Table 1. *Atomic coordinates for the non-*H *atoms and equivalent isotropic temperature factors* (Å$^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| C1 | 0.1936 (2) | 0.3392 (2) | −0.0070 (1) | 2.27 (3) |
| C2 | 0.2840 (2) | 0.5038 (2) | 0.0642 (1) | 2.12 (3) |
| C21 | 0.3718 (2) | 0.5287 (2) | 0.2183 (1) | 2.55 (3) |
| O21 | 0.3886 (3) | 0.3947 (1) | 0.2740 (1) | 4.48 (4) |
| O22 | 0.4210 (3) | 0.6806 (1) | 0.2744 (1) | 4.58 (4) |
| C3 | 0.2962 (2) | 0.6467 (2) | −0.0062 (1) | 2.28 (3) |
| C4 | 0.2178 (2) | 0.6203 (2) | −0.1470 (1) | 2.39 (3) |
| N41 | 0.2331 (2) | 0.7702 (2) | −0.2220 (1) | 3.16 (4) |
| O41 | 0.1234 (2) | 0.7516 (2) | −0.3427 (1) | 4.21 (4) |
| O42 | 0.3512 (2) | 0.9037 (1) | −0.1618 (1) | 4.62 (5) |
| C5 | 0.1265 (2) | 0.4590 (2) | −0.2209 (1) | 2.55 (3) |
| C6 | 0.1166 (2) | 0.3218 (2) | −0.1475 (1) | 2.38 (3) |
| N61 | 0.0149 (2) | 0.1494 (2) | −0.2244 (1) | 3.11 (3) |
| O61 | −0.0073 (2) | 0.1267 (1) | −0.3458 (1) | 4.17 (4) |
| O62 | −0.0458 (2) | 0.0367 (1) | −0.1638 (1) | 4.50 (4) |
| N11 | 0.6408 (2) | −0.1932 (2) | 0.5325 (1) | 2.91 (3) |
| C12 | 0.5048 (2) | −0.0648 (2) | 0.5502 (1) | 2.64 (4) |

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| C1—C2 | 1.389 (2) | C1—C6 | 1.383 (1) |
| C2—C21 | 1.516 (1) | C2—C3 | 1.393 (2) |
| C21—O21 | 1.239 (2) | C21—O22 | 1.233 (2) |
| C3—C4 | 1.385 (1) | C4—C5 | 1.378 (2) |
| N41—O41 | 1.230 (1) | N41—O42 | 1.215 (2) |
| C5—C6 | 1.374 (2) | N61—O61 | 1.224 (2) |
| N61—O62 | 1.226 (2) | N11—C12 | 1.480 (2) |
| C12—C12′ | 1.509 (2) | | |
| | | | |
| C2—C1—C6 | 118.6 (1) | C1—C2—C3 | 119.9 (1) |
| C1—C2—C21 | 120.3 (1) | C21—C2—C3 | 119.7 (1) |
| C2—C21—O22 | 116.2 (1) | C2—C21—O21 | 116.7 (1) |
| O21—C21—O22 | 127.1 (1) | C2—C3—C4 | 118.7 (1) |
| C3—C4—C5 | 123.0 (1) | C3—C4—N41 | 119.0 (1) |
| N41—C4—C5 | 118.1 (1) | C4—N41—O42 | 118.6 (1) |
| C4—N41—O41 | 117.2 (2) | O41—N41—O42 | 124.2 (1) |
| C4—C5—C6 | 116.5 (1) | C1—C6—C5 | 123.4 (1) |
| C5—C6—N61 | 117.3 (1) | C1—C6—N61 | 119.3 (1) |
| C6—N61—O62 | 118.2 (1) | C6—N61—O61 | 118.3 (1) |
| O61—N61—O62 | 123.5 (1) | N11—C12—C12′ | 112.6 (1) |

respect to the phenyl-group plane by 11.25 (9), 15.4 (1) and 162.0 (1)°, respectively. The molecules are stacked in the $x$ and $y$ directions, stacking distances being 3.650 (3) and 3.744 (2) Å between molecules related by the site symmetries $-x$, $1-y$, $-z$ and $1-x$, $1-y$, $-z$, respectively.

