Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Synthesis and absolute structure of (*R*)-2-(benzylselanyl)-1-phenylethanaminium hydrogen sulfate monohydrate: crystal structure and Hirshfeld surface analyses

H. R. Rajegowda,[a]‡ P. A. Suchetan,[a]‡ R. J. Butcher[b] and P. Raghavendra Kumar[a]*

[a]Department of Chemistry, University College of Science, Tumkur University, Tumkur-572 103, Karnataka, India, and [b]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC, 20059, USA. *Correspondence e-mail: raghukp1@gmail.com

Received 26 July 2021
Accepted 7 October 2021

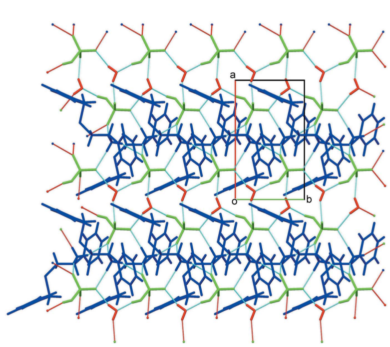
‡ These authors contributed equally.

Keywords: crystal structure; chiral; absolute structure; selenium; SBIs; Hirshfeld surface.

CCDC reference: 2114403

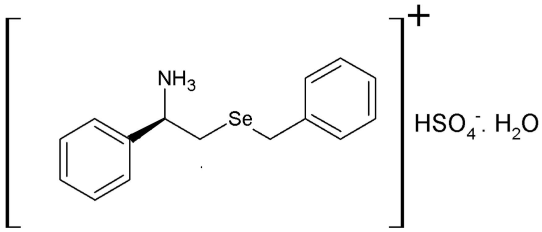
Supporting information: this article has supporting information at journals.iucr.org/e

A hydrogen sulfate salt, $C_{15}H_{18}NSe^+ \cdot HSO_4^- \cdot H_2O$ or $[BnSeCH_2CH(Ph)NH_3^+]$-(HSO_4^-), of a chiral selenated amine (*R*)-2-(benzylselanyl)-1-phenylethanamine ($BnSeCH_2CH(Ph)NH_2$) has been synthesized and characterized by elemental analysis, ^{1}H and $^{13}C\{^1H\}$ NMR, FT–IR analysis, and single-crystal X-ray diffraction studies. The title salt crystallizes in the monohydrate form in the non-centrosymmetric monoclinic $P2_1$ space group. The cation is somewhat W shaped with the dihedral angle between the two aromatic rings being 60.9 (4)°. The carbon atom attached to the amine nitrogen atom is chiral and in the *R* configuration, and, the –C—C– bond of the $–CH_2—CH–$ fragment has a staggered conformation. In the crystal structure, two HSO_4^- anions and two water molecules form an $R_4^4(12)$ tetrameric type of assembly comprised of alternating HSO_4^- anions and water molecules *via* discrete $D(2)$ O—H···O hydrogen bonds. This tetrameric assembly aggregates along the *b*-axis direction as an infinite one-dimensional tape. Adjacent tapes are interconnected *via* discrete $D(2)$ N—H···O hydrogen bonds between the three amino hydrogen atoms of the cation sandwiched between the two tapes and the three HSO_4^- anions of the nearest asymmetric units, resulting in a complex two-dimensional sheet along the *ab* plane. The pendant arrangement of the cations is stabilized by C—H···π interactions between adjacent cations running as chains down the [010] axis. Secondary Se···O [3.1474 (4) Å] interactions are also observed in the crystal structure. A Hirshfeld surface analysis, including d_{norm}, shape-index and fingerprint plots of the cation, anion and solvent molecule, was carried out to confirm the presence of various interactions in the crystal structure.



1. Chemical context

Selenium is an important bio-element (Schwarz *et al.*, 1957; Papp *et al.*, 2007). The hypervalent nature of selenium results in interesting secondary bonding interactions (SBIs), also known as non-bonded interactions, in organoselenium compounds (Musher *et al.*, 1969; Raghavendra Kumar *et al.*, 2006; Chivers & Laitinen, 2015; Bleiholder *et al.*, 2006). These structural aspects are worth exploring as weak SBIs (Iwaoka *et al.*, 2001, 2002*a*,*b*) in the compounds of heavy chalcogens (Se and Te) are ascribed important roles in structural chemistry, such as the stabilization of otherwise unstable organo-chalcogen compounds and supramolecular associations (Tiekink & Zukerman-Schpector, 2010; Werz *et al.*, 2002) and possessing biological activities (Reich *et al.*, 2016; Bartolini *et al.*, 2017; Engman *et al.*,1992; Mukherjee *et al.*, 2010). Some organoselenated alkyl/arylamines have been synthesized (Singh & Srivastava, 1990; Srivastava *et al.*, 1994; Revanna *et al.*, 2015), but further investigations on their single-crystal X-ray structures, especially of chiral derivatives, are limited (Musher *et al.*,1969; Raghavendra Kumar *et al.*, 2006; Chivers & Laitinen, 2015; Bleiholder *et al.*, 2006, Prabhu Kumar *et al.*, 2019). Therefore, the synthesis and discussions on the single-crystal structural features of (*R*)-2-(benzylselanyl)-1-phenylethanaminium hydrogen sulfate monohydrate, [BnSeCH$_2$CH(Ph)NH$_3$$^+$](HSO$_4$$^-$), are the subject of the present paper.



