Work is in progress to solve the structures of the NaDC crystal and macromolecular fibre as well as to check the helical model in aqueous solutions by means of small-angle X-ray scattering.

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Crystal Structures of Ethylenediaminetetraacetato Metal Complexes. V. Structures containing the $[Fe(C_{10}H_{12}N_2O_8)(H_2O)]^-$ Anion

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Abstract. (Na,Fe): $Na.[Fe(C_{10}H_{12}N_2O_8)(H_2O)].2H_2O$, $M_r = 421{\cdot}1$, monoclinic, Cc, $a = 8{\cdot}896$ (1), $b = 11{\cdot}931$ (2), $c = 15{\cdot}065$ (2) Å, $\beta = 100{\cdot}15$ (2)°, $V = 1574{\cdot}0$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}78$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7093$ Å, $\mu = 1{\cdot}083$ mm^{-1}, $F(000) = 868$, room temperature. (Ag,Fe): $Ag.[Fe(C_{10}H_{12}N_2O_8)(H_2O)].2H_2O$, $M_r = 506{\cdot}0$, monoclinic, Cc, $a = 8{\cdot}929$ (1), $b = 11{\cdot}871$ (2), $c = 15{\cdot}116$ (2) Å, $\beta = 99{\cdot}85$ (2)°, $V = 1578{\cdot}6$ (6) Å^3, $Z = 4$, $D_x = 2{\cdot}13$ Mg m^{-3}, μ(Mo $K\alpha$) $= 2{\cdot}219$ mm^{-1}, $F(000) = 1012$, room temperature. (K,Fe): $K.[Fe(C_{10}H_{12}N_2O_8)(H_2O)].H_2O$, $M_r = 419{\cdot}2$, monoclinic, $P2_1/n$, $a = 11{\cdot}361$ (2), $b = 13{\cdot}396$ (2), $c = 10{\cdot}484$ (1) Å, $\beta = 106{\cdot}00$ (2)°, $V = 1533{\cdot}8$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}82$ Mg m^{-3}, μ(Mo $K\alpha$) $= 1{\cdot}332$ mm^{-1}, $F(000) = 860$, room temperature. (Tl,Fe): $Tl.[Fe(C_{10}H_{12}N_2O_8)(H_2O)].H_2O$, $M_r = 584{\cdot}5$, triclinic, $P\bar{1}$, $a = 13{\cdot}825$ (2), $b = 8{\cdot}591$ (1), $c = 6{\cdot}807$ (1) Å, $\alpha = 87{\cdot}79$ (1), $\beta = 97{\cdot}75$ (1), $\gamma = 104{\cdot}34$ (2)°, $V = 776{\cdot}1$ (3) Å^3, $Z = 2$, $D_x = 2{\cdot}50$ Mg m^{-3}, μ(Mo $K\alpha$) $= 11{\cdot}458$ mm^{-1}, $F(000) = 554$, room temperature. Final R values are 0·047 (Na,Fe), 0·046 (Ag,Fe), 0·049 (K,Fe) and 0·040 (Tl,Fe) for 1403, 1438, 3229 and 2350 observed reflections, respectively. The Fe ion is coordinated to four O and two N atoms of one

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Table 1. *Experimental data*