The first interesting feature of this structure is that C—H$^+$ donors of ethylenediamine and N—O$^-$ acceptors of dinitrobenzoate are polarized to the extent that detectable C—H···O—N bonds operate towards stabilization of the structure in the lattice.

Though chelating, —COOH of dinitrobenzoic acid loses the proton to a neutral N atom of ethylenediamine which suggests a strong polarization of the N-atom lone pair by the proton of the carboxyl group and its ultimate transfer giving rise to 'self-complexation' (Poonia & Bajaj, 1979).

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## SHORT-FORMAT PAPERS

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# Structure of Pentakis(dimethyl sulfoxide)dioxouranium(VI) Bis(tetrafluoroborate)

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**Abstract.** $[(UO_2)(OSC_2H_6)_5][BF_4]_2$, $M_r = 834.28$, triclinic, $P\bar{1}$, $a = 11.455$ (3), $b = 12.401$ (4), $c = 12.549$ (3) Å, $\alpha = 72.64$ (2), $\beta = 64.20$ (2), $\gamma = 62.74$ (2)°, $V = 1415$ (2) Å$^3$, $Z = 4$, $D_x = 3.914$ g cm$^{-3}$, Mo $K\alpha_1$, $\lambda = 0.71073$ Å, $\mu = 117.3$ cm$^{-1}$, $F(000) = 1600$, $T = 295$ K, $R = 0.035$ for

0108-2701/92/122209-03$06.00

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å$^2$)

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

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| U | 0.21479 (5) | 0.47438 (4) | 0.22685 (4) | 2.93 (1) |
| S(1) | 0.2265 (4) | 0.7413 (3) | 0.0124 (3) | 4.5 (1) |
| S(2) | 0.2642 (3) | 0.4338 (3) | −0.0591 (3) | 3.99 (9) |
| S(3) | 0.2719 (3) | 0.1762 (3) | 0.2223 (3) | 4.4 (1) |
| S(4) | 0.2931 (3) | 0.2917 (3) | 0.4902 (3) | 4.18 (9) |
| S(5) | 0.1752 (5) | 0.6366 (4) | 0.4416 (4) | 7.4 (1) |
| F(1) | 0.2189 (9) | 0.911 (1) | 0.7536 (8) | 9.4 (3) |
| F(2) | 0.128 (1) | 0.7887 (9) | 0.749 (1) | 12.2 (5) |
| F(3) | 0.168 (1) | 0.924 (1) | 0.5957 (8) | 12.3 (5) |
| F(4) | −0.001 (1) | 0.019 (1) | 0.235 (1) | 10.9 (4) |
| F(5) | 0.4858 (9) | 0.8266 (9) | 0.1770 (8) | 8.4 (3) |
| F(6) | 0.320 (1) | 0.174 (1) | 0.825 (1) | 13.2 (5) |
| F(7) | 0.433 (1) | 0.281 (1) | 0.6733 (9) | 10.1 (4) |
| F(8) | 0.479 (1) | 0.9183 (9) | 0.3132 (9) | 11.0 (4) |
| O(1) | 0.2424 (9) | 0.6622 (7) | 0.1296 (7) | 5.2 (3) |
| O(2) | 0.1818 (8) | 0.5182 (7) | 0.0400 (6) | 3.8 (2) |
| O(3) | 0.1752 (8) | 0.3107 (6) | 0.2084 (7) | 4.1 (2) |
| O(4) | 0.2200 (8) | 0.3271 (7) | 0.4010 (7) | 4.3 (2) |
| O(5) | 0.2657 (8) | 0.5461 (7) | 0.3505 (7) | 4.4 (2) |
| O(11) | 0.0316 (8) | 0.5541 (8) | 0.2944 (8) | 5.0 (3) |
| O(12) | 0.3979 (7) | 0.3939 (7) | 0.1574 (7) | 3.9 (2) |
| C(1) | 0.283 (1) | 0.861 (1) | 0.001 (1) | 6.0 (5) |
| C(2) | 0.039 (2) | 0.829 (1) | 0.050 (1) | 6.1 (5) |
| C(3) | 0.397 (2) | 0.490 (1) | −0.166 (2) | 7.9 (6) |
| C(4) | 0.149 (1) | 0.489 (2) | −0.144 (1) | 7.4 (5) |
| C(5) | 0.279 (2) | 0.112 (1) | 0.107 (1) | 6.6 (5) |
| C(6) | 0.162 (1) | 0.107 (1) | 0.347 (1) | 5.7 (5) |
| C(7) | 0.473 (2) | 0.201 (2) | 0.411 (2) | 8.4 (7) |
| C(8) | 0.240 (2) | 0.173 (1) | 0.592 (1) | 6.5 (5) |
| C(9) | 0.189 (2) | 0.776 (1) | 0.377 (1) | 7.7 (6) |
| C(10) | 0.284 (2) | 0.594 (2) | 0.528 (2) | 15.3 (8) |
| B(1) | 0.133 (2) | 0.900 (1) | 0.713 (1) | 5.1 (5) |
| B(2) | 0.450 (2) | 0.176 (2) | 0.750 (2) | 6.8 (6) |

