max. $\Delta\rho = 0.33 \, e \, \text{Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *XSEN* system (Takenaka & Sasada, 1983) and *DABEX* (Ohba, 1986). Final atomic parameters are given in Table 1.*

Discussion. Fig. 1 shows the conformation of the molecule with the atom numbering. Bond distances and angles are in Table 2.

The dimensions of the glutamic acid moiety, the cysteine moiety and peptide moiety are in good agreement with those in related compounds (Wright, 1958; Kerr, Ashmore & Koetzle, 1975; Lehmann & Nunes, 1980; Lehmann, Koetzle & Hamilton, 1972; Takusagawa, Koetzle, Kou & Parthasarathy, 1981). As the thermal factors of the ethyl ester moiety are large due to disorder or large thermal vibrations, the C(4)—C(5) bond distance is seemingly shorter than the usual value. The molecule is in an extended form, and some conformation angles are: N(2)—C(9)—C(8)—C(7) 182·0 (4), C(9)—C(8)—C(7)—C(6) 182·9 (4), C(8)—C(7)—C(6)—N(1) 157·9 (4), C(7)—C(6)—N(1)—C(1) 177·2 (4), C(6)—N(1)—C(1)—C(3) 155·9 (5) and N(1)—C(1)—C(3)—O(2) 180·5 (5)°.

The crystal structure viewed along the c axis is shown in Fig. 2 and the hydrogen bonds are listed in

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* Lists of structure factors, anisotropic thermal parameters and atomic parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52955 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. The SH group of the cysteine residue is hydrogen bonded to a water molecule, $S\cdots O(W)$ 3·479 (5) and $H(S)\cdots OW$ 2·20 (6) Å. These values are similar to those found in the neutron diffraction study of L-cysteine (Takusagawa, Koetzle, Kou & Parthasarathy, 1981). The molecules are arranged so as to form ribbons along a twofold screw axis through $N(2)H\cdots O(4)^{iii}$. As seen from Fig. 2, the ribbons are connected to each other through $N(2)H\cdots O(4)^{iv}$ hydrogen bonds along the c axis and a double molecular sheet is formed parallel to the bc plane. In addition, there are some hydrogen bonds to reinforce the sheet structure. The double sheets are loosely packed, the shortest contact being $C(4)\cdots O(1)$ $(1 - x, -\frac{1}{2} + y, 3 - z) = 3.32 \, (1)$ Å.

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Structure of Potassium Difluorooctamolybdate Hexahydrate

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Abstract. $K_6[Mo_8O_{26}F_2].6H_2O$, $M_r = 1564·18$, triclinic, $P\bar{1}$, $a = 10·497$ (2), $b = 10·403$ (2), $c = 8·001$ (2) Å, $\alpha = 107·16$ (2), $\beta = 95·77$ (2), $\gamma = 105·53$ (2)°, $V = 788·9$ (3) Å³, $Z = 1$, $D_x = 3·29$ Mg m⁻³, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0·7107$ Å, μ(Mo $K\alpha$) = 36·63 cm⁻¹,

$F(000) = 736$, room temperature, final conventional $R = 0.025$, $wR = 0.030$ for 3932 observed reflections. The structure is built up of K^+ cations, $[Mo_8O_{26}F_2]^{6-}$ anions and H_2O molecules. The difluorooctamolybdate anions consist of eight centrosymmetrically condensed edge-sharing octahedra with 16 terminal positions. Of these 16 positions, 14 are occupied by O atoms with Mo=O bond lengths varying between 1.703 (3) and 1.724 (3) Å, and two F atoms at a distance of 1.970 (3) Å. Of the remaining 12 O atoms, six single bridge two Mo atoms with Mo—O bond lengths of between 1.755 (2) and 2.408 (2) Å, four triple bridge three Mo atoms with Mo—O distances of between 1.882 (2) and 2.291 (2) Å and two O atoms quadruple bridge four Mo atoms with Mo—O distances of between 1.971 (3) and 2.398 (2) Å. The coordination octahedra are significantly distorted, with the angles at the Mo atoms ranging from 69.7 (1) to 106.4 (1)°. K^+ cations are irregularly coordinated by O atoms from octamolybdates and water molecules as well as by F
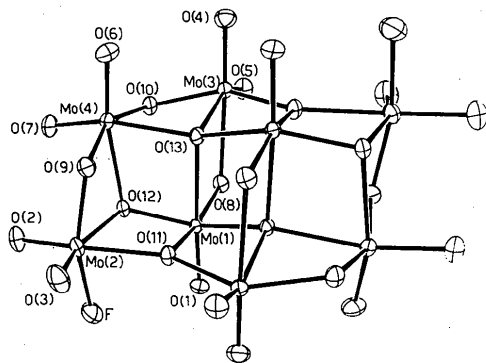

