Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS
ISSN 2056-9890

Crystal structure and Hirshfeld surface analysis of (*E*)-3-[(4-fluorobenzylidene)amino]-5-phenylthiazolidin-2-iminium bromide

Ali N. Khalilov,[a,b] Zeliha Atioğlu,[c] Mehmet Akkurt,[d] Gulnara Sh. Duruskari,[a] Flavien A. A. Toze[e]* and Afet T. Huseynova[a]

[a]Organic Chemistry Department, Baku State University, Z. Xalilov str. 23, Az 1148, Baku, Azerbaijan, [b]Department of Physics and Chemistry, "Composite Materials" Scientific Research Center, Azerbaijan State Economic University (UNEC), H. Aliyev str. 135, Az 1063, Baku, Azerbaijan, [c]İlke Education and Health Foundation, Cappadocia University, Cappadocia Vocational College, The Medical Imaging Techniques Program, 50420 Mustafapaşa, Ürgüp, Nevşehir, Turkey, [d]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and [e]Department of Chemistry, Faculty of Sciences, University of Douala, PO Box 24157, Douala, Republic of Cameroon. *Correspondence e-mail: toflavien@yahoo.fr

Received 2 April 2019
Accepted 11 April 2019

Edited by C. Rizzoli, Universita degli Studi di Parma, Italy

Keywords: crystal structure; charge assisted hydrogen bonding; thiazolidine ring; disorder; Hirshfeld surface analysis..

CCDC reference: 1909594

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

In the cation of the title salt, $C_{16}H_{15}FN_3S^+\cdot Br^-$, the phenyl ring is disordered over two sets of sites with a refined occupancy ratio of 0.503 (4):0.497 (4). The mean plane of the thiazolidine ring makes dihedral angles of 13.51 (14), 48.6 (3) and 76.5 (3)° with the fluorophenyl ring and the major- and minor-disorder components of the phenyl ring, respectively. The central thiazolidine ring adopts an envelope conformation. In the crystal, centrosymmetrically related cations and anions are linked into dimeric units *via* N—H···Br hydrogen bonds, which are further connected by weak C—H···Br hydrogen bonds into chains parallel to [110]. Hirshfeld surface analysis and two-dimensional fingerprint plots indicate that the most important contributions to the crystal packing are from H···H (44.3%), Br···H/H···Br (16.8%), C···H/H···C (13.9%), F···H/H···F (10.3%) and S···H/H···S (3.8%) interactions.

1. Chemical context

Noncovalent interactions, both intermolecular and intramolecular, occur in virtually every substance and play an important role in the synthesis, catalysis, design of materials and biological processes (Akbari *et al.*, 2017; Gurbanov *et al.*, 2018; Kopylovich *et al.*, 2011; Maharramov *et al.*, 2010; Mahmoudi *et al.*, 2018*a*,*b*,*c*; Mahmudov *et al.*, 2011, 2013, 2014*a*,*b*, 2015, 2017*a*,*b*, 2019; Shixaliyev *et al.*, 2013, 2018). On the other hand, Schiff bases and related hydrazone ligands and their complexes have attracted attention over the past decades due to their potential biological, pharmacological and analytical applications (Kopylovich *et al.*, 2011; Mahmoudi *et al.*, 2018*a*,*b*,*c*; Mahmudov *et al.*, 2013). Hetercyclic amines are also widely used in the synthesis of Schiff bases, which provide different kinds of noncovalent interactions. As a further study in this field, we report herein the crystal structure and Hirshfeld surface analysis of the title compound.

OPEN ACCESS

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···Br1	0.90	2.36	3.2557 (18)	172
N3—H3B···Br1^{i}	0.90	2.55	3.3552 (18)	150
C4—H4A···Br1^{ii}	0.93	2.99	3.726 (2)	137

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

2. Structural commentary

The thiazolidine ring (S1/N2/C1–C3) in the cation of the title salt (Fig. 1) adopts an envelope conformation, with puckering parameters of $Q(2)$ = 0.321 (3) Å and $\varphi(2)$ = 43.3 (5)°. The mean plane of the thiazolidine ring makes dihedral angles of 13.51 (14), 48.6 (3) and 76.5 (3)° with the fluorophenyl ring (C5–C10) and the major- and minor-disorder components (C11–C16 and C11′–C16′) of the phenyl ring, respectively. The N2—N1—C4—C5 bridge that links the thiazolidine and 4-fluorophenyl rings has a torsion angle of −177.36 (19)°.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, centrosymmetrically related cations and anions are linked *via* pairs of N—H···Br hydrogen bonds (Table 1) into dimeric units forming rings of $R_4^2(8)$ graph-set motif (Fig. 2). The dimers are further connected by weak C—H···Br interactions to form chains running parallel to [110].

Hirshfeld surface analysis was used to investigate the presence of hydrogen bonds and intermolecular interactions in the crystal structure. The Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) of the title salt was generated by *CrystalExplorer3.1* (Wolff *et al.*, 2012), and comprised d_{norm} surface plots and 2D fingerprint plots (Spackman & McKinnon, 2002). The plots of the Hirshfeld surface mapped over d_{norm} using a standard surface resolution with a fixed colour scale of −1.4747 (red) to 1.2166 a.u. (blue) is shown in Fig. 3. This plot was generated to quantify and visualize the intermolecular interactions and to explain the observed crystal packing.