2. Structural commentary

The title salt (Fig. 1) is formed by the transfer of a proton from sulfuric acid to the chiral selenated amine $C_{15}H_{17}SeN$. The asymmetric unit of the structure consists of one $(C_{15}H_{18}SeN)^+$ cation, one HSO_4^- anion and a solvent water molecule with no direct hydrogen-bonding interactions between them. In the HSO_4^- ion, three of the S—O bond lengths are almost the same, falling in the range of 1.447 (4)–1.452 (5) Å, while the fourth is slightly elongated at 1.527 (5) Å. This suggests that the three nearly identical S—O bonds have partial double-bond character owing to resonance, while the fourth S—O bond has single-bond character. This validates the formation of the salt *via* single proton transfer from sulfuric acid to the amine. The title salt crystallizes in the monohydrate form in the non-centrosymmetric monoclinic $P2_1$ space group. The cation is somewhat W shaped (Fig. 1) with the dihedral angle between the two aromatic rings being 60.9 (4)°. The carbon atom attached to the amine nitrogen atom is a chiral atom with an *R* configuration and the –C—C– bond of the –CH$_2$—CH– fragment has a staggered conformation.

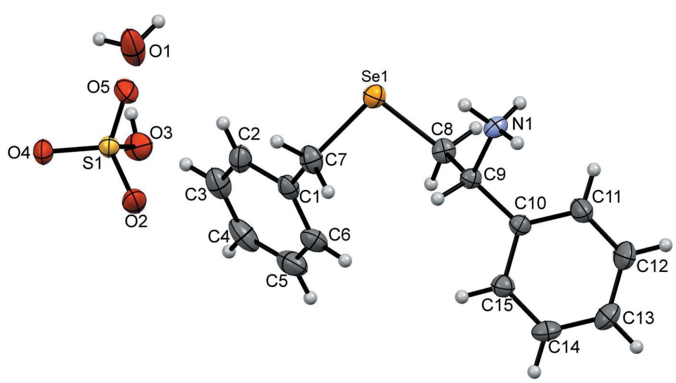


Figure 1
A view of the molecular structure of the title salt, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

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

Cg is the centroid of the C1–C6 aromatic ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.89	2.16	3.003 (7)	157
N1—H1B···O4^{ii}	0.89	2.05	2.893 (6)	159
N1—H1C···O2^{iii}	0.89	1.92	2.812 (6)	176
O1—H1D···O2^{iv}	0.85	1.91	2.726 (8)	161
O1—H1E···O5^{v}	0.85	1.95	2.730 (6)	152
O3—H3A···O1	0.82	1.68	2.483 (9)	167
C15—H15···Cg^{vi}	0.93	2.75	3.547 (7)	144

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

3. Supramolecular features

The crystal structure features, by virtue of its salt form, several strong-to-moderate hydrogen bonds, which are not seen to the same extent in the reported freebase structure of the closely related compound (*S*)-1-(benzylselanyl)-3-phenylpropan-2-amine (Prabhu Kumar *et al.*, 2019). The general rule that all strong hydrogen-bond donors participate in hydrogen bonding with strong hydrogen-bond acceptors is totally satisfied in this salt, with all the strong donors and acceptors in the cation, anion and the solvent being involved in at least one hydrogen bond. In the crystal structure, two HSO_4^- anions and two water molecules are interconnected to form a tetrameric type of assembly comprising of alternating HSO_4^- anions and water molecules *via* discrete $D(2)$ O1—H1D···O2, O1—H1E···O5 and O3—H3A···O1 hydrogen bonds (Fig. 2, Table 1), with the O1—H1E···O5 hydrogen bond appearing twice. This tetrameric type of assembly having a $R_4^4(12)$ graph-set notation aggregates along the *b*-axis direction as an infinite one dimensional tape, with adjacent tetrameric units in the tape glued to each other through the common O1—H1E···O5 hydrogen bonds (Fig. 2). The O1—H1D···O2 and O1—H1E···O5 hydrogen bonds have structure-directing features along the [010] axis. Adjacent tapes, which are 5.2133 (4) Å

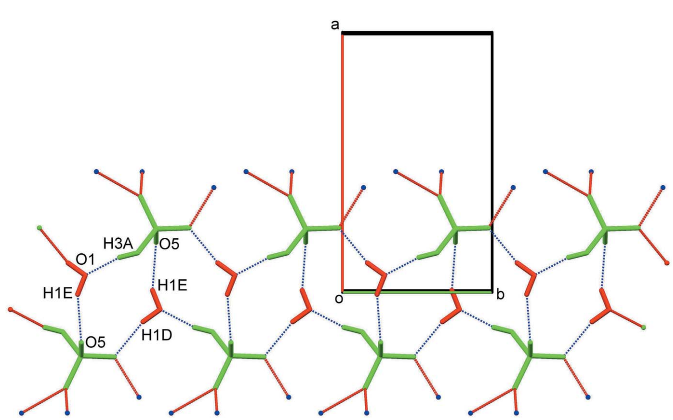


Figure 2
A partial view along the *c* axis of the crystal packing of the title salt, showing the propagation of the one-dimensional tape along the *b*-axis direction. The various intermolecular interactions (Table 1) are shown as dashed lines. Colour key: green, anions; red, water; blue spheres, cations.

