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Structure of (η^5-Cyclopentadienyl)(η^6-thianthrene)iron(II) Hexafluorophosphate

BY K. A. ABBOUD, V. M. LYNCH AND S. H. SIMONSEN*

Department of Chemistry, University of Texas at Austin, Austin, TX 78712, *USA*

AND A. PIÓRKO AND R. G. SUTHERLAND

Department of Chemistry, University of Saskatchewan, Saskatoon, Saskatchewan, S7N 0W0, Canada

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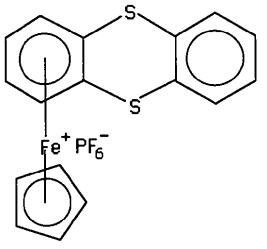
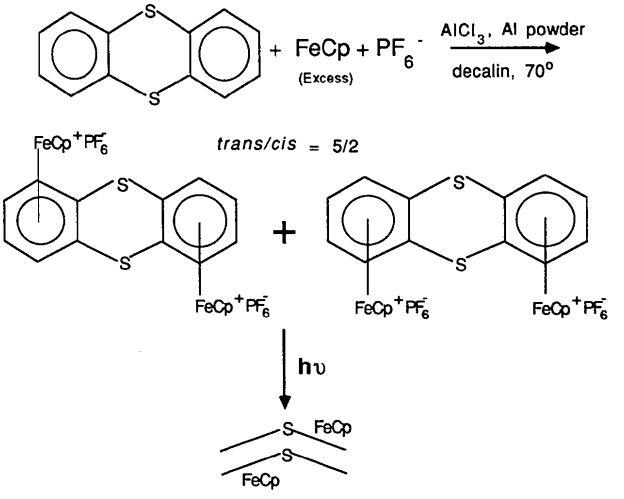
Abstract. $[Fe(C_5H_5)(C_{12}H_8S_2)][PF_6]$ (I), $M_r = 482{\cdot}22$, monoclinic, $P2_1/c$, $a = 14{\cdot}194$ (3), $b = 9{\cdot}617$ (2), $c = 25{\cdot}971$ (5) Å, $\beta = 100{\cdot}31$ (2)°, $V = 3488$ (1) Å^3, $Z = 8$ (two molecules per asymmetric unit), $D_x = 1{\cdot}836$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}70169$ Å), $\mu = 12{\cdot}429$ cm^{-1}, $F(000) = 1936$, $T = 163$ K. Block-matrix least-squares refinement of (I) converged to $R = 0{\cdot}044$ and $wR = 0{\cdot}040$ using 3785 reflections with $I > 3\sigma(I)$. The dihedral angles of the two molecules in the asymmetric unit are different. In molecule *A*, the FeCp moiety (Cp = cyclopentadiene) is inside the fold with a dihedral angle of 143·1 (2)°, whereas in molecule *B*, the FeCp moiety is located outside the fold with a dihedral angle of 136·3 (2)°.

Introduction. In a continuation of our study on the synthesis, structure and properties of the FeCp complexes of tricyclic heterocycles, the structure of (I)

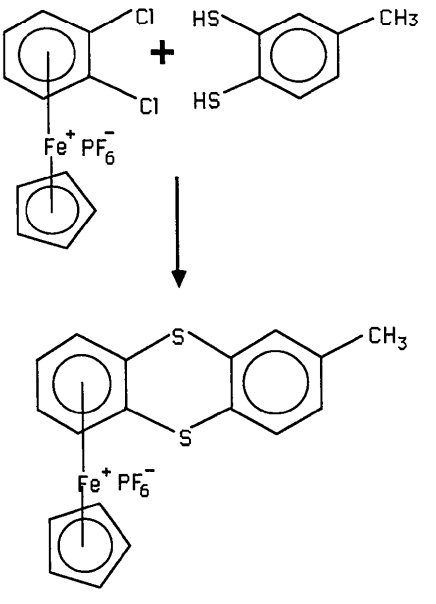


(I)

was determined. A method of synthesis (Scheme 1) different from the one used in the synthesis (Scheme 2) of the 2-methyl homologue (Sutherland, Piórko, Gill & Lee, 1982), for which the crystal structure has been elucidated (Simonsen, Lynch, Sutherland & Piórko, 1985), was employed. The objective of the present study was to determine whether the change in the method of synthesis, as well as the presence of a methyl substituent, would have any effect on the geometry of the heterocycle in the complex.



