

This is a self-archived version of an original article. This version may differ from the original in pagination and typographic details.

Author(s): Prakasam, Balasubramaniam Arul; Kumar, Chandran Udhaya; Lahtinen, Manu; Peuronen, Anssi; Sillanpää, Mika

Title: Single crystal X-ray structural dataset of 1,2,4-dithiazolium tetrafluoroborate

Year: 2022

Version: Published version

Copyright: © 2022 The Author(s). Published by Elsevier Inc.

Rights: CC BY 4.0

Rights url: <https://creativecommons.org/licenses/by/4.0/>

Please cite the original version:

Prakasam, B. A., Kumar, C. U., Lahtinen, M., Peuronen, A., & Sillanpää, M. (2022). Single crystal X-ray structural dataset of 1,2,4-dithiazolium tetrafluoroborate. *Data in Brief*, 41, Article 107924. <https://doi.org/10.1016/j.dib.2022.107924>



Data Article

Single crystal X-ray structural dataset of 1,2,4-dithiazolium tetrafluoroborate

Balasubramaniam Arul Prakasam^{a,*}, Chandran Udhaya Kumar^a,
Manu Lahtinen^{b,*}, Anssi Peuronen^c, Mika Sillanpää^{d,e,f,g}

^a Department of Chemistry, Annamalai University, Annamalainagar, 608002, India

^b Department of Chemistry, P.O.Box 35, FI-40014 University of Jyväskylä, Jyväskylä, Finland

^c Group of Intelligent Materials Chemistry, Department of Chemistry, University of Turku, FI-20014 Turku, Finland

^d Department of Chemical Engineering, School of Mining, Metallurgy and Chemical Engineering, University of Johannesburg, P. O. Box 17011, Doornfontein 2028, South Africa

^e Chemistry Department, College of Science, King Saud University, Riyadh 11451, Saudi Arabia

^f Faculty of Science and Technology, School of Applied Physics, University Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

^g Department of Biological and Chemical Engineering, Aarhus University, Nørrebrogade 44, 8000 Aarhus C, Denmark

ARTICLE INFO

Article history:

Received 30 September 2021

Revised 18 January 2022

Accepted 2 February 2022

Available online 4 February 2022

Keywords:

1,2,4-Dithiazolium

Crystal structure

Hydrogen bond

Heterocycle

ABSTRACT

Herein, we present the crystallographic dataset of 1,2,4-dithiazolium tetrafluoroborate. Single crystal X-ray structural analysis evidences that the 1,2,4-dithiazolium ring is almost planar. The 1,2,4-dithiazolium and tetrafluoroborate ions contribute in hydrogen bonding wherein the N-H-N hydrogen bonding in 1,2,4-dithiazolium dimer forms an eight-membered pseudo ring with the $R_2^2(8)$ Etter's graph set. The information provided in this data contributes to the understanding of structural chemistry and hydrogen bonding interactions in dithiazole derivatives.

© 2022 The Author(s). Published by Elsevier Inc.

This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>)

* Corresponding authors.

E-mail address: manu.k.lahtinen@jyu.fi (M. Lahtinen).

Specifications Table

Subject	Organic Chemistry
Specific subject area	Organic heterocyclic molecules and crystallographic structure
Type of data	Figures and Tables
How data were acquired	Single Crystal X-ray diffraction: Agilent Supernova (Cu/Mo dual flux micro-focus sources) diffractometer.
Data format	Raw data and Analyzed
Parameters for data collection	SXRD: Agilent Supernova (Cu/Mo dual flux micro-focus sources) diffractometer was used to acquire the data at 123 K using Cu-K α radiation ($\lambda = 1.54184 \text{ \AA}$).
Data source location	Department of Chemistry, Laboratories of Inorganic and Analytical Chemistry, University of Jyväskylä, Finland.
Data accessibility	The data can be accessed at https://data.mendeley.com/datasets/6zw4w4kvsc/1 or from the Cambridge Crystallographic Data Centre. CCDC No. 2109451. Copies of the data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html , or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk .

Value of the Data

- The presented data will be useful for the organic chemists and structural chemists.
- The data will be useful to analyze the hydrogen bonding pattern in crystal structures of dithiazolium salts.
- The data may be useful for the comparison of aromatic character of the heterocyclic cations.
- The data may provide information pertaining to single crystal X-ray structural analysis of ionic liquids.

