*Inorganica Chimica Acta*, 50 (1981) 111–120

# Iron(III) Compounds of Phenolic Ligands. The Crystal and Molecular Structure of Iron(III) Compounds of the Sexadentate Ligand N,N′-ethylene-bis-(*o*-hydroxyphenylglycine)

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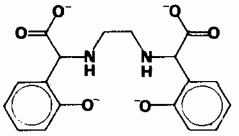
Received December 18, 1980

*The iron(III) compounds of N,N′-ethylene-bis (o-hydroxyphenylglycine),* $M^{n+}[Fe(ehpg)]^{-}]xH_2O$ *have been isolated and two distinct isomorphous series characterised; one (labelled α) for* $Li^+$, $Na^+$, $K^+$, $Ca^{2+}$, $Ba^{2+}$, *the other (labelled β) for* $Mg^{2+}$ *and* $Na^+$ *only. A full X-ray structural analysis of each type is reported. Crystals of* $Na[Fe(ehpg)]\cdot 4H_2O$ *(α) are orthorhombic* a = *37.66(2),* b = *7.12(1),* c = *24.44(1) Å, space group* Pbcn *(*$D_{2h}^{14}$*, no. 60), and those of* $Mg[Fe(ehpg)]_2\cdot 9H_2O$ *(β) are monoclinic* a = *17.074(15),* b = *13.099(11),* c = *20.868(11) Å, β = 113.57(5)°, space group* I2/c *(non-standard setting* $C_{2h}^{6}$*, no. 15). Data for each analysis were collected by diffractometer, the structures solved for 2191 and 3124 reflections, respectively, by conventional Patterson and Fourier techniques and were refined by block-diagonal least-squares to final* R*-values of 0.067 and 0.038, respectively. The asymmetric unit of the sodium salt contains one [Fe(*meso*-ehpg)]⁻ ion with* cis *phenoxy and carboxylate oxygens in a general position and one-half [Fe(*rac*-ehpg)]⁻ ion, with* trans *carboxylate oxygens, on a* $C_2$ *axis. In each case, the co-ordination polyhedron is distorted octahedral [mean Fe–N 2.149(2), Fe–O (phenolic) 1.906(15), Fe–O (carboxylate) 2.042(40) Å,* $N\text{–}\hat{Fe}\text{–}N'$ *79.9(1.0),* $N\text{–}\hat{Fe}\text{–}O$ *(phenolic) 88.1(1.3),* $N\text{–}\hat{Fe}\text{–}O$ *(carboxylate) 77.9(0.2)°]. The crystal lattice contains hydrophilic channels, with disordered water molecules and* $Na^+$ *ions, and hydrophobic regions bounded by phenyl groups. The magnesium salt contains only the [Fe(*rac*-ehpg)]⁻ ion in general positions, with the same configuration as the sodium salt [mean Fe–N 2.166(13), Fe–O (phenolic) 1.911(10), Fe–O (carboxylate) 2.042(7) Å,* $N\text{–}\hat{Fe}\text{–}O$ *(phenolic) 88.4(8) and* $N\text{–}\hat{Fe}\text{–}O$ *(carboxylate) 77.2(5)°. The crystal lattice again contains hydrophilic and hydrophobic regions, the former with octahedral hexaaquamagnesium ions on* $C_2$ *axes.*

## Introduction

We have recently taken an interest in the bonding to iron(III) of the tetra-anionic ligand I [1] {abbreviated $ehpg^{4-}$} because the stability constant of this compound [2] is of the same order of magnitude as that observed for transferrin [3] and the other biological 'iron carriers'.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In a preliminary study [4], various salts of the anion $[Fe(ehpg)]^-$ were isolated for X-ray structural studies using the commercially available ligand $\{ehpgH_4\}$. Powder patterns show that these compounds generally fall into two distinct isomorphous series: the first (labelled α) has been characterised for $Li^+$, $Na^+$, $K^+$, $Ca^{2+}$ and $Ba^{2+}$, the second (labelled β) has so far only been found for $Mg^{2+}$ and for $Na^+$ (in the latter case crystallising from the reaction mixture after the α-form). Here we report on the isolation of the compounds and the structures of one of each of the two main crystalline types.

## Experimental

### *Preparation of the Compounds* $M^{n+}[Fe(ehpg)]_n\cdot xH_2O$*, where* $M^{n+}$ = $Na^+$, $K^+$, $Li^+$, $Ca^{2+}$, $Mg^{2+}$ *and* $Ba^{2+}$

N,N′-ethylene-bis-(*o*-hydroxyphenylglycine) (1g) {obtained from Fluka AG} was suspended in refluxing alcohol (10 ml) and a solution of ferric chloride (0.74 g) in water (10 ml) was added very slowly with stirring. The mixture was refluxed for half an hour. The resulting purple solution was filtered hot to

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TABLE I. The Compounds Prepared, their Crystalline Forms and their Analyses.

| Compound | Source | Form | Analyses (%) | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Found | | | Calculated | | |
| | | | C | H | N | C | H | N |
| $Na[Fe(ehpg)]\cdot 4H_2O$ | 4 equivalents NaOH | α | 42.2 | 4.5 | 5.7 | 42.6 | 4.7 | 5.5 |
| | 3 equivalents NaOH | | | | | | | |
| | 1st crop | β | | | | | | |
| | 2nd crop | α | | | | | | |
| $K[Fe(ehpg)]\cdot 4H_2O$ | 4 or 3 equivalents KOH | α | 41.9 | 4.3 | 5.6 | 41.3 | 4.6 | 5.4 |
| $Li[Fe(ehpg)]\cdot 4H_2O$ | 3 equivalents LiOH | α | 42.1 | 4.5 | 5.6 | 44.0 | 4.9 | 5.7 |
| $Ca[Fe(ehpg)]_2\cdot 9H_2O$ | 3 equivalents $Ca(OH)_2$ | α | 41.4 | 4.7 | 5.6 | 42.1 | 4.9 | 5.5 |
| $Mg[Fe(ehpg)]_2\cdot 9H_2O$ | 4 or 3 equivalents $Mg(OH)_2$ | β | 42.9 | 4.9 | 5.8 | 42.7 | 5.0 | 5.5 |
| $Ba[Fe(ehpg)]_2\cdot 9H_2O$ | 4 equivalents $Ba(OH)_2$ | α | 36.8 | 4.1 | 5.1 | 38.5 | 4.5 | 5.0 |
| $NH_4[Fe(ehpg)]\cdot 4H_2O$ | 3 equivalents $NH_4OH$ | γ | 42.8 | 5.6 | 8.5 | 43.0 | 5.6 | 8.4 |

remove traces of unreacted ligand. On cooling, three or four equivalents of the appropriate metal hydroxide were added, giving a blood red solution which was left to stand until red crystals formed. These were filtered off and recrystallised from methanol. The compounds prepared are listed in Table I, together with the analytical data and the crystalline forms, identified by their X-ray powder diffraction patterns. The IR spectra, shown in Fig. 1, are also characteristic of the crystalline form.

### *X-ray Structure Analyses*

#### *(A) Sodium Salt*

This was crystallised from methanol as thin red rectangular plates. The one used for the analysis had dimensions *ca.* 0.32 X 0.28 X 0.03 mm.

