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Three anionic polymeric networks: sodium(I)–ruthenium(III), sodium(I)–rhodium(III) and sodium(I)–aluminium(III) μ-oxalato complexes with tris(2,2′-bipyridine)ruthenium(II) cations

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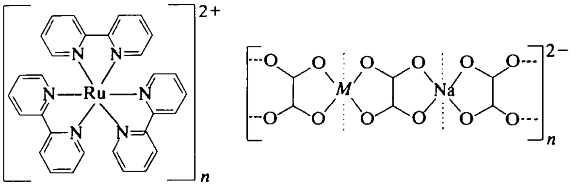
Abstract

The crystal structures of $[\mathrm{Ru(bpy)_3}]_n^{2+}[\mathrm{Na}M\mathrm{(ox)_3}]_n^{2n-}$, with M = Ru$^{\rm III}$ [tris(2,2′-bipyridine)ruthenium(II) tris(μ-oxalato)ruthenium(III)sodium(I), (1)], M = Rh$^{\rm III}$ [tris(2,2′-bipyridine)ruthenium(II) tris(μ-oxalato)rhodium(III)sodium(I), (2)] and M = Al$^{\rm III}$ [tris(2,2′-bipyridine)ruthenium(II) tris(μ-oxalato)aluminium(III)sodium(I), (3)], where bpy is 2,2′-bipyridine ($\mathrm{C_{10}H_8N_2}$) and ox is oxalate ($\mathrm{C_2O_4}$), represent examples of a specific polymeric three-dimensional network topology. These networks, formally built up from $[M\mathrm{(ox)_{3/2}}]$ subunits and $[\mathrm{Ru(bpy)_3}]^{2+}$ counter-ions which template the polymeric structure, are chiral. The absolute configurations of the measured crystals have been determined as the Λ form for (1), and as the Δ form for (2) and (3). The coordinated metal ions form trigonally distorted octahedra. The average distances are 2.060 (2) Å for Ru$^{\rm II}$—N in the three $[\mathrm{Ru(bpy)_3}]^{2+}$ counter-ions, 2.338 (3) Å for Na$^{\rm I}$—O, 2.031 (2) Å for Ru$^{\rm III}$—O, 2.005 (2) Å for Rh$^{\rm III}$—O and 1.890 (3) Å for Al$^{\rm III}$—O.

Comment

The anionic oxalate molecule can act as a mono-, bi-, tri- or tetradentate ligand, but is also well known for its ability to form bridged polynuclear complexes (Scott *et al.*, 1973). In particular, several two-dimensional heterometallic layer compounds with network stoichiometries $[M^{\rm II}M^{\rm III}\mathrm{(ox)_3}]_n^{n-}$ (Tamaki *et al.*, 1992; Decurtins, Schmalle, Oswald *et al.*, 1994; Pellaux *et al.*, 1997), as well as three-dimensional homometallic network compounds with stoichiometries $[M_2^{\rm II}\mathrm{(ox)_3}]_n^{2n-}$ (Decurtins, Schmalle, Schneuwly *et al.*, 1994; Decurtins *et al.*, 1996), have been reported. Both structure types have been realised for many different transition metals and they are of interest in the field of molecule-based magnets due to the ability of the oxalato ligand to act as mediator for magnetic exchange interactions. In addition, a three-dimensional heterometallic structure type with stoichiometries $[M^{\rm I}M^{\rm III}\mathrm{(ox)_3}]_n^{2n-}$, of which three examples are presented in this report, has been studied mainly because of its specific photophysical properties (Hauser *et al.*, 1996; von Arx *et al.*, 1996). For a general description of the $[M^{\rm I}M^{\rm III}\mathrm{(ox)_3}]_n^{2n-}$ structures, these anionic networks may be considered as built up from formal $[M\mathrm{(ox)_{3/2}}]$ subunits, each representing three-connected points (Wells, 1984). If all subunits have the same chiral configuration, a well defined three-dimensional three-connected decagon network is formed, where the $[\mathrm{Ru(bpy)_3}]^{2+}$ cations occupy the vacancies of the framework.