1. Data Description

This work describes the data with respect to the new crystal structure of 1,2,4-dithiazolium tetrafluoroborate. ORTEP drawing of 1,2,4-dithiazolium tetrafluoroborate is shown in Fig. 1 and the packing diagram is shown in Fig. 2. There are two molecules in the unit cell and the 1,2,4-dithiazolium ring is planar. The amino substituents at carbon atoms 3 and 5 are coplanar with the central ring. The bond lengths suggest that there is a delocalization of π electrons in the N-

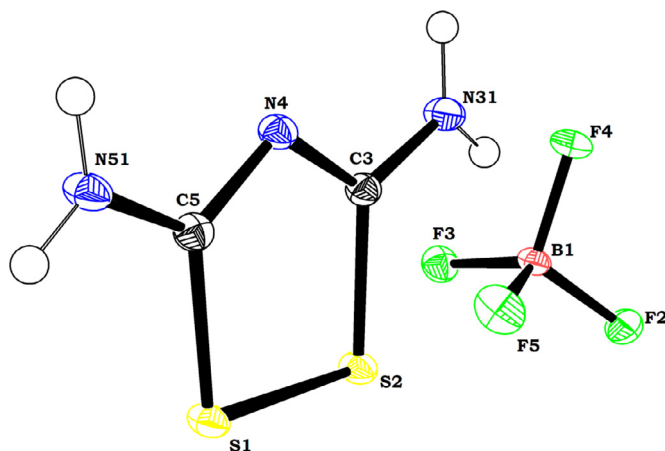


Fig. 1. ORTEP image of 1,2,4-dithiazolium tetrafluoroborate.

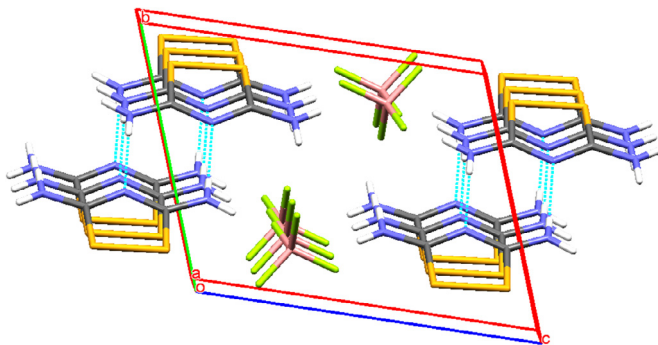


Fig. 2. Packing diagram of 1,2,4-dithiazolium tetrafluoroborate.

Table 1

Crystal data and structure refinement of 1,2,4-dithiazolium tetrafluoroborate.

Empirical formula	$C_2H_4BF_4N_3S_2$
FW	221.01
Crystal dimensions (mm)	$0.167 \times 0.1244 \times 0.072$
Crystal system	triclinic
Color	pale yellow
Space group	<i>P</i> -1
<i>a</i> /Å	5.2098(10)
<i>b</i> /Å	7.7233(15)
<i>c</i> /Å	9.742(16)
α /°	110.2(16)
β /°	90.664(15)
γ /°	93.314(16)
Volume/ Å ³	367.06(12)
<i>Z</i>	2
<i>D_c</i> /gcm ⁻³	2.000
μ /cm ⁻¹	6.906
<i>F</i> (000)	220
2θ range/°	9.68 to 137.954
Index ranges	$-6 \leq h \leq 6, -9 \leq k \leq 6, -9 \leq l \leq 11$
Reflections collected	2129
Observed reflections $F_0 > 4\sigma(F_0)$	1353
Number of parameters refined	122
Final <i>R</i> , <i>R_w</i> (obs, data)	0.0234, 0.0588
GOOF	1.051

C–N–C–N fragment. Nevertheless, the dithiazolium ring is devoid of aromatic character as there is no delocalization of π electrons throughout the ring. The 1,2,4-dithiazolium and tetrafluoroborate ions contributed in hydrogen bonding wherein, the N–H–N hydrogen bonding in 1,2,4-dithiazolium dimer forms an eight membered pseudo ring with the $R_2^2(8)$, Etter's graph set [1]. The details of crystal data with structure refinement [2–4], bond distances, bond angles, torsional angles, fractional atomic coordinates, anisotropic atomic displacement and hydrogen bonds are depicted in Tables 1–7 respectively. The data deposited with the repository Mendeley and can be accessed at <https://data.mendeley.com/datasets/6zw4w4kvcv/1> [5].