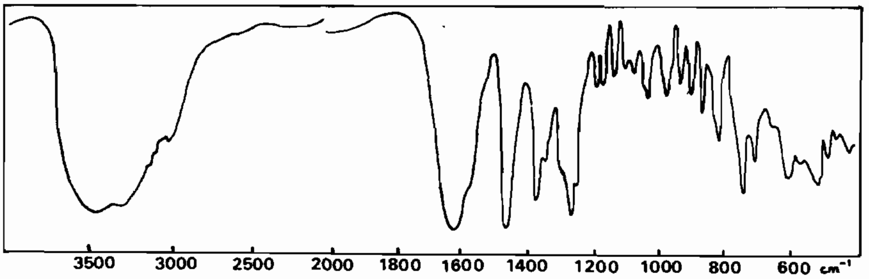
#### *Crystal Data*

$[C_{18}H_{16}N_2O_6Fe]Na\cdot 4H_2O$ (microanalysis); $M$ = 507.2. Orthorhombic, $a$ = 37.66(2), $b$ = 7.12(1), $c$ = 24.44(1) Å, $U$ = 6555(6) Å$^3$. $D_m$ (flotation) = 1.53 g cm$^{-3}$, $z$ = 12, $D_c$ = 1.542 g cm$^{-3}$. Space group *Pbcn* ($D_{2h}^{14}$, no. 60) from systematic absences. MoK$_\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu(MoK_\alpha) = 7.72$ cm$^{-1}$.

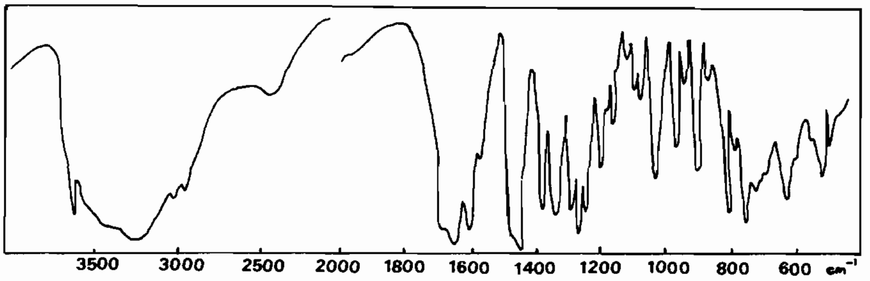
X-ray intensity data for $6.5° \leqslant 2\theta \leqslant 50°$ were measured in stationary counter/moving crystal scan mode on a Stoe Stadi-2 automatic diffractomer. For low layers ($k \leqslant 3$), the intensity profiles were rather asymmetric and some background differences (measured at each extremity of the scan) exceeded the normal acceptance criterion of $4\sigma_b$. Reflections with $I < 3\sigma_I$ were rejected, and Lorentz, polarisation and absorption corrections were applied. No gross extinction was observed for any strong low angle reflections at the end of the structure refinement, and a total of 2191 independent reflections was finally used.

The structure was solved using conventional Patterson and Fourier techniques and refined by block-diagonal least-squares methods. Non-hydrogen atoms of the molecular anions gave satisfactory refinement with anisotropic thermal parameters, and the remaining electron density maxima of Fourier syntheses were assigned as oxygen atoms of water molecules or sodium cations. Allowance was made for the anomalous dispersion of iron in the later stages of refinement.

The four sites in the asymmetric unit chosen for the sodium ions (on chemical grounds through co-ordination with oxygen atoms) are all in general positions, and form a disordered set with linked populations; the mean $Na^+$ population per asymmetric unit is 1.5 to maintain the charge balance with 1.5 molecular anions. Occupancies in these sites were initially refined independently with a common isotropic thermal parameter and when the shifts in these parameters were less than half of their e.s.d. s



α-form



β-form

Fig. 1. The Infrared Spectra of the α- and β- Forms of $[Fe(ehpg)]^-$.

(at a total population of 1.56), the occupancies were rescaled to total 1.5 and refinement continued with final populations and overall isotropic thermal parameter. Na(1) and Na(2) (separation 1.13(4) Å) are too close not to be mutually exclusive in occupation, and the present model may be an approximation to a non-harmonic, labile situation.

Four electron density maxima assigned to water molecules OW(1–4) appeared to be in fully occupied sites, and anisotropic thermal parameters refined satisfactorily for these. Three solvent-occupied sites remained; independent refinement of their populations gave occupancies of 0.5, 0.25 and 0.20 (within half the e.s.d.) respectively for OW(5)–OW(7), and these populations were fixed. Anisotropic thermal parameters were refined for OW(5). OW(6) is very close to the centre of symmetry at (O, ½, O) with OW(6)···OW(6′) = 1.2(1) Å and these two sites probably represent a vibrationally labile site around this centre of symmetry with an occupancy close to 0.5. OW(6) and OW(7) were finally refined with fixed populations of 0.25 and 0.2 and a common isotropic thermal parameter. The total solvent in the lattice, as represented by OW(1)–OW(7), gives a total of 3.3 $H_2O$ per Fe, compared to 4 $H_2O$ from good, internally consistent microanalysis. The remaining water may be sufficiently mobile in the crystal to prevent its reliable observation by diffraction methods.

Hydrogen atoms of the molecular anions were located close to calculated positions (C–H = 0.95, N–H = 0.87 Å) in a difference Fourier synthesis at a late stage of refinement. These were included, with isotropic thermal parameters 6.0 Å$^2$, but not varied in the final refinement to $R$ 0.067, when parameter shifts were all less than one-sixth of their e.s.d.s.

### *(b) Magnesium Salt*

The compound crystallised from methanol as dark red irregular polyhedra. The one used for the structure analysis had minimum and maximum transmission coefficients, respectively, 0.70 and 0.92.

### *Crystal Data*

$[C_{18}H_{16}N_2O_6Fe]_2Mg\cdot 9H_2O$; $M$ = 1010.8. Monoclinic, $a$ = 17.074(15), $b$ = 13.099(1), $c$ = 20.868(11) Å, $\beta$ = 113.57(5)°, $U$ = 4277(5) Å$^3$. Space group $I2/c$*, $D_m$ (flotation) = 1.54 g cm$^{-3}$, $z$ = 4, $D_c$ = 1.57 g cm$^{-3}$, Mo$K_\alpha$ radiation, $\lambda$ = 0.71069 Å, $\mu$(Mo-$K_\alpha$) = 7.75 cm$^{-1}$. $F(000)$ = 2120.

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*A non-standard setting of space group $C2/c$ ($C^6_{2h}$, No. 15); matrix transformation to $I2/c$ $\begin{bmatrix} 1 & 0 & 1 \\ 0 & 1 & 0 \\ -1 & 0 & 1 \end{bmatrix}$ for $C2/c$ unit cell $a$ = 31.811, $b$ = 13.099, $c$ = 20.868 Å, $\beta$ = 150.53°.

