

$|w = 1/\sigma^2(|F_o|)|$ ,  $S = 1.13$  and  $(\Delta/\sigma)_{\max} = 0.002$ . A final difference Fourier synthesis showed  $(\Delta\rho)_{\max} = 0.89$ ,  $(\Delta\rho)_{\min} = -0.78 \text{ e } \text{\AA}^{-3}$ . Minor disorder in ring C(13)–C(18) is indicated by slightly elevated temperature factors and two rather short C–C distances. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). All computer programs from the *TEXSAN* crystal structure analysis package (Molecular Structure Corporation, 1985). Table 1 presents atomic coordinates and  $B_{\text{eq}}$  temperature factors while Table 2\* lists selected interatomic distances and angles. Fig. 1 illustrates the molecular configuration, the thermal motion and the atom-numbering scheme.

**Related literature.** While other Os–As bonds have been reported and characterized (Bottomley, Lin & White, 1978), this compound is apparently the first triphenylarsine-containing Os complex to have been structurally characterized.

\* Lists of structure factors, anisotropic thermal parameters, distances and angles involving the phenyl rings, H-atom coordinates, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44446 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

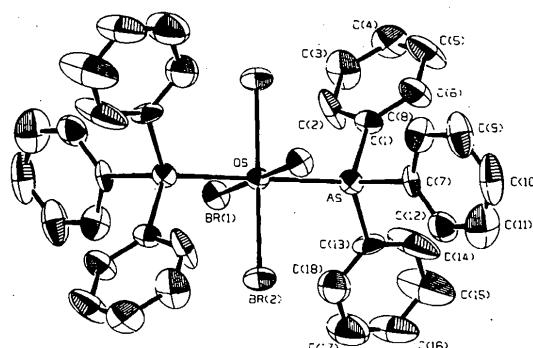


Fig. 1. Molecular structure and numbering scheme; thermal ellipsoids shown at the 50% probability level.

This research was supported by the Southern Illinois University Materials Technology Center.

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*Acta Cryst.* (1988). **C44**, 372–374

## Structure of Tetramethylammonium Cerium(III) Bis(sulfate) Trihydrate

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(Received 3 June 1987; accepted 21 October 1987)

**Abstract.**  $(\text{C}_4\text{H}_{12}\text{N})\text{Ce}(\text{SO}_4)_2 \cdot 3\text{H}_2\text{O}$ ,  $M_r = 460.44$ , orthorhombic,  $Pca2_1$ ,  $a = 11.670(2)$ ,  $b = 5.440(1)$ ,  $c = 21.841(3) \text{ \AA}$ ,  $V = 1386(2) \text{ \AA}^3$ ,  $Z = 4$ ,  $D_m = 2.22(5)$ ,  $D_x = 2.206 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } \text{K}\alpha) = 0.71069 \text{ \AA}$ ,  $\mu = 36.7 \text{ mm}^{-1}$ ,  $F(000) = 880$ ,  $T = 293(1) \text{ K}$ , final  $R = 0.030$  for 2034 observed reflexions. The Ce atom is coordinated by eight O atoms (five from sulfate groups, three from water molecules) in the form of an irregular polyhedron. The Ce–O distances range from 2.361(5) to 2.593(4)  $\text{\AA}$ . The  $\text{N}(\text{CH}_3)_4^+$  cation has the usual tetrahedral shape. The structure can be described as layers of  $\text{N}(\text{CH}_3)_4^+$  cations,  $\text{SO}_4$  anions, Ce and water molecules,  $\text{SO}_4$  anions,  $\text{N}(\text{CH}_3)_4^+$  cations, ... .

**Experimental.** Single crystals prepared by slow evaporation of aqueous solution containing equivalent quantities of tetramethylammonium sulfate and cerium(III) sulfate. Colourless prismatic crystals with well developed  $\{100\}$ ,  $\{010\}$  and  $\{001\}$  faces were obtained.  $D_m$  measured by flotation (mixture of diiodomethane and toluene). Crystal size  $0.33 \times 0.25 \times 0.28 \text{ mm}$ . Lattice parameters were determined by least-squares fit of  $\theta$  values of 60 reflexions ( $10 < \theta < 14^\circ$ ); Enraf–Nonius CAD-4 diffractometer; data collected using  $\omega/2\theta$  scans;  $[(\sin\theta)/\lambda]_{\max} = 0.70 \text{ \AA}^{-1}$ ; variable scan rate (min.  $1.68$ , max.  $20.1^\circ \text{ min}^{-1}$ ), scan time 40 s, scan width  $(2\theta) (0.6 +$