2465 independent observed reflections with $I > 3\sigma(I)$. The title compound is composed of discrete ions $[(UO_2)(dmso)_5]^{2+}$ (dmso = $OSC_2H_6$) and $BF_4^-$. The coordination polyhedron of the U atom is a pentagonal bipyramid with the pentagonal base made up of five dmso O atoms [U—O = 2.365 (5)–2.401 (5) Å]. The uranyl group is linear [U—O = 1.775 (5) and 1.779 (5) Å] and perpendicular to the equatorial plane.

**Experimental.** A yellow–green single crystal of dimensions 0.30 × 0.25 × 0.15 mm, grown during the reaction between $UO_3$ and fluoroboric acid in $H_2O$–dmso solution, was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite monochromator. Lattice parameters were determined by least-squares procedure from setting angles of 25 reflections with $\theta = 8–12°$. Data were collected up to $2\theta = 42°$, for $0 \leq h \leq 11$, $-12 \leq k \leq 12$, $-12 \leq l \leq 12$, using $\omega$–$2\theta$ scans with $\omega$-scan width $(0.80 + 0.35\tan\theta)°$, at 1.3–10° min$^{-1}$. Three standard reflections measured every hour (352, $\bar{2}\bar{1}\bar{5}$, 030) showed loss of 7.5% in 48 h, which was linearly corrected. Lorentz–polarization correction was applied, as well as an empirical absorption correction using the program *DIFABS* (Walker & Stuart, 1983), with maximum and minimum transmission of 1.205 and 0.802, respectively. 3233 reflections were measured, 3036 unique ($R_{int}$ = 0.025), of which 2465 with $I > 3\sigma(I)$ were labelled observed and used in structure solution and refinement. The structure was solved by the heavy-atom method, and refined by full-matrix least-squares procedures based on $F$ with anisotropic thermal parameters; 298 parameters refined; H atoms not introduced. Calculations were carried out on a MicroVAX II computer using the *SDP-Plus* suite of programs (Frenz, 1985). Atomic scattering factors including anomalous-scattering