TABLE II. Atomic Positions for $Na[Fe(ehpg)]\cdot 4H_2O$, with Estimated Standard Deviations in Parentheses.

| Atom | x/a | y/b | z/c |
|---|---|---|---|
| *Atomic positions ($\times 10^4$) of the non-hydrogen atoms in $[Fe($meso-*ehpg*$)]^-$* | | | |
| Fe(1) | 1540(1) | 1755(2) | 1109(1) |
| N(1) | 1398(2) | 450(12) | 1875(3) |
| N(2) | 1804(2) | –892(12) | 1032(3) |
| O(1) | 1916(2) | 3007(11) | 1495(3) |
| O(2) | 1700(2) | 2425(10) | 401(3) |
| O(3) | 1162(2) | 3497(11) | 1393(3) |
| O(4) | 1148(2) | 71(11) | 756(3) |
| O(5) | 914(2) | 4583(11) | 2157(3) |
| O(6) | 1086(2) | –2336(11) | 196(3) |
| C(1) | 1115(3) | 3526(16) | 1908(5) |
| C(2) | 1337(3) | 2106(16) | 2242(4) |
| C(3) | 1685(3) | 3074(15) | 2410(4) |
| C(4) | 1731(4) | 3639(18) | 2947(5) |
| C(5) | 2033(4) | 4585(17) | 3109(5) |
| C(6) | 2295(3) | 4961(18) | 2732(5) |
| C(7) | 2255(3) | 4438(15) | 2201(5) |
| C(8) | 1946(3) | 3479(15) | 2030(4) |
| C(9) | 1668(3) | –949(16) | 2021(5) |
| C(10) | 1719(3) | –2159(16) | 1505(5) |
| C(11) | 1676(3) | –1667(15) | 506(5) |
| C(12) | 1840(3) | –634(14) | 28(5) |
| C(13) | 1990(3) | –1617(18) | –400(5) |
| C(14) | 2130(3) | –770(19) | –851(5) |
| C(15) | 2118(3) | 1149(19) | –889(5) |
| C(16) | 1980(3) | 2198(17) | –465(4) |
| C(17) | 1836(3) | 1328(16) | –3(4) |
| C(18) | 1271(3) | –1272(16) | 492(5) |
| *Hydrogen atom positions ($\times 10^3$)$^a$ in $[Fe($meso-*ehpg*$)]^-$* | | | |
| H(1″) | 120 | –24 | 188 |
| H(2″) | 204 | –75 | 104 |
| H(2) | 122 | 168 | 257 |
| H(4) | 155 | 335 | 322 |
| H(5) | 206 | 500 | 348 |
| H(6) | 251 | 559 | 285 |
| H(7) | 244 | 471 | 193 |
| H(9) | 189 | –33 | 212 |
| H(9′) | 160 | –169 | 233 |
| H(10) | 150 | –282 | 143 |
| H(10′) | 190 | –307 | 156 |
| H(11) | 173 | –300 | 49 |
| H(13) | 200 | –300 | –37 |
| H(14) | 223 | –151 | –115 |
| H(15) | 221 | 177 | –121 |
| H(16) | 198 | 358 | –49 |
| *Atomic positions ($\times 10^4$) of the non-hydrogen atoms in $[Fe(R,R\text{-}ehpg)]^-$* | | | |
| Fe(2) | 0 | 1101(3) | 2500 |
| N(3) | 302(2) | 3405(13) | 2828(4) |
| O(7) | 385(2) | –486(10) | 2702(3) |
| O(8) | –196(2) | 1298(12) | 3271(3) |
| O(9) | –31(3) | 1884(18) | 4125(4) |
| C(19) | 27(4) | 1961(20) | 3617(5) |
| C(20) | 370(3) | 2833(18) | 3392(5) |
| C(21) | 651(3) | 1321(18) | 3418(5) |
| C(22) | 921(3) | 1484(20) | 3806(5) |
| C(23) | 1176(4) | 141(23) | 3861(5) |
| C(24) | 1176(3) | –1401(21) | 3514(6) |
| C(25) | 905(3) | –1590(19) | 3133(5) |
| C(26) | 641(3) | –218(17) | 3085(4) |
| C(27) | 83(3) | 5138(16) | 2784(5) |
| *Hydrogen atom positions ($\times 10^3$)$^a$ in $[Fe(R,R\text{-}ehpg)]^-$* | | | |
| H(3″) | 50 | 367 | 265 |
| H(20) | 44 | 394 | 360 |
| H(22) | 93 | 260 | 404 |
| H(23) | 136 | 28 | 414 |
| H(24) | 136 | –237 | 354 |
| H(25) | 90 | –270 | 289 |
| H(27) | –10 | 512 | 307 |
| H(27′) | 23 | 624 | 285 |
| *Sodium ion and water molecule positions ($\times 10^4$) in $Na[Fe(ehpg)]\cdot 4H_2O$* | | | |
| Na(1) | 501 | –623 | 928 |
| Na(2) | 588 | 850 | 823 |
| Na(3) | 567 | 4891 | 741 |
| Na(4) | –55 | 7254 | 886 |
| OW(1) | 2567 | –631 | 780 |
| OW(2) | 719 | –1882 | 1740 |
| OW(3) | 441 | –497 | –26 |
| OW(4) | 1107 | 4162 | –214 |
| OW(5) | 541 | 2876 | –453 |
| OW(6) | 76 | 5074 | 207 |
| OW(7) | 216 | 2879 | 32 |

[a] Hydrogen atoms are numbered according to the carbon or nitrogen to which they are attached with primes used to distinguish the pairs on the same carbon and double primes to indicate a nitrogen bonded hydrogen.

The crystals were previously reported as being triclinic [4]**. Layer $\{h', k', 0 \rightarrow 13\}$ with $6.5° < 2\theta < 50°$ (in the triclinic cell) were collected on the Stoe Stadi-2 automatic diffractometer as for (A). Reflections with intensity $I < 3\sigma_I$ were rejected as were those with background difference $\Delta > 4\sigma_B$. Lorentz and polarisation corrections were applied, but no allowance was made for absorption, nor was any significant extinction of strong low-angle reflections observed during structure refinement. The triclinic data-set was transformed to the data-set in $I2/c$ {which has a less obtuse β-angle than that

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**For footnote, please see facing page.

TABLE III. Atomic Positions for Mg[Fe(ehpg)]·$9H_2O$ with Estimated Standard Deviations in Parentheses.