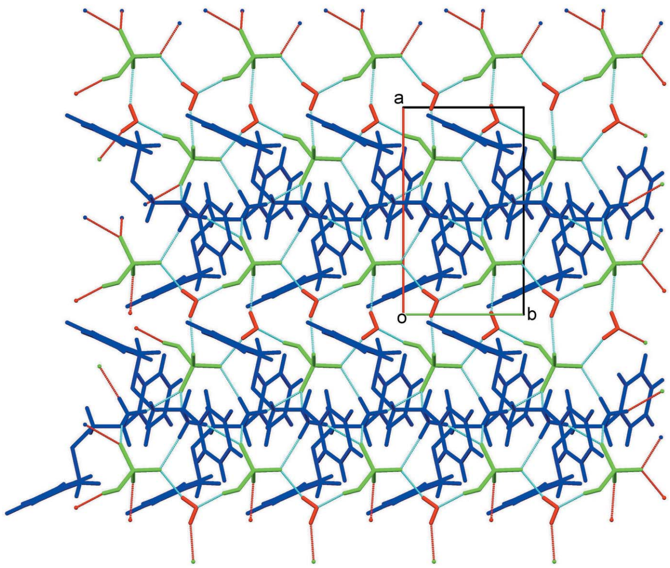


Figure 3
A partial view along the c axis of the crystal packing of the title salt, showing the formation of a two-dimensional sheet along the ab plane. The various intermolecular interactions (Table 1) are shown as dashed lines. Colour key: green, anions; red, water; blue, cations.

apart (*i.e.* half of the unit-cell length a) along the a axis, are interconnected *via* discrete $D(2)$ N1—H1A···O4, N1—H1B···O4 and N1—H1C···O2 hydrogen bonds (Fig. 3, Table 1) between the three amino hydrogen atoms of the cation sandwiched between the two tapes and the three HSO_4^- anions of the nearest asymmetric units (two HSO_4^- anions belong to one tape and two to the other), resulting in a complex two-dimensional sheet along the ab plane (Fig. 3). The cations serve as pendants to the complex sheet. The N1—H1A···O4, N1—H1B···O4 and N1—H1C···O2 interactions are not structure-directing hydrogen bonds of themselves, but structure-directional characteristics are induced to them *via* the O1—H1D···O2 and O1—H1E···O5 hydrogen bonds. The pendant-type arrangement of cations is stabilized by C15—H15···π (π electrons of the C1–C6 ring) interactions between adjacent cations running as chains down the [010] axis. Secondary Se1···O4($x - 1$, y, z) [3.1474 (4) Å] interactions are also observed in the crystal structure.

4. Hirshfeld surface analyses

The Hirshfeld surfaces including d_{norm} and shape-index and fingerprint (FP) analyses of the cation, anion and the solvent are shown in Figs. 4 and 5. In the d_{norm} surface of the cation (highlighting O···H/H···O contacts only; Fig. 4a), dark-red spots in the proximity of three amino hydrogen atoms are a result of strong N1—H1A···O4, N1—H1B···O4 and N1—H1C···O2 hydrogen bonds between the cation and HSO_4^- anions. Further, the Hirshfeld surface of the cation mapped over shape-index (highlighting C···H/H···C contacts only; Fig. 4b) shows a dark-red spot close to the centroid of the C1–C6 ring facing the H15 hydrogen atom, which is due to the C15—H15···π (π electrons of the C1–C6 ring) interactions

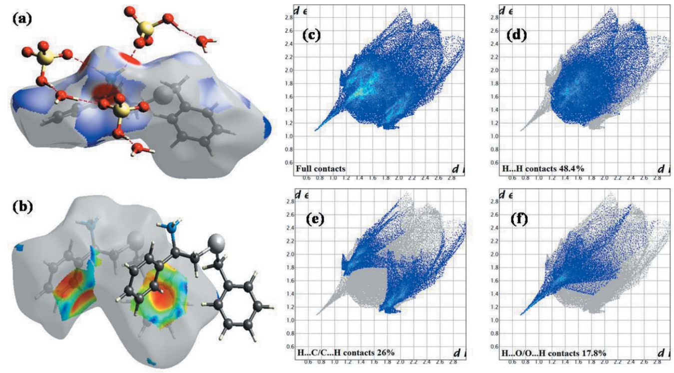


Figure 4
Hirshfeld surfaces comprising (a) d_{norm} surface, (b) shape-index and (c)–(f) fingerprint plots of the cation.