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

| | | | | | |
|---|---|---|---|---|---|
| Uranium coordination sphere | | | | | |
| U—O(11) | 1.775 (5) | U—O(12) | 1.779 (5) | | |
| U—O(1) | 2.379 (5) | U—O(2) | 2.401 (5) | | |
| U—O(3) | 2.365 (5) | U—O(4) | 2.397 (5) | | |
| U—O(5) | 2.372 (5) | | | | |
| O(11)—U—O(12) | 179.1 (3) | O(11)—U—O(1) | 90.4 (2) | | |
| O(1)—U—O(2) | 72.3 (2) | O(1)—U—O(5) | 71.7 (2) | | |
| O(2)—U—O(3) | 72.2 (2) | O(3)—U—O(4) | 70.8 (2) | | |
| O(4)—U—O(5) | 72.9 (2) | | | | |
| dmso | | | | | |
| S(1)—O(1) | 1.538 (5) | S(2)—O(2) | 1.543 (5) | S(3)—O(3) | 1.531 (5) |
| S(1)—C(1) | 1.814 (9) | S(2)—C(3) | 1.796 (9) | S(3)—C(5) | 1.799 (9) |
| S(1)—C(2) | 1.814 (9) | S(2)—C(4) | 1.813 (9) | S(3)—C(6) | 1.798 (8) |
| U—O(1)—S(1) | 132.7 (3) | U—O(2)—S(2) | 126.0 (3) | U—O(3)—S(3) | 124.0 (3) |
| S(4)—O(4) | 1.528 (5) | S(5)—O(5) | 1.520 (5) | | |
| S(4)—C(7) | 1.807 (9) | S(5)—C(9) | 1.724 (9) | | |
| S(4)—C(8) | 1.789 (8) | S(5)—C(10) | 1.79 (1) | | |
| U—O(4)—S(4) | 133.4 (3) | U—O(5)—S(5) | 133.0 (3) | | |
| $BF_4$ | | | | | |
| B(1)—F(1) | 1.36 (1) | B(2)—F(5) | 1.39 (1) | | |
| B(1)—F(2) | 1.34 (1) | B(2)—F(6) | 1.39 (1) | | |
| B(1)—F(3) | 1.32 (1) | B(2)—F(7) | 1.36 (1) | | |
| B(1)—F(4) | 1.37 (1) | B(2)—F(8) | 1.33 (1) | | |
| F(1)—B(1)—F(2) | 111 (1) | F(5)—B(2)—F(6) | 106 (1) | | |
| F(1)—B(1)—F(3) | 113.6 (9) | F(5)—B(2)—F(7) | 110 (1) | | |
| F(1)—B(1)—F(4) | 106.3 (9) | F(5)—B(2)—F(8) | 114 (1) | | |
| F(2)—B(1)—F(3) | 110 (1) | F(6)—B(2)—F(7) | 109 (1) | | |
| F(2)—B(1)—F(4) | 106.7 (9) | F(6)—B(2)—F(8) | 110 (1) | | |
| F(3)—B(1)—F(4) | 109 (1) | F(7)—B(2)—F(8) | 109 (1) | | |

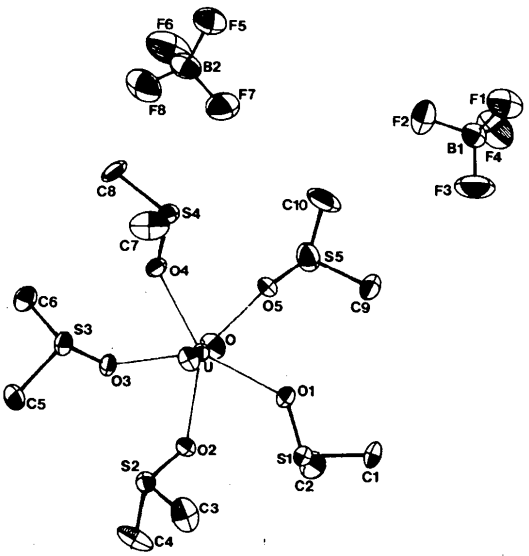

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the discrete ions.

contributions were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 4F_o/\sigma^2(I)$ with $\sigma^2(I) = [\sigma^2(I_c) + (pF_o)^2]$; final $R = 0.035$, $wR = 0.045$ ($w = 1$), $S = 3.0$, $(\Delta/\sigma)_{\max} = 0.10$; highest peak in the final difference Fourier map = 0.70 e Å$^{-3}$.