The shape index of the Hirshfeld surface is a tool to visualize π–π stacking interactions by the presence of adjacent red and blue triangles; if there are no adjacent red and/or blue

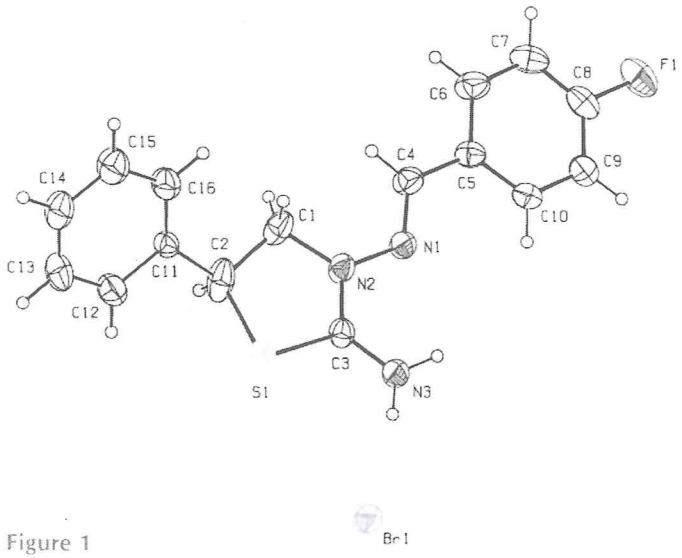

Figure 1
The molecular structure of the title salt. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radius. The minor-disorder component has been omitted for clarity

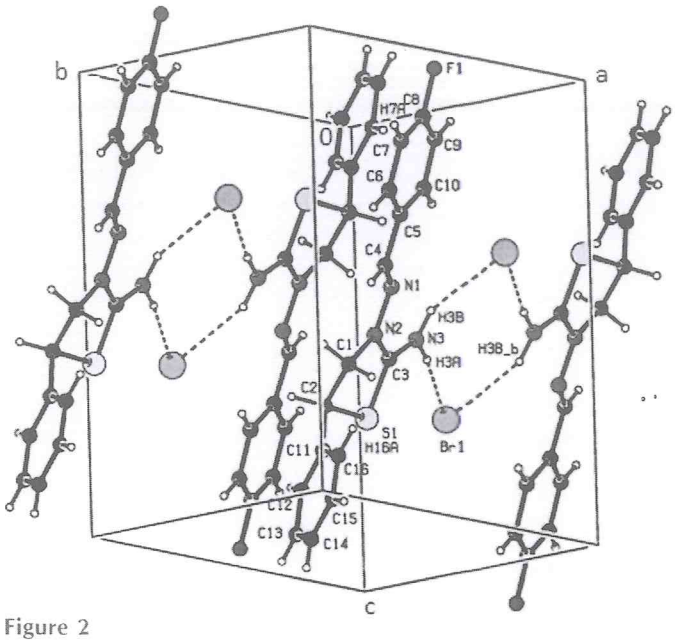

Figure 2
A view of the intermolecular N—H···Br hydrogen bonds of the title salt in the unit cell. The minor-disorder component has been omitted for clarity

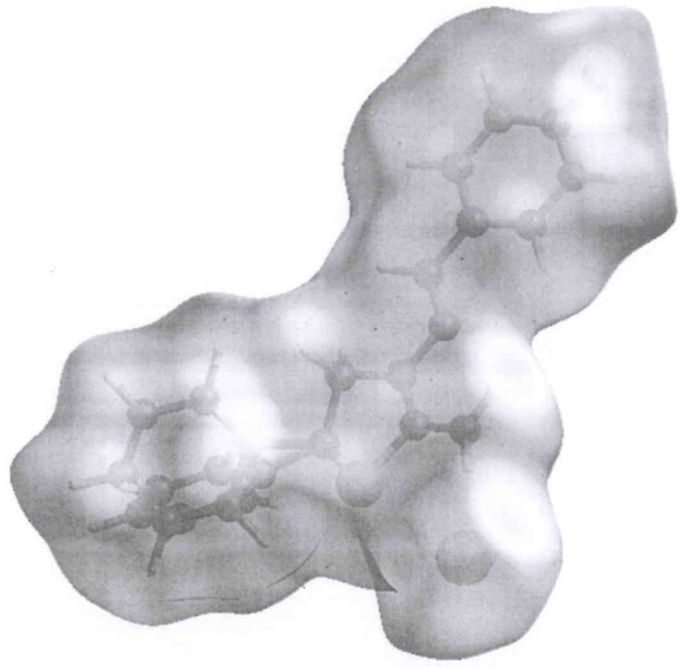

Figure 3
Hirshfeld surface of the title salt mapped with d_{norm}.

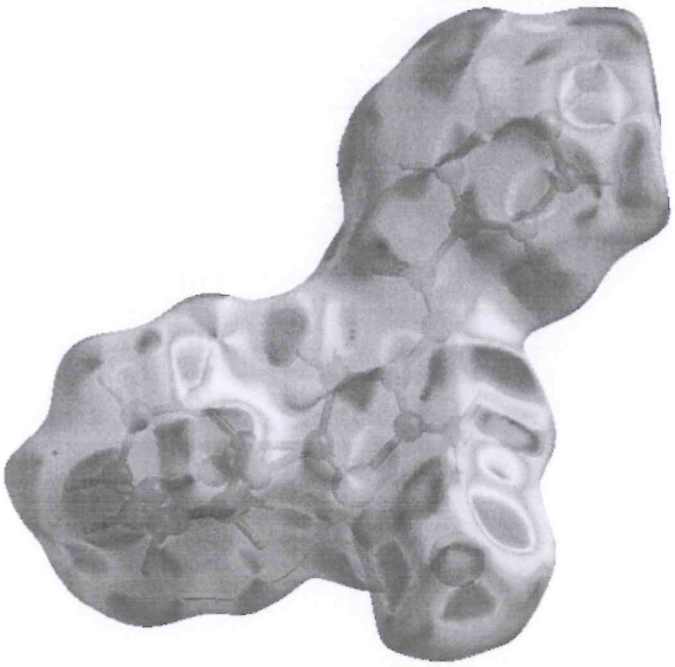

Figure 4
Hirshfeld surface of the title salt mapped with shape index.

