

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1001). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Potassium Aqua(3,4-toluenediamine-*N,N,N',N'*-tetraacetato)ferrate(III)–Water (1/1.5)

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Abstract

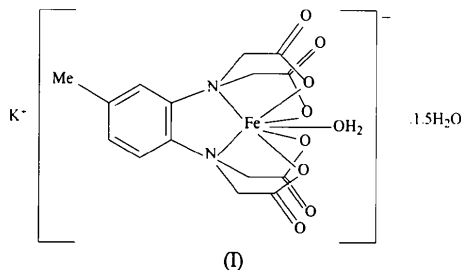
The ligand 3,4-toluenediamine-*N,N,N',N'*-tetraacetate in the title compound, K[Fe(C₁₅H₁₄N₂O₈)(H₂O)].1.5H₂O, is hexadentate, but the central iron(III) ion is seven-coordinate, with one coordinated water molecule. The complex has roughly pentagonal-bipyramidal geometry. The Fe—N bond lengths are 2.304(3) Å in the *para*

position and 2.349(3) Å in the *meta* position with respect to the methyl group. The presence of the CH₃ group increases the basicity of the N atom in the *para* position, shortening the length of the Fe—N bond.

Comment

The preparation of coordinating agents derived from aromatic diamines is of special interest since the use of N atoms for coordination to a single cation is directly related to their situation in *ortho*, *meta* or *para* positions. Thus, in the case of diaminetetramethylenecarboxylic acids, derived from *o*-phenylenediamines, the closer proximity of the N atoms permits the simultaneous coordination of both to the same metal cation, as has been established by the X-ray crystallographic determination of the structures of the complexes of *o*-phdta acid (*ortho*-phenylenediamine-*N,N,N',N'*-tetraacetic acid) with Co^{II} (McCandlish, Michael, Neal, Lingafelter & Rose, 1978), Mn^{II} (Nakasuka, Azuma, Katayama, Honda, Tanaka & Tanaka, 1985), Zn^{II} (Azuma, Nakasuka & Tanaka, 1986), Cu^{II} (Nakasuka, Azuma & Tanaka, 1986*a*), Cd^{II} (Nakasuka, Azuma & Tanaka, 1986*b*), Mg^{II} (Nakasuka & Shiro, 1989) and Fe^{III} (Mizuno, Funahashi, Nakasuka & Tanaka, 1991). Contrary to this is the behaviour of diaminetetramethylenecarboxylic acids derived from *meta*- or *para*-phenylenediamines, which can only coordinate one N atom of the ligand to the same metallic cation. Therefore, these ligands can bridge metal ions, as has been proven by the preparation of bimetallic species *M*₂*L* for *p*-phdta acid (*para*-phenylenediamine-*N,N,N',N'*-tetraacetic acid) (Ruíz-Pérez, Rodríguez, Rodríguez Romero, Mederos, Gili & Martín-Zarza, 1990) and dimer species *M*₂*L*₂ for *m*-phdta acid (*meta*-phenylenediamine-*N,N,N',N'*-tetraacetic acid) (Mederos *et al.*, 1990).

In the present work, the solid crystalline complex K[Fe(H₂O)(3,4-tdta)].1.5H₂O, (I), was prepared and characterized. 3,4-tdta (3,4-toluenediamine-*N,N,N',N'*-tetraacetic acid) is a potentially hexacoordinating ligand, analogous to *o*-phdta and edta.



The determination by X-ray diffraction of the structure of the complex [Fe(OH₂)(*o*-phdta)]⁻ (Mizuno, Funahashi, Nakasuka & Tanaka, 1991) shows that Fe^{III} is heptacoordinate, with roughly bipyramidal pentagonal