Table 1. Final atomic coordinates ( $\times 10^5$  for Ce and S;  $\times 10^4$  for O, N and C) and  $U_{eq}$  ( $\text{\AA}^2 \times 10^3$ ) (Hamilton, 1959) with e.s.d.'s in parentheses

|       | <i>x</i>   | <i>y</i>   | <i>z</i>  | $U_{eq}$ |
|-------|------------|------------|-----------|----------|
| Ce    | 2800 (2)   | 18296 (3)  | 25000     | 15.2 (1) |
| S(1)  | 7642 (11)  | 68176 (20) | 13824 (5) | 18.1 (5) |
| S(2)  | 44592 (11) | 38749 (22) | 34096 (6) | 19.0 (5) |
| O(1)  | 1958 (4)   | 5930 (8)   | 1314 (2)  | 28 (2)   |
| O(2)  | 836 (4)    | 9266 (8)   | 1679 (2)  | 33 (2)   |
| O(3)  | 201 (5)    | 6971 (11)  | 797 (2)   | 39 (3)   |
| O(4)  | 129 (5)    | 5161 (11)  | 1796 (3)  | 36 (2)   |
| O(5)  | 5657 (4)   | 4808 (8)   | 3303 (2)  | 28 (2)   |
| O(6)  | 3726 (3)   | 5629 (7)   | 3065 (2)  | 24 (2)   |
| O(7)  | 4334 (4)   | 1398 (7)   | 3134 (2)  | 24 (2)   |
| O(8)  | 4177 (6)   | 3828 (9)   | 4051 (2)  | 38 (2)   |
| N     | 1861 (5)   | -0202 (9)  | 4948 (2)  | 28 (2)   |
| C(1)  | 2488 (8)   | 1440 (14)  | 5382 (4)  | 40 (3)   |
| C(2)  | 1152 (7)   | 1354 (14)  | 4525 (3)  | 37 (3)   |
| C(3)  | 1094 (7)   | -1892 (12) | 5293 (4)  | 38 (3)   |
| C(4)  | 2695 (8)   | -1684 (16) | 4585 (4)  | 46 (4)   |
| O(10) | 2308 (3)   | 3182 (8)   | 2268 (2)  | 25 (2)   |
| O(11) | 1782 (4)   | -488 (8)   | 3107 (2)  | 30 (2)   |
| O(12) | -1541 (4)  | 1026 (9)   | 1971 (2)  | 30 (2)   |

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses

| (a) $\text{SO}_4$ groups            |            |                        |           |
|-------------------------------------|------------|------------------------|-----------|
| S(1)–O(1)                           | 1.481 (5)  | S(2)–O(5)              | 1.505 (5) |
| S(1)–O(2)                           | 1.484 (5)  | S(2)–O(6)              | 1.487 (4) |
| S(1)–O(3)                           | 1.440 (6)  | S(2)–O(7)              | 1.483 (4) |
| S(1)–O(4)                           | 1.475 (6)  | S(2)–O(8)              | 1.440 (5) |
| O(1)–S(1)–O(2)                      | 106.5 (3)  | O(5)–S(2)–O(6)         | 103.8 (2) |
| O(1)–S(1)–O(3)                      | 111.0 (3)  | O(5)–S(2)–O(7)         | 109.6 (2) |
| O(1)–S(1)–O(4)                      | 109.5 (3)  | O(5)–S(2)–O(8)         | 111.7 (3) |
| O(2)–S(1)–O(3)                      | 111.2 (3)  | O(6)–S(2)–O(7)         | 108.7 (2) |
| O(2)–S(1)–O(4)                      | 108.0 (3)  | O(6)–S(2)–O(8)         | 111.9 (3) |
| O(3)–S(1)–O(4)                      | 110.4 (3)  | O(7)–S(2)–O(8)         | 110.9 (3) |
| (b) $\text{N}(\text{CH}_3)_4$ group |            |                        |           |
| N–C(1)                              | 1.494 (10) | C(1)–N–C(2)            | 108.9 (5) |
| N–C(2)                              | 1.502 (9)  | C(1)–N–C(3)            | 110.0 (5) |
| N–C(3)                              | 1.489 (9)  | C(1)–N–C(4)            | 110.0 (6) |
| N–C(4)                              | 1.492 (11) | C(2)–N–C(3)            | 109.2 (6) |
|                                     |            | C(2)–N–C(4)            | 109.7 (6) |
|                                     |            | C(3)–N–C(4)            | 109.2 (5) |
| (c) Ce polyhedron                   |            |                        |           |
| Ce–O(4)                             | 2.384 (6)  | Ce–O(10)               | 2.529 (4) |
| Ce–O(11)                            | 2.534 (5)  | Ce–O(12)               | 2.458 (5) |
| Ce–O(2 <sup>ii</sup> )              | 2.362 (5)  | Ce–O(5 <sup>ii</sup> ) | 2.572 (5) |
| Ce–O(6 <sup>ii</sup> )              | 2.592 (4)  | Ce–O(7 <sup>ii</sup> ) | 2.495 (4) |