Scheme 1



Scheme 2

* To whom correspondence should be addressed.

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Experimental. The title compound was obtained from the monodemetallation reaction of a 2:5 mixture of *cis* and *trans*-(η^6,η^6-thianthrene)bis[(Cp)iron(II)] hexafluorophosphate by light in an acetone–diethylether–methylene chloride solution. The preparation of the dicationic mixture was reported by Lee, Piórko & Sutherland (1983). Crystals of the thianthrene monocations were grown from the resulting solution at *ca* 255 K. A needle-shaped orange-yellow crystal, 0·10 × 0·21 × 0·50 mm, was mounted and transferred to a Syntex $P2_1$ diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ (λ = 0·71069 Å) radiation, where it was maintained in an environment of dry N_2 at 163 K using a Syntex LT-1 low-temperature delivery system. Preliminary investigations revealed the crystal system to be monoclinic. 45 strong reflections, $20{\cdot}68 \le 2\theta \le 26{\cdot}87°$, were used to refine the unit-cell parameters. 13 497 reflections (h −17→17; k −11→11; l 0→32) were collected using the ω-scan method, 6849 unique reflections, R_{int} = 0·019; 2θ range 4→52°, 1° ω scan at 6–12° min^{-1}, depending upon intensity. Four reflections (006; 00$\bar{6}$; 200; $\bar{2}1\bar{1}$) were remeasured every 96 reflections to monitor instrument stability and crystal decay (maximum correction on I was < 1·75%). Absorption corrections were applied based on measured crystal faces (Riley & Davis, 1976); min. and max. transmission factors 0·773 and 0·900. The structure was solved by the heavy-atom method [Patterson, *SHELX*76 (Sheldrick, 1976)] which revealed the positions of the Fe atoms in both molecules of the asymmetric unit (molecules *A* and *B*). The positions of the rest of the non-H atoms were obtained from a difference Fourier map and all H atoms were obtained from a subsequent difference map. Refinement was carried out by block-matrix least squares (each molecule was refined in a separate block) in *SHELX*76 using anisotropic thermal parameters for the non-H atoms and isotropic thermal parameters for the H atoms. $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0{\cdot}5\,kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$, I(intensity) = $(I_{peak} - I_{bkg}) \times$ (scan rate), and $\sigma(I) = (I_{peak} + I_{bkg})^{1/2} \times$ (scan rate). k is the correction due to decay and Lp effects, and 0·02 is a factor used to downweight intense reflections and to account for instrument instability. Convergence was attained with R and wR of 0·044 and 0·040, respectively, for 3785 reflections of intensity larger than $3\sigma(I)$. In the last cycle of refinement, 591 variables were refined to a goodness-of-fit of 1·18; the largest shift/e.s.d. did not exceed 0·1; min. and max. peaks in the difference Fourier map were −0·44 and 0·54 e Å^{-3}, respectively. The linear absorption coefficient was calculated from values from *International Tables for X-ray Crystallography* (1974); scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965). The least-squares planes program was supplied by Cordes (1983); other programs used are cited in reference 11 of Gadol & Davis (1982). Positional parameters of all non-H atoms with equivalent isotropic thermal parameters are given in Table 1. Bond lengths and angles for the non-H atoms are listed in Table 2.* Figs. 1 and 2 show the thermal ellipsoid drawings (*SHELXTL-PLUS*) of molecules *A* and *B*, respectively, with the atomic labeling schemes. The packing diagram is shown in Fig. 3.

