and P—Co—P planes of 87·3 (4)° denoting a small deviation from the tetrahedral geometry. The N—Co—N and the Co—N—O angles are smaller and larger respectively than in the $[Co(NO)_2(PPh_3)_2]^+$ complex [136·7 (4) and 171·0 (5)° (Reichert, 1976)] and are consistent with a more evident NO^+ character of the nitrosyl with the phosphite than phosphine ligands, according to the higher π-acceptor character of the phosphite compared with the phosphine ligand.

Also, the Co—P bond distances in the complex with the phosphite ligand [2·214 (5) and 2·209 (4) Å] shorter than those found in the phosphine cation [2·266 (3) Å] are consistent with a greater π-acceptor character of the phosphite. In the phosphite complex the P—Co—P angle [99·5 (3)°] is smaller than in the phosphine complex [113·5 (2)°] probably because of the smaller steric demand of the $P(OMe)_3$ ligand than that of the bulky PPh_3 ligand.

The projection of the structure along [010] is shown in Fig. 2. The cations and the BPh_4^- anions are linked by normal van der Waals contacts, no non-bonded distances being particularly short.

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Structure of *fac*-(2,2′-Bipyridyl)trichloro(1*H*-1,2,4-triazole-*N*⁴)iron(III), $[Fe(C_2H_3N_3)(C_{10}H_8N_2)Cl_3]$

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Abstract. $M_r = 387{\cdot}46$, monoclinic, $P2_1/n$, $a = 9{\cdot}179$ (2), $b = 14{\cdot}288$ (5), $c = 12{\cdot}001$ (3) Å, $\beta = 98{\cdot}01$ (2)°, $V = 1558{\cdot}6$ (9) Å^3, $Z = 4$, $D_m = 1{\cdot}64$ (2), $D_x = 1{\cdot}651$ (1) g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 14{\cdot}84$ cm^{-1}, $F(000) = 770{\cdot}8$, 295 K, $R = 0{\cdot}022$ for 2012 significant $[I > 2\sigma(I)]$ reflexions. The iron(III) ion is coordinated by three chloride ions (in a *fac* arrangement), the two bipyridyl nitrogens, and the N(4) triazole atom in a distorted octahedral geometry. The bipyridyl and triazole molecules are planar. The least-squares planes of these molecules intersect at an angle of 91·05 (8)°.

Introduction. A large number of structures is possible for a species with molecular formula FeLCl$_3$ with L 2,2′-bipyridyl (bpy) or 1,10-phenanthroline (phen) but little is known about the chemistry of these compounds (Cotton, 1972). Fe(bpy)Cl$_3$ and Fe(phen)Cl$_3$ were first reported by Simon, Morgenstern & Albrecht (1937) and later formulated as $[FeL_2Cl_2]^+[FeCl_4]^-$ (Harris & Lockyer, 1958; Figgis & Lewis, 1964). Indeed, the crystal structure of $[Fe(phen)_2Cl_2]^+[FeCl_4]^-$ has recently been published (Goodwin, McPartlin & Goodwin, 1977). However, Mössbauer experiments pointed to the existence of another species with a dimeric halogen-bridged structure (Berrett, Fitzsimmons & Owusu, 1968). To obtain more information about this type of compound the preparation of Fe(bpy)Cl$_3$ was re-investigated, whereby polymorphism was established (Vos, 1983). Reaction of Fe(bpy)Cl$_3$ with various nitrogen donor ligands resulted in a number of mixed-ligand adduct compounds. We report the crystal structure of one of these complexes, Fe(bpy)(Htrz)Cl$_3$, where Htrz is 1H-1,2,4-triazole, the first structure of an iron(III)–bipyridyl compound to be reported.