geometry and C_{2v} symmetry. The two Fe—N bonds are equal [2.344 (3) Å], as expected. The introduction of a methyl radical into the aromatic ring in position 3 produces the following electron-donor effect: *para* > *ortho* > *meta* (Hehre, Random, Schleyer & Pople, 1986). In the complex $[\text{Fe}(\text{OH}_2)(3,4\text{-tdta})]^-$, the C_{2v} symmetry is lost and it is to be expected that the Fe—N bond with the N atom in the *para* position is shorter than the Fe—N bond with the N atom in the *meta* position. Indeed, Fig. 1 shows the loss of symmetry of the complex ion $[\text{Fe}(\text{OH}_2)(3,4\text{-tdta})]^-$, and the data in Table 2 show that the Fe—N2 bond with N in the *para* position is approximately 0.04 Å shorter than in the complex $[\text{Fe}(\text{OH}_2)(o\text{-phdta})]^-$, while the Fe—N bonds with N in the *meta* positions are practically equal. This effect is analogous to that observed in aqueous solution where the 3,4-tdta complexes are more stable than the *o*-phdta complexes (Brito, Mederos, Herrera, Domínguez & Hernández-Padilla, 1988; Mederos, Herrera & Felipe, 1987).

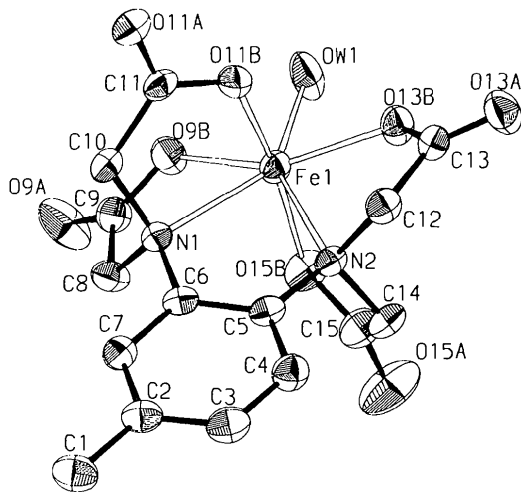


Fig. 1. ORTEPII (Johnson, 1976) view of the $[\text{Fe}(\text{OH}_2)(3,4\text{-tdta})]^-$ anionic chelate complex with 50% probability displacement ellipsoids. H atoms are excluded for clarity.

The $[\text{Fe}(\text{OH}_2)(3,4\text{-tdta})]^-$ complex is seven-coordinate with $[3,4\text{-tdta}]^{4-}$ as a hexadentate ligand and one water molecule filling the seventh position, similar to the anionic iron(III) chelates of *o*-phdta (Mizuno, Funahashi, Nakasuka & Tanaka, 1991), edta (Lind, Hamor, Hamor & Hoard, 1964; Nesterova, Polynova & Porai-Koshits, 1975; Solans, Font-Altaba & García-Orcaín, 1984; López-Alcalá, Puerta-Vizcaíno, González-Vilchez, Duesler & Tapscott, 1984) and cydta (1,2-cyclohexanediamine-*N,N,N',N'*-tetraacetic acid) (Cohen & Hoard, 1966).

The title complex has a roughly pentagonal bipyramidal shape, as observed in most Fe^{III} -edta complexes and in the Fe^{III} -*o*-phdta complex. In these cases, each Fe^{III} ion occupies the centre of the bipyramid; thus, Fe1

occupies the centre of the equatorial pentagonal plane whose vertices are O13B, O9B, O1W, N1 and N2; O11B and O15B occupy the apical positions.

The equatorial atoms form an approximate plane. The sum of the five angles around the central metal ion is $364.1(5)^\circ$ (362.9° in the Fe^{III} -*o*-phdta complex). The sum of the five endocyclic angles of the chelate ring of type *E* (pseudo ethylenediamine), Fe1—N1—C6—C5—N2, is $540(1)^\circ$. This is very close to that of the Fe^{III} -*o*-phdta complex, 539.5° , and an ideal pentagon, 540° , but considerably greater than that of the chelate ring in Fe^{III} -cydta, 518.7° .