observed between adjacent cations. The overall FP plot and those decomposed to individual atom···atom contacts contributing to the Hirshfeld surfaces of the cation are shown in Fig. 4c, 4d , 4e and 4f, respectively. The highest contribution to the Hirshfeld surface is from H···H dispersions, which contribute 48.4%, followed by C···H/H···C (26%), O···H/H···O (17.8%), Se···H/H···Se (5.7%) and others (2.1%). The symmetry about the $d_i = d_e$ axis passing through the origin observed in the FP plots for the H···H and C···H/H···C contacts suggests that these interactions exist only between the cationic species and not between cation–anion or cation–water. The asymmetric nature of the FP of the O···H/H···O contacts about the $d_i = d_e$ axis suggests that the O···H interactions are between unlike species, which is in agreement with the observed N—H···O interactions between cations and anions. A single spike observed in the FP of O···H/H···O contacts is characteristic of a strong or a moderate hydrogen bond. The spike observed at $d_i + d_e \sim 1.9$ Å is very close to the H1C···O2 distance of 1.92 Å (Table 1), thus supporting the participation of the cations in various N—H···O hydrogen bonds. Two blunt spikes (a characteristic of a weak interaction between like species) observed in the FP of C···H/H···C contacts at $d_i + d_e \sim 2.8$ Å is very close to the H15···Cg distance of 2.75 Å (Table 1), thereby confirming the presence

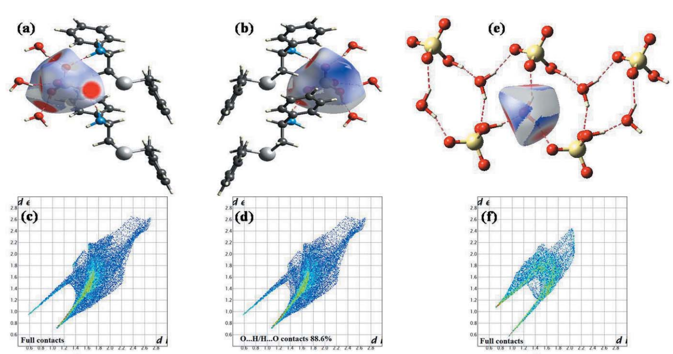


Figure 5
Hirshfeld surfaces: (a) and (b) two different views of the d_{norm} surface of the anion, (c) and (d) fingerprint plots of the anion, (e) d_{norm} surface and (f) fingerprint plot of the water molecule.

of C—H⋯π interactions between the cations. Thus, the Hirshfeld surface analysis provides adequate and reliable evidence, both qualitatively (in terms of pictorial depiction) and quantitatively, for the various interactions in which the cations participate. Analysis of the Hirshfeld surfaces of the anion and the solvent molecule gives similar results (Fig. 5). In the case of the anion, the highest contribution to the Hirshfeld surface is from O⋯H/H⋯O contacts, contributing 88.6%, while for the Hirshfeld surface of water, 61.6% is from O⋯H/H⋯O contacts and the remaining 38.4% is from H⋯H dispersions.

5. Database survey

The cation of the reported structure is somewhat similar to that observed in a closely related structure, (*S*)-1-(benzylselanyl)-3-phenylpropan-2-amine (Prabhu Kumar *et al.*, 2019), which is homologous to the cation of the title salt with one additional –CH_2– group between the chiral carbon atom and its nearest aromatic ring. The configurations of the chiral carbon atom are different in the two structures. The dihedral angle between the aromatic rings in the related molecule is 66.49 (12)°, which is very similar to that observed in the title structure. No intramolecular N—H⋯Se interaction is observed in the molecular cation of the present structure, unlike in the related molecule where one is observed. In the crystal of the related amine, the molecules are linked by weak N—H⋯N interactions, generating chains along the [100] direction.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{18}NSe^{+}\cdot HSO_4^{-}\cdot H_2O$
M_r	406.35
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	293
a, *b*, *c* (Å)	10.4266 (4), 6.0539 (2), 14.2168 (7)
β (°)	90.261 (4)
V (Å^3)	897.38 (6)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	2.23
Crystal size (mm)	0.22 × 0.18 × 0.16
Data collection	
Diffractometer	Bruker APEXII CCD area
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.624, 0.700
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4255, 3088, 2624
R_{int}	0.035
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.044, 0.113, 0.99
No. of reflections	3088
No. of parameters	213
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.48
Absolute structure	Flack *x* determined using 665 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.002 (16)

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXT2016/4* (Sheldrick, 2015*a*), *SHELXL2016/4* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2020).

6. Synthesis and crystallization

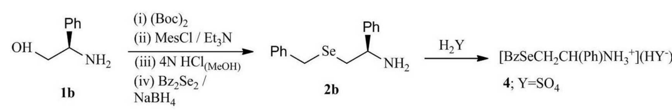
6.1. Materials and methods

Chemical reagents were purchased from Sigma–Aldrich (India) and used without further purification unless stated otherwise. For chemical synthesis, reactions were carried out in distilled water or in laboratory-grade solvents at room temperature. Melting points were determined in capillary tubes closed at one end and were reported uncorrected. IR spectra were recorded on a Jasco FT–IR-4100 spectrometer. Specific optical rotations (SOR) were measured on a Rudolph Autopol-I automatic polarimeter using a cell of 100 mm path length. ^{1}H and ^{13}C{^{1}H} NMR spectra were recorded on an AVANCE-II Bruker 400 MHz spectrometer. (*R*)-1-(Benzylselanyl)-2-phenylethan-2-amine was synthesized according to our reported literature procedure (Revanna *et al.*, 2015).