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

$0.2\tan\theta$ °, aperture  $(2.5 + 0.9\tan\theta)$  mm; background measured for 1/4 of scan time at each of scan limits. Three reflexions (304, 208, 219) monitored at intervals of 140 reflexions, no intensity decline ( $-0.6\%$ ); data corrected for intensity variation and Lp effects; an exact absorption correction was performed (12 faces, grid:  $8 \times 8 \times 8$ , transmission: max. 0.447, min. 0.377). 8044 ( $+h, \pm k, \pm l$ ) measured reflexions, 2077 unique reflexions [ $R_{int}(I) = 0.040$  for 7889 contributing reflexions], 2034 observed reflexions ( $h 0/16, k 0/7, l 0/30$ ) with  $I > 3\sigma(I)$ ,  $\sigma(I)$  based on counting statistics;  $Pca2_1$  chosen after examination of systematic absences,

distribution of  $|E|$  values and distribution of peaks in Patterson map. Ce atom located from Patterson map, subsequent Fourier map revealed positions of the other non-H atoms.  $Pca2_1$  confirmed by the successful structure analysis; the refinement in the centrosymmetric alternative  $Pcam$  ( $=Pbcm$ ) with the necessary disorder yielded  $R = 0.091$  with non-positive temperature factor for O(4) and S–O distances ranging from 1.23 (2) to 1.52 (2)  $\text{\AA}$ . Refinement by full-matrix least squares on  $F$  of positional and anisotropic thermal parameters of all non-H atoms converged to final  $R = 0.030$  and  $wR = 0.032$  with mean and max. values of  $\Delta/\sigma = 0.009$  and 0.047. 171 variables. Weighting function  $w = w_F w_S$  determined empirically from

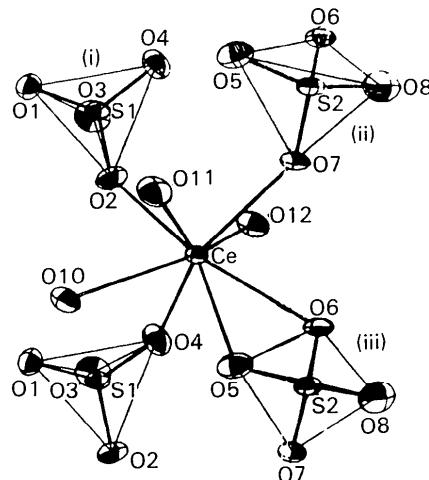


Fig. 1. The coordination around the Ce atom. Symmetry code: (i)  $x, -1 + y, z$ ; (ii)  $-\frac{1}{2} + x, -y, z$ ; (iii)  $-\frac{1}{2} + x, 1 - y, z$ . Thermal ellipsoids are drawn at the 50% probability level (Johnson, 1965).

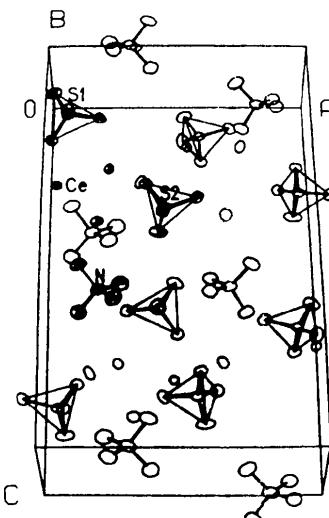


Fig. 2. A view of the unit-cell contents.

