Fig. 1. Constituent molecules and atomic numbering. Numbers in parentheses correspond to W and Se atoms. The intermolecular contacts (Å) are given: d^1(Se1···Se3^i) = 3.633 (6), d^2(Se2···Se4^i) = 3.613 (5), d^3(Se3···Se5^{ii}) = 3.909 (5), d^4(Se2···Se5^{ii}) = 3.898 (5), d^5(Se6···O17^{iii}) = 3.27 (2), d^6(O18···O18^{iv}) = 3.06 (3). Symmetry code: (i) $-x$, $-y$, $1-z$; (ii) $1-x$, $-y$, $1-z$; (iii) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$; (iv) $1-x$, $-y$, $-z$.

over-double-bond fashion to form dimers. The latter are separated along the [100] direction by molecule A. The overlaps between molecules A and B are of the criss-cross type (Fig. 2). This arrangement results in short Se···Se (3.60 Å) contacts between molecules of the B type. The contacts between mol-
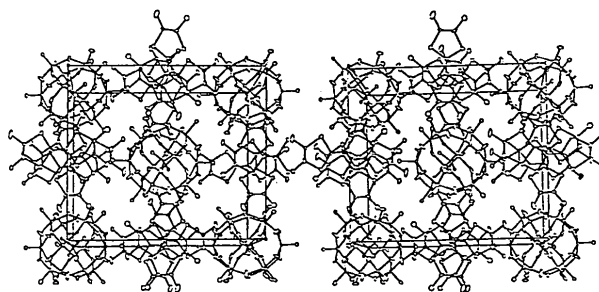

Fig. 2. Stereoscopic view of the crystal structure.

ecules A and B are equal to 3.90 Å (see Fig. 1). Additionally, strong organic–inorganic interactions are observed: Se1···O3 = 3.29 (3), Se2···O9($-x$, $-y$, $1-z$) = 3.00 (2), Se3···O12 ($-x$, $-y$, $1-z$) = 3.06 (2), Se4···O15 = 3.31 (3), Se5···O17 ($\frac{1}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$) = 3.25 (2), Se6···O17 ($\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$) = 3.27 (2) Å.

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Structure of Tetraethylammonium Permanganate

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Abstract. $(C_2H_5)_4N^+.MnO_4^-$, $M_r = 249.19$, monoclinic, $P2_1/c$, $a = 7.512$ (1), $b = 11.103$ (1), $c = 14.764$ (2) Å, $\beta = 91.44$ (1)°, $V = 1230.9$ (3) Å^3, $Z = 4$, $D_x = 1.34$ g cm^{-3}, λ(Mo $K\alpha_1$) = 0.7093 Å, $\mu = 10.2$ cm^{-1}, $F(000) = 528$, $T = 295$ K, final $R = 0.047$ for 983 reflections $[F_o > 3\sigma(F_o)]$. The average Mn—O bond length in the title permanganate is 1.610 (6) Å

which is similar to the values reported for the potassium and caesium salts.

Experimental. The crystals of the title compound were obtained accidentally from the reaction mixture of $MnCl_2$ and $KMnO_4$ in aqueous solution containing tetraethylammonium bromide. X-ray data for a black oblique crystal ($0.2 \times 0.2 \times 0.07$ mm) mounted on the top of a glass fiber were collected on an

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Table 1. *Fractional coordinates and equivalent iso-
tropic thermal parameters* ($\AA^2$) *for non-H atoms with
e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
Mn	-0.1870 (2)	0.2498 (1)	-0.13410 (7)	3.59 (2)
O1	-0.2399 (7)	0.3320 (5)	-0.0493 (4)	4.7 (1)
O2	-0.2340 (8)	0.3199 (6)	-0.2267 (4)	5.4 (1)
O3	-0.2976 (9)	0.1256 (5)	-0.1323 (4)	5.7 (2)
O4	0.0237 (7)	0.2205 (6)	-0.1283 (4)	5.6 (2)
N	0.2605 (7)	0.2223 (5)	0.6211 (4)	3.1 (1)
C1	0.158 (1)	0.3281 (7)	0.6635 (5)	3.5 (2)
C2	0.033 (1)	0.3928 (8)	0.5995 (6)	4.7 (2)
C3	0.3773 (9)	0.2679 (8)	0.5454 (5)	4.1 (2)
C4	0.514 (1)	0.3607 (9)	0.5736 (6)	5.4 (2)
C5	0.375 (1)	0.1670 (8)	0.6978 (5)	4.2 (2)
C6	0.490 (1)	0.0626 (8)	0.6680 (7)	5.6 (2)
C7	0.132 (1)	0.1296 (7)	0.5805 (5)	3.8 (2)
C8	-0.000 (1)	0.0777 (8)	0.6442 (6)	5.1 (2)