6.2. Synthesis of (1*R*)-2-(benzylselanyl)-1-phenylethan-1-ammoniumhydrogensulfate

The chiral selenated amine (*R*)-2-(benzylselanyl)-1-phenylethanamine was synthesized by a sequence of reactions shown in the reaction scheme starting from (2*R*)-2-amino-2-phenylethan-1-ol [derived from amino acid (*R*)-phenylglycinal] as per the literature procedure (Revanna *et al.*, 2015). The title salt of the above amine was obtained by treating it with sulfuric acid (5 *M*) in methanol under ice-cold conditions.

To an ice-cold methanolic (5 mL) solution of (2*R*)-1-(benzylselanyl)-2-phenylethan-2-amine (0.291 g, 1 mmol) was added 5 *M* of H_2SO_4 (2 mL) under stirring. The resulting precipitate was stirred for a further hour at the same temperature. Then the precipitate was filtered and washed twice with cold methanol (10 mL × 2). The white solid obtained was recrystallized from hot methanol (10 mL), which afforded colourless crystals of the title salt. The salt is soluble in water, dimethyl formamide (DMF) and dimethyl sulfoxide (DMSO), but insoluble in methanol, chloroform, dichloromethane, ether, tetrahydrofuran (THF) and hydrocarbon solvents such as *n*-hexane, benzene and toluene.



Yield: 92%; m.p. 469–472 K; (*c* 1.0 in MeOH). Elemental analysis: found C, 46.51; H, 4.88; N, 3.54. Calculated for $C_{15}H_{19}NO_4SSe$: C, 46.39; H, 4.93; N, 3.61%. FT–IR (KBr, ν cm^{-1}): 3452, 3027, 2925, 1615, 1537, 1361, 1186, 699, 556, 477; ^{1}H NMR (DMSO-d_6, 400.233 MHz, δ ppm): 2.867–3.060 (*dd*, 2H, *J* = 9.2, 6.0 Hz, CH_2Se), 3.648 (*s*, 2H, $SeCH_2$), 4.329–4.351 (*m*, 1H, CH), 7.166–7.288 (*m*, 5H, ArH), 7.373–7.440 (*m*, 5H, ArH), 8.412 (*bs*, 3H, NH_3);^{13}C{^{1}H} NMR (DMSO-d_6, 100.638 MHz, δ ppm): 26.99 (CH_2Se), 27.15 ($SeCH_2$), 54.75

(CH), 126.89 (C-7), 127.73 (C-13), 128.63 (C-11, C-15), 128.89 (C-6, C-8), 128.99 (C-12, C-14), 129.09 (C-5, C-9), 137.11 (C-4), 139.25 (C-10).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound H atoms were positioned with idealized geometry and refined using a riding model: C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms, C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene H atoms and C—H = 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methine H atoms. The amino H atoms and O-bound H atoms were also positioned geometrically and refined as riding: N—H = 0.89 Å with $U_{iso}(H) = 1.2U_{eq}(N)$; O_{water}—H = 0.85 Å with $U_{iso}(H) = 1.5U_{eq}(O_{water})$; O_{anion}—H = 0.82 Å with $U_{iso}(H) = 1.5U_{eq}(O_{anion})$.

Funding information

PRK thanks the Department of Science and Technology–SERB, New Delhi, India, for financial support in the form of project No. DST/SR/S1/IC-76/2010(G).

References

Bartolini, D., Sancineto, L., Bem, A. F. D., Tew, K. D., Santi, C., Radi, R., Toquato, P. & Galli, F. (2017). *Selenocompounds in Cancer Therapy: An Overview Advances in Cancer Research.* Amsterdam: Elsevier.
Bleiholder, C., Werz, D. B., Köppel, H. & Gleiter, R. (2006). *J. Am. Chem. Soc.* **128**, 2666–2674.
Bruker (2009). *APEX2*, *SAINT-Plus*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chivers, T. & Laitinen, R. S. (2015). *Chem. Soc. Rev.* **44**, 1725–1739.
Engman, L., Stern, D., Cotgreave, I. A. & Andersson, C. M. (1992). *J. Am. Chem. Soc.* **114**, 9737–9743.
Iwaoka, M., Takemoto, S., Okada, M. & Tomoda, S. (2001). *Chem. Lett.* **30**, 132–133.
Iwaoka, M., Takemoto, S., Okada, M. & Tomoda, S. (2002*a*). *Bull. Chem. Soc. Jpn*, **75**, 1611–1625.
Iwaoka, M., Takemoto, S. & Tomoda, S. (2002*b*). *J. Am. Chem. Soc.* **124**, 10613–10620.
Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
Mukherjee, A. J., Zade, S. S., Singh, H. B. & Sunoj, R. B. (2010). *Chem. Rev.* **110**, 4357–4416.
Musher, J. I. (1969). *Angew. Chem. Int. Ed. Engl.* **8**, 54–68.
Papp, L. V., Lu, J., Holmgren, A. & Khanna, K. K. (2007). *Antioxid. & Redox Signal.* **9**, 775–806.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Prabhu Kumar, K. M., Palakshamurthy, B. S., Jasinski, J. P., Butcher, R. J. & Raghavendra Kumar, P. (2019). *IUCrData*, **4**, x191029.
Raghavendra Kumar, P., Upreti, S. & Singh, A. K. (2006). *Inorg. Chim. Acta*, **359**, 4619–4626.
Reich, H. J. & Hondal, R. J. (2016). ACS *Chem. Biol.* 11, 821–841.
Revanna, R. H., Kumar, P. R., Hosamani, A. & Siddagangaiah, P. B. (2015). *J. Organomet. Chem.* **799–800**, 61–69.
Schwarz, K. & Foltz, C. M. (1957). *J. Am. Chem. Soc.* **79**, 3292–3293.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Singh, A. K. & Srivastava, V. (1990). *Phosphorus Sulfur Silicon*, **47**, 471–475.
Srivastava, V., Batheja, R. & Singh, A. K. (1994). *J. Organomet. Chem.* **484**, 93–96.
Tiekink, E. R. T. & Zukerman-Schpector, J. (2010). *Coord. Chem. Rev.* **254**, 46–76.
Werz, D. B., Gleiter, R. & Rominger, F. (2002). *J. Am. Chem. Soc.* **124**, 10638–10639.

