La cohésion entre les cycles est assurée par les cations Na^+. Si on se limite à une sphère de coordination de rayon 3 Å, les quatre atomes $2 \times Na(1)$ et $2 \times Na(2)$ possèdent des voisinages octaédriques. Les atomes $Na(1)$ s'entourent de six oxygènes provenant du groupement anionique et les atomes $Na(2)$ de cinq oxygènes appartenant au cycle P_3O_9 et d'une molécule d'eau. Les distances Na—O (Tableau 2) sont comprises:

—entre 2,35 et 2,59 Å dans les polyèdres $Na(1)O_6$;

—entre 2,37 et 2,58 Å dans les polyèdres $Na(2)O_6$.

Ces polyèdres s'enchaînent par des arêtes communes:

—O($E11$)–O($E32$) pour les polyèdres $Na(1)O_6$, $Na(2)O_6$;

—O($E11$)–O($E22$) pour les polyèdres $Na'(1)O_6$, $Na(2)O_6$;

—O($E'11$)–O($E'22$) pour les polyèdres $Na(1)O_6$, $Na'(2)O_6$;

—O($E'32$)–O($E'22$) pour les polyèdres $Na'(1)O_6$, $Na'(2)O_6$.

L'octaèdre de coordination du nickel est constitué uniquement des atomes d'oxygènes des molécules d'eau. Cette propriété est peu commune dans les phosphates hydratés (Jouini, Dabbabi & Durif, 1985; Seethanen et al., 1978). Les distances Ni–O($W2$) (cf. Tableau 2) sont comprises entre 2,035 et 2,080 Å. Les polyèdres $Na(2)O_6$, en plus des arêtes communes avec les polyèdres $Na(1)O_6$, possèdent un sommet commun O($W1$) avec les octaèdres de coordination des atomes de nickel $[Ni(H_2O)_6]^{2+}$. L'ensemble ainsi formé par tous les polyèdres de coordination des cations associés constitue un enchaînement bidimensionnel qui se développe selon la direction 'moyenne' $a + c$. Le schéma de cet enchaînement est représenté sur la Fig. 2.

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Structure of Potassium Hydrogenselenate

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Abstract. $KHSeO_4$, $M_r = 183.07$, orthorhombic, *Pbca*, $a = 8.690$ (9), $b = 10.053$ (9), $c = 19.47$ (2) Å, $V = 1701$ Å^3, $Z = 16$, $D_m = 2.84$, $D_x = 2.86$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 10.31$ mm^{-1}, $F(000) = 1376$, $T = 295$ (1) K, final $R = 0.054$ for 2163 non-zero reflexions. The crystal is isomorphous with $KHSO_4$ and displays a similar hydrogen-bonding scheme. However, the two crystallographically different hydrogen bonds are shorter than in the case of $KHSO_4$. The O···O distances utilized in hydrogen bonding are 2.51 (1) and 2.57 (1) Å for polymer and dimer, respectively.

Introduction. The structure of potassium hydrogenselenate is similar to that of potassium hydrogensulfate (Loopstra & MacGillavry, 1958; Madar, 1960; Cruickshank, 1964; Cotton, Frenz & Hunter, 1975; Payan & Haser, 1976). They belong to the same space-group type and contain simultaneously cyclic dimers and infinite polymeric chains of hydrogen-bonded HSO_4^- and $HSeO_4^-$ anions, respectively. However, IR spectra of $KHSO_4$ (Goypiron, de Villepin & Novak, 1980) and $KHSeO_4$ (Paetzold & Amoulong, 1962) are quite different in the region of hydrogen-bonding stretching vibrations. This suggests that the hydrogen bonds are not similar in these two crystals. Therefore we decided to determine an accurate crystal structure of $KHSeO_4$.

Experimental. Crystals of $KHSeO_4$ were grown from a saturated aqueous solution containing K^+ ions and selenic acid in a 1:1 stoichiometric ratio. The single crystals obtained look like truncated pyramids whose (113) and (001) faces are easily identified according to Groth's (1908) information concerning $KHSO_4$ crystals. The a and b axes are the bisectors of the (001) parallelogram perpendicular to the c axis.

D_m by flotation in $CHBr_3$, preliminary examination by rotation and Weissenberg photographs. A specimen $0.4 < 2R < 0.55$ mm was cut from a large crystal.