Table 2
Percentage contributions of interatomic contacts to the Hirshfeld surface for the title salt.

Contact	Percentage contribution
H···H	44.3
Br···H/H···Br	16.8
C···H/H···C	13.9
F···H/H···F	10.3
S···H/H···S	3.8
N···C/C···N	3.6
S···C/C···S	2.7
N···H/H···N	1.8
C···C	1.5
N···N	0.7
Br···C/C···Br	0.3
S···N/N···S	0.3
F···C/C···F	0.2

triangles, then there are no π–π interactions. Fig. 4 clearly suggest that there are no π–π interactions present in the title salt.

Fig. 5(*a*) shows the 2D fingerprint of the sum of the contacts contributing to the Hirshfeld surface represented in normal mode. These represent both the overall 2D fingerprint plots

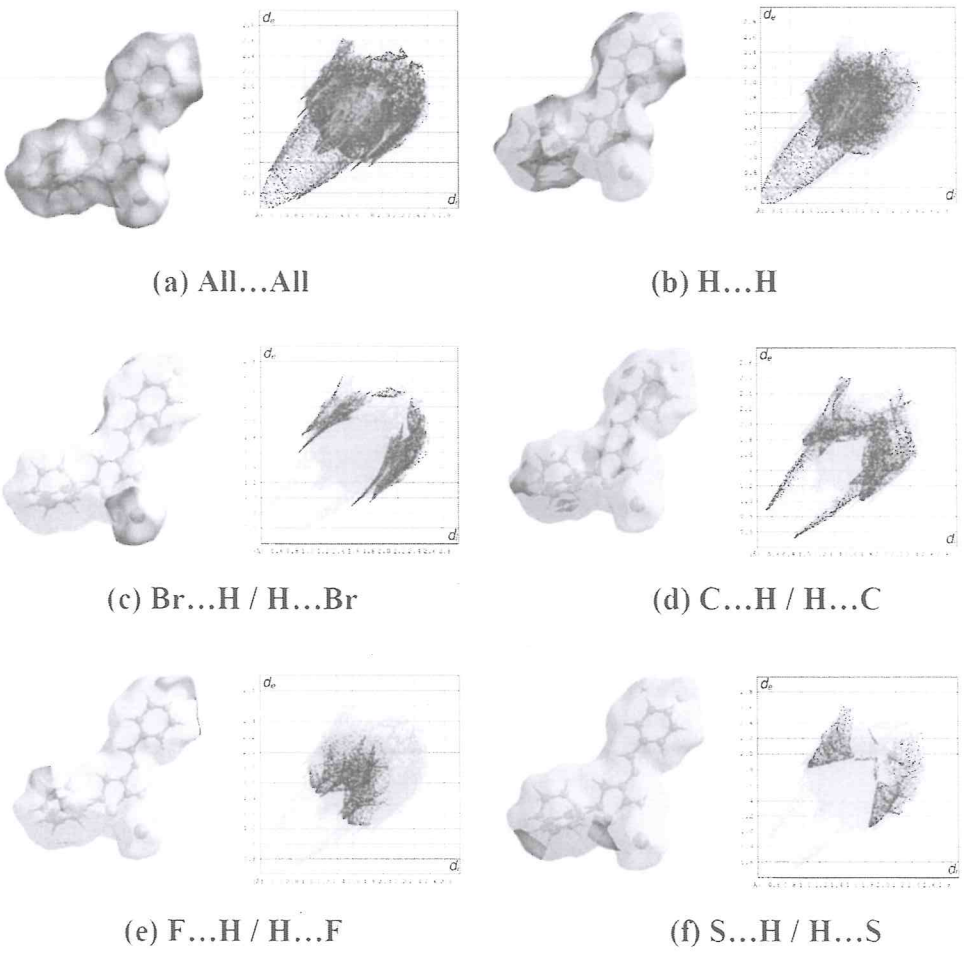

Figure 5
The 2D fingerprint plots of the title salt, showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) Br···H/H···Br, (*d*) C···H/H···C, (*e*) F···H/H···F and (*f*) S···H/H···S interactions [d_e and d_i represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively].

and those that represent H···H (44.3%), Br···H/H···Br (16.8%), C···H/H···C (13.9%), F···H/H···F (10.3%) and S···H/H···S (3.8%) contacts, respectively (Figs. 5*b–f*). The most significant intermolecular interactions are the H···H interactions (44.3%) (Fig. 6*b*). All the contributions to the Hirshfeld surface are given in Table 2.

3.1. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, update of November 2018; Groom *et al.*, 2016) for 2-thiazolidiniminium compounds gave seven hits, *viz.* UDELUN (Akkurt *et al.*, 2018), WILBIC (Marthi *et al.*, 1994), WILBOI (Marthi *et al.*, 1994), WILBOI01 (Marthi *et al.*, 1994), YITCEJ (Martem'yanova *et al.*, 1993*a*), YITCAF (Martem'yanova *et al.*, 1993*b*) and YOPLUK (Marthi *et al.*, 1995).