Fig. 1. ORTEP drawing of the $[Mo_8O_{26}F_2]^{6-}$ anion with the atomic labelling scheme. Thermal ellipsoids are at 50% probability level.
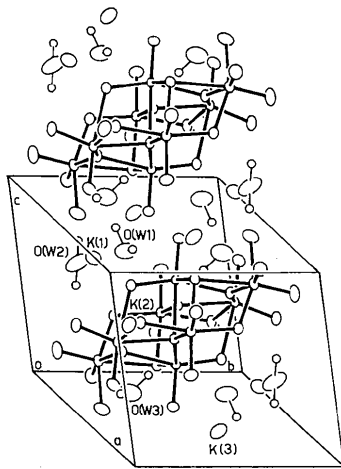

Fig. 2. Packing diagram showing the atomic labelling for K^+ ions and water molecules.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($\times 10^4$, $\times 10^5$ *for* Mo) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
Mo(1)	48951 (2)	47980 (2)	27535 (3)	1206 (4)
Mo(2)	17756 (3)	24346 (3)	14397 (4)	1799 (4)
Mo(3)	50144 (2)	79143 (2)	60542 (3)	1792 (4)
Mo(4)	20806 (2)	56085 (3)	40707 (3)	1982 (4)
F	2698 (2)	1919 (3)	−566 (3)	351 (5)
O(1)	5557 (2)	4115 (2)	949 (3)	204 (4)
O(2)	502 (3)	2591 (3)	53 (4)	291 (5)
O(3)	1157 (3)	816 (3)	1660 (4)	323 (6)
O(4)	4408 (3)	8628 (3)	7916 (3)	316 (5)
O(5)	6031 (2)	9353 (2)	5677 (4)	292 (5)
O(6)	1547 (3)	6090 (3)	6041 (3)	371 (5)
O(7)	927 (2)	5741 (3)	2495 (4)	335 (5)
O(8)	5687 (2)	6634 (2)	3441 (3)	222 (4)
O(9)	1501 (2)	3580 (2)	3634 (3)	192 (4)
O(10)	3427 (2)	7381 (2)	4266 (3)	230 (4)
O(11)	3737 (2)	2787 (2)	2862 (3)	166 (4)
O(12)	3113 (2)	4643 (2)	1854 (3)	161 (4)
O(13)	4033 (2)	5598 (2)	5402 (3)	184 (4)
K(1)	4891 (1)	1483 (1)	8256 (1)	347 (2)
K(2)	8646 (1)	1158 (1)	6626 (1)	383 (2)
K(3)	1705 (1)	5060 (1)	8990 (1)	367 (2)
O(W1)	7578 (4)	1926 (4)	9939 (5)	493 (8)
O(W2)	1298 (4)	2263 (5)	6254 (5)	641 (12)
O(W3)	8506 (5)	882 (5)	3109 (6)	658 (11)

atoms. Different types of coordination are exhibited by the two K^+ cations: K(1) is surrounded by eight O atoms [2.796 (2)–3.134 (3) Å] and one F atom [2.671 (3) Å], K(2) [2.728 (5)–3.107 (3) Å] and K(3) [2.694 (3)–2.971 (4) Å] by eight O atoms only. The distances $O(W1)\cdots O(4^i)$ 2.878 (5), $O(W1)\cdots O(2^{ii})$ 2.945 (5), $O(W2)\cdots O(2^{iii})$ 3.179 (5), $O(W2)\cdots O(9)$ 2.819 (6), $O(W3)\cdots F^{iv}$ 2.870 (4), $O(W3)\cdots O(3^v)$ 3.130 (6) and $O(W3)\cdots O(6^{vi})$ 3.038 (6) Å indicate that the difluorooctamolybdate anions are mutually hydrogen bonded through the water molecules [(i) 1 $- x$, 1 $- y$, 2 $- z$; (ii) 1 $+ x$, y, 1 $+ z$; (iii) x, y, 1 $+ z$; (iv) 1 $- x$, $- y$, $- z$; (v) 1 $+ x$, y, z; (vi) 1 $- x$, 1 $- y$, 1 $- z$].

