

dra are not regular: the bond distances Cr(1)—O(1) = 1.767 (4) and Cr(2)—O(1) = 1.787 (4) Å are longer than the other Cr—O lengths ranging between 1.570 and 1.607 Å for Cr(1) and between 1.505 and 1.568 Å for Cr(2); the bond angles O—Cr—O range between 104 and 112° for Cr(1) and between 103 and 115° for Cr(2). Most of the dichromate O atoms are mobile: O(12) with  $B_{\text{eq}} = 10$ , O(22) with  $B_{\text{eq}} = 10.8$ , O(23) with  $B_{\text{eq}} = 11.8$  and specially O(21) with  $B_{\text{eq}} = 18.7 \text{ \AA}^2$ : this compound is not very stable and decomposes when heated above 333 K.

The two pyramidal thioocations have neither symmetry axis nor symmetry plane but, as usual, they approximate to  $3m$  symmetry; they have the same geometrical features. Fig. 1 presents a projection of the compound.

**Related literature.** As we have noted in previous papers, very little is known about the structures of the trimethyloxosulfonium salts. In this series of papers, we have already reported the iodide, bro-

mide, chloride, nitrate, chromate, chlorocadmate, bromocadmate and thiocyanate structures (Jannin, Puget, de Brauer & Perret, 1991; de Brauer, Jannin, Puget & Perret, 1991; Puget, Jannin, de Brauer & Perret, 1991; Puget, Jannin, Perret & de Brauer, 1991).

This work was carried out at the Centre de Diffractométrie de l'Université de Bourgogne.

#### References

- BRAUER, C. DE, JANNIN, M., PUGET, R. & PERRET, R. (1991). *Acta Cryst.* C47, 2231–2232.  
 BRAUER, C. DE & PERRET, R. (1992). *Bull. Soc. Chim. Fr.* In preparation.  
 Enraf–Nonius (1977). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.  
 JANNIN, M., PUGET, R., DE BRAUER, C. & PERRET, R. (1991). *Acta Cryst.* C47, 1687–1689, 2228–2229, 2229–2230.  
 PUGET, R., JANNIN, M., DE BRAUER, C. & PERRET, R. (1991). *Acta Cryst.* C47, 1803–1805.  
 PUGET, R., JANNIN, M., PERRET, R. & DE BRAUER, C. (1991). *Acta Cryst.* C47, 2232–2234.

*Acta Cryst.* (1993). C49, 751–752

## Structure of Potassium Tribromo(diethyl sulfoxide)platinate(II)

BY VITALII K. BELSKY AND VADIM E. KONOVALOV

*L. Ya. Karpov Physico-Chemical Institute, Obukha st., 10, 103064, Moscow, Russia*

AND VADIM YU. KUKUSHKIN

*Department of Chemistry, St. Petersburg University, Universitetsky pr., 2, 198904, Stary Petergof, Russia*

(Received 20 January 1992; accepted 31 July 1992)

**Abstract.**  $\text{K}[\text{Pt}(\text{C}_4\text{H}_{10}\text{SO})\text{Br}_3]$ ,  $M_r = 580.09$ , monoclinic,  $P2_1/a$ ,  $a = 9.120$  (5),  $b = 9.157$  (5),  $c = 17.505$  (4) Å,  $\gamma = 123.98$  (3)°,  $V = 1212.2$  (8) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 3.18 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 219.61 \text{ cm}^{-1}$ ,  $F(000) = 1040$ ,  $T = 298 \text{ K}$ ,  $R = 0.063$  for 1674 unique observed reflections. The complex consists of discrete  $\text{K}^+$  and  $[\text{Pt}(\text{Et}_2\text{SO})\text{Br}_3]^-$  ions. Pt<sup>II</sup> adopts a nearly square-planar geometry. Angles around the Pt atom are close to 90 and 180°. The angle between SOPt and the coordination planes is 8.6°. Bond lengths and bond angles in the S-coordinated  $\text{Et}_2\text{SO}$  ligand have normal values. The complex is an isostructural analogue of the previously characterized  $\text{K}[\text{Pt}(\text{Et}_2\text{SO})\text{Cl}_3]$  complex.