In the crystal of UDELUN (Akkurt *et al.*, 2018), C—H···Br and N—H···Br hydrogen bonds link the components into a three-dimensional network with the cations and anions stacked along the *b*-axis direction. Weak C—H···π interactions, which only involve the minor-disorder component of the ring, also contribute to the molecular packing. In addition, there are also inversion-related Cl···Cl halogen bonds and C—Cl···π(ring) contacts.

In the remaining structures, the 3-N atom carries a C-substituent instead of an N-substituent, as found in the title compound. The first three crystal structures were determined for racemic (WILBIC; Marthi *et al.*, 1994) and two optically active samples (WILBOI and WILBOI01; Marthi *et al.*, 1994) of 3-(2′-chloro-2′-phenylethyl)-2-thiazolidiniminium *p*-toluenesulfonate. In all three structures, the most disordered fragment of these molecules is the asymmetric C atom and the Cl atom attached to it. The disorder of the cation in the racemate corresponds to the presence of both enantiomers at each site in the ratio 0.821 (3):0.179 (3). The system of hydrogen bonds connecting two cations and two anions into 12-membered rings is identical in the racemic and in the optically active crystals. YITCEJ (Martem'yanova *et al.*, 1993*a*) is a product of the interaction of 2-amino-5-methylthiazoline with methyl iodide, with alkylation at the endocylic N atom, while YITCAF (Martem'yanova *et al.*, 1993*b*) is a product of the reaction of 3-nitro-5-methoxy-, 3-nitro-5-chloro- and 3-bromo-5-nitrosalicylaldehyde with the heterocyclic base to form the salt-like complexes.

4. Synthesis and crystallization

To a solution of 3-amino-5-phenylthiazolidin-2-iminium bromide (1 mmol) in ethanol (20 ml) was added 4-fluorobenzaldehyde (1 mmol). The mixture was refluxed for 2 h and then cooled. The reaction product precipitated from the reaction mixture as colourless single crystals, was collected by filtration and washed with cold acetone (yield 64%; m.p. 544–545 K). Analysis calculated (%) for $C_{16}H_{15}BrFN_3S$: C 50.53, H 3.98, N 11.05; found: C 50.47, H 3.93, N 11.00. ^{1}H NMR (300 MHz, DMSO-d_6) : 4.56 (*k*, 1H, CH_2, $^3J_{H\text{-}H}$ = 6.6 Hz), 4.87 (*t*, 1H, CH_2, $^3J_{H\text{-}H}$ = 7.8 Hz), 5.60 (*t*, 1H, CH-Ar, $3J_{H\text{-}H}$ = 7.8 Hz), 7.32–8.16 (*m*, 9H, 9Ar-H), 8.45 (*s*, 1H, CH=), 10.37 (*s*, 2H, NH_2). ^{13}C NMR (75 MHz, DMSO-d_6): 45.39, 55.97, 116.05, 127.81, 128.91, 129.13, 129.60, 131.05, 131.17, 137.55, 150.00, 167.89. MS (ESI), *m*/*z*: 300.36 $[C_{16}H_{15}FN_3S]^+$ and 79.88 Br^-.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{15}FN_3S^+\cdot Br^-$
M_r	380.28
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, *b*, *c* (Å)	8.0599 (3), 8.6086 (4), 12.7608 (5)
α, β, γ (°)	96.548 (2), 92.518 (2), 111.065 (2)
V (Å^3)	817.39 (6)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.65
Crystal size (mm)	0.16 × 0.12 × 0.09
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2003)
T_{min}, T_{max}	0.664, 0.782
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12009, 3321, 2672
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.627
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.029, 0.077, 1.04
No. of reflections	3321
No. of parameters	254
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.48

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2003).

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were positioned geometrically and refined using a riding model, with N—H = 0.90 Å and C—H = 0.93–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N). The phenyl ring in the cation is disordered over two sets of sites with an occupancy ratio of 0.503 (4):0.497 (4). Seven outliers (001; $\overline{3}$05; $\overline{1}$43; 010; $\overline{2}$75; $\overline{2}$,$\overline{1}$,12; and 7$\overline{5}$3) were omitted in the final cycles of refinement.

Acknowledgements

Ali Khalilov is grateful to Baku State University for the '50+50' individual grant in support of this work.

