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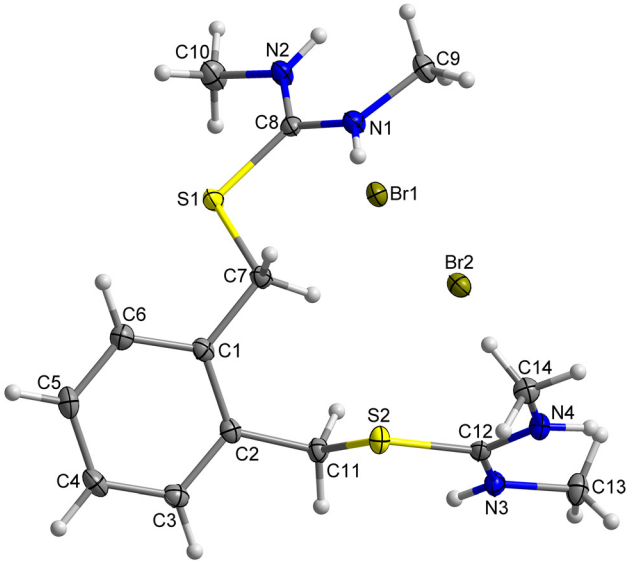
The crystal structure of 2,2′-(1,2-phenylenebis(methylene)) bis(1,3-dimethylisothiouronium) bromide, $C_{14}H_{24}Br_2N_4S_2$



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Abstract

$C_{14}H_{24}Br_2N_4S_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.1005(2) Å, b = 9.8627(3) Å, c = 12.5984(3) Å, α = 78.038(2)°, β = 84.8290(19)°, γ = 73.203(2)°, V = 942.16(4) Å^3, Z = 2, R_{gt} (F^2) = 0.0162, wR_{ref} (F^2) = 0.0486, T = 95 K [1–4].

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.19 × 0.10 × 0.07 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	7.55 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.3°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,622, 3705, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3557
N(param)$_{refined}$:	212
Programs:	CrysAlis^{PRO} [1], Superflip [2], Jana2006 [3], Diamond [4]

Source of material

To the solution of 1,2-bis(bromomethyl)benzene (1.119 g, 4.24 mmol) in 30 ml acetonitrile, the solution of *N*, *N*′-dimethylthiourea (0.883 g, 4.48 mmol) in 10 ml of acetonitrile was added. The reaction mixture was stirred at room temperature for 4 h. The resulting precipitate was filtered off, washed with 10 ml of cold acetonitrile and dried on air. The reaction yield was 1.914 g (95.7%). The crystals suitable for single crystal diffraction were obtained by slow evaporation of solution of the title compound (1 mg) in acetonitrile (20 ml).

Experimental details

H atoms bonded to C were placed in calculated positions, while H atoms bonded to N were refined with restrained bond lengths (N–H = 0.86 Å), C–H = 0.96 Å. U_{iso}(H) = 1.2 U_{eq}(C, N).

Comment

The title compound is an *N*,*N*′-dimethylisothiouronium salt. This group of compounds was recently studied for their proapoptotic effects in a human prostate adenocarcinoma [5]. They can also be used in sythesis of organic heterocyclic compounds [6].