(1) M=Ru$^{\rm III}$
(2) M=Rh$^{\rm III}$
(3) M=Al$^{\rm III}$

In Fig. 1, a stereoplot of the structure of (1) in the Λ configuration is presented. Strands of bridged metal ions in the oxalato network form left-handed helices along the 2_1 axes. According to the space-group symmetry, all metal ions occupy sites with D_3 point-group symmetry (Wyckoff letter *a*). Contrary to the Λ configuration found in the case of (1), the absolute configurations of the coordinated metal ions in the measured crystals of (2) and (3) represent the Δ form; compound (3) is shown in Fig. 2 as an example. The Na—O 3+3 coordination distances range between 2.315 (2) Å in (1) and 2.388 (3) Å in (3). The most pronounced differences within the Na—O distances are observed in (3). Furthermore, these bond lengths are significantly elongated compared with values in (1) and (2). In a similar way, the Na—O distances reported for $[\mathrm{Ni(bpy)_3}]$-

$[NaAl(ox)_3]$ [2.358 (2) and 2.397 (2) Å; Román *et al.*, 1996] are also clearly longer than the values found for $[Fe(bpy)_3][NaFe(ox)_3]$ [2.317 (3) and 2.320 (3) Å; Decurtins, Schmalle, Schneuwly *et al.*, 1994]. The average *M*—O bond lengths for the trivalent metals Ru, Rh and Al correspond to the values found in the monomeric tris(oxalato)metallates: 2.028 (7) (Kaziro *et al.*, 1989) and 2.028 (4) Å (Faure *et al.*, 1986) in $K_3[Ru(ox)_3]\cdot4.5H_2O$, 2.016 Å in $K_3[Rh(ox)_3]\cdot4.5H_2O$ (Dazell & Eriks, 1971) and 1.896 (15) Å in $K_3[Al(ox)_3]\cdot3H_2O$ (Taylor, 1978).

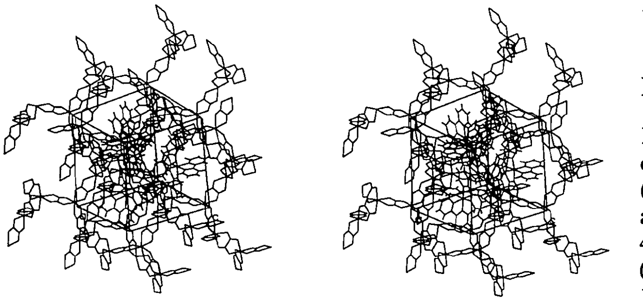


Fig. 1. Stereoview of the three-dimensional anionic network of (1) including some of the $[Ru(bpy)_3]^{2+}$ templates in the unit cell (*PLUTON*; Spek, 1990).

The bond distances and valence angles involving C and N atoms in the bpy ligand have normal values. The Ru atoms of the cations in the three structures exhibit trigonally distorted octahedral coordinations. The average Ru—N distances [2.067 (2) Å for (1), 2.061 (2) Å for (2) and 2.053 (3) Å for (3)] are in good agreement with the values found in $[Ru(bpy)_3](PF_6)_2$ [2.053 (2) (Biner *et al.*, 1992) and 2.056 (2) Å (Rillema *et al.*, 1992)] and $[Ru(bpy)_3](ClO_4)_2$ [2.058 (3) (Harrowfield & Sobolev, 1994) and 2.056 (3) Å (Krausz *et al.*, 1995)].

Experimental

$K_3[Ru(ox)_3]\cdot4.5H_2O$ was synthesized following a procedure described by Faure *et al.* (1986), but using $(NH_4)_2[Ru(H_2O)Cl_5]$ and $K_2C_2O_4\cdot H_2O$ as starting materials. The syntheses of $[Ru(bpy)_3]Cl_2\cdot6H_2O$, $K_3[Rh(ox)_3]\cdot4.5H_2O$ and $K_3[Al(ox)_3]\cdot3H_2O$ have been described previously (Palmer & Piper, 1966; Gillard *et al.*, 1969; Bailar & Jones, 1939). Single crystals of the title compounds were grown by topping a tetramethoxysilane gel of a 20 m*M* aqueous solution of the potassium tris(oxalato)metallate with equimolar amounts of 20 m*M* solutions of NaCl and $[Ru(bpy)_3]Cl_2$·-