Final positional parameters are given in Table 1,* with selected bond distances and angles in Table 2. The atomic labelling scheme and molecular structure are shown in Fig. 1.

**Related literature.** This work is part of structural studies of uranyl with poorly coordinating anions in various solvents (Alcock & Esperas, 1977).

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* Lists of structure factors, anisotropic thermal parameters, and bond angles and distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55326 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0328]

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# Structure of the Dibenzyl Sulfoxide Adduct of *N*-Triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-Dioxide

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**Abstract.** [1,2-Benzisothiazol-3(2*H*)-one 1,1-dioxide-*N*](dibenzyl sulfoxide-*O*)triphenyltin(IV), $[\mathrm{Sn(C_{14}H_{14}OS)(C_7H_4NO_3S)(C_6H_5)_3}]$, $[\mathrm{(C_6H_5)_3SnNC(O)C_6H_4SO_2.OS(CH_2C_6H_5)_2}]$, $M_r = 762.50$, monoclinic, $P2_1$, $a = 10.9026$ (7) $b = 19.614$ (1), $c = 16.8211$ (9) Å, $\beta = 94.512$ (5)°, $V = 3585.8$ (8) Å$^3$, $Z = 4$, $D_x = 1.412$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71073 Å, $\mu = 8.64$ cm$^{-1}$, $F(000) = 1552$, $T = 298$ K, $R = 0.038$ for 4729 reflections [$I \geq 3\sigma(I)$]. The Sn atoms in the two independent molecules exhibit a *trans*-C$_3$SnNO trigonal bipyramidal geometry. In molecule *A*, the Sn—N bond [2.237 (8) Å] is short and the Sn—O bond [2.413 (7) Å] long; in molecule *B*, the Sn—N bond [2.253 (9) Å] is longer whereas the Sn—O bond [2.376 (7) Å] is shorter.

**Experimental.** The adduct was prepared by reacting triphenyltin hydroxide, saccharin and dibenzyl sulfoxide in 1/1/1 stoichiometric ratio in ethanol (Ng, Kuthubutheen, Zainudin, Chen, Kumar Das, Schulze, Molloy, Yip & Mak, 1991). A crystal of triphenyltin saccharin–dibenzyl sulfoxide measuring approximately 0.14 × 0.22 × 0.22 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. Unit-cell parameters were fixed from 25 strong reflections in the $16 \leq \theta \leq 18°$ thin shell. Intensity data were collected to $2\theta_{\max} = 50°$ (collection range: $h$ 0–12, $k$ 0–21, $l$ −19–19). 6251 reflections were collected, of which 4729 obeyed the $I \geq 3\sigma(I)$ criterion. Three reflections (0,16,0, 5,3,$\overline{11}$ and 90$\bar{5}$) were used to monitor the intensity. Their intensities decreased by 5.7% over the 75 h of data collection, and a correction was applied to the data (average correction factor 1.02962). The two Sn atoms were obtained by direct methods, and the $y$ coordinate of Sn1 was fixed at 0.25. The non-H atoms were derived from successive difference Fourier syntheses, and all the non-H atoms were refined anisotropically. H atoms were generated geometrically (C—H = 0.95 Å, $B$ = 5 Å$^2$) and were included in the structure-factor calculations. Refinement based on $F$ converged at $R = 0.038$ ($R_2 = 0.043$ for all reflections); 846 variables were refined, and unit weights were used; $\Delta/\sigma = 0.04$; $S = 2.353$; $(\Delta\rho)_{\max} = 0.41$ (6) e Å$^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B

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