	(Na,Fe)	(Ag,Fe)	(K,Fe)	(Tl,Fe)
Crystal size (mm)	0·1 × 0·1 × 0·2	0·1 × 0·1 × 0·1	0·1 × 0·1 × 0·1	0·1 × 0·1 × 0·3
Colour	Yellow-brown	Yellow-brown	Brown	Yellow-brown
Diffractometer, radiation	Philips PW-1100, graphite monochromated Mo $K\alpha$			
Cell parameters from	25 reflections ($4 \le \theta \le 12°$)			
Scan technique	ω			
Number of reflections measured	1420	1488	3240	2363
Number of observed reflections [$\theta \le 25°, I \ge 2{\cdot}5\sigma(I)$]	1403	1438	3229	2350
Range of *hkl*	−10,10; 0,14; 0,17	−10,10; 0,14; 0,17	−15,15; 0,18; 0,14	−15,15; −10,10; 0,8
Variation of reference intensities	3 reflections, no variation			
Corrections of I_{hkl}	Lp, absorption ignored			
Determination	*MULTAN*80 (Main *et al.*, 1980)	Isomorphous replacement from (Na,Fe) coordinates	*MULTAN*80	Tl,Fe from Patterson, remaining from *F* synthesis
Refinements	Isotropic and anisotropic full-matrix least squares (*SHELX*76; Sheldrick, 1976)			
Scattering factors' source	f,f',f'' from *International Tables for X-ray Crystallography* (1974)			
Number of H atoms located (total H)	15(18)	13(18)	15(16)	11(16)
H refinement	Overall isotropic temperature factor			
R final (R_w)	0·047 (0·049)	0·046 (0·051)	0·049 (0·058)	0·040 (0·040)
Minimized function	$w\|\|F_o\| - \|F_c\|\|^2$, $w = [\sigma(F_o)^2 + k\|F_o\|]^{-1}$			
k value	0·0064	0·0041	0·0055	0·0002
Max. Δ/σ	0·4	3·2	4·6	1·3
in parameter	U_{13} of Ag	*x* of H(C21′)	*z* of H(C31′)	$U_{\rm iso}$ of H
Max. peak in final ΔF (e Å^{-3})	0·7	0·5	0·7	0·3
Min. peak (e Å^{-3})	−0·3	−0·3	−0·3	−0·3

ethylenediaminetetraacetato ligand and a water molecule in a pentagonal bipyramidal shape. Each cation is linked to six (Na and Ag) or seven (K and Tl) oxygen atoms, which produces variations in the Fe coordination polyhedra.

Introduction. A systematic crystal-structure study of compounds with ethylenediaminetetraacetato (EDTA) as ligand is being carried out by the authors' Departments (Solans, Font-Altaba, Oliva & Herrera, 1983, 1984; Solans, Galí, Font-Altaba, Oliva & Herrera 1983, 1984).

Crystal structures of X[EDTA(H_2O)Fe].$n H_2O$, with $X = Li^+$, Na^+, Rb^+ and $CH_6N_3^+$ (guanidinium) are known from the literature (Lind, Hamor, Hamor & Hoard, 1964; Novozhilova, Polynova, Porai-Koshits & Martynenko, 1974; Nesterova, Polynova, Martynenko & Pechurova, 1971). In order to determine the possible influence of the cation on the [EDTA(H_2O)Fe] coordination polyhedra, a refinement of the crystal structure with $X = Na^+$ and determinations of those with $X = Ag^+$, K^+ and Tl^+ have been carried out.

Experimental. All information is given in Table 1. Atomic parameters are listed in Table 2.* The atom numbering is defined in Fig. 1.

Discussion. The Fe^{3+} is surrounded by four O and two N atoms of an EDTA ligand, which acts in sexidentate fashion, and by a water molecule. The coordination polyhedron is of roughly pentagonal bipyramidal shape. The pentagonal bipyramid has been observed in the ethylenediaminetetraacetato metal complexes of (Li,Fe), (Rb,Fe) (Lind *et al.*, 1964), (Mg,Cd), (Mn,Mn) (Solans, Galí, Font-Altaba, Oliva & Herrera, 1983, 1984) and (Mn,Cd) (Solans, Galí, Font-Altaba, Oliva & Herrera, 1984), while the monocapped trigonal prism has been observed in (Mg,Mg) (Passer, White & Cheng, 1977) and (Mg,Mn) (Solans, Galí, Font-Altaba, Oliva & Herrera, 1984). According to the notation of Lind *et al.* (1964), we call O_G the EDTA oxygen atoms coordinated to Fe^{3+} lying nearly in the plane defined by the central atom and the two N atoms [O(11), O(41)], and O_R those O atoms located roughly along the normal to the Fe, N, N plane [O(21), O(31)]. From Table 3 it can be seen that the Fe—O_R bond distances [average 1·97 (1) Å] are shorter than the Fe—O_G bond distances [2·11 (1) Å], while the remaining bond distances are Fe—O(*W*) 2·12 (1) and Fe—N 2·33 (1) Å. This result is similar to that obtained by Lind *et al.* (1964) in the (Rb,Fe) and (Li,Fe) structures. The bond angles at Fe (Table 3) have similar values in the four title compounds and in the (Rb,Fe) and (Li,Fe) structures, with the exception of certain angles involving O_R. The cation displays six (Na and Ag) or seven (K and Tl) coordination [four (Li) and six (Rb) in (Li,Fe) and (Rb,Fe)]. The X—O mean distances are 2·41 (7), 2·50 (13), 2·92 (14) and 2·85 (13) Å for X = Na, Ag, Tl and K, respectively. These values are larger than the sum of ionic radii. The X—O distances near to the sum of ionic radii are, generally, the X—O(*W*) and X—O_R^* distances, where O_R^* is an oxygen atom linked to the acetato group of an O_R atom. Only the seven-coordinate ions are not linked to water