Experimental. The title compound was obtained by the reaction of MoO_3 with equivalent amounts of HF and KOH. The reaction product was evaporated to dryness and dissolved in hot water to yield transparent prism-shaped colourless crystals. A crystal of dimensions $0.32 \times 0.09 \times 0.07$ mm was used. Mo $K\alpha$ radiation was used with a graphite-crystal monochromator on a Philips PW1100 single-crystal diffractometer. The unit-cell parameters were determined from the angular settings of 20 reflections ($10 < \theta < 16°$). The intensities of 4026 reflections (up to $\theta = 30°$), hkl range from $-14, -14, 0$ to 14, 13, 11, were measured using the $\omega - 2\theta$ scan technique with a scan angle of $1.60°$ and a scan speed of $0.04°$ s^{-1}. 3932 unique observed reflections with $I \geq 3\sigma(I)$ were used in the structure determination. Three standard reflections (230, $\overline{3}45$, $\overline{3}7\overline{4}$) monitored every 2 h showed no significant intensity fluctuation.

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

Primed atoms are related to unprimed atoms by a centre of symmetry at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$).

Mo(1)—O(1)	1·712 (3)	Mo(2)—F	1·970 (3)
Mo(1)—O(8)	1·755 (2)	Mo(2)—O(2)	1·724 (3)
Mo(1)—O(11)	2·151 (2)	Mo(2)—O(3)	1·703 (3)
Mo(1)—O(12)	1·882 (2)	Mo(2)—O(9)	1·907 (2)
Mo(1)—O(13)	2·398 (2)	Mo(2)—O(11)	2·133 (2)
Mo(1)—O(13')	1·971 (3)	Mo(2)—O(12)	2·256 (2)
Mo(3)—O(4)	1·722 (3)	Mo(4)—O(6)	1·706 (3)
Mo(3)—O(5)	1·717 (1)	Mo(4)—O(7)	1·721 (3)
Mo(3)—O(8)	2·408 (2)	Mo(4)—O(9)	1·944 (2)
Mo(3)—O(10)	1·921 (2)	Mo(4)—O(10)	1·952 (2)
Mo(3)—O(13)	2·227 (2)	Mo(4)—O(12)	2·291 (2)
Mo(3)—O(11')	1·913 (3)	Mo(4)—O(13)	2·217 (2)
O(1)—Mo(1)—O(8)	104·7 (1)	F—Mo(2)—O(2)	90·7 (1)
O(1)—Mo(1)—O(11)	95·9 (1)	F—Mo(2)—O(3)	99·5 (1)
O(1)—Mo(1)—O(12)	106·4 (1)	F—Mo(2)—O(9)	157·1 (1)
O(1)—Mo(1)—O(13)	174·4 (1)	F—Mo(2)—O(11)	79·8 (1)
O(1)—Mo(1)—O(13')	99·8 (1)	F—Mo(2)—O(12)	83·1 (1)
O(8)—Mo(1)—O(11)	158·8 (1)	O(2)—Mo(2)—O(3)	105·5 (1)
O(8)—Mo(1)—O(12)	101·7 (1)	O(2)—Mo(2)—O(9)	97·3 (1)
O(8)—Mo(1)—O(13)	78·7 (1)	O(2)—Mo(2)—O(11)	160·6 (1)
O(8)—Mo(1)—O(13')	97·9 (1)	O(2)—Mo(2)—O(12)	92·6 (1)
O(11)—Mo(1)—O(12)	76·6 (1)	O(3)—Mo(2)—O(9)	98·9 (1)
O(11)—Mo(1)—O(13)	80·4 (1)	O(3)—Mo(2)—O(11)	92·8 (1)
O(11)—Mo(1)—O(13')	73·4 (1)	O(3)—Mo(2)—O(12)	161·7 (1)
O(12)—Mo(1)—O(13)	76·9 (1)	O(9)—Mo(2)—O(11)	85·9 (1)
O(12)—Mo(1)—O(13')	141·8 (1)	O(9)—Mo(2)—O(12)	75·2 (1)
O(13)—Mo(1)—O(13')	75·2 (1)	O(1)—Mo(2)—O(12)	69·7 (1)
O(4)—Mo(3)—O(5)	104·6 (1)	O(6)—Mo(4)—O(7)	106·1 (1)
O(4)—Mo(3)—O(8)	172·9 (1)	O(6)—Mo(4)—O(9)	96·8 (1)
O(4)—Mo(3)—O(10)	99·2 (1)	O(6)—Mo(4)—O(10)	100·9 (1)
O(4)—Mo(3)—O(11')	98·4 (1)	O(6)—Mo(4)—O(12)	163·3 (1)
O(4)—Mo(3)—O(13)	102·4 (1)	O(6)—Mo(4)—O(13)	92·5 (1)
O(5)—Mo(3)—O(8)	82·5 (1)	O(7)—Mo(4)—O(9)	101·5 (1)
O(5)—Mo(3)—O(10)	101·7 (1)	O(7)—Mo(4)—O(10)	93·9 (1)
O(5)—Mo(3)—O(13)	153·0 (1)	O(7)—Mo(4)—O(12)	89·4 (1)
O(5)—Mo(3)—O(11')	103·6 (1)	O(7)—Mo(4)—O(13)	159·1 (1)
O(8)—Mo(3)—O(10)	78·6 (1)	O(9)—Mo(4)—O(10)	152·3 (1)
O(8)—Mo(3)—O(13)	70·5 (1)	O(9)—Mo(4)—O(12)	73·7 (1)
O(8)—Mo(3)—O(11')	80·1 (1)	O(9)—Mo(4)—O(13)	85·2 (1)
O(10)—Mo(3)—O(13)	73·2 (1)	O(10)—Mo(4)—O(12)	83·7 (1)
O(10)—Mo(3)—O(11')	144·3 (1)	O(10)—Mo(4)—O(13)	72·9 (1)
O(13)—Mo(3)—O(11')	72·7 (1)	O(12)—Mo(4)—O(13)	73·3 (1)
Mo(1)—O(8)—Mo(3)	112·9 (1)	Mo(1)—O(12)—Mo(4)	111·8 (1)
Mo(2)—O(9)—Mo(4)	115·7 (1)	Mo(2)—O(12)—Mo(4)	91·6 (1)
Mo(3)—O(10)—Mo(4)	114·2 (1)	Mo(1)—O(13)—Mo(3)	97·8 (1)
Mo(1)—O(11)—Mo(2)	103·7 (1)	Mo(1)—O(13)—Mo(4)	97·1 (1)
Mo(1)—O(12)—Mo(2)	108·6 (1)	Mo(3)—O(13)—Mo(4)	94·0 (1)