References

Akbari Afkhami, F., Mahmoudi, G., Gurbanov, A. V., Zubkov, F. I., Qu, F., Gupta, A. & Safin, D. A. (2017). *Dalton Trans.* **46**, 14888–14896.
Akkurt, M., Duruskari, G. S., Toze, F. A. A., Khalilov, A. N. & Huseynova, A. T. (2018). *Acta Cryst.* E**74**, 1168–1172.
Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Gurbanov, A. V., Maharramov, A. M., Zubkov, F. I., Saifutdinov, A. M. & Guseinov, F. I. (2018). *Aust. J. Chem.* **71**, 190–194.
Kopylovich, M. N., Mahmudov, K. T., Haukka, M., Luzyanin, K. V. & Pombeiro, A. J. L. (2011). *Inorg. Chim. Acta*, **374**, 175–180.
Maharramov, A. M., Aliyeva, R. A., Aliyev, I. A., Pashaev, F. G., Gasanov, A. G., Azimova, S. I., Askerov, R. K., Kurbanov, A. V. & Mahmudov, K. T. (2010). *Dyes Pigments*, **85**, 1–6.
Mahmoudi, G., Seth, S. K., Bauzá, A., Zubkov, F. I., Gurbanov, A. V., White, J., Stilinović, V., Doert, Th. & Frontera, A. (2018*a*). *CrystEngComm*, **20**, 2812–2821.
Mahmoudi, G., Zangrando, E., Mitoraj, M. P., Gurbanov, A. V., Zubkov, F. I., Moosavifar, M., Konyaeva, I. A., Kirillov, A. M. & Safin, D. A. (2018*b*). *New J. Chem.* **42**, 4959–4971.
Mahmoudi, G., Zaręba, J. K., Gurbanov, A. V., Bauzá, A., Zubkov, F. I., Kubicki, M., Stilinović, V., Kinzhybalo, V. & Frontera, A. (2018*c*). *Eur. J. Inorg. Chem.* pp. 4763–4772.
Mahmudov, K. T., Guedes da Silva, M. F. C., Kopylovich, M. N., Fernandes, A. R., Silva, A., Mizar, A. & Pombeiro, A. J. L. (2014*a*). *J. Organomet. Chem.* **760**, 67–73.
Mahmudov, K. T., Guedes da Silva, M. F. C., Sutradhar, M., Kopylovich, M. N., Huseynov, F. E., Shamilov, N. T., Voronina, A. A., Buslaeva, T. M. & Pombeiro, A. J. L. (2015). *Dalton Trans.* **44**, 5602–5610.
Mahmudov, K. T., Gurbanov, A. V., Guseinov, F. I. & Guedes da Silva, M. F. C. (2019). *Coord. Chem. Rev.* **387**, 32–46.
Mahmudov, K. T., Kopylovich, M. N., Guedes da Silva, M. F. C. & Pombeiro, A. J. L. (2017*a*). *Coord. Chem. Rev.* **345**, 54–72.
Mahmudov, K. T., Kopylovich, M. N., Guedes da Silva, M. F. C. & Pombeiro, A. J. L. (2017*b*). *Dalton Trans.* **46**, 10121–10138.
Mahmudov, K. T., Kopylovich, M. N., Maharramov, A. M., Kurbanova, M. M., Gurbanov, A. V. & Pombeiro, A. J. L. (2014*b*). *Coord. Chem. Rev.* **265**, 1–37.
Mahmudov, K. T., Kopylovich, M. N. & Pombeiro, A. J. L. (2013). *Coord. Chem. Rev.* **257**, 1244–1281.
Mahmudov, K. T., Maharramov, A. M., Aliyeva, R. A., Aliyev, I. A., Askerov, R. K., Batmaz, R., Kopylovich, M. N. & Pombeiro, A. J. L. (2011). *J. Photochem. Photobiol. Chem.* **219**, 159–165.
Martem'yanova, N. A., Chunaev, Y. M., Przhiyalgovskaya, N. M., Kurkovskaya, L. N., Filipenko, O. S. & Aldoshin, S. M. (1993*a*). *Khim. Geterotsikl. Soedin.* pp. 415–419.
Martem'yanova, N. A., Chunaev, Y. M., Przhiyalgovskaya, N. M., Kurkovskaya, L. N., Filipenko, O. S. & Aldoshin, S. M. (1993*b*). *Khim. Geterotsikl. Soedin.* pp. 420–425.
Marthi, K., Larsen, M., Ács, M., Bálint, J. & Fogassy, E. (1995). *Acta Chem. Scand.* **49**, 20–27.
Marthi, K., Larsen, S., Ács, M., Bálint, J. & Fogassy, E. (1994). *Acta Cryst.* B**50**, 762–771.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Shixaliyev, N. Q., Ahmadova, N. E., Gurbanov, A. V., Maharramov, A. M., Mammadova, G. Z., Nenajdenko, V. G., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2018). *Dyes Pigments*, **150**, 377–381.
Shixaliyev, N. Q., Maharramov, A. M., Gurbanov, A. V., Nenajdenko, V. G., Muzalevskiy, V. M., Mahmudov, K. T. & Kopylovich, M. N. (2013). *Catal. Today*, **217**, 76–79.
Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.
Spackman, M. A. & McKinnon, J. J. (2002). *CrystEngComm*, **4**, 378–392.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wolff, S. K., Grimwood, D. J., McKinnon, J. J., Turner, M. J., Jayatilaka, D. & Spackman, M. A. (2012). *CrystalExplorer*. University of Western Australia.

supporting information

Acta Cryst. (2019). E**75**, 662-666 [https://doi.org/10.1107/S2056989019004973]

Crystal structure and Hirshfeld surface analysis of (*E*)-3-[(4-fluorobenzylidene)amino]-5-phenylthiazolidin-2-iminium bromide

Ali N. Khalilov, Zeliha Atioğlu, Mehmet Akkurt, Gulnara Sh. Duruskari, Flavien A. A. Toze and Afet T. Huseynova

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2003).