$w_F(|F_o| < 25) = (|F_o|/25)^2$ ,  $w_F(|F_o| > 30) = (30/|F_o|)^2$ , and  $w_F(25 < |F_o| < 30) = 1.0$ , and  $w_s(\sin\theta < 0.40) = (\sin\theta/0.40)^{2.5}$ ,  $w_s(\sin\theta > 0.42) = (0.42/\sin\theta)^2$ , and  $w_s(0.40 < \sin\theta < 0.42) = 1.0$ . H atoms were found in the difference map and were included with calculated positions and  $U = 0.06 \text{ \AA}^2$  as fixed atom contribution to the structure-factor calculation. Final difference Fourier map shows max. height of  $1.04 \text{ e \AA}^{-3}$  close to Ce atom, min.  $-0.83 \text{ e \AA}^{-3}$ . The hand of the crystal was established by the refinement of imaginary component of the anomalous dispersion [2.1 (7) e].

All calculations carried out using *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).\* Atomic scattering factors for non-H atoms from Cromer & Mann (1968). Anomalous-dispersion correction factors for non-H atoms from Cromer & Liberman (1970).

Table 1 gives the atomic coordinates and Table 2 bond lengths and angles. Fig. 1 shows the coordination

sphere around the Ce atom with the labelling of atoms, Fig. 2 shows the packing in the unit cell.

**Related literature.** The crystal structure of the title compound was determined as part of a systematic study concerning double salts of the rare earths of general formula  $M^3\text{Ln}^{III}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$  (Arhar, Golič, Jordanovska & Šiftar, 1981; Kaučič, Bukovec & Golič, 1985, and references therein).

Financial support of the Research Community of Slovenia is gratefully acknowledged.

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*Acta Cryst.* (1988). **C44**, 374–376

## 2-(2,5-Dimethoxybenzyl)-3,6-dimethoxy-4-methylbenzonitrile

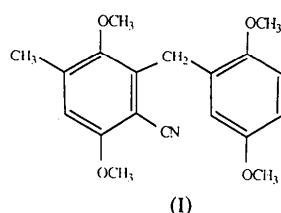
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(Received 16 September 1987; accepted 29 September 1987)

**Abstract.**  $C_{19}H_{21}NO_4$ ,  $M_r = 327.41$ , monoclinic,  $P2_1/c$ ,  $a = 8.152$  (3),  $b = 21.858$  (7),  $c = 9.872$  (3)  $\text{\AA}$ ,  $\beta = 103.83$  (2) $^\circ$ ,  $V = 1708.1$  (9)  $\text{\AA}^3$ ,  $Z = 4$ ,  $D_x = 1.27 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$ ,  $\mu(\text{Mo } K\alpha) = 0.83 \text{ cm}^{-1}$ ,  $F(000) = 696$ ,  $T = 295 \text{ K}$ , final  $R = 0.041$  for 1524 observed reflections. The bond distances are  $\text{H}_2\text{C}-\text{C}(\text{av.}) = 1.510$  (3),  $\text{C}=\text{N} = 1.144$  (4),  $\text{C}-\text{CN} = 1.427$  (4),  $\text{C}-\text{CH}_3 = 1.505$  (4),  $\text{H}_3\text{C}-\text{O}(\text{av.}) = 1.426$  (4),  $\text{O}-\text{C}(\text{benz.}, \text{av.}) = 1.372$  (4) and  $\text{C}-\text{C}(\text{benz.}, \text{av.}) = 1.386$  (4)  $\text{\AA}$ . The X-ray structure unambiguously confirms the 4-position of the methyl group. The benzene rings are planar and the angle between the normals to the planes of the rings is  $85.0$  (1) $^\circ$ .

**Experimental.** The title compound (I) was prepared in 20% yield by the reaction of lithio-2,5-dimethoxyphenylacetonitrile and 2-bromo-5-methyl-1,4-dimethoxybenzene (generated *in situ* with LDA in THF) involving a tandem addition rearrangement.



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