Table 2

Selected bond distances (Å) for 1,2,4-dithiazolium tetrafluoroborate.

S1–S2	2.0586(6)
S1–C5	1.7507(16)
S2–C3	1.7535(16)
F3–B1	1.40(2)
F4–B1	1.4005(19)
F5–B1	1.389(2)
F2–B1	1.385(2)
N31–C3	1.309(2)
N51–C5	1.312(2)
N4–C5	1.334(2)
N4–C3	1.337(2)

Table 3

Selected bond angles (°) for 1,2,4-dithiazolium tetrafluoroborate.

C5–S1–S2	92.86(5)
C3–S2–S1	93.29(6)
C5–N4–C3	114.53(14)
N51–C5–S1	118.30(12)
N51–C5–N4	121.73(15)
N4–C5–S1	119.96(12)
N31–C3–S2	118.77(12)
N31–C3–N4	121.93(15)
N4–C3–S2	119.30(12)

Table 4

Selected torsional angles (°) for 1,2,4-dithiazolium tetrafluoroborate.

S1–S2–C3–N31	177.60(12)
S1–S2–C3–N4	–2.46(12)
S2–S1–C5–N51	179.02(12)
S2–S1–C5–N4	–0.11(12)
C5–N4–C3–S2	2.80(18)
C5–N4–C3–N31	–177.26(13)
C3–N4–C5–S1	–1.54(18)
C3–N4–C5–N51	179.35(13)

Table 5Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 1,2,4-dithiazolium tetrafluoroborate. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	Z	$U(\text{eq})$
S1	3695.4(7)	1724.9(5)	9321.2(4)	12.94(13)
S2	3281.5(7)	1482.7(5)	7157.3(4)	12.81(14)
F3	28.5(18)	3487.9(13)	3468.8(10)	18.8(2)
F4	4125.4(19)	2802.7(14)	2769.0(11)	23.5(2)
F5	2170(2)	1544.7(15)	4304.1(11)	25.1(3)
F2	820.6(19)	609.1(13)	1918.0(10)	21.2(2)
N31	6895(3)	3037(2)	5994.1(15)	15.2(3)
N51	7706(3)	3712(2)	10875.9(15)	14.6(3)
N4	7524(2)	3540.4(18)	8460.3(14)	12.1(3)
C5	6567(3)	3108(2)	9573.0(16)	12.0(3)
C3	6155(3)	2802(2)	7201.0(17)	11.8(3)
B1	1774(3)	2101(2)	3109.8(18)	13.4(3)

Table 6Anisotropic atomic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1,2,4-dithiazolium tetrafluoroborate.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
S1	10.2(2)	17.4(2)	11.8(2)	6.37(15)	-0.23(13)	-2.78(14)
S2	10.4(2)	16.4(2)	11.0(2)	4.60(15)	-0.82(13)	-2.91(14)
F3	14.8(5)	21.7(5)	19.5(5)	6.5(4)	-0.1(4)	1.7(4)
F4	14.2(5)	24.2(5)	29.2(5)	6.4(4)	6.2(4)	-5.3(4)
F5	25.5(5)	35.6(6)	19.5(5)	16.1(5)	-1.9(4)	3.5(4)
F2	20.6(5)	20.5(5)	17.3(5)	1.1(4)	-0.9(4)	-5.8(4)
N31	14.5(7)	18.6(7)	12.2(6)	5.6(6)	-0.3(5)	-4.4(6)
N51	10.7(6)	21.6(7)	12.5(6)	8.0(6)	-1.3(5)	-3.8(5)
N4	9.0(6)	14.5(6)	12.6(6)	4.6(5)	-0.2(5)	-0.5(5)
C5	9.4(7)	12.4(7)	14.3(7)	4.7(6)	0.2(5)	1.7(6)
C3	9.5(7)	11.4(7)	14.2(7)	3.9(6)	0.9(5)	0.6(5)
B1	10.8(8)	17.4(8)	11.8(8)	5.5(7)	0.3(6)	-2.7(6)

Table 7

Hydrogen bonds for 1,2,4-dithiazolium tetrafluoroborate.