**Experimental.** The title compound was prepared as a result of interaction of  $\text{K}_2[\text{PtCl}_4]$ , KBr and diethyl sulfoxide with molar ratio 1:4:1.1. The dark orange

crystals [data crystal: size  $0.093 \times 0.093 \times 0.114 \text{ mm}$ ; faces (010), (1 $\bar{1}$ 3), (1 $\bar{1}$ 3)] were obtained by slow cooling of a solution in nitromethane–toluene mixture. Diffraction data were collected on a Syntex P1 diffractometer using Mo  $K\alpha$  radiation and  $\beta$ -filter. Cell parameters were determined from refined angles of 12 centred reflections with  $2\theta$  between 20 and 26°. 1799 independent reflections of which 1674 had  $I \geq 3\sigma(I)$  were measured for  $2\theta < 50^\circ$  by  $\theta/2\theta$  scans of speed  $3.4\text{--}12^\circ \text{ min}^{-1}$ , and for range of  $hkl$ :  $h = -10$  to 10,  $k = 0$  to 9,  $l = 0$  to 20. Standard reflections 010, 200 and 004, measured every 100 reflections, showed no change with time.

Diffractometer data were processed by the program PROFIT (Strel'tsov & Zavodnik, 1989) with profile analysis of reflections. The structure was solved by means of Fourier syntheses based on the Pt-atom coordinates obtained from Patterson syn-

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ) with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
Pt	2587 (1)	4155 (1)	2102 (1)	30 (1)
Br(1)	1608 (3)	1611 (2)	2901 (1)	47 (1)
Br(2)	3901 (2)	6817 (2)	1321 (1)	46 (1)
Br(3)	3874 (2)	5998 (2)	3245 (1)	47 (1)
K	2660 (4)	8614 (5)	2681 (2)	47 (2)
S	1259 (5)	2501 (4)	1079 (2)	33 (2)
O	39 (13)	579 (13)	1199 (5)	42 (5)
C(1)	79 (20)	3294 (19)	566 (10)	45 (7)
C(2)	1308 (25)	1934 (26)	53 (9)	52 (9)
C(3)	2874 (19)	2853 (18)	408 (8)	40 (7)
C(4)	4183 (24)	2451 (24)	732 (10)	55 (9)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) and K $\cdots$ O distances ( $\text{\AA}$ )

Pt—Br(1)	2.420 (2)	Pt—Br(2)	2.446 (2)
Pt—Br(3)	2.452 (2)	Pt—S	2.218 (3)
S—C(1)	1.83 (2)	S—O	1.48 (1)
C(3)—C(4)	1.54 (3)	S—C(3)	1.77 (2)
C(1)—C(2)	1.48 (2)		
Br(1)—Pt—Br(2)	173.8 (1)	Br(1)—Pt—Br(3)	88.2 (1)
Br(2)—Pt—Br(3)	89.5 (1)	Br(1)—Pt—S	92.4 (1)
Br(2)—Pt—S	90.3 (1)	Br(3)—Pt—S	175.9 (1)
Pt—S—C(1)	110.0 (6)	Pt—S—O	117.2 (4)
Pt—S—C(3)	109.8 (4)	O—S—C(1)	108.8 (7)
C(1)—S—C(3)	102.5 (8)	S—C(3)—C(4)	118.0 (11)
S—C(1)—C(2)	112.2 (15)		
K $\cdots$ Br(1 <sup>ii</sup> )	3.238 (3)	K $\cdots$ Br(3)	3.302 (6)
K $\cdots$ Br(1 <sup>i</sup> )	3.397 (6)	K $\cdots$ Br(3 <sup>iii</sup> )	3.323 (4)
K $\cdots$ Br(2)	3.426 (5)	K $\cdots$ O <sup>i</sup>	2.725 (9)
K $\cdots$ Br(2 <sup>iii</sup> )	3.348 (4)		