supporting information

Acta Cryst. (2021). E**77**, 1062-1066 [https://doi.org/10.1107/S2056989021010409]

Synthesis and absolute structure of (*R*)-2-(benzylselanyl)-1-phenylethanaminium hydrogen sulfate monohydrate: crystal structure and Hirshfeld surface analyses

H. R. Rajegowda, P. A. Suchetan, R. J. Butcher and P. Raghavendra Kumar

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009); program(s) used to solve structure: *SHELXT2016/4* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016/4* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *SHELXL2016/4* (Sheldrick, 2015b).

(*R*)-2-(Benzylselanyl)-1-phenylethanaminium hydrogen sulfate monohydrate

Crystal data

$C_{15}H_{18}NSe^{+}\cdot HSO_4^{-}\cdot H_2O$
$M_r = 406.35$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 10.4266$ (4) Å
$b = 6.0539$ (2) Å
$c = 14.2168$ (7) Å
$\beta = 90.261$ (4)°
$V = 897.38$ (6) Å^3
$Z = 2$
$F(000) = 416$
Prism
$D_x = 1.504$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1021 reflections
$\theta = 2.4–27.5°$
$\mu = 2.23$ mm^{-1}
$T = 293$ K
Prism, colourless
0.22 × 0.18 × 0.16 mm

Data collection

Bruker APEXII CCD area diffractometer
Radiation source: sealed X-ray tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
$T_{min} = 0.624$, $T_{max} = 0.700$
4255 measured reflections
3088 independent reflections
2624 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -12 \rightarrow 13$
$k = -6 \rightarrow 7$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.113$
$S = 0.99$
3088 reflections
213 parameters
1 restraint
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}

$\Delta\rho_{min} = -0.48$ e Å^{-3}

Absolute structure: Flack x determined using 665 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: 0.002 (16)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
O2	0.2505 (4)	−0.0248 (8)	−0.0946 (4)	0.0611 (14)
C3	−0.0762 (7)	−0.6548 (14)	−0.3497 (6)	0.067 (2)
H3	−0.038625	−0.793146	−0.342466	0.080*
C2	−0.1040 (6)	−0.5295 (12)	−0.2720 (6)	0.0556 (19)
H2	−0.086191	−0.584727	−0.212284	0.067*
N1	−0.5188 (4)	0.2116 (9)	−0.1128 (3)	0.0392 (13)
H1A	−0.531807	0.355694	−0.104787	0.047*
H1B	−0.466298	0.161851	−0.068113	0.047*
H1C	−0.593408	0.140600	−0.109508	0.047*
O4	0.3706 (4)	−0.3492 (8)	−0.0578 (3)	0.0476 (11)
O3	0.1539 (5)	−0.3689 (10)	−0.1198 (4)	0.0627 (14)
H3A	0.136250	−0.489162	−0.096649	0.094*
O1	0.0666 (5)	−0.7188 (11)	−0.0553 (7)	0.097 (2)
H1D	0.114172	−0.832572	−0.058680	0.145*
H1E	−0.007149	−0.769210	−0.041900	0.145*
SE1	−0.35634 (5)	−0.25057 (7)	−0.14135 (4)	0.04370 (19)
S1	0.24573 (11)	−0.2451 (4)	−0.05445 (9)	0.0386 (3)
O5	0.1938 (3)	−0.2455 (14)	0.0397 (3)	0.0577 (11)
C6	−0.1847 (6)	−0.239 (2)	−0.3707 (5)	0.0576 (17)
H6	−0.218500	−0.097722	−0.378626	0.069*
C10	−0.5289 (5)	0.3042 (9)	−0.2820 (4)	0.0377 (16)
C15	−0.4578 (6)	0.3885 (12)	−0.3559 (5)	0.0449 (15)
H15	−0.370318	0.359532	−0.359223	0.054*
C4	−0.1034 (7)	−0.5772 (18)	−0.4374 (7)	0.077 (3)
H4	−0.084348	−0.662521	−0.489933	0.092*
C1	−0.1588 (5)	−0.3192 (11)	−0.2815 (5)	0.0463 (16)
C9	−0.4594 (5)	0.1722 (10)	−0.2082 (4)	0.0360 (13)
H9	−0.370076	0.222507	−0.205826	0.043*
C12	−0.7151 (7)	0.4805 (15)	−0.3469 (6)	0.068 (2)
H12	−0.802097	0.513070	−0.342967	0.081*
C14	−0.5161 (7)	0.5156 (13)	−0.4248 (5)	0.0546 (18)
H14	−0.467590	0.568783	−0.474659	0.066*
C8	−0.4598 (6)	−0.0755 (10)	−0.2284 (5)	0.0404 (14)
H8A	−0.428439	−0.099360	−0.291714	0.048*
H8B	−0.547581	−0.128372	−0.226248	0.048*