Data were corrected for Lorentz and polarization effects but not for absorption. Atomic scattering factors and anomalous-dispersion corrections for Mo and K atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Patterson synthesis was used to locate Mo atoms and subsequent Fourier maps for non-H atoms. All H atoms were found in a difference synthesis map. The refinement of 219 parameters was by least-squares methods on F_o assuming anisotropic thermal parameters for all non-H atoms. In the final rounds of refinement H atoms were included in the structure-factor calculations with an overall U_{iso} of 0·45 Å^2. The final conventional agreement factors were $R = 0.025$ and $wR = 0.030$. The function minimized was

$\sum w(|F_o| - |F_c|)^2$ with $w = 1/[\sigma^2(F_o) + 0.0018F_o^2]$ with $\sigma(F_o)$ from counting statistics. The final difference Fourier map showed a residual density between -1.34 and 1.31 e Å^{-3}, maximum Δ/σ 0·155 for y of atom O(W3). An *ORTEP* (Johnson, 1971) representation of the octamolybdate anion is shown in Fig. 1. Final atomic coordinates are given in Table 1, selected bond lengths and angles in Table 2.* The crystal packing is shown in Fig. 2. A Univac 1110 computer of the Zagreb University Computing Centre, SRCE, was used to carry out all crystallographic computations with the program *SHELX*76 (Sheldrick, 1976). The final geometrical calculations were performed with the *CSU* program (Vickovic, 1988).

Related literature. So far several octamolybdates of a general formula $[Mo_8O_{26}(L)_2]^{(2n-4)}$ (where n is a formal charge of L) with L as an O atom (Isobe, Marumo, Yamase & Ikawa, 1978; Adams, Klemperer & Liu, 1979; You, Chen, Zheng & Huang, 1989; Kamenar, Korpar-Čolig, Penavić & Cindrić, 1989) and as nitrogen-donating ligands (McCarron, Whitney & Chase, 1984; Kamenar, Penavić & Marković, 1988) have been prepared and characterized structurally. The title compound is an example with F atoms as the ligand L, coordinated to an Mo_8O_{26} core (Kamenar, Kaitner & Strukan, 1989).

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* Lists of structure factors, H-atom coordinates, interatomic distances involving K$^+$ ions and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53073 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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