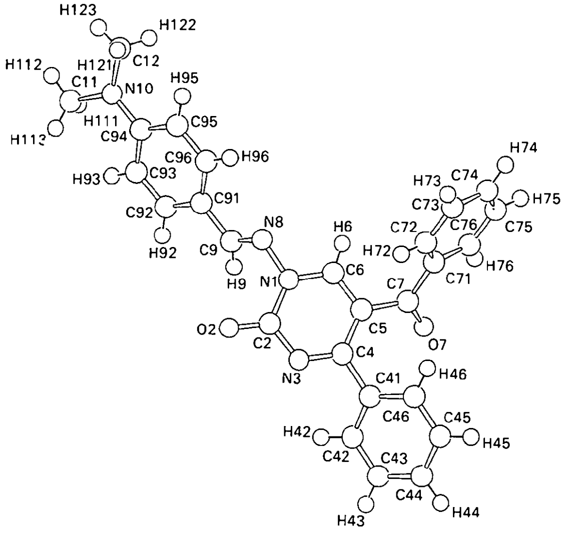

Fig. 1. A *SCHAKAL* (Keller, 1988) drawing of the title molecule with the atom-numbering scheme.

pınar, 1992). The N3—C2 and N1—C6 bond distances are respectively 1.367 (5) and 1.345 (6) Å, which are exceptional compared with the above compound [1.357 (3) and 1.332 (3) Å] and with 5-benzoyl-1-methyl-4-phenylpyrimidine-2-thione [1.385 (4) and 1.316 (4) Å (Özbey *et al.*, 1991)]. In the pyrimidine ring, the angle between the planes N1,C6,C5 and C2,N3,C4 is 7.5°; this angle appears sensitive to the type of substituents present on N(1) since in 5-benzoyl-1-(diphenylmethyleneamino)-4-phenyl-1*H*-pyrimidine-2-one it is 5.3° (Akçamur *et al.*, 1988) and in 1-amino-5-benzoyl-4-phenyl-1*H*-pyrimidine-2-thione it is 0.74° (Akkurt *et al.*, 1992).

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SHORT-FORMAT PAPERS

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Acta Cryst. (1993). C**49**, 749–751

Structures of Trimethyloxosulfonium Salts. VII. The Dichromate: $[(CH_3)_3SO]_2Cr_2O_7$

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Abstract. Trimethyloxosulfonium dichromate, $C_3H_9OS^+.Cr_2O_7^-$, $M_r = 402.32$, monoclinic, $P2_1$, $a = 8.917\,(2)$, $b = 10.319\,(2)$, $c = 8.418\,(1)$ Å, $\beta = 102.30\,(5)°$, $V = 756.8\,(6)$ Å^3, $Z = 2$, $D_m = 1.75\,(3)$, $D_x = 1.765$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.698$ mm^{-1}, $F(000) = 412$, $T = 293$ K, final $R =$

0.038, $wR = 0.038$ for 2571 independent observed reflections. As usual, the dichromate ion is formed by two non-regular CrO_4 tetrahedra, linked by the O(1) atom. The two pyramidal thiocations, $(CH_3)_3SO^+$, with approximate $3m$ symmetry, have the same geometrical features.

Experimental. The preparation of the crystals will be described elsewhere (de Brauer & Perret, 1992).

A red–yellow prismatic crystal (density measured in toluene) of size 0.19 × 0.19 × 0.21 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer. Unit-cell dimensions were determined from least-squares refinement of 25 reflections with $5 < \theta < 15°$. Systematic absences: $0k0$ $(k = 2n)$. Space group: $P2_1$ (No.4) or $P2_1/m$ (No.11). $\omega/2\theta$ scans of width 1.2° were used to measure reflections over $1 < \theta < 30°$, $-10 < h < 10$, $-12 < k < 12$, $0 < l < 9$. Four orientation reference reflections ($\bar{2}2\bar{1}$, $\bar{3}03$, $\bar{2}\bar{2}\bar{2}$ and $\bar{1}3\bar{1}$), monitored every 200 scans, showed no significant variations; the same intensity reference reflections recorded every 2 h showed a fading of 5.3% during 159 h; decay corrections were applied. Of 5312 measured reflections, 5120 had $I > 3\sigma(I)$. Lorentz and polarization corrections were applied as well as absorption corrections from ψ scans (relative transmission factors between 0.944 and 0.999). 2592 reflections were obtained after averaging ($R_{\rm int} = 0.018$).