Table 1. *The final atom coordinates and equivalent isotropic thermal parameters of potassium hydrogenselenate*

$$B_{eq} = \tfrac{1}{3}(B_{11} + B_{22} + B_{33}).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Se(1)	0·25433 (11)	0·42428 (8)	0·00284 (5)	0·65
Se(2)	−0·01870 (9)	0·01642 (8)	0·20493 (5)	0·80
K(1)	0·3851 (3)	0·1760 (3)	0·1255 (2)	1·37
K(2)	−0·1247 (3)	0·3199 (2)	0·1189 (2)	1·14
O(11)	0·2963 (8)	0·5931 (6)	0·0004 (5)	1·54
O(12)	0·4176 (7)	0·3432 (6)	0·0067 (4)	1·34
O(13)	0·1667 (9)	0·3929 (8)	−0·0672 (4)	2·29
O(14)	0·1537 (9)	0·4023 (8)	0·0700 (4)	2·31
O(21)	0·0723 (8)	0·0030 (8)	0·2792 (4)	1·98
O(22)	−0·1715 (7)	0·1182 (6)	0·2229 (4)	1·15
O(23)	−0·0832 (9)	−0·1220 (8)	0·1778 (5)	2·49
O(24)	0·0785 (9)	0·0985 (8)	0·1497 (4)	1·97

Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflexions in range $15 < 2\theta < 26°$) and intensity measurements; 2θ–ω scan technique, 3661 reflexions measured below $\theta = 35°$ ($h\ 0{\to}13$, $k\ 0{\to}16$, $l\ 0{\to}31$), after each group of 50 reflexions two standards measured, variation 6%; most of calculations performed on a Nova 1200 computer with locally modified *XTL/XTLE* programs (Syntex, 1976); neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); real and imaginary components of anomalous dispersion included for all atoms; final coordinates of KHSO$_4$ (Cotton *et al.*, 1975) used as starting parameters of K, Se and O for the refinement. Refinement based on F (K, Se and O anisotropic) for 2163 reflexions with $I \geq 3\sigma(I)$, $w = 1/\sigma^2(F_o)$ gave $R = 0·054$ and $wR = 0·062$; maximum least-squares Δ/σ 0·01 (the 020 and 202 reflexions suffered from strong extinction and were omitted from the calculations). An absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) applied on isotropically refined data. Residual electron density in final difference Fourier map within −0·6 and 0·7 e Å^{-3}, but H atoms were not uniquely determined.

The final atomic parameters are given in Table 1.*

Discussion. The general features of the KHSeO$_4$ crystal structure are similar to those of the sulfate analogue (Payan & Haser, 1976). A listing of some interatomic distances and angles is given in Table 2. A projection of the crystal structure is shown in Fig. 1.

The asymmetric unit contains two crystallographically independent formula units. One forms a polymer and the other a dimer. The polymeric unit

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42616 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

containing Se(2) is hydrogen-bonded along an a-glide plane, the O(22)$\cdots$O(21)(x−0·5, y, 0·5−z) distance being 2·510 (9) Å. The two Se(1) tetrahedra are linked by a double hydrogen bridge on both sides of a symmetry centre, forming a dimer. The O(11)$\cdots$O(12)-(1−x, 1−y, −z) distance of the dimer hydrogen bond is 2·571 (9) Å. The plane of the dimer is almost parallel to the (001) crystallographic plane. The equation of the dimer plane is: $0·0387X + 0·0651Y + 0·9971Z − 0·4958 = 0$.

Table 2. *Principal interatomic distances* (Å) *and bond angles* (°)*for* KHSeO$_4$

Se(1)—O(11)	1·737 (6)	Se(2)—O(21)	1·654 (7)
Se(1)—O(12)	1·638 (6)	Se(2)—O(22)	1·712 (6)
Se(1)—O(13)	1·594 (8)	Se(2)—O(23)	1·591 (7)
Se(1)—O(14)	1·589 (8)	Se(2)—O(24)	1·598 (7)
O(11)$\cdots$O(12^i)	2·57 (1)	O(22)$\cdots$O(21^{ii})	2·51 (1)
O(11)—Se(1)—O(12)	107·8 (4)	O(21)—Se(2)—O(22)	103·9 (4)
O(11)—Se(1)—O(13)	105·7 (4)	O(21)—Se(2)—O(23)	112·8 (4)
O(11)—Se(1)—O(14)	105·9 (4)	O(21)—Se(2)—O(24)	112·3 (4)
O(12)—Se(1)—O(13)	110·8 (4)	O(22)—Se(2)—O(23)	108·5 (4)
O(12)—Se(1)—O(14)	111·7 (4)	O(22)—Se(2)—O(24)	103·8 (4)
O(13)—Se(1)—O(14)	114·5 (4)	O(23)—Se(2)—O(24)	114·5 (4)

Symmetry code: (i) $1−x$, $1−y$, $−z$; (ii) $x−0·5, y, 0·5−z$.