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

Mn—O1	1.608 (6)	N—C5	1.534 (9)
Mn—O2	1.605 (6)	N—C7	1.52 (1)
Mn—O3	1.611 (6)	C1—C2	1.50 (1)
Mn—O4	1.616 (6)	C3—C4	1.51 (1)
N—C1	1.546 (9)	C5—C6	1.52 (1)
N—C3	1.524 (9)	C7—C8	1.50 (1)

O1—Mn—O2	109.6 (3)	C1—N—C7	111.0 (5)
O1—Mn—O3	109.6 (3)	C3—N—C5	110.6 (5)
O1—Mn—O4	109.5 (3)	C3—N—C7	107.9 (5)
O2—Mn—O3	109.0 (3)	C5—N—C7	111.0 (6)
O2—Mn—O4	109.7 (3)	N—C1—C2	114.7 (6)
O3—Mn—O4	109.4 (3)	N—C3—C4	115.2 (6)
C1—N—C3	110.2 (6)	N—C5—C6	113.8 (6)
C1—N—C5	106.3 (5)	N—C7—C8	115.8 (6)

Enraf–Nonius CAD-4 diffractometer using mono-
chromated Mo $K\alpha$ radiation. Lattice parameters and
their e.s.d.'s were derived from the scattering angles
of 25 reflections ($10 < \theta < 14°$). The space group was
determined from observed systematic absences.
Intensity data were collected using an $\omega/2\theta$ scan to a
maximum $\sin\theta/\lambda$ of 0.5958 $\AA^{-1}$, $-8 \le h \le 8$, $0 \le k$
≤ 13, $0 \le l \le 17$, with ω-scan width of (0.70 +
$0.34\tan\theta$)°. A total of 2364 reflections were collected;
symmetry-equivalent reflections were averaged
(agreement: 2.5% on I, 2.1% on F) leaving 2277
unique reflections of which 983 were considered
observed at the $3\sigma(I)$ level. The intensities of four
standard reflections (032, $\bar{2}20$, 104, $\bar{1}12$), recorded
every 3 h of X-ray exposure, showed no systematic
changes. All the calculations were carried out with
the Enraf–Nonius (1982) *Structure Determination
Package*. The data were corrected for crystal decay
(5.8% over 48 h of data collection), Lorentz-
polarization and absorption effects. The empirical
absorption correction (relative transmission coeffi-
cients 0.889–0.998) was based on azimuthal (ψ) scans
of five reflections. The structure was solved by a
combination of Patterson and difference Fourier

methods. The structure model was refined by full-
matrix least squares (on F). The function minimized
was $\Sigma w(|F_o| - |F_c|)^2$ where the weights were defined
as $w = 4F_o^2/\sigma^2(I)$ with $\sigma(I) = [\sigma(I)^2 + (pF_o^2)^2]^{1/2}$ ($p =$
0.04). All the non-H atoms were refined anisotrop-
ically. The H atoms were positioned according to
idealized geometry (C—H = 0.95 $\AA$) and given iso-
tropic B values of 1.2 times the B value of the
attached atom. $R = 0.047$, $wR = 0.052$, $S = 0.42$ (217
parameters refined). In the final difference map the
highest and lowest peaks were 0.29 and
-0.29 e $\AA^{-3}$, respectively. Final $(\Delta/\sigma)_{max} = 0.28$.
The atomic scattering factors and anomalous-
dispersion corrections were taken from *International
Tables for X-ray Crystallography* (1974, Vol. IV) for
the non-H atoms and from Stewart, Davidson &
Simpson (1965) for H atoms. Table 1 gives atomic
coordinates and equivalent isotropic thermal
parametres of the non-H atoms and Table 2 bond
distances and angles.* Fig. 1 shows an *ORTEP*II
(Johnson, 1976) drawing of the title compound with
adopted numbering.