| Atom | x/a | y/b | z/c |
|---|---|---|---|
| *Atomic positions ($\times 10^4$) of the non-hydrogen atoms* | | | |
| Fe | –1418(0) | –923(0) | –1123(0) |
| N(1) | –1321(2) | –526(2) | –91(1) |
| N(2) | –1870(2) | 646(2) | –1302(1) |
| O(1) | –754(2) | –2105(2) | –721(1) |
| O(2) | –1815(2) | –1102(2) | –2115(2) |
| O(3) | –2549(1) | –1455(2) | –1129(1) |
| O(4) | –359(1) | –124(2) | –1040(1) |
| O(5) | –3154(1) | –2028(2) | –430(1) |
| O(6) | 5(2) | 1020(2) | –1647(2) |
| C(1) | –2557(2) | –1649(3) | –531(2) |
| C(2) | –1714(2) | –1413(3) | 103(2) |
| C(3) | –1156(2) | –2353(3) | 252(2) |
| C(4) | –1116(2) | –2988(3) | 798(2) |
| C(5) | –620(3) | –3859(3) | 967(2) |
| C(6) | –124(3) | –4080(3) | 602(2) |
| C(7) | –153(3) | –3472(3) | 54(2) |
| C(8) | –695(2) | –2621(3) | –153(2) |
| C(9) | –1770(2) | 446(3) | –114(2) |
| C(10) | –1597(2) | 1165(3) | –611(2) |
| C(11) | –1456(2) | 1033(3) | –1759(2) |
| C(12) | –1951(2) | 649(3) | –2495(2) |
| C(13) | –2276(2) | 1340(3) | –3049(2) |
| C(14) | –2771(2) | 1007(3) | –3725(2) |
| C(15) | –2932(2) | –25(4) | –3849(2) |
| C(16) | –2623(2) | –720(3) | –3311(2) |
| C(17) | –2119(2) | –394(3) | –2624(2) |
| C(18) | –525(2) | 634(3) | –1462(2) |
| *Hydrogen atom postions ($\times 10^3$)*[a] | | | |
| H(1″) | –77 | –43 | 23 |
| H(2″) | –239 | 69 | 150 |
| H(2) | –181 | –123 | 55 |
| H(4) | –141 | –282 | 104 |
| H(5) | –66 | –434 | 135 |
| H(6) | 21 | –472 | 69 |
| H(7) | 16 | –364 | –23 |
| H(9) | –240 | 29 | –30 |
| H(9′) | –160 | 72 | 32 |
| H(10) | –98 | 129 | –46 |
| H(10′) | –188 | 183 | –69 |
| H(11) | –144 | 183 | –177 |
| H(13) | –272 | –150 | –340 |
| H(14) | –324 | –29 | –430 |
| H(15) | –300 | 152 | –411 |
| H(16) | –218 | 209 | –298 |
| *Magnesium ion and water molecule positions ($\times 10^4$)* | | | |
| Mg | 5000 | 2369(1) | –7500 |
| OW(1) | 5000 | 767(3) | –7500 |
| OW(2) | 5000 | 4011(3) | –7500 |
| OW(3) | 5324(2) | 2495(3) | –6437(1) |
| OW(4) | 3740(1) | 2264(2) | –7644(1) |
| OW(5) | 0 | 2612(3) | –2500 |
| OW(6) | 3802(2) | 857(3) | –2617(2) |

[a] Hydrogen atoms are numbered according to the carbon or nitrogen to which they are attached, with primes used to distinguish the pairs on the same carbon and double primes to indicate a nitrogen bonded hydrogen.

for the standard setting} and symmetry equivalent reflections averaged accordingly. A total of 3124 independent reflections was used in the final refinement.

The structure was solved by conventional Patterson and Fourier techniques and refined by block-diagonal least-squares methods. Positional and anisotropic thermal parameters were refined for all non-hydrogen atoms, and positional parameters only for the hydrogen atoms of the molecular anion with isotropic thermal parameters fixed at 4.0 $Å^2$. Hydrogen atoms of water molecules, both coordinated with $Mg^{2+}$ and hydrogen-bonded in the lattice, could not all be located, and since their positions are not satisfactorily predictable (even for the $Mg(H_2O)_6^{2+}$ ion [5]) they were not included in the final refinement. Allowance was made for anomalous dispersion of iron in the final refinement to $R$ 0.038.

Tables II and III list the atomic positional parameters with estimated standard deviations for the sodium and magnesium salts respectively. Tables of anisotropic thermal vibrational parameters, bond-angles within the hydrogen bonding network, and of observed structure amplitudes and calculated structure factors are available from the Editor. Atomic scattering factors were taken from reference 6; unit weights were used throughout both least-squares refinements.

## Results and Discussion

Figure 2 shows the crystal packing of the sodium salt, (A), viewed down the *b*-axis. The asymmetric unit contains one [Fe(*meso*-ehpg)]$^-$ ion in a general

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**Cell constants were originally measured in space group $I\bar{1}$ a non-standard setting of $P\bar{1}$ ($C_i^1$, No. 2), for $a$ = 17.201(12), $b$ = 21.517(15), $c$ = 12.388(9) A, $\alpha$ = 92.434(5), $\beta$ = 91.865(5), $\gamma$ = 110.768(4)°. Matrix transformation to $C2/c \begin{bmatrix} 1.5 & 0.5 & 0.5 \\ 0.5 & 0.5 & -0.5 \\ -1 & 0 & -1 \end{bmatrix}$ and to $I2/c \begin{bmatrix} 0.5 & -0.5 & -0.5 \\ 0.5 & 0.5 & -0.5 \\ -1 & 0 & -1 \end{bmatrix}$. Data were collected for the triclinic cell, and were later reindexed to the correct monoclinic cell, which had initially been overlooked because of non-standard crystal mounting.

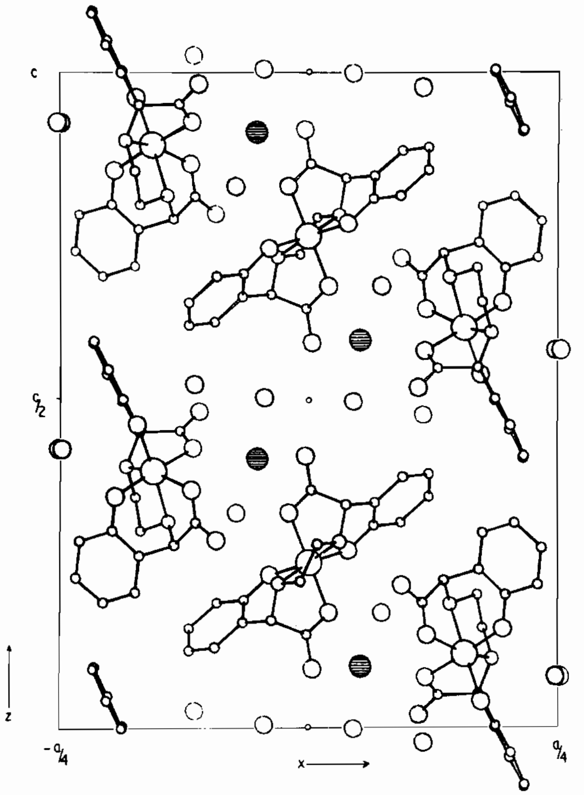

Fig. 2. The crystal packing in Na[Fe(ehpg)]·4$H_2O$ (structure A), showing half the unit cell viewed down the *b* axis. The empty circles represent Fe, O, N and C atoms in descending order of size. The shaded circles represent three sites occupied by $Na^+$, all of which have closely similar x and z co-ordinates. Only the major sites occupied by waters of crystallization are shown.

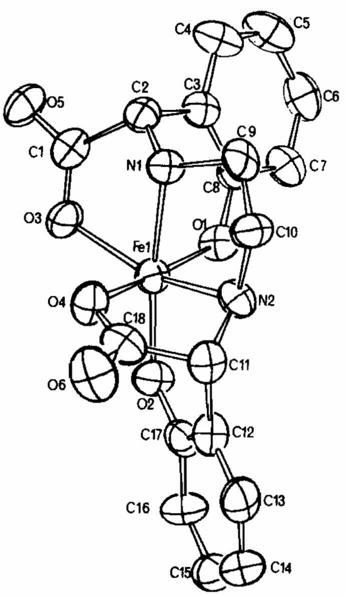

Fig. 3. The molecular structure and atom-labelling scheme of the [Fe(*meso*-ehpg)]$^-$ ion in Na[Fe(ehpg)]·4$H_2O$ (Structure A).