(*E*)-3-[(4-Fluorobenzylidene)amino]-5-phenylthiazolidin-2-iminium bromide

Crystal data

$C_{16}H_{15}FN_3S^+\cdot Br^-$
$M_r = 380.28$
Triclinic, $P\overline{1}$
$a = 8.0599$ (3) Å
$b = 8.6086$ (4) Å
$c = 12.7608$ (5) Å
$\alpha = 96.548$ (2)°
$\beta = 92.518$ (2)°
$\gamma = 111.065$ (2)°
$V = 817.39$ (6) Å^3
$Z = 2$
$F(000) = 384$
$D_x = 1.545$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5655 reflections
$\theta = 2.6$–$26.3°$
$\mu = 2.65$ mm^{-1}
$T = 296$ K
Plate, colorless
0.16 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
$T_{min} = 0.664$, $T_{max} = 0.782$
12009 measured reflections
3321 independent reflections
2672 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.7°$
$h = -10 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.077$
$S = 1.03$
3321 reflections
254 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 0.2228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	0.28837 (4)	−0.06846 (3)	0.65093 (2)	0.07274 (12)	
S1	0.50535 (9)	0.40641 (8)	0.72511 (4)	0.06317 (17)	
F1	1.1652 (2)	0.6889 (3)	0.07197 (13)	0.0994 (6)	
N1	0.7677 (2)	0.5288 (2)	0.48291 (13)	0.0510 (4)	
C1	0.7491 (4)	0.6768 (3)	0.66453 (19)	0.0676 (7)	
H1A	0.765957	0.780305	0.635640	0.081*	
H1B	0.859205	0.688530	0.704392	0.081*	
N2	0.6981 (3)	0.5342 (2)	0.57951 (13)	0.0540 (4)	
C2	0.5974 (5)	0.6381 (4)	0.73431 (19)	0.0803 (9)	
H2A	0.506784	0.680819	0.710523	0.096*	
N3	0.5389 (3)	0.2468 (2)	0.54077 (14)	0.0564 (5)	
H3A	0.472588	0.153018	0.566419	0.068*	
H3B	0.598218	0.240158	0.483319	0.068*	
C3	0.5851 (3)	0.3881 (3)	0.60334 (16)	0.0489 (5)	
C4	0.8962 (3)	0.6588 (3)	0.46468 (18)	0.0583 (6)	
H4A	0.943400	0.751707	0.516854	0.070*	
C5	0.9692 (3)	0.6619 (3)	0.36204 (18)	0.0532 (5)	
C6	1.0980 (3)	0.8092 (3)	0.3405 (2)	0.0691 (7)	
H6A	1.139096	0.902914	0.392330	0.083*	
C7	1.1660 (4)	0.8188 (4)	0.2431 (2)	0.0767 (8)	
H7A	1.254083	0.916970	0.229104	0.092*	
C8	1.1011 (3)	0.6810 (4)	0.1683 (2)	0.0665 (7)	
C9	0.9756 (3)	0.5314 (3)	0.1862 (2)	0.0659 (6)	
H9A	0.935350	0.438646	0.133610	0.079*	
C10	0.9115 (3)	0.5229 (3)	0.28380 (19)	0.0592 (6)	
H10A	0.827792	0.422230	0.298028	0.071*	
C11	0.6893 (9)	0.7289 (7)	0.8505 (5)	0.0447 (13)	0.503 (4)
C12	0.5575 (7)	0.6983 (7)	0.9197 (5)	0.0691 (15)	0.503 (4)
H12A	0.439229	0.635805	0.894732	0.083*	0.503 (4)
C13	0.6019 (12)	0.7611 (13)	1.0269 (6)	0.074 (2)	0.503 (4)
H13A	0.513251	0.739287	1.073481	0.089*	0.503 (4)
C14	0.7747 (12)	0.8546 (10)	1.0639 (5)	0.0740 (16)	0.503 (4)
H14A	0.804349	0.898834	1.135130	0.089*	0.503 (4)
C15	0.9041 (8)	0.8822 (8)	0.9946 (4)	0.0811 (17)	0.503 (4)
H15A	1.022734	0.943608	1.019215	0.097*	0.503 (4)
C16	0.8600 (7)	0.8201 (7)	0.8893 (4)	0.0660 (14)	0.503 (4)
H16A	0.949586	0.841027	0.843322	0.079*	0.503 (4)
C11′	0.5982 (9)	0.7008 (8)	0.8494 (5)	0.0489 (14)	0.497 (4)
C12′	0.5168 (7)	0.8137 (6)	0.8805 (4)	0.0631 (13)	0.497 (4)