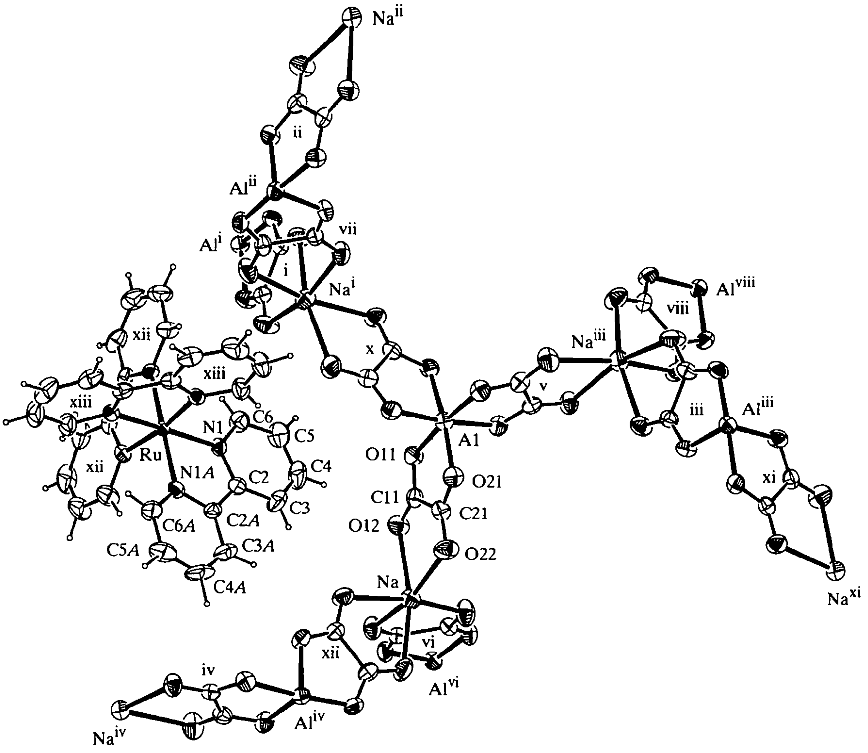


Fig. 2. *PLATON* displacement ellipsoid plot (Spek, 1990) at the 50% level of probability of (3), exhibiting the Δ configuration of the $[Ru(bpy)_3]^{2+}$ cation and a section of the $[NaAl(ox)_3]_n^{2n-}$ network. H atoms are shown as small circles of arbitrary radii. Symmetry codes: (i) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (ii) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iii) $\frac{1}{2}+x, \frac{3}{2}-y, -z$; (iv) $z, x, -1+y$; (v) $\frac{1}{2}-z, 1-x, -\frac{1}{2}+y$; (vi) $-z, \frac{1}{2}+x, \frac{1}{2}-y$; (vii) $\frac{1}{2}+z, \frac{1}{2}-x, 1-y$; (viii) $y, 1+z, x$; (ix) $-\frac{1}{2}+y, \frac{1}{2}-z, -x$; (x) $1-y, \frac{1}{2}+z, \frac{1}{2}-x$; (xi) $\frac{3}{2}-y, 1-z, -\frac{1}{2}+x$; (xii) z, x, y; (xiii) y, z, x.

$6H_2O$. After two weeks, tetrahedal-shaped dark-orange crystals were separated mechanically from the gel. Analysis calculated for $C_{36}H_{24}N_6NaO_{12}Ru_2$, (1): C 45.15, H 2.53, N 8.77%; found: C 44.43, H 2.65, N 9.00%; analysis calculated for $C_{36}H_{24}N_6NaO_{12}RhRu$, (2): C 45.06, H 2.52, N 8.76%; found: C 44.96, H 2.63, N 8.92%; analysis calculated for $C_{36}H_{24}AlN_6NaO_{12}Ru$, (3): C 48.93, H 2.74, N 9.51%; found: C 48.17, H 2.76, N 9.66%.

Compound (1)

Crystal data

$[Ru(C_{10}H_8N_2)_3][NaRu(C_2O_4)_3]$
$M_r = 957.74$
Cubic
$P2_13$
$a = 15.5560\,(12)$ Å
$V = 3764.4\,(5)$ Å^3
$Z = 4$
$D_x = 1.690$ Mg m^{-3}
$D_m = 1.67$ Mg m^{-3}
D_m measured by flotation in diiodomethane/toluene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5000 reflections
$\theta = 2.4$–$30.95°$
$\mu = 0.885$ mm^{-1}
$T = 193\,(2)$ K
Tetrahedral
$0.52 \times 0.52 \times 0.52$ mm
Dark orange