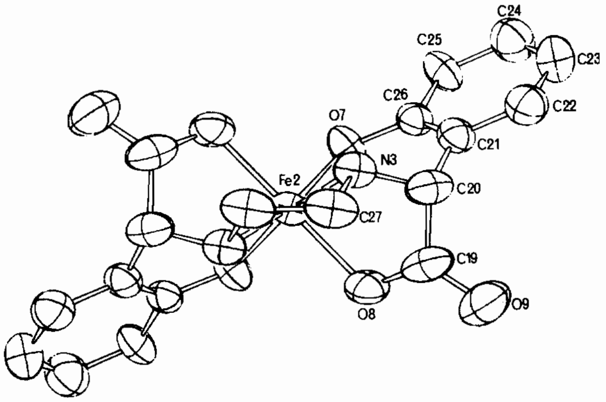

Fig. 4. The molecular structure and atom-labelling scheme of the [Fe(*R,R*-ehpg)]$^-$ ion in Na[Fe(ehpg)]·4$H_2O$ (Structure A) viewed along the crystallographic 2-fold axis.

position, with the all-*cis* configuration of the ligand, and also a [Fe(*rac*-ehpg)]$^-$ ion with *trans* carboxylate groups lying across a crystallographic 2-fold axis. The molecular structures of these two ions, together with their atom-labelling schemes, are shown in Figs. 3 and 4 respectively. Details of the various bond-lengths and -angles are given in Table IV. Mean bond-lengths within the co-ordination polyhedron are Fe–N 2.149(2), Fe–O (phenolic) 1.906(15) and Fe–O (carboxylate) 2.042(40) Å, with only minor differences between the bond-lengths of the same type. In each case, the coordination polyhedron is distorted octahedral, the mean bond-angles are N–$\widehat{\mathrm{Fe}}$–N′ 79.9(1.0), N–$\widehat{\mathrm{Fe}}$–O (phenolic) 88.1(1.3), and N–$\widehat{\mathrm{Fe}}$–O (carboxylate) 77.9(0.2)° [where ′ refers to the equivalent atom in the second half ligand molecule] and, clearly, is the result of ligand steric restraints. Hydrogen bonding distances are given in Table VI and equations of relevant least-squares planes in Table VII.

The crystal lattice contains distinct hydrophilic channels, bounded by the carboxylate fragments at, for example, (0, y, 0.5), which contain disordered water molecules and $Na^+$ ions (three sites): the former in positions suitable for hydrogen-bonding and the latter in sites allowing co-ordination by oxygen atoms. The remainder of the structure comprises an extended, essentially hydrophobic region bounded by phenyl groups, but containing localised small hydrophilic pockets occupied by full-population water molecules, which are hydrogen-bonded to NH groups of the [Fe(*meso*-ehpg)]$^-$ ion.

Figure 5 shows the crystal packing of the magnesium salt, (B), viewed down the *b*-axis. This structure contains only the [Fe(*rac*-ehpg)]$^-$ ion, in general

TABLE IV. Bond Lengths (Å) and Angles (°) in the Molecular Anions. Corresponding Parameters are on the Same Line for the two Halves of the Pseudosymmetric [Fe(*R*,*R*-ehpg)]$^-$ in the Mg Salt, and the Symmetric [Fe(*R*,*R*-ehpg)]$^-$ and the Two Distinct Halves of the [Fe(*meso*-ehpg)]$^-$ in the Na Salt.