H12B	0.447047	0.841350	0.831371	0.076*	0.497 (4)
C13′	0.5408 (8)	0.8852 (7)	0.9865 (5)	0.0759 (18)	0.497 (4)
H13B	0.488252	0.962201	1.007970	0.091*	0.497 (4)
C14′	0.6390 (15)	0.8437 (12)	1.0570 (7)	0.079 (3)	0.497 (4)
H14B	0.655646	0.893516	1.127240	0.094*	0.497 (4)
C15′	0.7133 (14)	0.7321 (12)	1.0286 (5)	0.0843 (19)	0.497 (4)
H15B	0.779040	0.702274	1.078940	0.101*	0.497 (4)
C16′	0.6923 (8)	0.6598 (8)	0.9226 (5)	0.0793 (17)	0.497 (4)
H16B	0.744737	0.582013	0.902974	0.095*	0.497 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0842 (2)	0.05494 (16)	0.05450 (16)	−0.00131 (12)	0.02403 (12)	−0.00748 (11)
S1	0.0911 (4)	0.0681 (4)	0.0419 (3)	0.0401 (3)	0.0203 (3)	0.0126 (3)
F1	0.1003 (12)	0.1287 (15)	0.0746 (10)	0.0375 (11)	0.0439 (9)	0.0375 (10)
N1	0.0588 (10)	0.0484 (10)	0.0428 (9)	0.0154 (9)	0.0093 (8)	0.0067 (8)
C1	0.1015 (19)	0.0518 (13)	0.0448 (12)	0.0257 (13)	−0.0005 (12)	−0.0010 (10)
N2	0.0706 (12)	0.0494 (10)	0.0385 (9)	0.0183 (9)	0.0088 (8)	0.0032 (8)
C2	0.145 (3)	0.0759 (18)	0.0422 (13)	0.0652 (19)	0.0157 (15)	0.0117 (12)
N3	0.0696 (11)	0.0478 (10)	0.0492 (10)	0.0162 (9)	0.0199 (9)	0.0087 (8)
C3	0.0593 (12)	0.0513 (12)	0.0400 (10)	0.0240 (10)	0.0079 (9)	0.0079 (9)
C4	0.0643 (14)	0.0516 (13)	0.0492 (12)	0.0108 (11)	0.0016 (10)	0.0044 (10)
C5	0.0504 (12)	0.0514 (12)	0.0535 (12)	0.0119 (10)	0.0028 (10)	0.0139 (10)
C6	0.0733 (16)	0.0558 (14)	0.0663 (15)	0.0074 (12)	0.0053 (13)	0.0159 (12)
C7	0.0708 (16)	0.0692 (17)	0.0845 (19)	0.0101 (13)	0.0177 (14)	0.0362 (16)
C8	0.0585 (14)	0.0891 (19)	0.0617 (15)	0.0311 (14)	0.0196 (12)	0.0303 (14)
C9	0.0575 (14)	0.0732 (16)	0.0636 (15)	0.0204 (12)	0.0141 (11)	0.0049 (12)
C10	0.0515 (12)	0.0560 (13)	0.0627 (14)	0.0092 (10)	0.0148 (11)	0.0103 (11)
C11	0.052 (3)	0.043 (3)	0.043 (3)	0.018 (3)	0.015 (3)	0.012 (2)
C12	0.060 (3)	0.084 (4)	0.059 (4)	0.021 (3)	0.014 (3)	0.005 (3)
C13	0.084 (6)	0.101 (6)	0.045 (4)	0.042 (5)	0.018 (4)	0.009 (4)
C14	0.100 (5)	0.083 (5)	0.042 (3)	0.038 (4)	0.006 (3)	0.001 (3)
C15	0.085 (4)	0.096 (4)	0.055 (3)	0.029 (3)	0.003 (3)	0.001 (3)
C16	0.066 (3)	0.077 (3)	0.049 (3)	0.020 (3)	0.011 (2)	0.003 (2)
C11′	0.051 (4)	0.053 (3)	0.036 (3)	0.009 (3)	0.016 (3)	0.007 (2)
C12′	0.066 (3)	0.058 (3)	0.065 (3)	0.019 (2)	0.009 (2)	0.020 (2)
C13′	0.092 (4)	0.057 (3)	0.083 (4)	0.031 (3)	0.041 (3)	0.006 (3)
C14′	0.095 (7)	0.073 (5)	0.042 (4)	0.002 (5)	0.013 (4)	−0.004 (3)
C15′	0.087 (5)	0.111 (7)	0.055 (4)	0.037 (5)	−0.011 (3)	0.012 (4)
C16′	0.084 (4)	0.096 (4)	0.072 (4)	0.053 (4)	0.008 (3)	0.002 (3)

Geometric parameters (Å, °)

S1—C3	1.720 (2)	C9—C10	1.369 (3)
S1—C2	1.848 (3)	C9—H9A	0.9300
F1—C8	1.353 (3)	C10—H10A	0.9300
N1—C4	1.276 (3)	C11—C16	1.353 (8)

N1—N2	1.380 (2)	C11—C12	1.383 (7)
C1—N2	1.465 (3)	C12—C13	1.393 (10)
C1—C2	1.506 (4)	C12—H12A	0.9300
C1—H1A	0.9700	C13—C14	1.364 (14)
C1—H1B	0.9700	C13—H13A	0.9300
N2—C3	1.339 (3)	C14—C15	1.370 (9)
C2—C11′	1.505 (6)	C14—H14A	0.9300
C2—C11	1.607 (7)	C15—C16	1.370 (7)
C2—H2A	0.9800	C15—H15A	0.9300
N3—C3	1.297 (3)	C16—H16A	0.9300
N3—H3A	0.9001	C11′—C16′	1.333 (9)
N3—H3B	0.9000	C11′—C12′	1.389 (8)
C4—C5	1.458 (3)	C12′—C13′	1.394 (8)
C4—H4A	0.9300	C12′—H12B	0.9300
C5—C6	1.386 (3)	C13′—C14′	1.334 (12)
C5—C10	1.390 (3)	C13′—H13B	0.9300
C6—C7	1.379 (4)	C14′—C15′	1.329 (15)
C6—H6A	0.9300	C14′—H14B	0.9300
C7—C8	1.358 (4)	C15′—C16′	1.399 (9)
C7—H7A	0.9300	C15′—H15B	0.9300
C8—C9	1.373 (4)	C16′—H16B	0.9300

C3—S1—C2	90.65 (11)	C10—C9—H9A	121.0
C4—N1—N2	117.98 (19)	C8—C9—H9A	121.0
N2—C1—C2	105.8 (2)	C9—C10—C5	121.2 (2)
N2—C1—H1A	110.6	C9—C10—H10A	119.4
C2—C1—H1A	110.6	C5—C10—H10A	119.4
N2—C1—H1B	110.6	C16—C11—C12	118.7 (5)
C2—C1—H1B	110.6	C16—C11—C2	133.2 (5)
H1A—C1—H1B	108.7	C12—C11—C2	108.2 (5)
C3—N2—N1	116.37 (17)	C11—C12—C13	120.0 (6)
C3—N2—C1	115.67 (18)	C11—C12—H12A	120.0
N1—N2—C1	127.52 (19)	C13—C12—H12A	120.0
C11′—C2—C1	129.4 (4)	C14—C13—C12	120.3 (7)
C1—C2—C11	104.7 (3)	C14—C13—H13A	119.8
C11′—C2—S1	104.9 (3)	C12—C13—H13A	119.8
C1—C2—S1	105.04 (17)	C13—C14—C15	119.0 (6)
C11—C2—S1	112.7 (2)	C13—C14—H14A	120.5
C1—C2—H2A	111.4	C15—C14—H14A	120.5
C11—C2—H2A	111.4	C16—C15—C14	120.5 (6)
S1—C2—H2A	111.4	C16—C15—H15A	119.7
C3—N3—H3A	117.3	C14—C15—H15A	119.7
C3—N3—H3B	119.6	C11—C16—C15	121.5 (5)
H3A—N3—H3B	120.5	C11—C16—H16A	119.2
N3—C3—N2	123.59 (19)	C15—C16—H16A	119.2
N3—C3—S1	123.18 (17)	C16′—C11′—C12′	118.8 (6)
N2—C3—S1	113.23 (16)	C16′—C11′—C2	119.7 (5)
N1—C4—C5	120.0 (2)	C12′—C11′—C2	121.2 (5)

