Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Pyridinium 2,2′-dithiodisalicylate

Yao Kang, Rui-Feng Hu, Ye-Yan Qin, Zhao-Ji Li, Yu-Biao Chen, Yi-Hang Wen, Jian-Kai Cheng and Yuan-Gen Yao*

The State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: yyg@ms.fjirsm.ac.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.071
wR factor = 0.185
Data-to-parameter ratio = 13.7

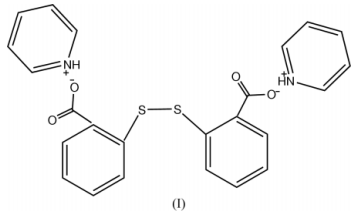
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 July 2002
Accepted 26 July 2002
Online 31 July 2002

The title compound, $2C_5H_6N^+\cdot[C_6H_4(COO)_2S]_2^{2-}$ or $2C_5H_6N^+\cdot C_{14}H_8O_4S_2^{2-}$, has a twofold axis of symmetry passing through the centre of the S—S bond.

Comment

It is well known that transition metal ions can function as electrophiles in the cleavage of the S—S bond in organic disulfides. For this reason, transition metal ion/organic disulfide compounds, such as those of nickel(II) (Ottersen *et al.*, 1974; Ramalingam *et al.*, 1987) and osmium (Lee & Wong, 1996), have been synthesized. In our systematic investigation of trimolybdenum clusters, we have synthesized successfully $Mo_3S_4(DTP)_3(p\text{-}NO_2C_6H_4COO)(NC_5H_5)$ (Tang *et al.*, 2001) from the reaction of $Mo_3S_4(DTP)_4(H_2O)$ (DTP is diethyl dithiophosphate) with p-$NO_2C_6H_4COOH$ and pyridine. However, when we used $Mo_3S_4(DTP)_4(H_2O)$ and 2,2′-dithiodisalicylic acid as the starting materials for the reaction in a mixed solvent of dichloromethane, ethanol and pyridine, the title compound, $2C_5H_6N^+\cdot[C_6H_4(COO)_2S]_2^{2-}$, (I), was unexpectedly obtained.



(I)

The title compound (Fig. 1) is composed of 2,2′-dithiodisalicylate and pyridinium ions, in a ratio of 1:2; the asymmetric unit consists of one-half molecule of the disalicylate and one pyridinium cation. A twofold axis of symmetry passes through the centre of the S—S bond. There is a hydrogen bond; O1···N 2.653 (6) Å, H0*A*···O1 1.79 Å and O1···H0*A*—N 178°. The C2—S—S′—C2′ torsion angle and S—S′ bond length are 87.3 (3)° and 2.067 (2) Å, respectively. These values compare well with corresponding angles and bond lengths of 86 (1)° and 2.039 (10) Å (Lee *et al.*, 1996), and 86.7° and 2.047 (3) Å (Ottersen *et al.*, 1974). The dihedral angle between the plane defined by atoms C1–C7/O1/O2 and the plane defined by the atoms symmetry-related to these nine is 87.6 (1)°. Bond lengths are within expected ranges.

Experimental

The title compound was prepared by the following two methods.

Method 1: $Mo_3S_4(DTP)_4(H_2O)$ (0.085 mmol) and 2,2′-dithiodisalicylic acid (0.042 mmol) were dissolved in a mixed solvent (20 ml dichloromethane, 20 ml ethanol and 1 ml pyridine). After refluxing over an oil bath at 35 3 K for 1 h, the hot dark-brown solution was

filtered into another flask. Rectangular yellow crystals precipitated over a period of 20 d (yield: 0.01 g, 52%).

Method 2: 2,2′-dithiodisalicylic acid (1 mmol) was dissolved in 20 ml dichloromethane, 20 ml ethanol and 1 ml pyridine and refluxed over an oil bath at 353 K for 1 h. Rectangular, yellow crystals precipitated over a period of 15 d (yield: 0.34 g, 73%).

Crystal data

$2C_5H_6N^+\cdot C_{14}H_8O_4S_2^{2-}$	$D_x = 1.350$ Mg m^{-3}
$M_r = 464.54$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 1954 reflections
$a = 8.0936$ (4) Å	$\theta = 1.9$–$25.0°$
$b = 12.9825$ (6) Å	$\mu = 0.27$ mm^{-1}
$c = 21.9157$ (10) Å	$T = 293$ (2) K
$\beta = 96.882$ (1)°	Rectangular, yellow
$V = 2286.20$ (19) Å^3	0.68 × 0.48 × 0.30 mm
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	1406 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.035$
Absorption correction: none	$\theta_{max} = 25.0°$
3703 measured reflections	$h = -9 \rightarrow 9$
1980 independent reflections	$k = -15 \rightarrow 10$
	$l = -21 \rightarrow 25$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 6.9607P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.071$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.185$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
$S = 1.15$	$\Delta\rho_{min} = -0.29$ e Å^{-3}
1980 reflections	
145 parameters	
H-atom parameters not refined	

Table 1
Selected geometric parameters (Å, °).