Data collection

Stoe IPDS diffractometer
φ-rotation scans
Absorption correction: numerical (Coppens *et al.*, 1965)
$T_{min} = 0.607$, $T_{max} = 0.685$
27 319 measured reflections
2169 independent reflections (plus 1753 Friedel-related reflections)

3210 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 30.79°$
$h = -22 \rightarrow 21$
$k = -22 \rightarrow 15$
$l = -22 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.063$
$S = 1.026$
3922 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0300P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.507$ e Å^{-3}
$\Delta\rho_{min} = -1.030$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = $-0.03\,(3)$

Table 1. *Selected geometric parameters (Å, °) for (1)*

Ru1—N1*A*	2.065 (2)	Na—O11*A*	2.317 (3)
Ru1—N1	2.0687 (19)	Ru2—O11	2.027 (2)
Na—O21*A*	2.315 (2)	Ru2—O21	2.034 (2)
Na—O21A^{i}	2.315 (2)		
N1A^{ii}—Ru1—N1A^{iii}	95.38 (9)	O21A^{i}—Na—O11A^{iv}	163.22 (11)
N1A^{ii}—Ru1—N1	171.85 (8)	O11A^{iv}—Na—O11*A*	88.69 (11)
N1A^{iii}—Ru1—N1	90.90 (9)	O11^{v}—Ru2—O11	92.90 (9)
N1*A*—Ru1—N1	78.84 (8)	O11^{v}—Ru2—O21^{vi}	175.48 (9)
N1—Ru1—N1^{ii}	95.40 (8)	O11—Ru2—O21^{vi}	88.31 (10)
O21A^{iv}—Na—O21*A*	103.28 (9)	O11^{vi}—Ru2—O21^{vi}	82.69 (8)
O21A^{iv}—Na—O11A^{iv}	74.72 (8)	O21^{vi}—Ru2—O21	96.16 (8)
O21*A*—Na—O11A^{iv}	93.33 (9)		
O11*A*—C11—C21—O21*A*	−1.1 (5)		
O11—C11—C21—O21*A*	179.6 (3)		
O11*A*—C11—C21—O21	178.7 (3)		
O11—C11—C21—O21	−0.6 (4)		

Symmetry codes: (i) $2 - z, x - \frac{1}{2}, \frac{3}{2} - y$; (ii) y, z, x; (iii) z, x, y; (iv) $\frac{1}{2} + y, \frac{3}{2} - z, 2 - x$; (v) $\frac{3}{2} - z, 1 - x, \frac{1}{2} + y$; (vi) $1 - y, z - \frac{1}{2}, \frac{3}{2} - x$.

Compound (2)

Crystal data

$[Ru(C_{10}H_8N_2)_3][NaRh(C_2O_4)_3]$
$M_r = 959.58$
Cubic
$P2_13$
$a = 15.515\,(2)$ Å
$V = 3734.7\,(8)$ Å^3
$Z = 4$
$D_x = 1.707$ Mg m^{-3}
$D_m = 1.68$ Mg m^{-3}
D_m measured by flotation in diiodomethane/toluene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.9$–$16.0°$
$\mu = 0.930$ mm^{-1}
$T = 296\,(2)$ K
Tetrahedral
$0.45 \times 0.45 \times 0.45$ mm
Dark orange

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: numerical (Coppens *et al.*, 1965)
$T_{min} = 0.481$, $T_{max} = 0.706$
10 892 measured reflections
2671 independent reflections (plus 680 Friedel-related reflections)

2985 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 33.93°$
$h = -16 \rightarrow 24$
$k = -16 \rightarrow 24$
$l = -16 \rightarrow 24$
3 standard reflections every 400 reflections
frequency: 180 min
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.065$
$S = 1.126$
3441 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0320P)^2 + 0.8643P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.691$ e Å^{-3}
$\Delta\rho_{min} = -0.502$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: $2.9\,(9) \times 10^{-4}$
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.00 (3)