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.023826 (17)	1.123364 (14)	0.213988 (10)	0.01433 (6)
Br2	0.501046 (17)	0.750287 (15)	0.455853 (11)	0.01591 (6)
S1	0.54877 (4)	0.80954 (4)	0.10636 (3)	0.01703 (11)
S2	0.07020 (4)	0.67110 (4)	0.36948 (3)	0.01512 (10)
N1	0.47263 (15)	1.02130 (13)	0.22356 (9)	0.0156 (4)
N2	0.75095 (16)	0.94991 (13)	0.15450 (10)	0.0160 (4)
N3	0.20975 (16)	0.55630 (13)	0.56482 (9)	0.0152 (4)
N4	−0.00181 (15)	0.77312 (13)	0.54964 (9)	0.0150 (4)
C1	0.30681 (17)	0.66432 (15)	0.13806 (11)	0.0131 (4)
C2	0.26635 (17)	0.55017 (15)	0.21227 (11)	0.0134 (4)
C3	0.21590 (18)	0.44618 (15)	0.17318 (11)	0.0157 (4)
C4	0.20715 (19)	0.45213 (16)	0.06255 (12)	0.0178 (4)
C5	0.24846 (19)	0.56467 (17)	−0.01089 (11)	0.0188 (5)
C6	0.29733 (18)	0.66941 (16)	0.02699 (11)	0.0164 (4)
C7	0.34833 (18)	0.78653 (15)	0.17565 (11)	0.0147 (4)
C8	0.59106 (18)	0.93933 (15)	0.16796 (10)	0.0139 (4)
C9	0.51350 (19)	1.11475 (16)	0.28734 (12)	0.0187 (4)
C10	0.88668 (19)	0.86082 (17)	0.09377 (12)	0.0215 (5)
C11	0.26640 (18)	0.53923 (15)	0.33384 (11)	0.0149 (4)
C12	0.09829 (17)	0.66474 (15)	0.50709 (10)	0.0136 (4)
C13	0.24203 (18)	0.55265 (16)	0.67732 (11)	0.0195 (5)
C14	−0.12903 (18)	0.89572 (16)	0.48922 (11)	0.0172 (4)
H1c3	0.186591	0.368808	0.223851	0.0188*
H1c4	0.172938	0.379301	0.037134	0.0214*
H1c5	0.243208	0.569905	−0.087507	0.0225*
H1c6	0.325228	0.74702	−0.024173	0.0197*
H1c7	0.363621	0.762139	0.252638	0.0176*
H2c7	0.257109	0.873641	0.156515	0.0176*
H1c9	0.41296	1.155442	0.329034	0.0225*
H2c9	0.604688	1.059285	0.335425	0.0225*
H3c9	0.549692	1.190908	0.239461	0.0225*
H1c10	0.989224	0.892117	0.089595	0.0258*
H2c10	0.910796	0.761778	0.129982	0.0258*
H3c10	0.849189	0.870047	0.021819	0.0258*
H1c11	0.36545	0.563525	0.352131	0.0179*
H2c11	0.263134	0.444132	0.369789	0.0179*
H1c13	0.32723	0.464601	0.704663	0.0234*
H2c13	0.283218	0.633381	0.681412	0.0234*
H3c13	0.136933	0.557568	0.720074	0.0234*
H1c14	−0.176681	0.965984	0.534349	0.0206*
H2c14	−0.07417	0.938534	0.425631	0.0206*
H3c14	−0.219663	0.863048	0.468058	0.0206*
H1n1	0.3683 (9)	1.015 (2)	0.2295 (15)	0.0188*
H1n2	0.780 (2)	1.0110 (16)	0.1836 (14)	0.0192*
H1n3	0.274 (2)	0.4878 (13)	0.5345 (14)	0.0182*
H1n4	0.009 (2)	0.779 (2)	0.6157 (5)	0.018*

The asymmetric unit of the title compound, see figure, consists of one organic dication, and two bromide anions. The bond lengths and angles of both isothiouronium branches are almost identical, with the largest deviation from average value 0.003 Å for N2–C10 and N4–C11 bonds, and 0.675° for C1–C7–S1 and C2–C11–S4 angles. The significant differences are observed in overall tilting of both branches, with one branch observed above the benzene plane and the other below it. The tilting is described by the difference in torsion angles C6–C1–C7–S1 at −54.26 (16)° and C3–C2–C11–S2 at −101.27 (13)°. The remaining significant torsion angles C(ar)–C(me)–S–C(th) and C(me)–S–

C(th)–N have very similar values, with the largest difference from average value of 0.63° for C(ar)–C(me)–S–C(th).

As the structure has four good donors of proton, the hydrogen bonds play a significant role in the crystal structure packing. Three hydrogen bonds with Br1 anion N1–H1n1···Br1, N2–H1n2···Br1, and N4–H1n4···Br1 form ribbons in the direction of <1 0 0>. This is achieved by combination of $C_2^1(6)$ chain and two $R_4^2(26)$ rings. The ribbons are connected into layers parallel to the crystallographic plane (0 0 1) by combination of N3–H1n3···Br2 and C–H···Br2 weak hydrogen bonds. As stronger N–H hydrogen bond donors are used in layer formation, the layers are connected by weaker C10–H3c10···Br1 hydrogen bond.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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