| Mg salt | | | | Na salt | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | [Fe(*meso*-ehpg)]$^-$ | | | | |
| Fe–O(1) | 1.904(3) | Fe–O(2) | 1.918(3) | Fe(2)–O(7) | 1.904(8) | Fe(1)–O(2) | 1.893(7) | Fe(1)–O(1) | 1.922(7) | | |
| Fe–N(1) | 2.157(3) | Fe–N(2) | 2.175(3) | Fe(2)–N(3) | 2.151(9) | Fe(1)–N(2) | 2.140(9) | Fe(1)–N(1) | 2.157(8) | | |
| Fe–O(3) | 2.047(2) | Fe–O(4) | 2.037(3) | Fe(2)–O(8) | 2.029(8) | Fe(1)–O(4) | 2.087(8) | Fe(1)–O(3) | 2.011(8) | | |
| O(3)–C(1) | 1.280(4) | O(4)–C(18) | 1.281(5) | O(8)–C(19) | 1.28(2) | O(4)–C(18) | 1.24(1) | O(3)–C(1) | 1.27(1) | | |
| O(5)–C(1) | 1.225(5) | O(6)–C(18) | 1.226(5) | O(9)–C(19) | 1.26(2) | O(6)–C(18) | 1.26(1) | O(5)–C(1) | 1.23(1) | | |
| C(1)–C(2) | 1.547(5) | C(18)–C(11) | 1.548(5) | C(19)–C(20) | 1.54(2) | C(18)–C(11) | 1.55(2) | C(1)–C(2) | 1.55(2) | | |
| C(2)–N(1) | 1.476(5) | C(11)–N(2) | 1.485(5) | C(20)–N(3) | 1.46(2) | C(11)–N(2) | 1.48(1) | C(2)–N(1) | 1.50(1) | | |
| C(2)–C(3) | 1.511(5) | C(11)–C(12) | 1.512(5) | C(20)–C(21) | 1.51(2) | C(11)–C(12) | 1.51(2) | C(2)–C(3) | 1.54(2) | | |
| C(3)–C(8) | 1.410(5) | C(12)–C(17) | 1.401(5) | C(21)–C(26) | 1.37(2) | C(12)–C(17) | 1.40(2) | C(3)–C(8) | 1.38(1) | | |
| C(3)–C(4) | 1.389(5) | C(12)–C(13) | 1.397(5) | C(21)–C(22) | 1.40(2) | C(12)–C(13) | 1.38(2) | C(3)–C(4) | 1.38(2) | | |
| C(4)–C(5) | 1.380(6) | C(13)–C(14) | 1.393(6) | C(22)–C(23) | 1.36(2) | C(13)–C(14) | 1.36(2) | C(4)–C(5) | 1.38(2) | | |
| C(5)–C(6) | 1.378(6) | C(14)–C(15) | 1.383(6) | C(23)–C(24) | 1.39(2) | C(14)–C(15) | 1.37(2) | C(5)–C(6) | 1.37(2) | | |
| C(6)–C(7) | 1.378(6) | C(15)–C(16) | 1.376(6) | C(24)–C(25) | 1.39(2) | C(15)–C(16) | 1.38(2) | C(6)–C(7) | 1.36(2) | | |
| C(7)–C(8) | 1.403(6) | C(16)–C(17) | 1.410(5) | C(25)–C(26) | 1.40(2) | C(16)–C(17) | 1.40(2) | C(7)–C(8) | 1.41(2) | | |
| C(8)–O(1) | 1.334(4) | C(17)–O(2) | 1.348(4) | C(26)–O(7) | 1.36(1) | C(17)–O(2) | 1.36(1) | C(8)–O(1) | 1.35(1) | | |
| N(1)–C(9) | 1.478(5) | N(2)–C(10) | 1.491(5) | N(3)–C(27) | 1.49(1) | N(2)–C(10) | 1.50(1) | N(1)–C(9) | 1.47(1) | | |
| C(9)–C(10) | 1.515(6) | | | C(27)–C(27′) | 1.53(2) | C(10)–C(9) | 1.54(2) | | | | |
| O(3)–C(1)–O(5) | 125.7(3) | O(4)–C(18)–O(6) | 123.1(4) | O(8)–C(19)–O(9) | 121(1) | O(4)–C(18)–O(6) | 124(1) | O(3)–C(1)–O(5) | 126(1) | | |
| O(3)–C(1)–C(2) | 115.1(3) | O(4)–C(18)–C(11) | 116.5(3) | O(8)–C(19)–C(20) | 118(1) | O(4)–C(18)–C(11) | 119(1) | O(3)–C(1)–C(2) | 115.9(9) | | |
| O(5)–C(1)–C(2) | 119.1(3) | O(6)–C(18)–C(11) | 120.3(3) | O(9)–C(19)–C(20) | 121(1) | O(6)–C(18)–C(11) | 117(1) | O(5)–C(1)–C(2) | 118(1) | | |
| C(1)–C(2)–C(3) | 107.9(3) | C(18)–C(11)–C(12) | 111.2(3) | C(19)–C(20)–C(21) | 107(1) | C(18)–C(11)–C(12) | 107.2(9) | C(1)–C(2)–C(3) | 108.0(9) | | |
| C(1)–C(2)–N(1) | 107.0(3) | C(18)–C(11)–N(2) | 107.7(3) | C(19)–C(20)–N(3) | 108(1) | C(18)–C(11)–N(2) | 105.9(9) | C(1)–C(2)–N(1) | 106.3(8) | | |
| C(3)–C(2)–N(1) | 112.3(3) | C(12)–C(11)–N(2) | 108.8(3) | C(21)–C(20)–N(3) | 111(1) | C(12)–C(11)–N(2) | 110.9(9) | C(3)–C(2)–N(1) | 112.6(8) | | |
| C(2)–N(1)–C(9) | 113.6(3) | C(11)–N(2)–C(10) | 114.9(3) | C(20)–N(3)–C(27) | 113.5(9) | C(11)–N(2)–C(10) | 112.0(8) | C(2)–N(1)–C(9) | 119.6(8) | | |
| C(2)–C(3)–C(8) | 122.4(3) | C(11)–C(12)–C(17) | 120.6(3) | C(20)–C(21)–C(26) | 122(1) | C(11)–C(12)–C(17) | 122(1) | C(2)–C(3)–C(8) | 121.4(9) | | |
| C(2)–C(3)–C(4) | 118.6(3) | C(11)–C(12)–C(13) | 120.1(3) | C(20)–C(21)–C(22) | 119(1) | C(11)–C(12)–C(13) | 120(1) | C(2)–C(3)–C(4) | 119(1) | | |
| C(8)–C(3)–C(4) | 119.0(3) | C(13)–C(12)–C(17) | 119.3(3) | C(26)–C(21)–C(22) | 119(1) | C(17)–C(12)–C(13) | 118(1) | C(8)–C(3)–C(4) | 119(1) | | |
| C(3)–C(4)–C(5) | 122.0(4) | C(12)–C(13)–C(14) | 121.0(4) | C(21)–C(22)–C(23) | 122(1) | C(12)–C(13)–C(14) | 123(1) | C(3)–C(4)–C(5) | 121(1) | | |
| C(4)–C(5)–C(6) | 118.8(4) | C(13)–C(14)–C(15) | 119.4(4) | C(22)–C(23)–C(24) | 120(1) | C(13)–C(14)–C(15) | 119(1) | C(4)–C(5)–C(6) | 120(1) | | |
| C(5)–C(6)–C(7) | 120.7(4) | C(14)–C(15)–C(16) | 120.7(4) | C(23)–C(24)–C(25) | 119(1) | C(14)–C(15)–C(16) | 120(1) | C(5)–C(6)–C(7) | 120(1) | | |
| C(6)–C(7)–C(8) | 120.0(4) | C(15)–C(16)–C(17) | 120.5(4) | C(24)–C(25)–C(26) | 121(1) | C(15)–C(16)–C(17) | 121(1) | C(6)–C(7)–C(8) | 120(1) | | |
| C(7)–C(8)–C(3) | 118.1(3) | C(16)–C(17)–C(12) | 119.1(3) | C(25)–C(26)–C(21) | 119(1) | C(16)–C(17)–C(12) | 119(1) | C(7)–C(8)–C(3) | 119(1) | | |
| C(7)–C(8)–O(1) | 118.8(3) | C(16)–C(17)–O(2) | 118.6(3) | C(25)–C(26)–O(7) | 118(1) | C(16)–C(17)–O(2) | 119(1) | C(7)–C(8)–O(1) | 118.4(9) | | |
| C(3)–C(8)–O(1) | 123.1(3) | C(12)–C(17)–O(2) | 122.3(3) | C(21)–C(26)–O(7) | 123(1) | C(12)–C(17)–O(2) | 123(1) | C(3)–C(8)–O(1) | 122.4(9) | | |
| N(1)–C(9)–C(10) | 108.5(3) | N(2)–C(10)–C(9) | 107.9(3) | N(3)–C(27)–C(27′) | 107.1(9) | N(2)–C(10)–C(9) | 108.7(9) | N(1)–C(9)–C(10) | 105.5(9) | | |

*(continued overleaf)*

TABLE IV. *(continued)*

| Mg salt | | | | Na salt | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | [Fe(*meso*-ehpg)]$^-$ | | | |
| O(1)–Fe–O(3) | 96.9(1) | O(2)–Fe–O(4) | 94.1(1) | O(7)–Fe(2)–O(8) | 94.4(3) | O(2)–Fe(1)–O(4) | 89.5(3) | O(1)–Fe(1)–O(3) | 93.8(3) |
| O(1)–Fe–O(2) | 105.5(1) | | | O(7)–Fe(2)–O(7′) | 107.2(3) | O(2)–Fe(1)–O(1) | 95.5(3) | | |
| O(1)–Fe–N(1) | 87.8(1) | O(2)–Fe–N(2) | 88.9(1) | O(7)–Fe(2)–N(3) | 87.3(3) | O(2)–Fe(1)–N(2) | 89.6(3) | O(1)–Fe(1)–N(1) | 87.5(3) |
| O(1)–Fe–N(2) | 162.2(1) | O(2)–Fe–N(1) | 163.8(1) | O(7)–Fe(2)–N(3′) | 162.1(3) | O(2)–Fe(1)–N(1) | 168.7(3) | O(1)–Fe(1)–N(2) | 96.2(3) |
| O(1)–Fe–O(4) | 91.6(1) | O(3)–Fe–O(2) | 91.5(1) | O(7)–Fe(2)–O(8′) | 90.3(3) | O(2)–Fe(1)–O(3) | 112.6(3) | O(4)–Fe(1)–O(1) | 172.1(3) |
| O(3)–Fe–N(1) | 77.5(1) | O(4)–Fe–N(2) | 76.8(1) | O(8)–Fe(2)–N(3) | 78.0(3) | O(4)–Fe(1)–N(2) | 77.7(3) | O(3)–Fe(1)–N(1) | 78.0(3) |
| O(3)–Fe–N(2) | 93.0(1) | O(4)–Fe–N(1) | 94.6(1) | O(8)–Fe(2)–N(3′) | 95.9(3) | O(4)–Fe(1)–N(1) | 86.3(3) | O(3)–Fe(1)–N(2) | 154.6(3) |
| O(3)–Fe–O(4) | 168.2(1) | | | O(8)–Fe(2)–O(8′) | 172.1(3) | O(4)–Fe(1)–O(3) | 89.8(3) | | |
| N(1)–Fe–N(2) | 79.9(1) | | | N(3)–Fe(2)–N(3′) | 80.6(3) | N(2)–Fe(1)–N(1) | 79.2(3) | | |
| Fe–O(1)–C(8) | 128.5(2) | Fe–O(2)–C(17) | 128.7(2) | Fe(2)–O(7)–C(26) | 129.5(7) | Fe(1)–O(2)–C(17) | 129.7(7) | Fe(1)–O(1)–C(8) | 130.6(6) |
| Fe–O(3)–C(1) | 116.1(2) | Fe–O(4)–C(18) | 113.3(2) | Fe(2)–O(8)–C(19) | 113.6(7) | Fe(1)–O(4)–C(18) | 113.3(7) | Fe(1)–O(3)–C(1) | 116.7(7) |
| Fe–N(1)–C(2) | 102.8(2) | Fe–N(2)–C(11) | 101.9(2) | Fe(2)–N(3)–C(20) | 103.5(7) | Fe(1)–N(2)–C(11) | 104.7(6) | Fe(1)N(1)–C(2) | 102.6(6) |
| Fe–N(1)–C(9) | 110.1(2) | Fe–N(2)–C(10) | 108.2(2) | Fe(2)–N(3)–C(27) | 108.3(7) | Fe(1)–N(2)–C(10) | 111.3(6) | Fe(1)–N(1)–C(9) | 109.4(6) |