Discussion. In both molecules of the asymmetric unit, the Fe atom is centered above the Cp ring; however, bonding between Fe and the arene rings is asymmetrical. The longest Fe—C distances are to the C atoms adjacent to the S atoms. The Fe—C4*a* and Fe—C10*a* distances are 2·100 (5) and 2·098 (6) Å in *A*, and 2·092 (5) and 2·081 (5) Å in *B*, as compared to the average 2·078 (6) Å Fe—arene C distance in *A* and 2·076 (6) Å in *B*. This indicates that the Fe atoms are not exactly centered on the arene rings. The C—C distances of the coordinated arenes are longer than the C—C distances of the uncoordinated rings in both molecules. This phenomenon was also observed in similar compounds reported by Simonsen *et al.* (1985) and Lynch, Thomas, Simonsen, Piórko & Sutherland (1986).

The distances between Fe and the Cp rings, 1·6702 (7) and 1·6650 (8) Å in *A* and *B*, respectively, are longer than the distances between Fe and the coordinated arene ring planes [1·5385 (7) and 1·5336 (8) Å in *A* and *B*, respectively]. All these distances are well within the range of distances observed in similar compounds (Lynch *et al.*, 1986; Simonsen *et al.*, 1985). The dihedral angles between the Cp ring planes and the coordinated arenes [2·5 (2), 1·5 (2)° in *A* and *B*, respectively] are both less than 3° which was observed in compounds reported by Simonsen *et al.* (1985) and Lynch *et al.* (1986). The largest angle is in *A* where the FeCp moiety is inside the fold.

In a previous paper, the crystal structure of (5a,6,7,8,9,9a-η^6-2-methylthianthrene)(η^5-Cp)iron(II) hexafluorophosphate (II), prepared in a double nucleophilic substitution reaction from an *o*-chlorobenzene complex as a starting material, was described (Simonsen *et al.*, 1985). It was found that

