	0.898207	0.482141	0.186*
H10C	0.114123	0.744633	0.439896	0.186*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0584 (2)	0.1095 (4)	0.0856 (3)	0.01372 (19)	−0.00560 (18)	−0.0048 (2)
S1	0.0493 (4)	0.0394 (4)	0.0605 (4)	0.0027 (3)	0.0184 (3)	0.0051 (3)
O1	0.0721 (13)	0.0571 (12)	0.0766 (13)	0.0052 (10)	0.0354 (11)	0.0223 (11)
N1	0.0505 (12)	0.0283 (10)	0.0788 (16)	0.0045 (10)	0.0285 (12)	0.0093 (11)
C1	0.0395 (14)	0.070 (2)	0.0567 (16)	0.0047 (14)	0.0149 (13)	−0.0058 (15)

O2	0.0633 (11)	0.0485 (11)	0.0599 (11)	0.0019 (10)	0.0118 (10)	−0.0062 (9)
N2	0.0567 (14)	0.0405 (12)	0.0777 (16)	0.0001 (11)	0.0308 (13)	0.0032 (12)
C7	0.0330 (12)	0.0342 (14)	0.0648 (16)	−0.0019 (11)	0.0091 (12)	0.0005 (13)
C2	0.0507 (16)	0.064 (2)	0.075 (2)	−0.0164 (15)	0.0188 (15)	−0.0123 (17)
O3	0.0663 (13)	0.0297 (10)	0.0885 (14)	−0.0019 (9)	0.0349 (11)	0.0027 (9)
C3	0.0571 (17)	0.0459 (16)	0.0736 (19)	−0.0083 (14)	0.0262 (16)	0.0022 (15)
C4	0.0442 (13)	0.0360 (14)	0.0596 (16)	0.0011 (11)	0.0245 (12)	0.0003 (12)
C5	0.0492 (16)	0.0419 (16)	0.0746 (19)	−0.0030 (13)	0.0122 (14)	0.0007 (14)
C6	0.0560 (17)	0.0503 (16)	0.0736 (19)	0.0099 (15)	0.0125 (15)	0.0091 (16)
C8	0.077 (2)	0.066 (2)	0.077 (2)	0.0027 (18)	0.0361 (18)	0.0017 (18)
C9	0.089 (3)	0.084 (3)	0.133 (3)	0.004 (2)	0.066 (3)	0.025 (2)
C10	0.153 (4)	0.078 (3)	0.201 (5)	0.008 (3)	0.133 (4)	0.013 (3)

Geometric parameters (Å, º)

Br1—C1	1.887 (3)	C3—C4	1.378 (4)
S1—O1	1.4207 (19)	C3—H3	0.9300
S1—O2	1.4225 (19)	C4—C5	1.372 (3)
S1—N1	1.634 (2)	C5—C6	1.379 (4)
S1—C4	1.763 (3)	C5—H5	0.9300
N1—C7	1.388 (3)	C6—H6	0.9300
N1—H1	0.8600	C8—C9	1.494 (4)
C1—C2	1.363 (4)	C8—H8A	0.9700
C1—C6	1.371 (4)	C8—H8B	0.9700
N2—C7	1.319 (3)	C9—C10	1.498 (5)
N2—C8	1.460 (4)	C9—H9A	0.9700
N2—H2	0.8600	C9—H9B	0.9700
C7—O3	1.228 (3)	C10—H10A	0.9600
C2—C3	1.374 (4)	C10—H10B	0.9600
C2—H2A	0.9300	C10—H10C	0.9600
O1—S1—O2	119.76 (13)	C3—C4—S1	119.8 (2)
O1—S1—N1	103.90 (12)	C4—C5—C6	120.1 (3)
O2—S1—N1	109.80 (12)	C4—C5—H5	119.9
O1—S1—C4	108.87 (12)	C6—C5—H5	119.9
O2—S1—C4	108.00 (12)	C1—C6—C5	119.2 (3)
N1—S1—C4	105.65 (11)	C1—C6—H6	120.4
C7—N1—S1	123.94 (17)	C5—C6—H6	120.4
C7—N1—H1	118.0	N2—C8—C9	113.9 (3)
S1—N1—H1	118.0	N2—C8—H8A	108.8
C2—C1—C6	121.1 (3)	C9—C8—H8A	108.8
C2—C1—Br1	119.8 (2)	N2—C8—H8B	108.8
C6—C1—Br1	119.1 (2)	C9—C8—H8B	108.8
C7—N2—C8	123.5 (2)	H8A—C8—H8B	107.7
C7—N2—H2	118.3	C8—C9—C10	111.7 (3)
C8—N2—H2	118.3	C8—C9—H9A	109.3
O3—C7—N2	124.7 (3)	C10—C9—H9A	109.3
O3—C7—N1	120.4 (3)	C8—C9—H9B	109.3

N2—C7—N1	114.8 (2)	C10—C9—H9B	109.3
C1—C2—C3	119.8 (3)	H9A—C9—H9B	107.9
C1—C2—H2A	120.1	C9—C10—H10A	109.5
C3—C2—H2A	120.1	C9—C10—H10B	109.5
C2—C3—C4	119.7 (3)	H10A—C10—H10B	109.5
C2—C3—H3	120.1	C9—C10—H10C	109.5
C4—C3—H3	120.1	H10A—C10—H10C	109.5
C5—C4—C3	120.0 (2)	H10B—C10—H10C	109.5
C5—C4—S1	120.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.86	1.94	2.791 (3)	172
N2—H2···O2^{i}	0.86	2.24	2.998 (3)	147
N2—H2···O3^{i}	0.86	2.64	3.351 (3)	141

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