Patterson functions were used for structure determination in $P2_1$. H atoms were found from a difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) was performed for non-H atoms, isotropic for H atoms. Unit weights were used in the refinement of 243 parameters. Final refinement with 2571 reflections (21 rejected as bad) gave $R = 0.038$, $wR = 0.038$; $S = 0.813$. Maximum heights in the final difference Fourier synthesis were 0.995 and -0.696 e Å^{-3}; maximum Δ/σ was 0.01.

Scattering factors for neutral atoms and f' f'' values were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The Enraf-Nonius (1977) *SDP* was used for all calculations on a VAX 730 computer.

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{\rm eq} = (4/3)\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Cr(1)	0.80658 (8)	0.500*	0.46655 (9)	2.84 (1)
Cr(2)	0.52455 (9)	0.52208 (9)	0.1613 (1)	3.61 (2)
O(1)	0.6956 (5)	0.4500 (4)	0.2769 (6)	6.3 (1)
O(11)	0.9637 (4)	0.4194 (4)	0.4871 (5)	4.54 (9)
O(12)	0.7287 (6)	0.4641 (6)	0.6113 (6)	10.0 (1)
O(13)	0.8372 (5)	0.6521 (3)	0.4576 (6)	5.4 (1)
O(21)	0.5362 (9)	0.500 (1)	−0.0123 (7)	18.7 (3)
O(22)	0.3831 (6)	0.4464 (6)	0.192 (1)	10.8 (2)
O(23)	0.5014 (7)	0.6663 (5)	0.210 (1)	11.8 (2)
S(1)	0.0022 (1)	0.3587 (1)	0.0222 (1)	2.76 (2)
O(10)	0.0070 (5)	0.4975 (4)	0.0442 (5)	4.57 (8)
C(11)	−0.1851 (6)	0.3003 (5)	−0.0399 (7)	3.9 (1)
C(12)	0.0832 (6)	0.2738 (5)	0.1981 (6)	3.7 (1)
C(13)	0.1028 (6)	0.3099 (6)	−0.1241 (6)	4.1 (1)
S(2)	0.3058 (1)	0.5809 (1)	0.6290 (1)	2.85 (2)
O(20)	0.2139 (4)	0.6011 (4)	0.7469 (4)	4.11 (8)
C(21)	0.2096 (6)	0.6284 (6)	0.4327 (7)	4.2 (1)
C(22)	0.3592 (6)	0.4188 (5)	0.6129 (7)	3.9 (1)
C(23)	0.4752 (7)	0.6691 (5)	0.6702 (8)	4.5 (1)

* Coordinate fixed to define origin of structure with non-centrosymmetric space group $P2_1$.

Table 2. *Main interatomic distances* (Å) *and bond angles* (°)

Dichromate anion

Cr(1)—O(1)—Cr(2) 128.3 (3)

Cr(1)	O(1)	O(11)	O(12)	O(13)
O(1)	1.767 (4)	2.673 (5)	2.772 (7)	2.728 (6)
O(11)	104.7 (3)	1.607 (4)	2.575 (7)	2.643 (5)
O(12)	112.2 (2)	108.4 (2)	1.570 (6)	2.628 (7)
O(13)	108.2 (3)	111.1 (2)	112.2 (3)	1.599 (4)

Cr(2)	O(1)	O(21)	O(22)	O(23)
O(1)	1.787 (4)	2.596 (7)	2.725 (7)	2.808 (7)
O(21)	103.8 (4)	1.505 (7)	2.48 (1)	2.60 (1)
O(22)	109.2 (3)	108.4 (5)	1.552 (6)	2.493 (8)
O(23)	113.5 (3)	115.8 (5)	106.1 (4)	1.568 (6)