TABLE V. Bond-Lengths (Å) and -Angles (°) in the Hexaaquamagnesium(II) Ion of $Mg[Fe(ehpg)]_2 \cdot 9H_2O$

| | |
|---|---|
| Mg–OW(1) | 2.098(3) |
| Mg–OW(2) | 2.150(3) |
| Mg–OW(3) | 2.068(3) |
| Mg–OW(4) | 2.054(3) |
| OW(1)–Mg–OW(2) | 180.0(1) |
| OW(1)–Mg–OW(3) | 94.6(1) |
| OW(1)–Mg–OW(4) | 86.2(1) |
| OW(2)–Mg–OW(3) | 85.4(1) |
| OW(2)–Mg–OW(4) | 93.8(1) |
| OW(3)–Mg–OW(3′)[a] | 170.9(1) |
| OW(3)–Mg–OW(4) | 88.7(1) |
| OW(3)–Mg–OW(4′)[a] | 91.9(1) |
| OW(4)–Mg–OW(4′)[a] | 172.3(1) |

[a] Prime indicates the appropriate $C_2$ related water molecule.

TABLE VI. Hydrogen-Bonding Distances (Å) with Estimated Standard Deviations in Parentheses.

| *Na[Fe(ehpg)]·4H$_2$O* | |
|---|---|
| N(1)–OW(2) | 3.065(13) |
| N(2)–OW(1) | 2.943(15) |
| N(3)–O(5) | 2.952(12) |
| O(1)–OW(1) | 2.792(14) |
| O(2)–OW(4) | 2.960(21) |
| O(4)–Na(1) | 2.523(14) |
| O(4)–Na(2) | 2.186(43) |
| O(5)–OW(2) | 2.812(12) |
| O(6)–OW(3) | 2.811(21) |
| O(6)–Na(3) | 3.080(28) |
| O(6)–OW(4) | 2.689(21) |
| O(7)–OW(2) | 2.845(12) |
| O(8)–Na(1) | 2.649(15) |
| O(8)–Na(2) | 2.681(43) |
| O(8)–OW(2) | 3.002(13) |
| O(9)–OW(6) | 2.803(81) |
| O(9)–OW(7) | 2.888(93) |
| O(9)–Na(1) | 2.516(16) |
| O(9)–Na(2) | 2.227(44) |
| Na(1)–OW(2) | 2.328(16) |
| Na(1)–OW(3) | 2.345(23) |
| Na(2)–OW(3) | 2.351(47) |
| Na(3)–OW(6) | 2.268(85) |
| Na(3)–OW(7) | 2.608(96) |
| Na(3)–OW(4) | 3.139(33) |
| Na(4)–OW(6) | 2.325(90) |
| Na(4)–OW(7) | 2.327(100) |
| Na(4)–OW(5) | 2.117(63) |
| OW(3)–OW(7) | 2.553(94) |
| OW(3)–OW(5) | 2.645(53) |
| OW(4)–OW(5) | 2.394(53) |
| OW(5)–OW(6) | 2.848(94) |

*(continued on facing page)*

TABLE VI. *(continued)*

| *Mg[Fe(ehpg)]$_2$·9$H_2O$* | |
|---|---|
| O(1)–OW(3) | 2.844(4) |
| O(2)–OW(4) | 2.745(4) |
| O(3)–OW(4) | 2.767(4) |
| O(5)–OW(3) | 2.679(4) |
| O(6)–OW(5) | 2.741(4) |
| N(2)–OW(6) | 3.177(4) |
| OW(1)–OW(6) | 2.894(4) |
| OW(5)–OW(6) | 2.805(4) |

TABLE VII. Equations to Some Least Squares Planes Given in the Form lX + mY + nZ = d (where, X, Y, Z are Co-ordinates in Å Referred to the Axes a, b and c). Deviations (Å) of the Various Atoms from these Planes are Given in Square Brackets.

| | l | m | n | d |
|---|---|---|---|---|
| *Na[Fe(ehpg)]·4$H_2O$* | | | | |
| Plane (1): Fe(1), N(2), O(4) | −0.3207 | −0.2606 | 0.9106 | 0.2837 |
| [C(11) −0.872, C(18) −0.488, O(6) −0.725] | | | | |
| Plane (2): Fe(1), N(1), O(3) | 0.6990 | 0.5408 | 0.4679 | 5.9969 |
| [C(1) 0.477, C(2) 0.898, O(5) 0.639] | | | | |
| Plane (3): Fe(1), N(1), N(2) | −0.8166 | −0.3892 | −0.4262 | −6.3764 |
| [C(9) −0.596, C(10) 0.121, C(2) −0.655, C(11) 1.157] | | | | |
| Plane (4): C(2)–C(8), O(1) | 0.4329 | −0.8789 | 0.2005 | 1.9797 |
| [N(1) 0.936, Fe(1) −0.025] | | | | |
| Plane (5): C(11)–C(17), O(2) | −0.8916 | −0.0289 | −0.4519 | −6.1842 |
| [N(2) −0.995, Fe(1) −0.246] | | | | |
| Plane (6): C(1), C(2), O(3), O(5) | 0.7334 | 0.6742 | 0.0877 | 5.1838 |
| [N(1) −0.706, Fe(1) 0.148] | | | | |
| Plane (7): C(11), C(18), O(4), O(6) | −0.1367 | −0.6069 | 0.7830 | 0.8285 |
| [N(2) 0.603, Fe(1) −0.257] | | | | |
| Plane (8): Fe(2), O(8), N(3) | 0.6981 | −0.6402 | 0.3207 | 1.4579 |
| [C(19) 0.553, C(20) 0.883, O(9) 0.835] | | | | |
| Plane (9): Fe(2), N(3), N(3′) | −0.5771 | 0.0000 | 0.8167 | 4.9899 |
| [C(27) 0.387] | | | | |
| Plane (10): C(20)–C(26), O(7) | −0.5587 | −0.4903 | 0.6690 | 3.7710 |
| [N(3) −0.970, Fe(2) −0.068] | | | | |
| *Mg[Fe(ehpg)]$_2$·9$H_2O$* | | | | |
| Plane (1) Fe, N(2), O(4) | −0.1584 | 0.1159 | 0.9806 | −2.0105 |
| [C(11) −0.970, C(18) −0.687, O(6) −1.142] | | | | |
| Plane (2): Fe, N(1), O(3) | −0.3174 | 0.8835 | −0.3445 | 0.1427 |
| [C(1) −0.457, C(2) −0.889, O(5) −0.612] | | | | |
| Plane (3): Fe, N(1), N(2) | −0.9175 | −0.3175 | −0.2396 | 2.2606 |
| [C(9) 0.292, C(10) −0.431, C(2) 1.044, C(11) −0.950] | | | | |
| Plane (4): C(2)–C(8), O(1) | 0.5748 | 0.5492 | 0.6066 | −2.6733 |
| [N(1) 0.936, Fe −0.147] | | | | |
| Plane (5): C(11)–C(17), O(2) | −0.9821 | 0.0892 | 0.1660 | 0.5286 |
| [N(2) 1.202, Fe 0.465] | | | | |
| Plane (6): C(1), C(2), O(3), O(5) | −0.3903 | 0.9196 | 0.0446 | −0.5136 |
| [N(1) 0.723, Fe −0.115] | | | | |
| Plane (7): C(18), C(11), O(4), O(6) | −0.0643 | 0.6404 | 0.7654 | −1.6400 |
| [N(2) 0.411 Fe −0.682] | | | | |