Symmetry code: (none) x, y, z; (i) x, 1.0 + y, z; (ii) 0.5 + x, 1.0 + y, 0.5 - z; (iii) -0.5 + x, y, 0.5 - z.

thesis and was refined by full-matrix least squares based on  $F$  with weights  $w = 1/[\sigma^2(F) + 0.005542F^2]$ . All non-H atoms were treated anisotropically; H atoms were not refined.  $R = 0.063$ ,  $wR = 0.067$ ,  $S = 1.07$ ,  $(\Delta/\sigma)_{\max} = 0.18$ ; in the final  $\Delta F$  map,  $(\Delta\rho)_{\max} = 3.38 \text{ e \AA}^{-3}$  near the Pt atom,  $(\Delta\rho)_{\min} = -2.79 \text{ e \AA}^{-3}$ . Lorentz, polarization and absorption corrections were made (for the latter, transmission factors were in the range 0.0078–0.0123). Calculations were carried out with *SHELXTL* (Sheldrick, 1984) on a Nova-3 computer. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1 shows atomic parameters and their equivalent isotropic thermal parameters, Table 2 the bond lengths, bond angles, and the K $\cdots$ Br and K $\cdots$ O distances.\* Fig. 1 shows a view of the [Pt(Et<sub>2</sub>SO)-

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55631 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: VS1000]

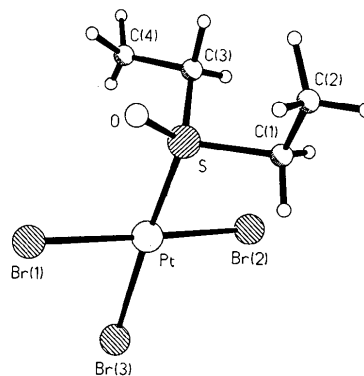


Fig. 1. View of the [Pt(Et<sub>2</sub>SO)Br<sub>3</sub>]<sup>-</sup> ion, showing the atomic numbering.

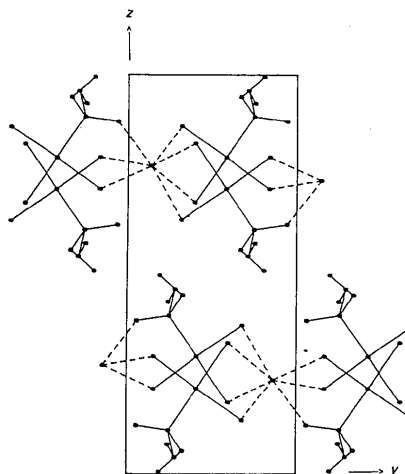


Fig. 2. Packing diagram of K[Pt(Et<sub>2</sub>SO)Br<sub>3</sub>]. Dotted lines indicate the interatomic bonds.

Br<sub>3</sub>]<sup>-</sup> ion with the atomic numbering scheme, and crystal packing is shown in Fig. 2.

**Related literature.** The title complex is isostructural with the K[Pt(Et<sub>2</sub>SO)Cl<sub>3</sub>] complex (Belsky, Kononov, Kukushkin & Aleksandrova, 1992). The crystal structure of the K[Pt(Me<sub>2</sub>SO)Cl<sub>3</sub>] complex has been published (Melanson, Hubert & Rochon, 1976).

#### References

- BELSKY, V. K., KONOVALOV, V. E., KUKUSHKIN, V. YU. & ALEKSANDROVA, E. A. (1992). *Phosphorus Sulfur Silicon*, **69**, 103–117.
- MELANSON, R., HUBERT, J. & ROCHON, F. D. (1976). *Acta Cryst.* **B32**, 1914–1916.
- SHELDRIK, G. M. (1984). *SHELXL User's Manual*. Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.
- STREL'TSOV, V. A. & ZAVODNIK, V. E. (1989). *Kristallografia*, **34**, 1369–1375.