Experimental. Pale orange-brown needles, grown at room temperature from an acetone/toluene solution containing Fe(bpy)Cl$_3$ and three equivalents of 1H-1,2,4-triazole; D_m by flotation in chloroform–dibromoethane; approx. 0·50 × 0·35 × 0·10 mm; Enraf–Nonius CAD-4 four-circle diffractometer,

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graphite-monochromated Mo *Kα*; cell constants from setting angles of 24 reflexions, systematic absences *h*0*l* *h*+*l* odd, 0*k*0 *k* odd, space group $P2_1/n$ (non-standard setting of $P2_1/c$); corrections for Lorentz and polarization effects and absorption (transmission coefficients 0·81 to 1·09); $\theta_{\max} = 25°$; $h = 0$ to 10, $k = 0$ to 17, $l = -14$ to 14; standard reflexions 068, 2,10,$\bar{4}$ and 632 with intensity variation of 5·9%: intensities corrected for crystal damage effect (using a fifth-order polynomial in the exposure time), residual error 1·4%; 3049 measured reflexions, 2862 independent, 2012 with $I > 2\sigma(I)$; direct methods; *F* used in LS refinement; positions of H atoms from difference Fourier map; least-squares refinement of positional (H and non-H) and isotropic (H) and anisotropic (non-H) thermal parameters; $R_w = 0.028$, $S = 1.695$, $w = 1/\sigma^2(F)$; $\Delta_{\max}/\sigma < 0.005$; max., min. $\Delta\rho$ excursions in final difference synthesis 0·24, −0·15 e Å^{-3}; scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); Leiden University Computers: IBM 370/158 and Amdahl V7B; *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1976) and programs written and modified by Mrs E. W. Rutten-Keulemans and Dr R.A.G. de Graaff.

Discussion. Positional parameters and isotropic thermal parameters for the non-hydrogen atoms are listed in Table 1.* Bond distances and angles are given in Table 2. The stereochemistry of the molecular entity Fe(bpy)(Htrz)Cl_3 and the atomic labelling are depicted in Fig. 1.

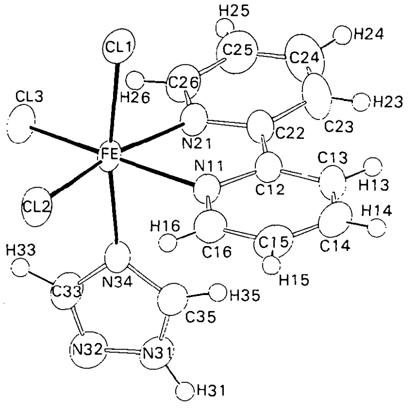

Fig. 1. *ORTEP* projection (Johnson, 1965) and atomic numbering of Fe(bpy)(Htrz)Cl_3.

The iron(III) ion is surrounded by three Cl^- ions in a facial arrangement and three N atoms, together forming a distorted octahedron. The N(11)—Fe—N(21) angle of 74·6° shows the largest deviation from a regular

Table 1. *Fractional coordinates* (Fe, Cl ×10^5, others ×10^4) *and isotropic thermal parameters* (Fe Å^2 ×10^3, *others* Å^2 ×10^2) *of the non-hydrogen atoms in* Fe(bpy)(Htrz)Cl_3, *with e.s.d.'s in parentheses*

$B_{eq} = (8/3)\pi^2 \text{ trace } \tilde{\mathbf{U}}$.

