& Lorberth, 1980)]. They are significantly longer than those in trialkyllead compounds R_3PbX (X = carboxylate) [$Me_3PbOOCC_6H_4(2\text{-}NH_2)$ 2.18 (2) Å (Fehlberg-Sternemann, 1992); $Me_3PbOOC(2\text{-}C_5H_3O)$ 2.17 (2) Å (Preut, Röhm & Huber, 1986)] and dialkyllead compounds R_2PbX_2 [dimethyllead bis(4-pyrimidinecarboxylate) trihydrate 2.142 (6) Å (Hoffmann, 1988)]. No other data on Pb—C bond distances in solid alkyllead compounds are available. A Pb—C bond distance in $Pb(CH_3)_4$ of 2.238 (9) Å was determined by gas-phase electron diffraction (Oyamada, Iijima & Kimura, 1971).

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Structure of Tetrabutylammonium Bis[benzene-1,2-dithiolato(2−)]oxorhenate(V)

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Abstract. $[C_{16}H_{36}N][Re(C_6H_4S_2)_2O]$, $M_r = 725.19$, orthorhombic, $Pna2_1$, $a = 37.912$ (3), $b = 9.286$ (1), $c = 17.482$ (3) Å, $V = 6154.6$ Å^3, $Z = 8$, $D_x = 1.565$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.709300 Å, $\mu = 4.284$ mm^{-1}, $F(000) = 2928$, $T = 215$ K. The final R value is 0.046 for 4236 significant [$I > 3\sigma(I)$] reflections. The structure consists of discrete $[ReO(bdt)_2]^{2-}$ anions, where bdt^{2-} is the benzene-1,2-dithiolato ligand, and $(Bu_4N)^+$ cations. The Re atom is pentacoordinate with the four S atoms in the basal positions and the oxo ligand at the apex of a square pyramid.

Introduction. In recent years, radioactive rhenium compounds (^{186}Re and ^{188}Re: β^--emitter with β^- energies between 0.17 and 1.12 MeV and half lives of 90 and 17 h, respectively) have been considered as potential radiopharmaceuticals in therapeutic nuclear medicine (Vanderheyden, Heeg & Deutsch, 1985). Therefore, there is considerable interest in detailed structural information on rhenium complexes, especially in comparison with technetium compounds, the biological distribution pattern of which is well known.

Experimental. The title compound was formed as a by-product (about 20%) during the reaction of $[Bu_4N][ReOBr_4]$ with 2,2′-(thioethylenethio)dibenzenethiol. Benzene-1,2-dithiol was formed by partial decomposition of the potentially terdentate organic ligand. An orange–brown single crystal of approximate dimensions 0.1 × 0.15 × 0.5 mm was mounted on a glass fiber. The complex crystallizes in the space group $Pna2_1$ with two independent formula units per unit cell. In the final full-matrix least-squares refinement, all non-H atoms were assigned anisotropic atomic displacement parameters. H atoms were included in calculated positions. 2000 Friedel pairs have been measured to confirm the non-centrosymmetric space group. The absolute structure has not been determined. A summary of data collection and structure refinement parameters is given in Table 1.† Final atomic coordinates are listed in Table 2. Table 3 contains selected bond

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† Full lists of structure factors, H-atom positions, anisotropic atomic displacement parameters, bond angles and distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55953 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1033]

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Table 1. *Data collection and structure refinement parameters*

Crystal shape	Column
Diffractometer used	CAD-4, Enraf–Nonius
Method of intensity measurement	ω scan
No.; θ range (°) of reflections for lattice parameters	25; 8–14
Method used for absorption correction	*DIFABS* (Walker & Stuart, 1983)
Min. absorption correction	0.713
Max. absorption correction	1.109
Average absorption correction	0.987
Range of h, k and l	$-10\rightarrow0$, $-20\rightarrow0$, $-43\rightarrow0$
Standard reflections	0,2,22, $\bar{5}$12, $\bar{4}0\bar{1}$
Interval, standard reflections measured (h)	1, no intensity variation
Total No. of reflections measured; θ range (°)	5411; $3\rightarrow24$
No. of observed reflections	4236 with $I > 3\sigma(I)$
Method used to solve structure	Patterson
Use of F or F^2 in LS refinement	F
Method of locating H atoms	Calculated in idealized positions, included in structure-factor calculation
Weighting scheme	$1/\sigma_F^2$
Parameters refined	626
R	0.046
wR	0.055
Ratio of max. LS shift to e.s.d. (Δ/σ)	0.02
Max. height in final ΔF map (e Å^{-3})	1.56
Secondary-extinction coefficient	4.85 (1) × 10^{-9} (Zachariasen, 1963)
Source of atomic scattering factors	*International Tables for X-ray Crystallography* (1974, Vol. IV)
Computer used	DECVAX station 3100
Programs used	*VAXSDP* (B. A. Frenz & Associates, Inc., 1986)