D	H	A	d(D-H)/\AA	d(H-A)/\AA	d(D-A)/\AA	D-H-A/\u00b0
N31	H31A	F3 ¹	0.85(2)	2.15(2)	2.9258(19)	151.1(19)
N51	H51A	F4 ²	0.84(2)	2.08(2)	2.8631(17)	155.7(19)
N51	H51B	N4 ³	0.81(2)	2.28(2)	3.082(2)	172(2)
N31	H31B	F5	0.84(2)	2.14(2)	2.8926(19)	148.0(19)

¹ 1-X,1-Y,1-Z;² +X,+Y,1+Z;³ 2-X,1-Y,2-Z

2. Experimental Design, Materials and Methods

2.1. Synthesis of 1,2,4-dithiazolium tetrafluoroborate

The title compound was prepared by refluxing a mixture of dithiobiuret (1 mmol) and BF₃:Et₂O (1.2 mmol) in 95% ethanol for a period of 12h. The reaction mixture was then filtered and left to evaporate. After two days, the solid obtained was recrystallized from dichloromethane:methanol (1:1) solvent mixture.

2.2. Single crystal X-ray structural analysis

Single crystals suitable for X-ray structural analysis were obtained by slow evaporation using dichloromethane:methanol (1:1) solvent mixture. Single crystal dataset used in structure determination was acquired with dual source (Cu/Mo) Agilent SuperNova diffractometer equipped with multilayer optics for generating monochromatized Cu K α radiation, and Atlas CCD detector for recording data. A crystal was mounted in a MiTeGen MicroMountTM loop (100 μ m), and data collection was made at -150°C under N₂ stream. Data collection, reduction and analytical numeric absorption corrections by multifaceted crystal models were all made using CrysAlisPRO program [2]. By using Olex² (v 1.3) [3], the crystal structure was solved with Superflip and refined on F² by full matrix least squares techniques with ShelXL [4] program. All non-hydrogen atoms were refined with anisotropic displacement parameters, whereas hydrogen atoms were located from the electron density map and refined freely except of using isotropic displacement parameters 1.2 times of the host atom.

Ethics Statement

The work was not involved with human subjects or animal experiments and the data was not collected from social media platforms.

CRediT Author Statement

Balasubramaniam Arul Prakasam: Conceptualization, Investigation, Methodology, Writing – Original Draft; **Chandran Udhaya Kumar:** Data Curation, Writing – Review & Editing; **Manu Lahtinen:** Investigation, Data Curation, Validation, Writing – Review & Editing. **Anssi Peuronen:** Investigation, Data Curation, Validation, Writing – Review & Editing; **Mika Sillanpää:** Methodology, Validation.

Declaration of Competing Interest

The authors state that they have no known competing financial interests or personal relationships that could have appeared to influence of the work reported in this paper.

Acknowledgements

The authors gratefully acknowledge the support of University of Jyväskylä.

Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:[10.1016/j.dib.2022.107924](https://doi.org/10.1016/j.dib.2022.107924).

References

- [1] M.C. Etter, J. Macdonald, J. Bernstein, *Acta Cryst. B* 46 (1990) 256–262, doi:[10.1107/S0108768189012929](https://doi.org/10.1107/S0108768189012929).
- [2] Rigaku Oxford Diffraction, *CrysAlisPro Software system, version 1.171.39.46*, Rigaku Corporation, Oxford, UK, 2019.
- [3] O.V. Dolomanov, L.J. Bourhis, R.J. Gildea, J.A.K. Howard, H. Puschmann, *J. Appl. Cryst.* 42 (2009) 339–341, doi:[10.1107/S0021889808042726](https://doi.org/10.1107/S0021889808042726).
- [4] G.M. Sheldrick, *Acta Cryst. C* 71 (2015) 3–8 <http://dx.doi.org/10.1107/S2053229614024218>.
- [5] Balasubramaniam Arul Prakasam, Chandran Udhaya Kumar, Anssi Peuronen, Manu Lahtinen, Mika Sillanpää, Single crystal X-ray structural dataset of 1,2,4-dithiazolium tetrafluoroborate, *Mendeley Data* (2022) V1, doi:[10.17632/6zw4w4kvsc.1](https://doi.org/10.17632/6zw4w4kvsc.1).