K(1)—O(11^i)	3·020 (8)	K(2)—O(11^v)	2·895 (8)
—O(12)	2·873 (7)	—O(12^{vi})	2·967 (7)
—O(13^{ii})	2·785 (8)	—O(13^v)	3·079 (8)
—O(14)	3·223 (8)	—O(13^{vi})	2·979 (8)
—O(14^i)	2·976 (8)	—O(14)	2·728 (8)
—O(21^{iii})	3·018 (8)	—O(21^{vii})	2·744 (7)
—O(22^{iii})	3·047 (7)	—O(22)	2·896 (7)
—O(23^{iv})	2·849 (8)	—O(23^{viii})	2·846 (8)
—O(24)	2·816 (8)	—O(24)	2·903 (8)

Symmetry code: (i) $0·5−x$, $−0·5+y$, z; (ii) $0·5+x$, $0·5−y$, $−z$; (iii) $0·5+x$, y, $0·5−z$; (iv) $0·5−x$, $0·5+y$, z; (v) $−x$, $1−y$, $−z$; (vi) $−0·5+x$, $0·5−y$, $−z$; (vii) $−x$, $0·5+y$, $0·5−z$; (viii) $−0·5−x$, $0·5+y$, z.
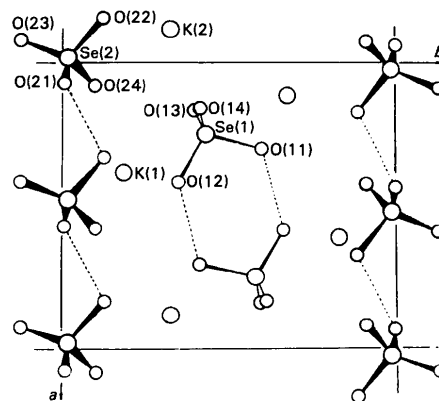
Fig. 1. The projection of the crystal structure of potassium hydrogenselenate on the ab plane.

Hence, the hydrogen bonds in the $KHSeO_4$ crystal are much stronger than in the $KHSO_4$ crystal, where the O···O distances are 2·619 (3) and 2·573 (3) Å for dimer and polymer, respectively (Payan & Haser, 1976).

Although the positions of the H atoms were not found in our work, taking into account the Se—O distances one may suppose that the H is closer to the O(11) atom [the Se—O(11) length is 1·737 (6) Å] in the dimer hydrogen bond and closer to the O(22) atom [the Se—O(22) distance is 1·712 (6) Å] in the polymer-chain hydrogen bond. The other O atoms involved in the hydrogen bonding (acceptors) make shorter bonds with the Se atoms of lengths 1·638 (6) and 1·654 (7) Å for the dimer and chain, respectively. The lengths of the remaining two Se—O bonds are very similar in both SeO_4 groups (Table 2). The Se—O distances obtained correspond to the distances observed in $RbHSeO_4$ (Waśkowska, Olejnik, Łukaszewicz & Głowiak, 1978; Waśkowska, Olejnik, Łukaszewicz & Czapla, 1980; Roziere, Brach, & Jones, 1983) and NH_4HSeO_4 (Kruglik, Misjul & Aleksandrov, 1980; Aleksandrov, Kruglik, Misjul & Simonov, 1980).

The environments of both K^+ cations are similar to those observed in the sulfate analogue (Loopstra & MacGillavry, 1958). Each K^+ cation is surrounded by nine O atoms and only the K(1)—O(14) distance is significantly longer than 3 Å (see Table 2).

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Structure of a Homoleptic Bis(diphenylphosphino)methanide Complex, [Pt{(C₆H₅)₂PCHP(C₆H₅)₂}₂].C₆H₆

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Abstract. Bis[bis(diphenylphosphino)methanido]platinum(II) benzene solvate, $[Pt(C_{25}H_{21}P_2)_2].C_6H_6$, $M_r = 1040·0$, triclinic, $P\bar{1}$, $a = 9·780$ (1), $b = 9·817$ (2), $c = 12·898$ (1) Å, $\alpha = 93·09$ (1), $\beta = 109·51$ (1), $\gamma = 95·68$ (1)° (reduced cell), $V = 1157$ (1) Å³, $Z = 1$, $D_x = 1·493$ g cm⁻³, λ(Mo Kα) = 0·71069 Å, $\mu = 32·4$ cm⁻¹, $F(000) = 522$, $T = 295$ K, $R = 0·016$ for 6723 unique observed intensities. The structure is built from molecules of the bis(chelate) square-planar platinum(II) complex and of benzene, both of which straddle sites of crystallographic $\bar{1}$ symmetry. The ligand is attached to Pt through both P atoms and contains a delocalized P⋯C⋯P system with a mean P—C(methanido) bond length of 1·729 (2) Å. The PtP_2C chelate rings are significantly non-planar.

Introduction. The title complex (1) contains the first example of a transition-metal ion stabilized only by chelating bis(diphenylphosphino)methanide ligands. Products of composition $[M\{(C_6H_5)_2PCHP(C_6H_5)_2\}_2]$, $M = $ Pd, Pt, have been reported previously but were deemed intractable and probably polymeric (Schmidbauer & Mandl, 1977; Bassett, Mandl & Schmidbauer, 1980). The preparation and spectroscopic characterization of (1), together with preliminary details of its structure, have been described earlier (Brown, Yavari, Manojlović-Muir, Muir, Moulding & Seddon, 1982).