S—C2	1.799 (4)	C6—C5	1.378 (7)
S—S^{i}	2.067 (2)	N—C8	1.314 (6)
O2—C7	1.208 (5)	N—C12	1.313 (7)
C1—C6	1.389 (6)	C4—C5	1.381 (7)
C1—C2	1.415 (6)	C12—C11	1.394 (8)
C1—C7	1.499 (6)	C9—C10	1.360 (9)
O1—C7	1.313 (5)	C9—C8	1.380 (8)
C2—C3	1.391 (6)	C10—C11	1.343 (9)
C3—C4	1.376 (6)		
C2—S—S^{i}	104.52 (14)	O1—C7—C1	114.5 (4)
C6—C1—C2	119.7 (4)	C5—C6—C1	121.5 (5)
C6—C1—C7	119.4 (4)	C8—N—C12	117.9 (5)
C2—C1—C7	120.9 (4)	C3—C4—C5	121.6 (5)
C3—C2—C1	118.1 (4)	N—C12—C11	123.6 (6)
C3—C2—S	121.4 (3)	C6—C5—C4	118.4 (5)
C1—C2—S	120.5 (3)	C10—C9—C8	117.4 (6)
C4—C3—C2	120.7 (4)	C11—C10—C9	121.6 (7)
O2—C7—O1	123.0 (4)	N—C8—C9	123.0 (6)
O2—C7—C1	122.4 (4)	C10—C11—C12	116.5 (7)
C2—S—S^{i}—C2^{i}	87.3 (3)		

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

H atoms were placed in calculated positions and not refined.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *XPREP* in *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

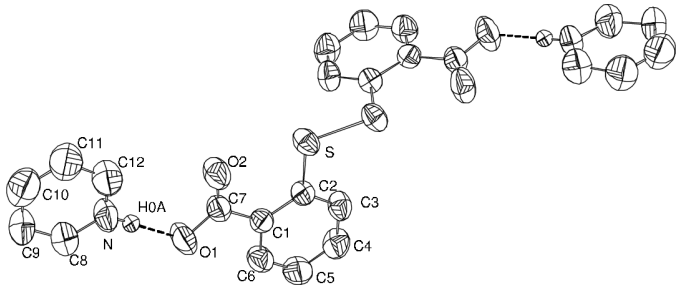


Figure 1
The structure of the title compound, showing 40% probability displacement ellipsoids. H atoms have been omitted for clarity. Hydrogen bonds are indicated by dashed lines.

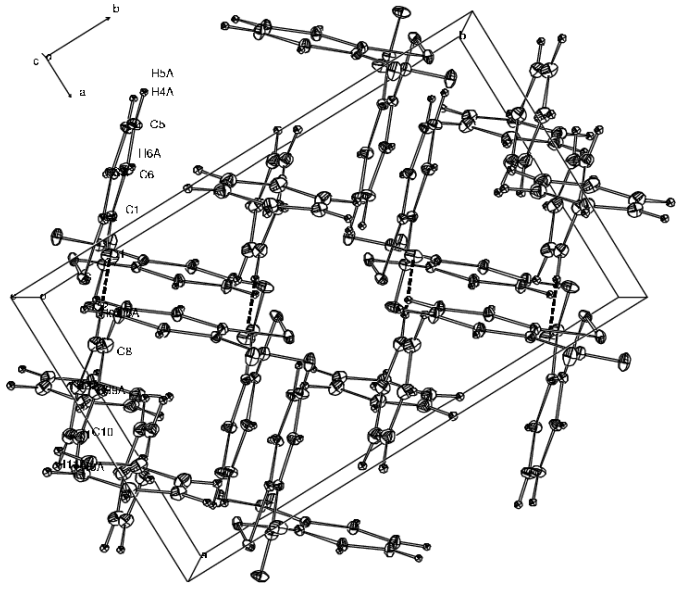


Figure 2
A view of the packing within the unit cell, viewed along [001]. Probability ellipsoids are drawn at the 30% level.

The authors are grateful to the State Key Basic Research and Development Plan of China (001-CB-108906), the NNSF of China (Nos. 29733090 and 20173063), Key Project in KIP of the CAS (KJCX2-H3) and the NNSF of Fujian Province (E0020001) for financial support of this work.

References

Lee, S. M. & Wong, W. T. (1996). *J. Cluster Sci.* **7**, 37–47.
Ottersen, T., Warner, L. G. & Seff, K. (1974). *Acta Cryst.* B**30**, 1188–1191.
Ramalingam, K., Aravamudan, G. & Seshasayee, M. (1987). *Z. Kristallogr.* **181**, 215–222.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *SAINT* and *SHELXTL* (Version 5). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tang, Y. H., Qin, Y. Y., Wu, L., Li, Z. J., Kang, Y. & Yao, Y. G. (2001). *Polyhedron*, **20**, 1–6.