Table 2. *Selected geometric parameters (Å, °) for (2)*

Ru—N1	2.0601 (18)	Rh—O22	2.0075 (18)
Ru—N1*A*	2.0624 (17)	Na—O11	2.327 (2)
Rh—O12	2.0018 (17)	Na—O21	2.335 (2)
N1^{i}—Ru—N1	95.62 (7)	O12^{iv}—Rh—O22^{iii}	176.43 (8)
N1^{i}—Ru—N1A^{ii}	90.87 (8)	O22^{iii}—Rh—O22	94.65 (8)
N1—Ru—N1A^{ii}	171.84 (7)	O11^{v}—Na—O11	89.04 (10)
N1^{ii}—Ru—N1A^{ii}	78.78 (7)	O11^{v}—Na—O21^{vi}	93.70 (8)
N1A^{ii}—Ru—N1A^{i}	95.25 (7)	O11—Na—O21^{vi}	162.16 (9)
O12^{iii}—Rh—O12	92.35 (8)	O11—Na—O21	73.40 (7)
O12^{iii}—Rh—O22^{iii}	84.24 (7)	O21^{vi}—Na—O21	103.98 (8)
O12—Rh—O22^{iii}	88.82 (9)		

O11—C11—C21—O21	1.4 (4)	O11—C11—C21—O22	−177.7 (3)
O12—C11—C21—O21	−179.0 (3)	O12—C11—C21—O22	1.8 (4)

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

Compound (3)

Crystal data

$[Ru(C_{10}H_8N_2)_3][NaAl(C_2O_4)_3]$
$M_r = 883.65$
Cubic
$P2_13$
$a = 15.407$ (4) Å
$V = 3657.2$ (16) Å^3
$Z = 4$
$D_x = 1.605$ Mg m^{-3}
$D_m = 1.60$ Mg m^{-3}
D_m measured by flotation in diiodomethane/toluene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.0$–16.1°
$\mu = 0.538$ mm^{-1}
$T = 296$ (2) K
Tetrahedral
0.45 × 0.45 × 0.45 mm
Dark orange

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: numerical (Coppens *et al.*, 1965)
$T_{\min} = 0.812$, $T_{\max} = 0.862$
10 055 measured reflections
2708 independent reflections (plus 499 Friedel-related reflections)

2089 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.083$
$\theta_{\max} = 33.94°$
$h = -14 \rightarrow 24$
$k = -14 \rightarrow 24$
$l = -14 \rightarrow 24$
3 standard reflections every 400 reflections frequency: 180 min intensity decay: 1.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.107$
$S = 1.145$
3207 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 2.9632P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.040$ e Å^{-3}
$\Delta\rho_{\min} = -0.787$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.09 (6)

Table 3. *Selected geometric parameters (Å, °) for (3)*

Ru—N1*A*	2.050 (3)	Na—O22	2.388 (3)
Ru—N1	2.056 (3)	Al—O21	1.882 (3)
Na—O12	2.347 (4)	Al—O11	1.899 (3)
N1*A*$^{\rm i}$—Ru—N1*A*	95.75 (14)	O12$^{\rm iv}$—Na—O22	162.96 (15)
N1*A*$^{\rm i}$—Ru—N1$^{\rm ii}$	172.11 (13)	O22—Na—O22$^{\rm iv}$	104.82 (13)
N1*A*—Ru—N1$^{\rm ii}$	90.51 (14)	O21—Al—O21$^{\rm v}$	92.72 (16)
N1*A*$^{\rm ii}$—Ru—N1$^{\rm ii}$	78.82 (13)	O21—Al—O11	84.86 (12)
N1$^{\rm ii}$—Ru—N1	95.41 (14)	O21$^{\rm v}$—Al—O11	93.10 (15)
O12$^{\rm iii}$—Na—O12	90.25 (15)	O21$^{\rm vi}$—Al—O11	173.80 (14)
O12$^{\rm iii}$—Na—O22	91.98 (14)	O11—Al—O11$^{\rm vi}$	89.57 (16)
O12—Na—O22	72.85 (10)		
O12—C11—C21—O22	2.1 (8)	O12—C11—C21—O21	−175.9 (5)
O11—C11—C21—O22	−177.2 (5)	O11—C11—C21—O21	4.7 (6)

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

The positions of the bpy H atoms were determined from difference electron-density maps; however, in all three structures, they were refined as riding models with isotropic displacement parameters set at 1.2 times the $U_{\rm eq}$ values of their adjacent C atoms.

Data collection: *EXPOSE* (Stoe & Cie, 1997*a*) for (1); *CAD*-4 *Software* (Enraf–Nonius, 1989) for (2) and (3). Cell refinement: *CELL* (Stoe & Cie, 1997*b*) for (1); *CAD*-4 *Software* for (2) and (3). Data reduction: *INTEGRATE* (Stoe & Cie, 1997*c*) for (1); *MolEN* (Fair, 1990) for (2) and (3). For all compounds, program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *PLUTON* (Spek, 1990) for (1); *PLATON* (Spek, 1990) for (2) and (3). For all compounds, software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1212). Services for accessing these data are described at the back of the journal.