positions, and has the same configuration as the corresponding molecular anion in the sodium salt. Figure 6 shows the molecular structure of the ion together with its atom-labelling scheme. Bond-lengths and -angles are listed in Tables IV and V, {Mean bond-lengths Fe–N 2.166(13), Fe–O (phenolic) 1.911(10) and Fe–O (carboxylate) 2.042(7) Å, and mean bond angles N–$\widehat{\mathrm{Fe}}$–O (phenolic) 88.4(8) and N–$\widehat{\mathrm{Fe}}$–O (carboxylate) 77.2(5)°}. Hydrogen-bonding distances are summarised in Table VI and equations of relevant least-squares planes in Table VII.

Once again the crystal structure contains distinct hydrophilic and hydrophobic regions, with an ordered octahedral hexaaquomagnesium(II) cation lying on the $C_2$-axis {Mg–O 2.054 to 2.150 Å, mean 2.082 Å, average e.s.d. 0.009 Å} and waters of crystallisation which are hydrogen-bonded at three other sites in the crystal lattice.

Molecular models indicate two possible configurations of similar energy for the [Fe(*rac*-ehpg)]$^-$ ion involving either *trans* carboxylates or *trans*

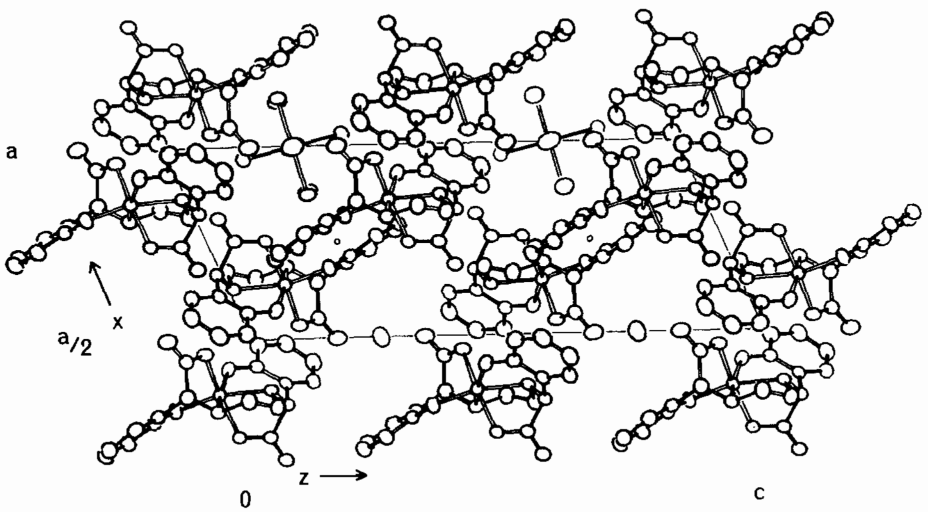

Fig. 5. The crystal packing in Mg[Fe(ehpg)]$_2$·9H$_2$O (Structure B), showing half the unit cell viewed down the *b* axis.

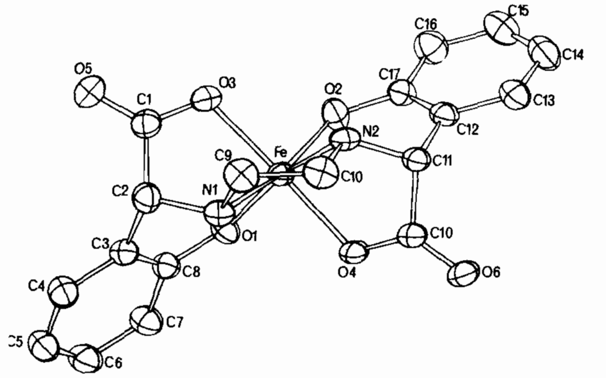

Fig. 6. Molecular structure and atom-labelling scheme for the [Fe(*R,R*-ehpg)]$^-$ ion in Mg[Fe(ehpg)]$_2$·9H$_2$O viewed down the pseudo (non-crystallographic) 2-fold axis.

phenoxyls*. We see no evidence of the latter in the solid state, but this does not preclude its existence in solution, nor possibly in the crystals of the NH$_4^+$ salt. The observation of the former in both structures A and B may well be a result of more favourable crystal-packing forces.

We saw no significant differences in the electronic spectra of the different crystalline forms (diffuse reflectance all had peaks at 20.2 kK) nor in the spectra of their aqueous solutions ($\lambda_{max}$ = 20.5 kK, $\epsilon \cong 4 \times 10^3$ $M^{-1}$ cm$^{-1}$). This is, perhaps, not unexpected if the phenoxys are always *cis*:undoubtedly the bands in the visible region have a phenoxy → metal charge-transfer origin.

Spiro and coworkers [7] have recently explored the system, concentrating on resonance raman measurements. However, together with the authors of a recent study of the terbium compounds [8], they were apparently unaware of the admixture of *meso* and *racemic* isomers.

The preparation of the magnesium salt provides a method of obtaining a pure *rac* form of the ligand.

Such crystals (especially the α-form) may offer useful models for the study of passive ion transport through channels which may in some of their aspects, resemble those within cell membranes.

## Acknowledgement

We thank the SRC for financial support, and Dr. P. M. Bailey for help with some crystallographic programs.

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*The ehpg$^{4-}$ ligand, having two chiral nitrogen and two chiral carbon atoms, can in theory give rise to seven sexadentate geometrical isomers of [Fe(ehpg)]$^-$. If the two nitrogens have the same chirality, the ethylenediamine bridge can adopt a gauche conformation and the four oxygen donors may bind without undue strain in the ligand with a) all like donor atoms in *cis* positions, or b) *trans* carboxylate oxygens or c) *trans* phenolic oxygens. If the nitrogens have opposite chirality there are another four possible isomers; two with the *racemic* and two with the *meso* ligand. However, all of these are strained to an extent that renders their existence unlikely, unless the ligand co-ordinates as a quinquedentate.