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39142 (45 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates* [$\times 10^4$; Tl *of* (Tl,Fe) *and* Fe *of* (K,Fe) $\times 10^5$] *and equivalent isotropic temperature factors* (Å^2)

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

	x	y	z	$B_{\rm eq}$
(*a*) (Na,Fe)				
Fe	9996	892 (1)	2499	1·34 (4)
Na	13930 (3)	316 (2)	1075 (2)	1·32 (10)
O(*W*1)	10010 (7)	−475 (6)	1594 (4)	2·0 (2)
O(*W*2)	6010 (10)	223 (8)	5435 (5)	3·7 (3)
O(*W*3)	10432 (12)	−2483 (7)	5062 (6)	3·8 (4)
N(10)	8555 (8)	1850 (6)	3392 (4)	1·7 (2)
C(10)	9503 (11)	2197 (8)	4263 (6)	2·1 (3)
N(20)	11697 (8)	1960 (5)	3495 (5)	1·7 (3)
C(20)	10938 (10)	2728 (9)	4074 (6)	2·4 (3)
C(11)	7346 (11)	1068 (8)	3549 (7)	2·1 (3)
C(12)	6767 (9)	434 (6)	2701 (6)	1·9 (3)
O(11)	7742 (7)	312 (5)	2170 (4)	2·0 (2)
O(12)	5467 (7)	36 (6)	2567 (4)	2·5 (2)
C(21)	7878 (11)	2817 (7)	2842 (6)	2·4 (3)
C(22)	8296 (9)	2885 (6)	1899 (6)	1·6 (2)
O(21)	9350 (7)	2222 (5)	1737 (4)	2·0 (2)
O(22)	7678 (7)	3572 (5)	1372 (4)	2·2 (2)
C(31)	12620 (11)	1109 (9)	4051 (7)	2·3 (3)
C(32)	11724 (9)	33 (7)	4128 (5)	1·7 (2)
O(31)	10548 (7)	−151 (5)	3516 (4)	1·8 (2)
O(32)	12194 (8)	−631 (5)	4750 (4)	2·3 (2)
C(41)	12627 (11)	2610 (7)	2953 (6)	2·1 (3)
C(42)	12868 (9)	1924 (7)	2150 (6)	1·7 (4)
O(41)	12025 (7)	1049 (5)	1975 (5)	1·9 (2)
O(42)	13818 (8)	2221 (6)	1690 (5)	2·7 (2)
(*b*) (Ag,Fe)				
Fe	9996	898 (1)	2499	0·90 (5)
Ag	14037 (2)	369 (1)	1002 (1)	2·95 (5)
O(*W*1)	10015 (10)	−474 (6)	1615 (6)	1·6 (3)
O(*W*2)	6191 (13)	206 (11)	5439 (7)	3·4 (5)
O(*W*3)	10471 (13)	−2553 (9)	5127 (7)	3·2 (5)
N(10)	8544 (9)	1863 (7)	3406 (6)	1·3 (3)
C(10)	9496 (13)	2185 (9)	4257 (7)	1·5 (4)
N(20)	11686 (10)	1946 (7)	3535 (6)	1·5 (3)
C(20)	10953 (14)	2712 (9)	4092 (8)	1·9 (4)
C(11)	7330 (11)	1089 (9)	3548 (8)	1·7 (3)
C(12)	6764 (13)	426 (7)	2713 (8)	1·2 (4)
O(11)	7742 (10)	320 (6)	2200 (6)	1·8 (3)
O(12)	5495 (8)	14 (7)	2581 (5)	1·8 (3)
C(21)	7934 (15)	2858 (10)	2862 (8)	2·0 (4)
C(22)	8289 (12)	2864 (8)	1927 (7)	1·4 (4)
O(21)	9392 (8)	2244 (6)	1778 (5)	1·7 (3)
O(22)	7628 (8)	3548 (6)	1382 (5)	1·7 (3)
C(31)	12574 (12)	1059 (8)	4077 (8)	1·7 (3)
C(32)	11680 (10)	8 (7)	4141 (6)	0·9 (3)
O(31)	10497 (10)	−160 (5)	3540 (5)	1·6 (3)