* Tables of the crystallographic data, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52632 (50 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* ($Å^2$)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Molecule *A*				
Fe	0·26044 (5)	0·13282 (8)	−0·15713 (3)	0·0205 (2)
P	−0·00443 (10)	−0·1215 (2)	0·33224 (5)	0·0244 (5)
F1	−0·0240 (2)	−0·0195 (4)	0·28284 (13)	0·0422 (13)
F2	0·0148 (2)	−0·2248 (4)	0·38179 (13)	0·0404 (13)
F3	0·1070 (2)	−0·1271 (4)	0·32906 (12)	0·0358 (12)
F4	0·0146 (2)	0·0094 (4)	0·37065 (14)	0·0431 (13)
F5	−0·1162 (2)	−0·1162 (3)	0·33585 (12)	0·0359 (12)
F6	−0·0235 (2)	−0·2536 (4)	0·29446 (14)	0·0484 (14)
S5	0·34874 (10)	0·05936 (15)	−0·02258 (5)	0·0251 (5)
S10	0·11644 (10)	0·0306 (2)	−0·06471 (6)	0·0282 (5)
C1	0·1661 (4)	−0·0328 (6)	−0·1590 (2)	0·028 (2)
C2	0·2300 (4)	−0·0593 (6)	−0·1927 (2)	0·030 (2)
C3	0·3288 (4)	−0·0481 (6)	−0·1749 (2)	0·029 (2)
C4	0·3644 (4)	−0·0070 (6)	−0·1231 (2)	0·026 (2)
C4*A*	0·2998 (4)	0·0166 (5)	−0·0882 (2)	0·021 (2)
C5*A*	0·2655 (4)	0·1812 (5)	−0·0064 (2)	0·021 (2)
C6	0·2992 (4)	0·2860 (6)	0·0290 (2)	0·027 (2)
C7	0·2366 (4)	0·3748 (6)	0·0472 (2)	0·030 (2)
C8	0·1403 (4)	0·3656 (7)	0·0285 (2)	0·034 (2)
C9	0·1057 (4)	0·2637 (7)	−0·0079 (2)	0·033 (2)
C9*A*	0·1674 (4)	0·1688 (5)	−0·0244 (2)	0·022 (2)
C10*A*	0·2004 (4)	0·0040 (5)	−0·1064 (2)	0·022 (2)
C11	0·1704 (4)	0·2917 (6)	−0·1884 (2)	0·028 (2)
C12	0·2227 (4)	0·3320 (6)	−0·1396 (2)	0·027 (2)
C13	0·3223 (4)	0·3248 (6)	−0·1424 (2)	0·029 (2)
C14	0·3292 (4)	0·2819 (6)	−0·1931 (2)	0·029 (2)
C15	0·2356 (4)	0·2607 (6)	−0·2216 (2)	0·027 (2)
Molecule *B*				
Fe	0·22338 (5)	0·90093 (8)	0·15244 (3)	0·0203 (2)
P	0·53346 (10)	0·9057 (2)	0·30340 (5)	0·0237 (5)
F1	0·5727 (2)	0·9038 (4)	0·36483 (12)	0·0468 (13)
F2	0·4946 (2)	0·9068 (4)	0·24162 (12)	0·0452 (13)
F3	0·5363 (2)	1·0705 (3)	0·30309 (14)	0·0470 (14)
F4	0·6411 (2)	0·9017 (4)	0·29322 (12)	0·0428 (13)
F5	0·5307 (2)	0·7408 (4)	0·3028 (2)	0·0545 (15)
F6	0·4263 (2)	0·9088 (4)	0·31401 (12)	0·0419 (13)
S5	0·39338 (9)	0·7434 (2)	0·09111 (5)	0·0257 (5)
S10	0·17270 (9)	0·73099 (15)	0·03439 (5)	0·0247 (5)
C1	0·1543 (4)	0·9832 (6)	0·0817 (2)	0·023 (2)
C2	0·1855 (4)	1·0907 (6)	0·1165 (2)	0·030 (2)
C3	0·2806 (4)	1·0950 (6)	0·1422 (2)	0·028 (2)
C4	0·3451 (4)	0·9926 (6)	0·1333 (2)	0·024 (2)
C4*A*	0·3145 (3)	0·8819 (6)	0·0978 (2)	0·020 (2)
C5*A*	0·3613 (4)	0·7088 (5)	0·0237 (2)	0·021 (2)
C6	0·4326 (4)	0·6819 (6)	−0·0054 (2)	0·025 (2)
C7	0·4096 (4)	0·6484 (6)	−0·0581 (2)	0·031 (2)
C8	0·3146 (4)	0·6466 (6)	−0·0827 (2)	0·028 (2)
C9	0·2434 (4)	0·6754 (6)	−0·0543 (2)	0·027 (2)
C9*A*	0·2663 (4)	0·7047 (5)	−0·0012 (2)	0·021 (2)
C10*A*	0·2169 (3)	0·8775 (6)	0·0722 (2)	0·019 (2)
C11	0·1202 (5)	0·7776 (7)	0·1755 (2)	0·039 (2)
C12	0·2057 (5)	0·7031 (6)	0·1773 (2)	0·038 (3)
C13	0·2784 (5)	0·7695 (6)	0·2120 (2)	0·030 (2)
C14	0·2373 (4)	0·8860 (7)	0·2323 (2)	0·030 (2)
C15	0·1404 (4)	0·8913 (7)	0·2094 (2)	0·035 (2)

the FeCp moiety was located inside the heterocyclic fold and that the dihedral angle of the thianthrene skeleton in that complex was 127·4 (3)°. In the present study, (I) was obtained by a different synthesis, *via* a photolytic monodemetallation reaction. A 1:1 mixture of conformers of the thianthrene complex with the FeCp moiety located inside and outside the heterocyclic fold was obtained. The dihedral angles of conformers *A* and *B* are 143·1 (2) and 136·3 (2)° for the in- and out-of-fold FeCp moieties. A significant increase in the dihedral angle (*ca* 7°) is thus observed for the conformer having the FeCp inside the fold which could be attributed to a repulsion between the uncoordinated arene ring and the

Table 2. *Bond lengths* (Å) *and angles* (°)

