($d \rightarrow d$) π-bonding component in the Mo—P bond in (I) is less important than in these other derivatives. The P—Si length of 2·266 (1) Å is similar to that of 2·248 (3) Å in $P(SiH_3)_3$ (Beagley, Robiette & Sheldrick, 1968). The Si—Si distance of 2·378 (2) Å is normal.

Fig. 2 shows the unit-cell contents viewed perpendicular to **a**.

Calculations were carried out with *SHELX* 76 (G. M. Sheldrick, Cambridge) and local ancillary programs (WSS). We are indebted to Dr O. Stelzer for suggesting the problem and for the provision of crystals.

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Potassium Perchlorate

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Abstract. $KClO_4$, orthorhombic, *Pnma*, a = 8·866 (2), b = 5·666 (1), c = 7·254 (1) Å, Z = 4, V = 364·4 (1) Å^3, M_r = 138·56. The final R is 0·031 for 533 independent reflexions. The K atom is twelvefold coordinated with K—O distances of 2·862 (3)—3·453 (3) Å. The Cl—O distances are 1·427 (4)—1·441 (3) Å.

Introduction. Crystals of $KClO_4$ were obtained from water. The structure has been solved from powder data by Gottfried & Schusterius (1933). Mani (1957) refined the structure with data obtained from two zero-layer Weissenberg films. In the present investigation intensities were collected on a four-circle Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The crystal was 0·012 × 0·025 × 0·018 mm. An $\omega/2\theta$ scan was used with scan intervals of 2·4–3·2° ($\Delta 2\theta$) and a scanning speed of 2·0–12·0° min^{-1}. The intensities were measured out to 2θ = 70° and those 533 independent reflexions with $I/\sigma(I) > 3$ were used in the structure refinement. The background and integrated intensities were calculated with the Lehmann–Larsen profile-analysis method (local program; Lindqvist & Ljungström, 1977). The intensities were corrected for Lorentz, polarization and absorption effects (program *DATAPH*; Coppens, Leiserowitz & Rabinovich, 1965); μ(Mo $K\alpha$) = 10·03 cm^{-1} (*International Tables for X-ray Crystallography*, 1962). The final refinement included anisotropic temperature factors and an isotropic extinction parameter (*LINUS*; Coppens & Hamilton, 1970) and yielded an R of 0·031. The resulting positional parameters are given in Table 1.*

Table 1. *Final positional parameters*

Standard deviations are given in parentheses.

	x	y	z
K	0·1809 (1)	0·25	0·3374 (1)
Cl	0·0679 (1)	0·25	0·8105 (1)
O(1)	0·1885 (4)	0·25	0·9419 (4)
O(2)	0·4197 (2)	0·5438 (4)	0·1952 (3)
O(3)	0·4253 (4)	0·25	0·5981 (4)

Table 2. *Bond distances* (Å) *and angles* (°) *in* $KClO_4$

Cl—O(3)	1·427 (4)	2× O(1)—O(2)	2·343 (4)
Cl—O(1)	1·433 (4)	O(1)—O(2)	2·351 (6)
2× Cl—O(2)	1·441 (3)	2× O(2)—O(3)	2·346 (4)
2× K—O(2)	2·862 (3)	O(2)—O(2)	2·337 (5)
K—O(1)	2·870 (4)		
K—O(3)	2·877 (4)	O(1)—Cl—O(2)	109·2 (1)
2× K—O(2)	2·884 (3)	O(1)—Cl—O(3)	110·6 (2)
2× K—O(2)	2·983 (3)	O(2)—Cl—O(2)	108·3 (2)
2× K—O(1)	3·153 (2)	O(2)—Cl—O(3)	108·7 (2)
2× K—O(2)	3·453 (3)		

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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 32684 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. Interatomic distances and angles are given in Table 2. The structural arrangement is in agreement with previous work (Gottfried & Schusterius, 1933; Mani, 1957). However, the precision in the present work is better by more than one order of magnitude. The Cl–O distances [mean 1·433 (2) Å] compare well with values found in six recent investigations (Berglund, Thomas & Tellgren, 1975).

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Caesium Triaquabis(oxalato)titanate(III) Dihydrate

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Abstract. $Cs[Ti(C_2O_4)_2(H_2O)_3].2H_2O$, $C_4H_{10}CsTiO_3$, $M_r = 446{\cdot}92$, monoclinic, $a = 6{\cdot}543$ (8), $b = 11{\cdot}490$ (11), $c = 8{\cdot}015$ (9) Å, $\beta = 96{\cdot}51$ (8)°, $U = 598{\cdot}7$ Å^3, $Z = 2$, $d_m = 2{\cdot}54$ (3), $d_c = 2{\cdot}48$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 39{\cdot}0$ cm^{-1}, space group $P2_1/m$ (from systematic absences $0k0$, $k = 2n + 1$ and the successful structure determination). The anion has crystallographically imposed C_s symmetry. The Ti atom is in a seven-coordinate pentagonal-bipyramidal environment with two water molecules in axial sites [2·101 (5), 2·070 (5) Å], and one in the equatorial girdle [2·130 (5) Å] together with both oxalates [2·314 (5), 2·115 (3) Å]. 1510 independent reflexions collected by counter methods have been refined to $R = 0{\cdot}042$.

Introduction. $Cs[Ti(ox)_2(H_2O)_3].2H_2O$ (ox = oxalate) was prepared by adding 0·5 g $TiCl_3$ to a hot solution of 0·7 g of oxalic acid hydrate in 20 cm^3 water and subsequently adding a solution of 0·6 g of Cs carbonate and 0·5 g oxalic acid in 10 cm^3 water. Reactions were carried out under nitrogen. Crystals of a lower hydrate, possibly $Cs[Ti(ox)_2(H_2O)_2]$, were first formed as fine gold/orange plates but these recrystallized after several hours to form much coarser brown crystals of the title compound. A crystal with dimensions *ca* 0·25 × 0·3 × 0·4 mm was mounted with the a^* axis parallel to the instrument axis of a GE XRD 5 apparatus, which was used to measure cell dimensions *via* least-squares refinement of high-angle reflexions and diffraction intensities by the stationary-crystal–stationary-counter method. It was equipped with a manual goniostat, a scintillation counter and a pulse-height discriminator. Zr-filtered Mo radiation was used with a 4° take-off angle and a counting time of 10 s. 1830 independent reflexions were measured with $2\theta < 60°$ and, of these, 1510 with $I > 3\sigma(I)$ were used in subsequent calculations. An absorption correction was applied using the *ABSORB* program (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The positions of the Cs atoms were obtained from a Patterson function and the positions of the Ti, C and O atoms from a Fourier map. After these atoms were refined anisotropically *via* full-matrix least squares, a difference Fourier map was calculated and the positions of four of the five independent H atoms were located. These were included in the refinement and given isotropic thermal parameters. The final R value was 0·042. In the final cycle no shift was $>0{\cdot}01\sigma$. The weighting scheme, chosen to give constant values of $w\Delta^2$ over ranges of F_o and sin θ/λ, was $\sqrt{w} = 1$ for $F_o < 12{\cdot}5$ and $12{\cdot}5/F_o$ for $F_o > 12{\cdot}5$. Calculations were performed on a CDC 7600 computer at the University of London Computer Centre with the XRAY system (Stewart *et al.*, 1972).