Related literature. The structures of methyltriphenyl-
phosphonium permanganate and *n*-heptyltriphenyl-
phosphonium permanganate have been reported
(Karaman, Barton, Robertson & Lee, 1984). The
average Mn—O bond distances in their structures
were substantially short [1.47 (2)–1.54 (1) $\AA$], presu-
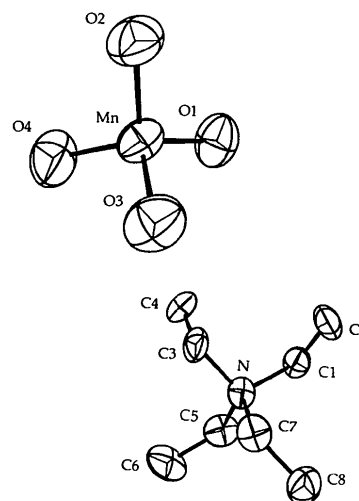mably because of large thermal motion. The average

Fig. 1. *ORTEP*II diagram of tetraethylammonium permanganate
with atom labelling (50% probability themal ellipsoids). H
atoms are omitted for clarity.

Mn—O bond length in the present structure is 1.610 (6) Å which is closer to the reported values for the potassium (Palenik, 1967) and caesium (Prout & Nassimbeni, 1966) salts [1.607 (5) (without libration correction) and 1.63 Å, respectively].

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Structure of 3-Methyl-3,4-dihydro-1*H*-2,3-benzothiazine 2,2-Dioxide

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Abstract. $C_9H_{11}NO_2S$, $M_r = 197.26$, triclinic, $P\bar{1}$, $a = 9.355$ (4), $b = 10.001$ (3), $c = 10.787$ (5) Å, $\alpha = 86.93$ (3), $\beta = 88.88$ (3), $\gamma = 69.56$ (3)°, $V = 944$ (1) Å^3, $Z = 4$, $D_x = 1.387$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.294$ mm^{-1}, $F(000) = 416$, $T = 293$ K, $R = 0.045$ for 2057 observed reflections. There are two crystallographically independent molecules per asymmetric unit and the heterocyclic ring in both takes a twisted half-chair conformation. The S atoms are tetrahedrally coordinated with a dihedral angle between the C—S—N and O—S—O planes of 89.51 (14)°. The methyl groups are almost axial with torsional angles C(1*A,B*)—S(2*A,B*)—N(3*A,B*)—C(11*A,B*) of 70.73 (31) and 74.52 (35)°, respectively.

Experimental. The synthesis of the title compound has been reported previously (Orazi, Corral & Bravo, 1986). Crystals obtained by slow cooling (330–300 K) of a solution in ethanol. The crystal used for

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data collection had dimensions $0.1 \times 0.3 \times 0.5$ mm. Intensities measured with a Nonius CAD-4 diffractometer; ω–2θ scanning mode, with scan width $(0.80 + 0.35\tan\theta)°$ and scan speed of 2.50–6.67° min^{-1}; graphite-monochromated Mo $K\alpha$ radiation. Unit-cell parameters determined from least squares on setting angles of 24 reflections with $8.5 < 2\theta < 19°$; range of hkl: $-1 < h < 11$, $-10 < k < 11$, $-12 < l < 12$, $\theta_{max} = 25°$. One standard reflection ($\bar{1}07$) varied $\pm 2.1\%$ of mean intensity over data collection; 3965 reflections measured, 2945 unique, $R_{int} = 0.018$, 2057 considered observed on the criterion $I > 3\sigma(I)$; Lorentz–polarization and geometrical absorption corrections were applied (average transmission factor 0.95, with maximum 0.97 and minimum 0.92 for 120 and $\bar{1},0,12$ reflections, respectively). Structure solved by centrosymmetric direct methods and refined by least squares. After a full-matrix least-squares refinement, with all non-H atoms anisotropic, the H atoms in the heterocyclic ring and the benzene ring were included at positions based on those found in a difference synthesis; refined in the final run with two common isotropic thermal parameters for CH$_2$ and fused-benzene H atoms. The methyl-group H atoms in both molecules were geometrically placed (C—H 1.08 Å); the methyl group was treated as a rigid group with a common isotropic factor for its H atoms. Function minimized $\sum[w(|F_o| - |F_c|)^2]$ with