The planes of the 'glycine' chelate rings Fe—O—C—C—N have been calculated and classified according to Hoard's proposal (Weakliem & Hoard, 1959). Rings Fe1—N1—C8—C9—O9B and Fe1—N2—C12—C13—O13B are of type *G* (nearly parallel to the NMN plane), and rings Fe1—N1—C10—C11—O11B and Fe1—N2—C14—C15—O15B are of type *R* (nearly perpendicular to the NMN plane). The average of the sum of the interior angles of the glycinate ring of type *R* is $533(1)^\circ$, which is very close to the corresponding values in complexes Fe^{III} -*o*-phdta and Fe^{III} -edta, which are 532.1 and 535.1° , respectively. There is also an agreement between the sum of the endocyclic angles of the ring of type *G*, which is $522(1)^\circ$ in the complex Fe^{III} -3,4-tdta and 520.7 and 524.8° in complexes Fe^{III} -*o*-phdta and Fe^{III} -edta, respectively. The difference between the two sums of the angles is practically constant: $11(2)$, 11.4 and 10.3° for Fe^{III} -3,4-tdta, Fe^{III} -*o*-phdta and Fe^{III} -edta, respectively. Comparison of C—N bond lengths reveals that the ring of type *R* has a longer distance [1.487 (5) Å] than the ring of type *G* [1.474 (5) Å]. This is also fulfilled for the complex Fe^{III} -*o*-phdta (1.492 and 1.483 Å, respectively).

A significant difference also exists between the Fe—O distances, according to whether they are situated in the ring of type *R* or that of type *G* [1.975 (3) and 2.062 (3) Å, respectively]. This difference is also

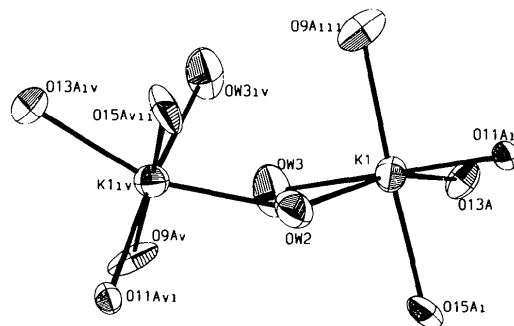


Fig. 2. ORTEPII (Johnson, 1976) plot of the coordination geometry of the K^+ atoms in $\text{K}[\text{Fe}(\text{OH}_2)(3,4\text{-tdta})].1.5\text{H}_2\text{O}$ with the atomic numbering schemes shown in Table 2 and the transformations: (iv) $1-x, y, \frac{1}{2}-z$; (v) $\frac{1}{2}+x, \frac{1}{2}+y, z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (vii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$. Ellipsoids are plotted at the 50% probability level.

present in the complex of *o*-phdta (1.990 and 2.087 Å). It is somewhat smaller (1.969 and 2.119 Å) in the edta complex. The Fe—O distance in the complex of 3,4-tdta is somewhat greater than in that of *o*-phdta [2.101 (3) and 2.057 Å, respectively], the former being closer to the corresponding value in complexes of edta and cydta (2.107 and 2.090 Å, respectively).

The C—O and glycinate ring C—C bond distances are virtually independent of ring type. The C—O bond distances are greater for the carboxylate O atom bonded to the Fe atom than for the non-complexed O atom [1.280 (5) and 1.222 (5) Å, respectively]. This fact is also verified for the complexes Fe^{III}-*o*-phdta (1.287 and 1.231 Å) and Fe^{III}-cydta (1.273 and 1.233 Å).

The KO₆ polyhedron may be described as a distorted octahedron. The K—O distances are listed in Table 2, showing a mean distance of 2.772 (4) and a range of 2.711 (4)–2.828 (3) Å. The OW2 atom bridges two K⁺ ions (Fig. 2).

Experimental

The potassium salt of 3,4-tdta was prepared by the method of Mederos, Herrera, Felipe & Quesada (1984). The potassium salt of the Fe^{III}-3,4-tdta complex was prepared by mixing aqueous solutions of iron(III) nitrate and KH₃(3,4-tdta). The final pH was adjusted to 4.5 by addition of KOH. Addition of 2-propanol to the solution resulted in a polycrystalline precipitate. Crystals suitable for X-ray crystallography were grown by liquid–vapour diffusion, using water as solvent and 2-propanol as precipitant. Elemental analysis: calculated for C₁₅H₁₉N₂O_{10.5}FeK C 36.75, N 5.71, H 3.91, K 7.97%; found C 36.40, N 5.61, H 3.81, K 6.91% (flame photometry).