lengths and angles of the complex anions. The tetrabutylammonium cations show no unusual structural features. A plot (Keller, 1988) of the $[ReO(bdt)_2]^{2-}$ anion is shown in Fig. 1, with the unit-cell packing in Fig. 2.

Discussion. The metal in the title complex is pentacoordinate. This is due to the strong *trans* effect of the oxo ligand which labilizes the potential coordination position *trans* to O^{2-}. The coordination geometry about Re is distorted square pyramidal with the S atoms occupying the positions in the basal plane, and the O atom at the apex. The Re atom is situated 0.72 Å above the plane of the four S atoms [which are coplanar within 0.032 (5) Å as is generally observed for complexes with MOS_4 and MNS_4 cores (Melnik & van Lier, 1987; Colmanet & Mackay, 1987)]. The atoms in each benzene-1,2-dithiolato ligand are almost coplanar and the dihedral angle between the normal to the two planes is 166.5 (9)°. The Re=O bond length of 1.71 (3) Å lies within the expected range for Re=O double bonds (1.66–1.76 Å) and the mean C—S bond length of 1.775 (6) Å indicates a C—S single bond, in good agreement with a mainly dithiolato conformation of the organic ligand. Interligand S···S distances of 3.052 (8) and 3.124 (8) Å rule out significant interactions between the equatorial ligands.