1	2	3	1—2	1—2—3
Molecule *A*				
C1	Fe		2·076 (6)	
C2	Fe		2·077 (6)	
C3	Fe		2·084 (6)	
C4	Fe		2·073 (5)	
C4*A*	Fe		2·100 (5)	
C10*A*	Fe		2·098 (6)	
C11	Fe		2·063 (6)	
C12	Fe		2·061 (6)	
C13	Fe		2·051 (6)	
C14	Fe		2·052 (6)	
C15	Fe		2·056 (6)	
F1	P	F2	1·600 (4)	179·7 (2)
F1	P	F3		90·6 (2)
F1	P	F4		90·2 (2)
F1	P	F5		89·8 (2)
F2	P	F3	1·610 (4)	89·6 (2)
F2	P	F4		90·1 (2)
F2	P	F5		90·0 (2)
F2	P	F6		89·2 (2)
F3	P	F4	1·600 (3)	90·0 (2)
F3	P	F5		179·6 (2)
F3	P	F6		90·1 (2)
F4	P	F5	1·599 (4)	89·8 (2)
F4	P	F6		179·3 (2)
F5	P	F6	1·606 (3)	90·0 (2)
F6	P	F1	1·598 (4)	90·5 (2)
C4*A*	S5	C5*A*	1·769 (5)	102·7 (2)
C5*A*	S5		1·767 (6)	
C9*A*	S10	C10*A*	1·764 (5)	102·8 (3)
C10*A*	S10		1·766 (6)	
C2	C1	C10*A*	1·392 (9)	120·3 (5)
C10*A*	C1		1·412 (8)	
C3	C2	C1	1·400 (8)	120·3 (5)
C4	C3	C2	1·406 (8)	120·3 (6)
C4*A*	C4	C3	1·419 (8)	119·6 (5)
C10*A*	C4*A*	S5	1·410 (7)	122·6 (4)
C10*A*	C4*A*	C4		119·6 (5)
S5	C4*A*	C4		117·8 (4)
C6	C5*A*	C9*A*	1·389 (7)	119·3 (5)
C6	C5*A*	S5		118·2 (4)
C9*A*	C5*A*	S5	1·393 (7)	122·3 (4)
C7	C6	C5*A*	1·377 (9)	120·7 (5)
C8	C7	C6	1·369 (8)	120·1 (5)
C9	C8	C7	1·390 (9)	119·8 (6)
C9*A*	C9	C8	1·385 (8)	120·6 (5)
S10	C9*A*	C5*A*		122·9 (4)
S10	C9*A*	C9		117·7 (4)
C5*A*	C9*A*	C9		119·3 (5)
S10	C10*A*	C1		118·4 (4)
S10	C10*A*	C4*A*		121·8 (4)
C1	C10*A*	C4*A*		119·8 (5)
C12	C11	C15	1·403 (8)	108·2 (5)
C15	C11		1·406 (9)	
C13	C12	C11	1·429 (9)	108·0 (5)
C14	C13	C12	1·400 (9)	107·4 (5)
C15	C14	C13	1·415 (8)	108·4 (6)
C11	C15	C14		108·0 (5)
Molecule *B*				
1	2	3	1—2	1—2—3
C1	Fe		2·079 (5)	
C2	Fe		2·077 (6)	
C3	Fe		2·072 (6)	
C4	Fe		2·077 (6)	
C4*A*	Fe		2·092 (5)	
C10*A*	Fe		2·081 (5)	
C11	Fe		2·055 (7)	
C12	Fe		2·038 (6)	
C13	Fe		2·042 (6)	
C14	Fe		2·054 (6)	
C15	Fe		2·052 (6)	
F1	P	F2	1·593 (3)	179·6 (2)
F1	P	F3		90·7 (2)
F1	P	F4		89·6 (2)
F1	P	F5		90·1 (2)
F2	P	F3	1·601 (3)	89·6 (2)
F2	P	F4		90·1 (2)
F2	P	F5		89·6 (2)
F2	P	F6		90·3 (2)
F3	P	F4	1·585 (4)	89·8 (2)
F3	P	F5		179·1 (2)
F3	P	F6		90·5 (2)
F4	P	F5	1·597 (3)	89·8 (2)
F4	P	F6		179·5 (2)

Table 2 (*cont.*)