O(32)	12123 (10)	−679 (7)	4751 (6)	2·0 (3)
C(41)	12643 (11)	2584 (9)	3009 (7)	1·6 (4)
C(42)	12880 (11)	1934 (8)	2195 (6)	1·3 (3)
O(41)	12035 (8)	1044 (6)	2020 (5)	1·3 (3)
O(42)	13837 (9)	2227 (7)	1763 (6)	2·3 (3)
(*c*) (K,Fe)				
Fe	82471 (4)	17431 (4)	6523 (5)	1·68 (2)
K	8985 (1)	470 (1)	6403 (1)	3·56 (5)
C(10)	9606 (4)	3332 (3)	2817 (4)	2·3 (1)
N(10)	8460 (3)	2727 (2)	2515 (3)	1·8 (1)
C(20)	9749 (4)	3757 (3)	1548 (4)	2·3 (1)
N(20)	9751 (3)	2930 (2)	598 (3)	1·9 (1)
C(11)	8459 (4)	2047 (3)	3605 (5)	2·1 (1)
C(12)	7523 (3)	1225 (3)	3082 (4)	2·1 (1)
O(11)	7241 (3)	1069 (2)	1839 (3)	2·4 (1)
O(12)	7119 (3)	744 (2)	3884 (3)	3·0 (1)
C(21)	7358 (4)	3363 (3)	2198 (5)	2·3 (1)
C(22)	6698 (3)	3422 (3)	730 (4)	2·0 (1)
O(21)	7033 (2)	2797 (2)	−36 (3)	2·2 (1)
O(22)	5877 (2)	4026 (2)	364 (3)	2·7 (1)
C(31)	10921 (4)	2376 (3)	982 (5)	2·6 (1)
C(32)	10836 (3)	1353 (3)	1567 (4)	1·9 (1)
O(31)	9771 (2)	1039 (2)	1613 (3)	2·1 (1)
O(32)	11783 (3)	874 (2)	2002 (3)	2·6 (1)
C(41)	9458 (4)	3295 (3)	−779 (4)	2·1 (1)
C(42)	9074 (4)	2415 (3)	−1687 (4)	2·3 (1)
O(41)	8646 (3)	1670 (2)	−1211 (3)	2·8 (1)
O(42)	9196 (4)	2438 (2)	−2829 (4)	3·5 (1)
O(*W*1)	7355 (3)	494 (3)	−417 (4)	3·9 (1)
O(*W*2)	−451 (4)	4182 (3)	5860 (4)	3·4 (1)
(*d*) (Tl,Fe)				
Tl	41259 (3)	42509 (5)	21248 (6)	2·75 (2)
Fe	3053 (1)	8885 (2)	4739 (2)	1·85 (6)
N(10)	2275 (5)	10153 (9)	6757 (11)	1·9 (3)
C(10)	1801 (8)	9033 (12)	8299 (14)	2·3 (4)
C(20)	1293 (8)	7461 (13)	7363 (16)	2·6 (5)
N(20)	2033 (5)	6849 (9)	6444 (11)	2·1 (3)
C(11)	3035 (7)	11518 (11)	7667 (14)	2·4 (4)
C(12)	3692 (7)	12311 (11)	6131 (14)	2·3 (4)
O(11)	3766 (5)	11366 (8)	4800 (10)	2·8 (3)
O(12)	4108 (5)	13757 (8)	6190 (11)	3·2 (3)
C(21)	1503 (7)	10724 (13)	5336 (13)	2·4 (4)
C(22)	1254 (7)	9862 (11)	3399 (14)	2·4 (4)
O(21)	1857 (5)	9021 (8)	2978 (9)	2·7 (3)
O(22)	500 (5)	9994 (10)	2280 (11)	3·9 (4)
C(31)	2761 (7)	6273 (12)	7946 (14)	2·6 (5)
C(32)	3783 (7)	7428 (11)	8342 (14)	2·3 (5)
O(31)	4020 (4)	8606 (8)	7085 (9)	2·3 (2)
O(32)	4374 (5)	7197 (8)	9799 (10)	2·8 (3)
C(41)	1547 (7)	5597 (12)	4977 (16)	2·5 (4)
C(42)	2287 (7)	5446 (11)	3584 (15)	2·7 (5)
O(41)	3020 (5)	6664 (8)	3493 (10)	2·8 (3)
O(42)	2133 (6)	4193 (9)	2603 (12)	3·7 (5)
O(*W*1)	4064 (5)	9363 (8)	2607 (10)	2·8 (3)
O(*W*2)	−800 (9)	6933 (13)	1046 (17)	7·7 (6)