Crystal data

K[Fe(C ₁₅ H ₁₄ N ₂ O ₈)(H ₂ O)].- 1.5H ₂ O	Mo K α radiation
$M_r = 490.27$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 25 reflections
$C2/c$	$\theta = 10\text{--}25^\circ$
$a = 11.018$ (7) Å	$\mu = 1.058$ mm ⁻¹
$b = 14.184$ (7) Å	$T = 293$ K
$c = 24.780$ (10) Å	Prism
$\beta = 94.50$ (5)°	$0.5 \times 0.20 \times 0.10$ mm
$V = 3861$ (4) Å ³	Orange
$Z = 8$	
$D_x = 1.687$ Mg m ⁻³	
D_m not measured	

Data collection

Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.1288$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 29^\circ$
Absorption correction: none	$h = -13 \rightarrow 13$
7142 measured reflections	$k = -16 \rightarrow 16$
3340 independent reflections	$l = 0 \rightarrow 29$
2676 observed reflections	3 standard reflections
$[I > 2\sigma(I)]$	frequency: 120 min
	intensity decay: 8%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = -0.008$
$R(F) = 0.0530$	$\Delta\rho_{\text{max}} = 0.91$ e Å ⁻³
$wR(F^2) = 0.1432$	$\Delta\rho_{\text{min}} = -0.89$ e Å ⁻³
$S = 1.115$	Extinction correction: none
3340 reflections	Atomic scattering factors
280 parameters	from <i>International Tables</i>
H-atom parameters not refined	for <i>X-ray Crystallography</i>
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 10.1515P]$	(1974, Vol. IV)
where $P = [\max(F_o^2) + 2F_o^2]/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{eq}
Fe1	0.2906 (1)	0.0456 (1)	0.1619 (1)	0.026 (1)
OW1	0.2544 (4)	0.1150 (2)	0.2338 (1)	0.048 (1)
C1	0.4325 (4)	-0.2802 (3)	-0.0357 (2)	0.040 (1)
C2	0.3923 (3)	-0.1919 (3)	-0.0090 (2)	0.032 (1)
C3	0.3372 (4)	-0.1189 (3)	-0.0390 (2)	0.035 (1)
C4	0.2973 (4)	-0.0386 (3)	-0.0147 (2)	0.033 (1)
C5	0.3100 (3)	-0.0296 (3)	0.0417 (1)	0.027 (1)
C6	0.3670 (3)	-0.1008 (3)	0.0719 (1)	0.026 (1)
C7	0.4080 (3)	-0.1812 (3)	0.0465 (2)	0.031 (1)
N1	0.3814 (3)	-0.0914 (2)	0.1303 (1)	0.026 (1)
C8	0.3232 (4)	-0.1693 (3)	0.1582 (2)	0.032 (1)
C9	0.3046 (4)	-0.1392 (3)	0.2152 (2)	0.033 (1)
O9A	0.2826 (4)	-0.1976 (2)	0.2492 (1)	0.061 (1)
O9B	0.3120 (3)	-0.0520 (2)	0.2244 (1)	0.036 (1)
C10	0.5122 (3)	-0.0833 (3)	0.1494 (2)	0.031 (1)
C11	0.5506 (3)	0.0178 (3)	0.1594 (1)	0.029 (1)
O11A	0.6599 (2)	0.0367 (2)	0.1654 (1)	0.035 (1)
O11B	0.4656 (2)	0.0789 (2)	0.1619 (1)	0.034 (1)
N2	0.2603 (3)	0.0496 (2)	0.0689 (1)	0.025 (1)
C12	0.3126 (3)	0.1403 (3)	0.0535 (2)	0.031 (1)
C13	0.2748 (3)	0.2145 (3)	0.0930 (2)	0.031 (1)
O13A	0.2658 (3)	0.2969 (2)	0.0797 (1)	0.045 (1)
O13B	0.2536 (3)	0.1824 (2)	0.1399 (1)	0.034 (1)
C14	0.1255 (3)	0.0522 (3)	0.0584 (2)	0.034 (1)
C15	0.0614 (4)	0.0093 (4)	0.1029 (2)	0.040 (1)
O15A	-0.0464 (3)	-0.0100 (4)	0.0948 (2)	0.072 (1)
O15B	0.1251 (2)	-0.0053 (2)	0.1482 (1)	0.039 (1)
K1	0.3421 (1)	0.3958 (1)	0.1722 (1)	0.045 (1)
OW2	1/2	0.4854 (5)	1/4	0.065 (2)
OW3	0.5263 (4)	0.2673 (3)	0.1877 (2)	0.076 (2)