	x	*y*	*z*	B_{eq}
Fe	21884 (3)	26124 (2)	97658 (2)	2482 (9)
Cl(1)	23372 (7)	32827 (4)	80210 (5)	347 (2)
Cl(2)	7091 (7)	37584 (5)	103700 (6)	401 (2)
Cl(3)	3662 (7)	15565 (5)	91652 (6)	369 (2)
N(11)	4228 (2)	3304 (1)	10431 (2)	251 (5)
C(12)	5492 (2)	2899 (2)	10228 (2)	273 (6)
C(13)	6834 (3)	3306 (2)	10573 (2)	362 (8)
C(14)	6894 (3)	4156 (2)	11108 (2)	407 (8)
C(15)	5624 (3)	4558 (2)	11326 (2)	390 (8)
C(16)	4310 (3)	4118 (2)	10986 (2)	318 (7)
N(21)	3935 (2)	1666 (1)	9413 (2)	266 (5)
C(22)	5317 (2)	1991 (2)	9628 (2)	291 (6)
C(23)	6472 (3)	1503 (2)	9285 (3)	481 (10)
C(24)	6213 (4)	677 (3)	8716 (3)	577 (11)
C(25)	4810 (4)	341 (2)	8511 (3)	484 (9)
C(26)	3696 (3)	845 (2)	8873 (2)	374 (8)
N(31)	3282 (3)	1395 (2)	13058 (2)	375 (6)
N(32)	1871 (3)	1108 (2)	12895 (2)	483 (8)
C(33)	1392 (3)	1465 (2)	11899 (2)	459 (9)
N(34)	2420 (2)	1950 (1)	11431 (2)	272 (5)
C(35)	3578 (3)	1895 (2)	12202 (2)	338 (7)

Table 2. *Bond distances* (Å) *and bond angles* (°) *for* Fe(bpy)(Htrz)Cl_3

Fe—Cl(1)	2·324 (1)	N(31—N(32)	1·346 (3)
Fe—Cl(2)	2·308 (1)	N(31)—C(35)	1·311 (3)
Fe—Cl(3)	2·293 (1)	N(32)—C(33)	1·317 (3)
Fe—N(11)	2·168 (2)	C(33)—N(34)	1·354 (3)
Fe—N(21)	2·184 (2)	N(34)—C(35)	1·310 (3)
Fe—N(34)	2·195 (2)	C(12)—C(22)	1·481 (4)
N(11)—C(12)	1·349 (3)	N(21)—C(22)	1·341 (3)
N(11)—C(16)	1·338 (3)	N(21)—C(26)	1·344 (3)
C(12)—C(13)	1·373 (3)	C(22)—C(23)	1·380 (4)
C(13)—C(14)	1·372 (4)	C(23)—C(24)	1·367 (5)
C(14)—C(15)	1·357 (4)	C(24)—C(25)	1·364 (5)
C(15)—C(16)	1·372 (4)	C(25)—C(26)	1·370 (4)
Cl(1)—Fe—Cl(2)	95·83 (3)	N(31)—N(32)—C(33)	102·0 (2)
Cl(1)—Fe—Cl(3)	96·88 (3)	N(32)—C(33)—N(34)	114·2 (3)
Cl(1)—Fe—N(11)	89·32 (5)	C(33)—N(34)—C(35)	102·8 (2)
Cl(1)—Fe—N(21)	86·78 (5)	N(34)—C(35)—N(31)	110·4 (2)
Cl(1)—Fe—N(34)	171·06 (5)	C(35)—N(31)—N(32)	110·6 (2)
Cl(2)—Fe—Cl(3)	97·48 (3)	N(11)—C(12)—C(13)	121·5 (2)
Cl(2)—Fe—N(11)	94·40 (5)	C(12)—C(13)—C(14)	119·5 (2)
Cl(2)—Fe—N(21)	168·66 (5)	C(13)—C(14)—C(15)	119·1 (3)
Cl(2)—Fe—N(34)	90·38 (6)	C(14)—C(15)—C(16)	119·5 (3)
Cl(3)—Fe—N(11)	165·95 (5)	C(15)—C(16)—N(11)	122·2 (3)
Cl(3)—Fe—N(21)	93·14 (5)	C(16)—N(11)—C(12)	118·2 (2)
Cl(3)—Fe—N(34)	88·65 (5)	C(13)—C(12)—C(22)	123·3 (2)
N(11)—Fe—N(21)	74·56 (7)	N(11)—C(12)—C(22)	115·2 (2)
N(11)—Fe—N(34)	83·79 (7)	N(21)—C(22)—C(23)	120·8 (3)
N(21)—Fe—N(34)	85·90 (7)	C(22)—C(23)—C(24)	119·9 (3)
Fe—N(11)—C(12)	117·3 (2)	C(23)—C(24)—C(25)	119·3 (3)
Fe—N(11)—C(16)	124·4 (2)	C(24)—C(25)—C(26)	119·0 (3)
Fe—N(21)—C(22)	116·6 (2)	C(25)—C(26)—N(21)	122·2 (3)
Fe—N(21)—C(26)	124·0 (2)	C(26)—N(21)—C(22)	118·8 (2)
Fe—N(34)—C(33)	127·8 (2)	C(12)—C(22)—C(23)	123·5 (2)
Fe—N(34)—C(35)	129·4 (2)	C(12)—C(22)—N(21)	115·7 (2)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38795 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