Table 2. *Atomic positional and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	B_{eq}
Molecule (1)				
Re1	0.23047 (2)	0.16477 (9)	0.000	3.25 (1)
S1	0.2914 (1)	0.1493 (6)	0.0042 (4)	4.0 (1)
S2	0.2400 (2)	0.3952 (6)	−0.0454 (3)	3.4 (1)
S3	0.1838 (1)	0.1686 (6)	−0.0844 (3)	3.2 (1)
S4	0.2337 (2)	−0.0775 (6)	−0.0281 (3)	3.6 (1)
O1	0.2116 (5)	0.182 (2)	0.0877 (8)	6.5 (4)
C11	0.3062 (5)	0.329 (3)	−0.002 (1)	4.7 (4)
C12	0.2829 (5)	0.436 (2)	−0.022 (1)	3.7 (4)
C13	0.2948 (6)	0.581 (3)	−0.028 (1)	4.2 (5)
C14	0.3298 (6)	0.612 (3)	−0.011 (1)	5.3 (5)
C15	0.3536 (6)	0.504 (3)	0.010 (2)	6.0 (6)
C16	0.3418 (6)	0.362 (3)	0.015 (1)	5.2 (6)
C21	0.1710 (5)	−0.014 (2)	−0.096 (1)	3.7 (4)
C22	0.1921 (6)	−0.121 (2)	−0.067 (1)	3.5 (4)
C23	0.1818 (7)	−0.266 (2)	−0.075 (1)	4.8 (5)
C24	0.1492 (6)	−0.300 (3)	−0.109 (1)	5.1 (6)
C25	0.1281 (6)	−0.190 (3)	−0.139 (1)	5.2 (6)
C26	0.1389 (6)	−0.047 (3)	−0.132 (1)	4.6 (5)
N1	0.0128 (4)	0.738 (2)	−0.0048 (9)	3.5 (3)
C51	−0.0132 (6)	0.641 (3)	0.029 (1)	5.0 (6)
C52	−0.0390 (7)	0.575 (3)	−0.026 (1)	6.1 (6)
C53	−0.0679 (7)	0.487 (3)	0.012 (2)	7.9 (7)
C54	−0.0956 (9)	0.570 (5)	0.048 (3)	13 (1)
C61	−0.0038 (5)	0.864 (2)	−0.039 (1)	3.6 (4)
C62	−0.0254 (6)	0.964 (2)	0.010 (2)	5.5 (5)
C63	−0.0396 (7)	1.087 (3)	−0.032 (1)	5.7 (6)
C64	−0.0128 (8)	1.192 (4)	−0.060 (2)	7.5 (8)
C71	0.0382 (6)	0.780 (3)	0.057 (1)	4.3 (5)
C72	0.0671 (7)	0.880 (3)	0.036 (1)	5.1 (6)
C73	0.0935 (6)	0.905 (4)	0.101 (1)	6.1 (7)
C74	0.1227 (7)	1.004 (4)	0.080 (1)	7.3 (8)
C81	0.0332 (6)	0.660 (2)	−0.066 (1)	3.9 (4)
C82	0.0535 (6)	0.533 (2)	−0.043 (1)	4.1 (5)
C83	0.0647 (7)	0.440 (3)	−0.110 (1)	5.0 (5)
C84	0.0860 (8)	0.304 (3)	−0.094 (2)	6.6 (7)
Molecule (2)				
Re2	0.52487 (2)	0.22270 (9)	0.19967 (6)	3.42 (1)
S5	0.5151 (2)	0.4554 (6)	0.2431 (3)	3.9 (1)
S6	0.4642 (1)	0.2054 (7)	0.2017 (5)	4.7 (1)
S7	0.5227 (2)	−0.0164 (6)	0.2365 (3)	4.0 (1)
S8	0.5724 (1)	0.2367 (6)	0.2819 (3)	3.6 (1)
O2	0.5401 (4)	0.232 (2)	0.107 (1)	5.7 (4)
C31	0.4717 (6)	0.489 (3)	0.223 (1)	4.7 (5)
C32	0.4489 (5)	0.384 (2)	0.204 (1)	4.1 (4)
C33	0.4137 (7)	0.413 (3)	0.184 (1)	6.3 (7)
C34	0.4004 (7)	0.551 (3)	0.186 (1)	7.2 (7)
C35	0.4256 (8)	0.670 (3)	0.210 (2)	10.2 (7)
C36	0.4589 (7)	0.635 (3)	0.228 (1)	6.1 (6)
C41	0.5644 (6)	−0.055 (3)	0.279 (1)	4.4 (5)
C42	0.5858 (6)	0.056 (2)	0.298 (1)	4.1 (5)
C43	0.6182 (6)	0.031 (3)	0.335 (1)	4.7 (5)
C44	0.6288 (6)	−0.116 (3)	0.348 (1)	5.5 (6)
C45	0.6067 (7)	−0.224 (3)	0.326 (2)	6.2 (7)
C46	0.5744 (7)	−0.199 (2)	0.291 (1)	5.3 (6)
N2	0.2381 (4)	0.662 (1)	0.1973 (8)	2.8 (3)
C91	0.2539 (6)	0.530 (2)	0.234 (1)	3.7 (4)
C92	0.2747 (5)	0.433 (2)	0.183 (1)	3.6 (4)
C93	0.2910 (5)	0.306 (2)	0.228 (1)	4.0 (5)
C94	0.2647 (6)	0.201 (3)	0.262 (1)	5.0 (5)
C101	0.2671 (6)	0.756 (3)	0.162 (1)	4.2 (5)
C102	0.2956 (5)	0.803 (2)	0.213 (1)	4.0 (4)
C103	0.3217 (7)	0.890 (3)	0.172 (1)	5.3 (6)
C104	0.3492 (7)	0.952 (3)	0.226 (2)	7.1 (8)
C111	0.2196 (5)	0.749 (2)	0.256 (1)	3.8 (4)
C112	0.1978 (6)	0.871 (2)	0.227 (1)	3.9 (5)
C113	0.1879 (6)	0.970 (2)	0.293 (1)	4.5 (5)
C114	0.1649 (7)	1.095 (3)	0.274 (1)	6.1 (6)
C121	0.2130 (6)	0.621 (2)	0.134 (1)	3.9 (5)
C122	0.1821 (6)	0.527 (2)	0.155 (1)	3.8 (5)
C123	0.1567 (6)	0.498 (2)	0.089 (1)	4.3 (5)
C124	0.1251 (7)	0.418 (3)	0.113 (1)	5.7 (6)

The structure of the complex anion is very similar to that of the analogous technetium complex $[TcO(bdt)_2]^-$ in its tetraphenylarsonium salt form, the structure of which has been reported recently (Colmanet & Mackay, 1987). In both compounds, the ligand bite angles are about 2° larger than the interligand S—*M*—S angles of adjacent S atoms. The O—*M*—S angles in the title complex, however, vary between 104.6 (6) and 113.5 (6)°, whereas for the technetium compound values have been found