Trimethyloxosulfonium cations

S(1)	O(10)	C(11)	C(12)	C(13)
O(10)	1.444 (4)	2.656 (6)	2.663 (6)	2.645 (8)
C(11)	112.4 (2)	1.748 (5)	2.788 (7)	2.807 (8)
C(12)	113.3 (2)	106.1 (3)	1.740 (5)	2.780 (7)
C(13)	111.8 (3)	106.9 (3)	105.8 (3)	1.745 (7)

S(2)	O(20)	C(21)	C(22)	C(23)
O(20)	1.431 (5)	2.652 (7)	2.667 (7)	2.642 (7)
C(21)	112.0 (2)	1.761 (6)	2.810 (7)	2.786 (7)
C(22)	113.4 (3)	106.2 (3)	1.752 (5)	2.786 (7)
C(23)	112.9 (3)	105.7 (3)	106.1 (3)	1.734 (6)

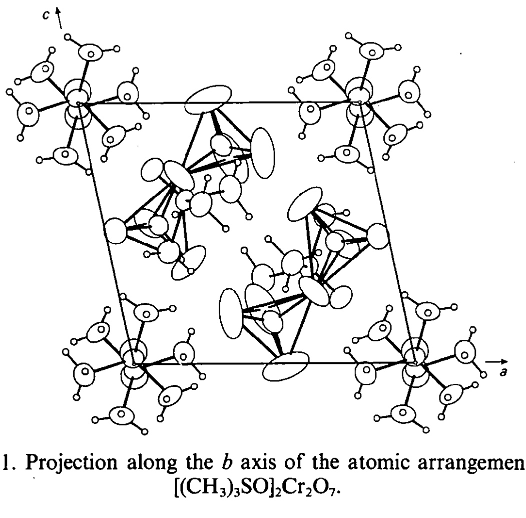

Fig. 1. Projection along the b axis of the atomic arrangement of $[(CH_3)_3SO]_2Cr_2O_7$.

The final atomic coordinates are reported in Table 1 and the main interatomic distances and bond angles are listed in Table 2.* The dichromate ion is formed, as usual, by two non-regular CrO_4 tetrahedra, linked by an apex, here the O(1) atom, with Cr(1)—O(1)—Cr(2) = 128.3 (3)°. These two tetrahe-

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55605 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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 $B_{eq} = 10$, O(22) with $B_{eq} = 10.8$, O(23) with $B_{eq} = 11.8$ and specially O(21) with $B_{eq} = 18.7$ Å^2: this compound is not very stable and decomposes when heated above 333 K.

The two pyramidal thiocations 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, bromide, 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.

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

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Abstract. K[Pt($C_4H_{10}SO$)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^3, $Z = 4$, $D_x = 3.18$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 219.61$ cm^{-1}, $F(000) = 1040$, $T = 298$ K, $R = 0.063$ for 1674 unique observed reflections. The complex consists of discrete K^+ and $[Pt(Et_2SO)Br_3]^-$ ions. Pt^{II} 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 Et_2SO ligand have normal values. The complex is an isostructural analogue of the previously characterized K[Pt(Et_2SO)Cl_3] complex.

Experimental. The title compound was prepared as a result of interaction of $K_2[PtCl_4]$, KBr and diethyl sulfoxide with molar ratio 1:4:1.1. The dark orange crystals [data crystal: size 0.093 × 0.093 × 0.114 mm; faces (010), ($1\bar{1}\bar{3}$), ($1\bar{1}3$)] were obtained by slow cooling of a solution in nitromethane–toluene mixture. Diffraction data were collected on a Syntex $P\bar{1}$ diffractometer using Mo $K\alpha$ radiation and β-filter. Cell parameters were determined from refined angles of 12 centred reflections with 2θ between 20 and 26°. 1799 independent reflections of which 1674 had $I \geq 3\sigma(I)$ were measured for $2\theta < 50°$ by $\theta/2\theta$ scans of speed 3.4–12° 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-

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