octahedral angle, obviously imposed by the Fe—N distances and the rather rigid bipyridyl conformation. In similar complexes the same 'deviation' in bonding angles has been observed, as for instance in $[\mathrm{phenH}]^+[\mathrm{Fe(phen)Cl_4}]^-$, where an angle of 75·0° is found (Veidis, Witten, Reiff, Garafalo & Brennan, 1981), and in two copper(II)–bipyridyl complexes where the Cu—N distances are shorter than the Fe—N distances in our compound and consequently the N—Cu—N angle is somewhat larger (80°) (Harrison, Hathaway & Kennedy, 1979). All Cl—Fe—Cl angles are about 96°. The $\mathrm{Cl^-}$ ions, in a *fac* arrangement, occupy more space than they would have in the case of a regular octahedral coordination of the iron(III) ion.

The bipyridyl ligand is planar to within ± 0·05 Å. The triazole ligand is also planar; all triazole atoms lie within 0·01 Å of the least-squares plane. The least-squares planes through the bipyridyl and triazole ligands are mutually perpendicular, intersecting at an angle of 91·05 (8)°.

The triazole ligand is coordinated through its N(4) atom. The only other examples containing monodentate N(4)-bonded triazole are $\mathrm{Mn(Htrz)(H_2O)_4(SO_4)}$ (Gorter & Engelfriet, 1981) and $\mathrm{Cd(NCS)_2(Htrz)_2}$ (Haasnoot, de Keyzer & Verschoor, 1983). For the first compound this was considered to be unusual and due to hydrogen bonding of H(N1) to the anion. The second compound showed intermolecular N(1)—H to N(2′) hydrogen bridges. However, in our case hydrogen bonding does not occur, as distances from the parent nitrogen atom [N(31)] to the nearest $\mathrm{Cl^-}$ ions or N atoms in neighbouring molecules are larger than 3·3 Å. This implies that coordination through N(4) is independent of hydrogen-bond formation by H(N1), and should not be considered unusual.

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Structure of 2-Amino-4,5-dihydro-3H^+-1,3-thiazolium Trichlorocadmate(II), $\mathrm{C_3H_7N_2S^+.CdCl_3^-}$

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Abstract. $M_r = 321·9$, monoclinic, $P2_1/c$, $a = 14·438\,(5)$, $b = 3·922\,(2)$, $c = 20·952\,(7)$ Å, $\beta = 131·72\,(5)°$, $U = 885·6$ Å^3, $Z = 4$, $D_m = 2·40\,(3)$, $D_x = 2·41$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 3·50$ mm^{-1}, $F(000) = 616$, room temperature, final $R = 0·029$ for 2064 reflections. The polymeric structure consists of $\mathrm{ATH^+}$ and $\mathrm{CdCl_3^-}$ anions. The Cd atoms are octahedrally coordinated and linked into infinite chains by double Cl bridges. Two such chains running parallel to each other are themselves linked by Cd—Cl bonds. There is no direct bonding between the metal atom and the organic molecule; each $\mathrm{ATH^+}$ cation is associated with $(\mathrm{CdCl_3^-})_n$ chains through hydrogen bonds involving all of the H atoms attached to N atoms.

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