Table 2. Selected geometric parameters (Å, °)

Fe1—N1	2.349 (3)	Fe1—N2	2.304 (3)
Fe1—O9B	2.078 (3)	Fe1—O11B	1.984 (3)
Fe1—O13B	2.047 (3)	Fe1—O15B	1.966 (3)
Fe1—OW1	2.101 (3)	C6—N1	1.449 (4)
N1—C8	1.476 (5)	N1—C10	1.486 (5)
C5—N2	1.439 (5)	N2—C12	1.472 (5)
N2—C14	1.488 (4)	C9—O9A	1.220 (5)
C9—O9B	1.259 (5)	C11—O11A	1.231 (5)
C11—O11B	1.282 (5)	C13—O13A	1.217 (5)
C13—O13B	1.287 (5)	C15—O15A	1.219 (5)
C15—O15B	1.293 (5)	K1—OW2	2.798 (4)
K1—OW3	2.732 (5)	K1—O13A	2.762 (3)
K1—O15A ⁱ	2.711 (4)	K1—O9A ⁱⁱⁱ	2.803 (4)
K1—O11A ⁱⁱ	2.828 (3)		
C5—N2—Fe1	114.7 (2)	C5—N2—C12	113.0 (3)
C5—N2—C14	110.4 (3)	C5—C6—N1	118.9 (3)
C6—N1—Fe1	113.1 (2)	C8—N1—Fe1	104.5 (2)
C6—N1—C10	110.7 (3)	C8—N1—C10	110.8 (3)
C12—N2—Fe1	104.5 (2)	C14—N2—Fe1	103.9 (2)
C12—N2—C14	109.8 (3)	C6—C5—N2	119.5 (3)
N1—C8—C9	108.9 (3)	N1—C10—C11	112.2 (3)

N2—C12—C13	107.9 (3)	N2—C14—C15	112.7 (3)
O9B—C9—C8	116.0 (4)	O11B—C11—C10	117.0 (3)
O13B—C13—C12	114.6 (3)	O15B—C15—C14	116.9 (3)
C9—O9B—Fe1	121.1 (2)	C11—O11B—Fe1	123.6 (3)
C13—O13B—Fe1	121.9 (3)	C15—O15B—Fe1	121.6 (3)
C10—N1—Fe1	104.9 (2)		

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}+y, z$; (ii) $x-\frac{1}{2}, \frac{1}{2}+y, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *XRAY72* (Stewart, Krüger, Ammon, Dickinson & Hall, 1972). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1280). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5,5'-(2-Hydroxy-1,3-propandiyldiamino)bis(1-phenyl-1,3-hexanedionato-O,O')oxovanadium(IV), [VO(C₂₇H₂₈N₂O₅)]

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Abstract

Schiff base ligands derived from triketones form stable complexes with VO. In the mononuclear vanadyl complex of 5,5'-(2-hydroxy-1,3-propandiyldiamino)bis(1-phenyl-1,3-hexanedione), vanadium is five-coordinate, equatorially bonded to four carbonyl O atoms in a nearly square-planar arrangement and axially to one other O atom. N atoms are not involved in complex formation. The hydroxy group is disordered.

Comment

It is well known that complexes such as molybdenum or vanadyl acetylacetonate catalyze the epoxidation of alkenes in the presence of organic hydroperoxides. (Sheldon & Kochi, 1981; Talsi, Chinakov, Babenko & Zamarev, 1993). Previously, we prepared the dinuclear Mo^{VI} complex of a triketone (Borer & Sinn, 1990) and reported the epoxidation of some bicyclic olefins using this complex (DiAmico, Rasmussen, Sisneros, Magnussen, Wade, Russell & Borer, 1992). Results of epoxidation reactions indicated that only one of the olefin bonds was oxidized in the ring, even though two metal sites were available on the catalyst.