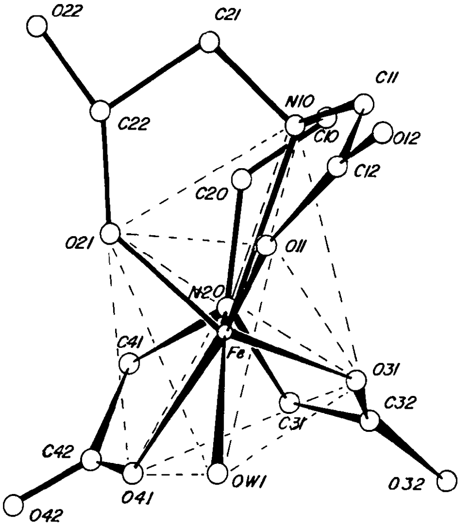

Fig. 1. View of the Fe coordination polyhedron, with the numbering of the atoms.

molecules, while the shorter distances are X—O_G^*. This would explain the differences in the coordination polyhedra.

Fig. 2 shows the packing arrangements in the (Ag,Fe), (K,Fe) and (Tl,Fe) structures. The anions form layers along a crystallographic plane. Single layers are loosely linked by the cations into electrically neutral double layers. Cohesion between double layers depends largely on van der Waals interactions supplemented by weak hydrogen bonds formed by water molecules with carboxylate O atoms. In each structure water molecules play the limited role of dielectric filler, which has been observed also in the (Rb,Fe) structure.

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Table 3. *Selected bond distances* (Å) *and angles* (°), *with weighted average values; averages in square brackets have been calculated without the* (K,Fe) *and* (Tl,Fe) *values*

	(Na,Fe)	(Ag,Fe)	(K,Fe)	(Tl,Fe)	Average	
Fe–N(10)	2·316 (5)	2·341 (8)	2·313 (3)	2·307 (7)	2·33 (1)	
Fe–N(20)	2·316 (5)	2·338 (8)	2·346 (3)	2·336 (7)		
Fe–O(11)	2·096 (4)	2·101 (8)	2·110 (2)	2·115 (6)	2·11 (1)	
Fe–O(41)	2·102 (5)	2·079 (6)	2·126 (2)	2·105 (7)		
Fe–O(21)	1·983 (4)	1·957 (7)	1·967 (2)	1·932 (7)	1·97 (2)	
Fe–O(31)	1·968 (4)	2·004 (7)	1·983 (2)	1·987 (7)		
Fe–O(*W*1)	2·128 (5)	2·109 (7)	2·111 (2)	2·109 (6)	2·121 (8)	
N(20)–Fe–N(10)	73·6 (2)	72·9 (3)	74·2 (1)	73·8 (3)	73·8 (5)	
O(11)–Fe–N(10)	72·3 (2)	71·3 (3)	72·2 (1)	71·4 (2)	71·6 (6)	
O(41)–Fe–N(20)	71·8 (2)	71·7 (3)	70·6 (1)	71·7 (3)		
O(21)–Fe–N(20)	78·9 (2)	78·5 (3)	78·5 (1)	79·4 (3)	78·5 (6)	
O(31)–Fe–N(20)	78·4 (2)	77·0 (3)	78·2 (1)	79·3 (3)		
O(21)–Fe–N(20)	91·4 (2)	91·4 (3)	86·9 (1)	87·7 (3)	88 (2)	
O(31)–Fe–N(20)	87·0 (2)	85·3 (3)	88·2 (1)	88·6 (3)		
O(21)–Fe–O(11)	87·7 (2)	89·1 (3)	94·8 (1)	97·2 (3)	92·7 (36)	[88·3 (7)]
O(31)–Fe–O(11)	94·2 (2)	92·3 (3)	92·1 (1)	88·1 (3)	92·0 (19)	[93·4 (9)]
O(21)–Fe–O(41)	83·8 (2)	84·9 (3)	90·1 (1)	90·3 (3)	87·9 (29)	[84·2 (5)]
O(31)–Fe–O(41)	103·6 (2)	103·6 (3)	93·8 (1)	93·8 (3)	97·6 (48)	[103·6 (1)]
O(11)–Fe–O(*W*1)	73·1 (2)	73·9 (3)	73·4 (1)	71·4 (3)	78·2 (14)	
O(41)–Fe–O(*W*1)	74·0 (2)	75·0 (3)	70·6 (1)	73·1 (3)		
O(21)–Fe–O(*W*1)	106·0 (2)	108·0 (3)	100·9 (1)	97·3 (3)	102·6 (36)	[106·8 (10)]
O(31)–Fe–O(*W*1)	89·2 (2)	89·4 (3)	96·7 (1)	98·3 (3)	94·1 (38)	[89·3 (1)]