C11	−0.6591 (6)	0.3503 (14)	−0.2794 (5)	0.0573 (19)
H11	−0.708984	0.292124	−0.231368	0.069*
C13	−0.6443 (7)	0.5641 (14)	−0.4206 (5)	0.061 (2)
H13	−0.682844	0.651632	−0.466410	0.073*
C7	−0.1873 (6)	−0.1864 (11)	−0.1955 (5)	0.0531 (19)
H7A	−0.183063	−0.030709	−0.211244	0.064*
H7B	−0.121754	−0.215704	−0.148436	0.064*
C5	−0.1593 (8)	−0.3717 (18)	−0.4485 (6)	0.075 (3)
H5	−0.180219	−0.321648	−0.508514	0.090*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.050 (3)	0.044 (3)	0.089 (4)	0.003 (2)	0.009 (3)	0.018 (3)
C3	0.048 (4)	0.058 (5)	0.094 (7)	0.000 (4)	0.012 (4)	−0.013 (5)
C2	0.044 (4)	0.048 (4)	0.074 (5)	0.005 (3)	−0.001 (3)	0.001 (4)
N1	0.050 (2)	0.034 (4)	0.033 (2)	0.002 (2)	−0.0048 (19)	−0.002 (2)
O4	0.038 (2)	0.052 (3)	0.053 (3)	0.012 (2)	0.0011 (19)	0.001 (2)
O3	0.057 (3)	0.068 (3)	0.064 (3)	−0.017 (3)	−0.014 (3)	0.002 (3)
O1	0.050 (3)	0.044 (4)	0.197 (7)	0.001 (3)	0.022 (4)	0.008 (4)
SE1	0.0484 (3)	0.0345 (3)	0.0482 (3)	0.0023 (4)	0.0009 (2)	−0.0026 (4)
S1	0.0342 (6)	0.0356 (6)	0.0460 (7)	0.0004 (10)	0.0002 (5)	0.0031 (11)
O5	0.048 (2)	0.077 (3)	0.049 (2)	0.010 (3)	0.0103 (18)	0.004 (4)
C6	0.050 (3)	0.060 (4)	0.064 (4)	−0.014 (5)	0.011 (3)	0.006 (6)
C10	0.038 (3)	0.037 (4)	0.038 (3)	−0.002 (2)	0.001 (2)	−0.001 (2)
C15	0.045 (3)	0.048 (4)	0.041 (4)	−0.002 (3)	0.002 (3)	0.002 (3)
C4	0.056 (5)	0.089 (7)	0.084 (7)	−0.011 (5)	0.028 (5)	−0.032 (6)
C1	0.034 (3)	0.042 (4)	0.062 (4)	−0.005 (3)	0.007 (3)	−0.002 (3)
C9	0.039 (3)	0.032 (3)	0.037 (3)	−0.001 (2)	0.002 (2)	−0.001 (2)
C12	0.044 (4)	0.093 (7)	0.065 (5)	0.006 (4)	−0.014 (4)	0.015 (5)
C14	0.072 (5)	0.055 (4)	0.037 (4)	−0.005 (4)	0.004 (3)	0.009 (3)
C8	0.044 (3)	0.036 (3)	0.041 (3)	−0.003 (3)	−0.003 (3)	−0.012 (3)
C11	0.039 (3)	0.079 (5)	0.054 (4)	0.000 (3)	0.003 (3)	0.009 (4)
C13	0.065 (5)	0.065 (5)	0.053 (5)	0.008 (4)	−0.017 (4)	0.010 (4)
C7	0.043 (3)	0.051 (5)	0.065 (4)	−0.004 (3)	0.000 (3)	−0.011 (3)
C5	0.060 (5)	0.103 (7)	0.063 (5)	−0.019 (5)	0.012 (4)	0.008 (5)

Geometric parameters (Å, º)

O2—S1	1.452 (5)	C10—C11	1.386 (8)
C3—C4	1.361 (13)	C10—C9	1.503 (8)
C3—C2	1.373 (10)	C15—C14	1.384 (9)
C3—H3	0.9300	C15—H15	0.9300
C2—C1	1.402 (10)	C4—C5	1.383 (14)
C2—H2	0.9300	C4—H4	0.9300
N1—C9	1.512 (7)	C1—C7	1.495 (9)
N1—H1A	0.8900	C9—C8	1.527 (8)
N1—H1B	0.8900	C9—H9	0.9800