N1—C4—H4A	120.0	C11′—C12′—C13′	119.1 (5)
C5—C4—H4A	120.0	C11′—C12′—H12B	120.4
C6—C5—C10	118.6 (2)	C13′—C12′—H12B	120.4
C6—C5—C4	119.1 (2)	C14′—C13′—C12′	120.3 (6)
C10—C5—C4	122.3 (2)	C14′—C13′—H13B	119.9
C7—C6—C5	120.8 (3)	C12′—C13′—H13B	119.9
C7—C6—H6A	119.6	C15′—C14′—C13′	121.0 (8)
C5—C6—H6A	119.6	C15′—C14′—H14B	119.5
C8—C7—C6	118.3 (2)	C13′—C14′—H14B	119.5
C8—C7—H7A	120.9	C14′—C15′—C16′	119.7 (8)
C6—C7—H7A	120.9	C14′—C15′—H15B	120.1
F1—C8—C7	119.0 (2)	C16′—C15′—H15B	120.1
F1—C8—C9	117.9 (3)	C11′—C16′—C15′	121.0 (6)
C7—C8—C9	123.1 (2)	C11′—C16′—H16B	119.5
C10—C9—C8	118.0 (3)	C15′—C16′—H16B	119.5

C4—N1—N2—C3	−169.1 (2)	C6—C5—C10—C9	−2.0 (4)
C4—N1—N2—C1	3.0 (3)	C4—C5—C10—C9	176.7 (2)
C2—C1—N2—C3	−26.1 (3)	C1—C2—C11—C16	−1.1 (7)
C2—C1—N2—N1	161.8 (2)	S1—C2—C11—C16	112.5 (6)
N2—C1—C2—C11′	155.8 (4)	C1—C2—C11—C12	−179.8 (4)
N2—C1—C2—C11	150.2 (3)	S1—C2—C11—C12	−66.2 (5)
N2—C1—C2—S1	31.4 (2)	C16—C11—C12—C13	0.1 (10)
C3—S1—C2—C11′	−163.6 (3)	C2—C11—C12—C13	178.9 (6)
C3—S1—C2—C1	−24.92 (19)	C11—C12—C13—C14	0.8 (14)
C3—S1—C2—C11	−138.3 (3)	C12—C13—C14—C15	−1.5 (15)
N1—N2—C3—N3	−0.7 (3)	C13—C14—C15—C16	1.4 (12)
C1—N2—C3—N3	−173.7 (2)	C12—C11—C16—C15	−0.2 (9)
N1—N2—C3—S1	179.87 (14)	C2—C11—C16—C15	−178.7 (5)
C1—N2—C3—S1	6.8 (3)	C14—C15—C16—C11	−0.6 (10)
C2—S1—C3—N3	−168.1 (2)	C1—C2—C11′—C16′	−64.5 (7)
C2—S1—C3—N2	11.35 (19)	S1—C2—C11′—C16′	59.9 (7)
N2—N1—C4—C5	−177.36 (19)	C1—C2—C11′—C12′	109.3 (6)
N1—C4—C5—C6	174.4 (2)	S1—C2—C11′—C12′	−126.3 (5)
N1—C4—C5—C10	−4.2 (4)	C16′—C11′—C12′—C13′	2.2 (9)
C10—C5—C6—C7	0.8 (4)	C2—C11′—C12′—C13′	−171.6 (5)
C4—C5—C6—C7	−177.9 (2)	C11′—C12′—C13′—C14′	−0.9 (9)
C5—C6—C7—C8	1.2 (4)	C12′—C13′—C14′—C15′	−1.0 (13)
C6—C7—C8—F1	179.3 (2)	C13′—C14′—C15′—C16′	1.5 (16)
C6—C7—C8—C9	−2.3 (4)	C12′—C11′—C16′—C15′	−1.7 (11)
F1—C8—C9—C10	179.6 (2)	C2—C11′—C16′—C15′	172.2 (6)
C7—C8—C9—C10	1.1 (4)	C14′—C15′—C16′—C11′	−0.2 (14)
C8—C9—C10—C5	1.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···Br1	0.90	2.36	3.2557 (18)	172

N3—H3*B*···Br1^{i}	0.90	2.55	3.3552 (18)	150
C4—H4*A*···Br1^{ii}	0.93	2.99	3.726 (2)	137

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

Journal Search

Search Terms

Database

Search Type Title Word

Master Journal List

Search

Search Term(s): *2056-9890 · The following title(s) matched your request

1-1 of 1 journals

Format for print

- ACTA CRYSTALLOGRAPHICA SECTION E-CRYSTALLOGRAPHIC COMMUNICATIONS
  Monthly
  ISSN: 2056-9890
  E-ISSN: 2056-9890
  INT UNION CRYSTALLOGRAPHY, 2 ABBEY SQ, CHESTER, ENGLAND, CH1 2HU
  Coverage
  - Emerging Sources Citation Index

1-1 of 1 journals

Format for print