(Na,Fe)		(Ag,Fe)		(K,Fe)		(Tl,Fe)	
Na–O(*W*2)	2·324 (5)	Ag–O(*W*2)	2·334 (7)	K–O(12)	2·918 (3)	Tl–O(12)	2·786 (8)
Na–O(12)	2·439 (5)	Ag–O(12)	2·551 (7)	K–O(42)	2·749 (3)	Tl–O(12′)	2·744 (8)
Na–O(42)	2·463 (5)	Ag–O(42)	2·508 (7)	K–O(41)	3·086 (3)	Tl–O(42)	2·805 (7)
Na–O(41)	2·507 (5)	Ag–O(41)	2·673 (6)	K–O(32)	2·757 (3)	Tl–O(11)	2·998 (6)
Na–O(32)	2·438 (5)	Ag–O(32)	2·614 (7)	K–O(22′)	2·712 (3)	Tl–O(41)	3·099 (7)
Na–O(22)	2·329 (5)	Ag–O(22)	2·351 (7)	K–O(22)	2·749 (7)	Tl–O(32)	2·905 (6)
				K–O(31)	2·965 (3)	Tl–O(32′)	3·115 (7)

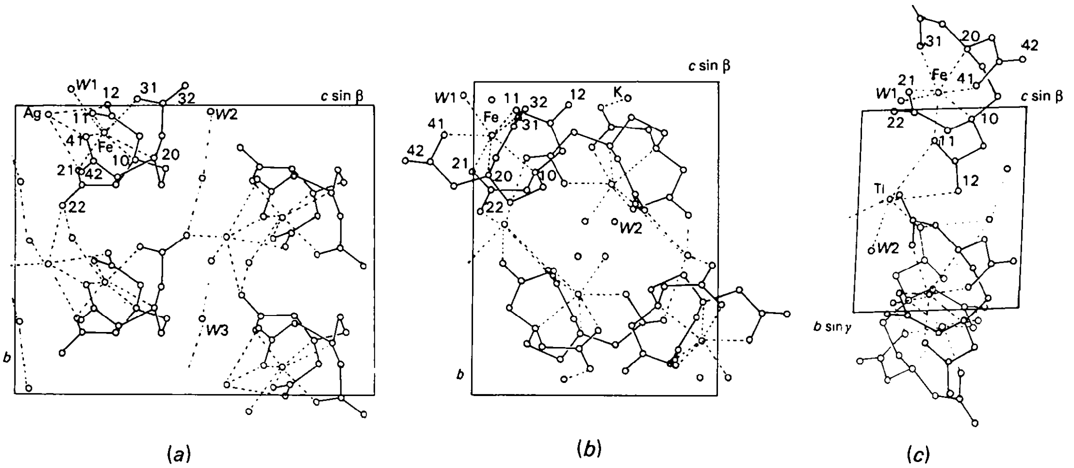

Fig. 2. Projection of the unit-cell contents for the (*a*) (Ag,Fe), (*b*) (K,Fe) and (*c*) (Tl,Fe) crystal structures.

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