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Intermolecular (pyrrole)N—H···π(pyrrole) interactions in bis(2-pyrrolyl)methylferrocene, an organometallic porphyrin precursor

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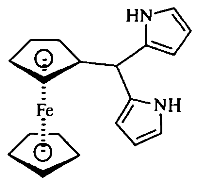
Abstract

The title compound, $C_{19}H_{18}FeN_2$ or $[Fe(C_5H_5)(C_{14}H_{13}N_2)]$, crystallizes in space group $P2_1/n$ with two molecules in the asymmetric unit. The pyrrole groups take part in intermolecular (pyrrole)N—H···π(pyrrole) interactions through a hydrogen-bonded ring system involving four pyrrole groups, with N···(pyrrole centroid) distances in the range 3.209 (3)–3.427 (3) Å and N—H···(pyrrole centroid) angles in the range 127–139°.

Comment

Dipyrromethane derivatives are useful building blocks in the synthesis of porphyrin model systems, with direct approaches to *trans*-substituted porphyrins available by condensation of dipyrromethanes with aldehydes (Lee & Lindsey, 1994). In contrast to porphyrin chemistry, there has been a paucity of structural reports on dipyrromethanes and their hydrogen-bonding interactions (Lin *et al.*, 1996; Bennis & Gallagher, 1998). The influence of weak hydrogen bonds in crystal packing has been commented on (Gallagher, Ferguson, Böhmer & Kraft, 1994; Ferguson *et al.*, 1996; Braga *et al.*, 1998), although a fundamental understanding of the control and exploitation of X—H···π interactions (X = C, N, O) remains an undeveloped field in crystal engineering (Steiner, 1997). This contrasts with the systematic study and exploitation of strong donors and acceptors in molecular-recognition systems (Etter *et al.*, 1990). In molecules where several different potential hydrogen-bond donors and acceptors are present (with cooperativity and/or competition among these interactions), the ability to deduce in advance the molecular-packing arrangements largely remains an unrealized vision (Wolff, 1996) and is principally due to the unpredictable nature of weaker hydrogen bonds in stabilizing three-dimensional structures.

Recent reports on N—H···π interactions include aliphatic N—H donors with conventional aromatic acceptors, *e.g.* phenyl rings (Allen *et al.*, 1997; Starikov & Steiner, 1998). Atypical heteroaromatic N—H donor and acceptor systems have also been described, where indole groups (Krishna *et al.*, 1999), pyrrole groups (Lin *et al.*, 1996; Goddard *et al.*, 1997; Bennis & Gallagher, 1998) and pyrazole ligands (Beringhelli *et al.*, 1998) take part in (heteroaromatic)N—H···π(heteroaromatic) interactions. These unusual examples of hydrogen bonds are important in facilitating our understanding of important molecular-recognition processes in biological systems, as well as in advancing crystal engineering studies. We report herein the structure of bis(2-pyrrolyl)methylferrocene, (I), where intermolecular N—H···π(pyrrole) interactions arise in a system with pyrrole N—H donors and pyrrole groups as acceptors.



(I)

Compound (I) crystallizes in space group $P2_1/n$ with two independent molecules, *A* and *B*, in the asymmetric unit (Fig. 1). Bond lengths and angles are in accord with anticipated values (Orpen *et al.*, 1994). The molecules differ in conformation, and torsion-angle differences are evident from the C11—C1—C41—N45 values of −170.0 (3) (*A*) and 175.5 (3)° (*B*) (Table 1). The weighted r.m.s. fit for the superposition of the non-disordered non-H atoms of molecules *A* and *B* (inverted) is 0.35 Å (Spek, 1998). The unsubstituted cyclopentadienyl ring of molecule *B* is disordered over two sites with occupancies of 0.68 (2) and 0.32 (2). Rotational disorder is often observed in the unsubstituted C_5 ring of ferrocene derivatives, *e.g.* 1-ferrocenyl-1-phenylethanol (Ferguson *et al.*, 1993). The dihedral angle between the C_5 planes are 1.1 (3) (*A*) and 3.6 (9)° (*B*), with the rings staggered by 15.7 (3) and 33.6 (7)° from an eclipsed

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