N1—H1C	0.8900	C12—C11	1.370 (10)
O4—S1	1.447 (4)	C12—C13	1.380 (11)
O3—S1	1.527 (5)	C12—H12	0.9300
O3—H3A	0.8200	C14—C13	1.370 (10)
O1—H1D	0.8500	C14—H14	0.9300
O1—H1E	0.8500	C8—H8A	0.9700
Se1—C8	1.951 (6)	C8—H8B	0.9700
Se1—C7	1.965 (6)	C11—H11	0.9300
S1—O5	1.447 (4)	C13—H13	0.9300
C6—C1	1.384 (10)	C7—H7A	0.9700
C6—C5	1.395 (13)	C7—H7B	0.9700
C6—H6	0.9300	C5—H5	0.9300
C10—C15	1.387 (8)		

C4—C3—C2	120.2 (8)	C2—C1—C7	119.5 (7)
C4—C3—H3	119.9	C10—C9—N1	110.1 (4)
C2—C3—H3	119.9	C10—C9—C8	112.9 (5)
C3—C2—C1	120.8 (8)	N1—C9—C8	108.8 (5)
C3—C2—H2	119.6	C10—C9—H9	108.3
C1—C2—H2	119.6	N1—C9—H9	108.3
C9—N1—H1A	109.5	C8—C9—H9	108.3
C9—N1—H1B	109.5	C11—C12—C13	120.9 (7)
H1A—N1—H1B	109.5	C11—C12—H12	119.5
C9—N1—H1C	109.5	C13—C12—H12	119.5
H1A—N1—H1C	109.5	C13—C14—C15	120.9 (7)
H1B—N1—H1C	109.5	C13—C14—H14	119.6
S1—O3—H3A	109.5	C15—C14—H14	119.6
H1D—O1—H1E	104.5	C9—C8—Se1	114.4 (4)
C8—Se1—C7	98.0 (3)	C9—C8—H8A	108.7
O5—S1—O4	111.7 (3)	Se1—C8—H8A	108.7
O5—S1—O2	112.3 (4)	C9—C8—H8B	108.7
O4—S1—O2	110.8 (3)	Se1—C8—H8B	108.7
O5—S1—O3	109.0 (3)	H8A—C8—H8B	107.6
O4—S1—O3	109.1 (3)	C12—C11—C10	120.8 (6)
O2—S1—O3	103.6 (3)	C12—C11—H11	119.6
C1—C6—C5	119.1 (10)	C10—C11—H11	119.6
C1—C6—H6	120.4	C14—C13—C12	118.7 (7)
C5—C6—H6	120.4	C14—C13—H13	120.6
C15—C10—C11	118.2 (6)	C12—C13—H13	120.6
C15—C10—C9	117.8 (5)	C1—C7—Se1	113.3 (4)
C11—C10—C9	123.9 (5)	C1—C7—H7A	108.9
C10—C15—C14	120.4 (6)	Se1—C7—H7A	108.9
C10—C15—H15	119.8	C1—C7—H7B	108.9
C14—C15—H15	119.8	Se1—C7—H7B	108.9
C3—C4—C5	120.1 (9)	H7A—C7—H7B	107.7
C3—C4—H4	120.0	C4—C5—C6	120.7 (9)
C5—C4—H4	120.0	C4—C5—H5	119.7
C6—C1—C2	119.1 (8)	C6—C5—H5	119.7

C6—C1—C7	121.5 (7)

C4—C3—C2—C1	−0.9 (12)	C10—C15—C14—C13	1.2 (11)
C11—C10—C15—C14	0.1 (10)	C10—C9—C8—Se1	174.2 (4)
C9—C10—C15—C14	−178.5 (6)	N1—C9—C8—Se1	−63.2 (5)
C2—C3—C4—C5	−0.1 (13)	C13—C12—C11—C10	1.8 (13)
C5—C6—C1—C2	2.0 (10)	C15—C10—C11—C12	−1.7 (11)
C5—C6—C1—C7	−178.4 (6)	C9—C10—C11—C12	176.9 (7)
C3—C2—C1—C6	−0.1 (10)	C15—C14—C13—C12	−1.1 (12)
C3—C2—C1—C7	−179.7 (6)	C11—C12—C13—C14	−0.4 (13)
C15—C10—C9—N1	145.4 (5)	C6—C1—C7—Se1	93.7 (6)
C11—C10—C9—N1	−33.1 (8)	C2—C1—C7—Se1	−86.7 (7)
C15—C10—C9—C8	−92.7 (6)	C3—C4—C5—C6	2.1 (13)
C11—C10—C9—C8	88.7 (7)	C1—C6—C5—C4	−3.1 (11)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 aromatic ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.89	2.16	3.003 (7)	157
N1—H1*B*···O4^{ii}	0.89	2.05	2.893 (6)	159
N1—H1*C*···O2^{iii}	0.89	1.92	2.812 (6)	176
O1—H1*D*···O2^{iv}	0.85	1.91	2.726 (8)	161
O1—H1*E*···O5^{v}	0.85	1.95	2.730 (6)	152
O3—H3*A*···O1	0.82	1.68	2.483 (9)	167
C15—H15···*Cg*^{vi}	0.93	2.75	3.547 (7)	144

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