Table 3. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s of the two independent complex anions in* $[Bu_4N][ReO(bdt)_2]$

Re1—O1	1.70 (2)	Re1—S1	2.316 (5)
Re1—S2	2.311 (6)	Re1—S3	2.304 (5)
Re1—S4	2.307 (6)	S1—C11	1.77 (2)
S2—C12	1.72 (2)	S3—C21	1.77 (2)
S4—C22	1.77 (2)		
O1—Re1—S1	113.5 (6)	O1—Re1—S2	106.7 (6)
O1—Re1—S3	104.6 (6)	O1—Re1—S4	107.9 (6)
S1—Re1—S2	84.9 (3)	S3—Re1—S4	85.4 (2)
S1—Re1—S3	142.0 (2)	S2—Re1—S4	145.3 (2)
S1—Re1—S4	83.9 (3)	S2—Re1—S3	83.5 (2)
Re1—S1—C11	104.8 (7)	Re1—S2—C12	105.6 (8)
Re1—S3—C21	105.4 (7)	Re1—S4—C22	105.1 (7)
Re2—O2	1.72 (2)	Re2—S5	2.321 (6)
Re2—S6	2.306 (5)	Re2—S7	2.314 (6)
Re2—S8	2.309 (6)	S5—C31	1.71 (2)
S6—C32	1.76 (2)	S7—C41	1.78 (2)
S8—C42	1.77 (2)		
O2—Re2—S5	108.3 (7)	O2—Re2—S6	110.7 (6)
O2—Re2—S7	108.9 (7)	O2—Re2—S8	108.6 (6)
S5—Re2—S6	84.3 (2)	S7—Re2—S8	84.7 (2)
S5—Re2—S7	142.9 (3)	S6—Re2—S8	140.7 (3)
S5—Re2—S8	82.5 (3)	S6—Re2—S7	83.9 (2)
Re2—S5—C31	104.8 (8)	Re2—S6—C32	105.4 (7)
Re2—S7—C41	106.2 (9)	Re2—S8—C42	105.7 (7)

only between 108.2 (2) and 108.7 (2)°. The benzene-1,2-dithiolato ligands exhibit an almost ideal planarity in the latter complex.

The structure of the tetrabutylammonium cations corresponds fully to the bonding feature which is observed in other structures. The geometry about the N atom is only slightly distorted from the regular tetrahedral [a mean value of 109.5 (25)° was observed for the C—N—C angles]. The mean C—N bond length is 1.49 (4) Å.

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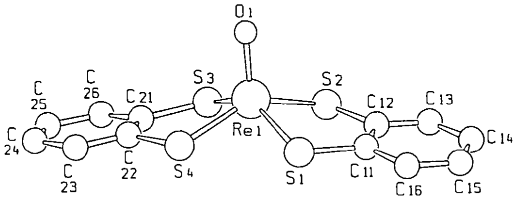

Fig. 1. Plot of the complex anion along with the atomic numbering scheme.

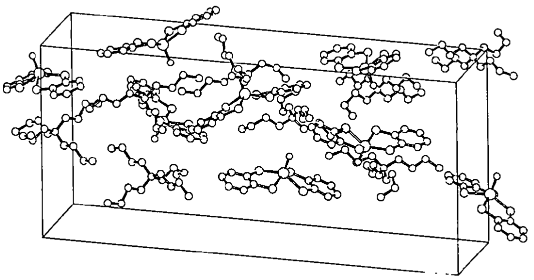

Fig. 2. Unit-cell packing; projection parallel to **a**.

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Structure of *N*-(2-Aminophenyl)-*N*′-methylurea

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Abstract. $C_8H_{11}N_3O$, $M_r = 165.19$, monoclinic, $P2_1/n$, $a = 10.3997$ (14), $b = 4.6395$ (12), $c = 18.2211$ (11) Å, $\beta = 100.994$ (7)°, $V = 868.0$ (2) Å^3, $Z = 4$, $D_x = 1.2271$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å (graphite monochromator), $\mu = 0.682$ mm^{-1}, $F(000) = 352$, $T = 293$ K, 1471 unique reflections, $R = 0.054$, $wR = 0.045$ for 887 reflections with $I > 2\sigma(I)$. The ureido moiety is approximately planar and makes a dihedral angle of 58.92 (16)° with the least-squares plane of the aromatic ring. The carbonyl

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