1	2	3	1—2	1—2—3
F5	P	F6	1·587 (4)	89·8 (2)
F6	P	F1	1·594 (3)	90·0 (2)
C4*A*	S5	C5*A*	1·769 (5)	100·6 (2)
C5*A*	S5		1·760 (5)	
C9*A*	S10	C10*A*	1·767 (6)	100·4 (2)
C10*A*	S10		1·767 (5)	
C2	C1	C10*A*	1·392 (8)	121·0 (5)
C10*A*	C1		1·401 (8)	
C3	C2	C1	1·396 (8)	120·0 (5)
C4	C3	C2	1·392 (8)	120·5 (5)
C4*A*	C4	C3	1·423 (8)	120·2 (5)
C10*A*	C4*A*	S5	1·426 (6)	120·7 (4)
C10*A*	C4*A*	C4		118·8 (5)
S5	C4*A*	C4		120·1 (4)
C6	C5*A*	C9*A*	1·391 (8)	118·8 (5)
C6	C5*A*	S5		119·4 (4)
C9*A*	C5*A*	S5	1·388 (7)	121·7 (4)
C7	C6	C5*A*	1·387 (8)	120·9 (5)
C8	C7	C6	1·385 (8)	119·8 (6)
C9	C8	C7	1·382 (9)	119·7 (5)
C9*A*	C9	C8	1·387 (8)	120·6 (5)
S10	C9*A*	C5*A*		120·7 (4)
S10	C9*A*	C9		119·0 (4)
C5*A*	C9*A*	C9		120·3 (5)
S10	C10*A*	C1		120·2 (4)
S10	C10*A*	C4*A*		120·1 (4)
C1	C10*A*	C4*A*		119·4 (5)
C12	C11	C15	1·403 (10)	107·4 (5)
C15	C11		1·402 (9)	
C13	C12	C11	1·397 (8)	108·9 (6)
C14	C13	C12	1·410 (9)	107·3 (5)
C15	C14	C13	1·399 (8)	108·1 (5)
C11	C15	C14		108·3 (6)

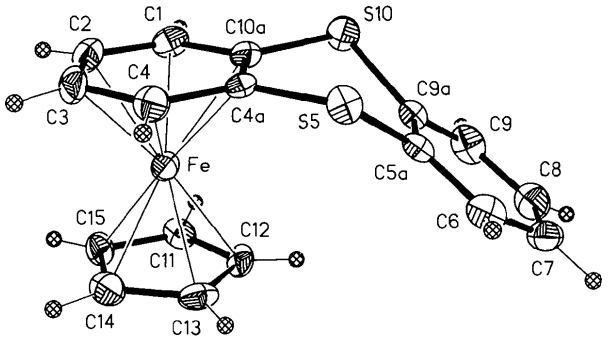

Fig. 1. 50% thermal ellipsoid drawing of molecule *A*.

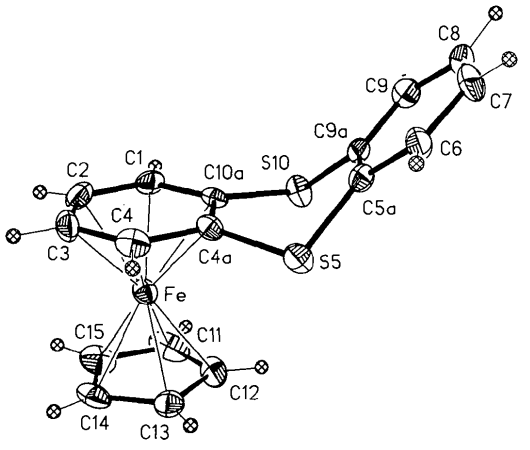

Fig. 2. 50% thermal ellipsoid drawing of molecule *B*.

Cp ring. The dihedral angle of the 2-methylthianthrene complex, for which a similar repulsion should exist, was slightly smaller than that of thianthrene (Larson, Simonsen, Martin, Smith & Puig-Torres, 1984) [127·4 (3) *vs* 128·7 (7)°]. Three other factors should be considered: (1) FeCp is an electron-withdrawing group which would tend to flatten the heterocycle, (2) the methyl substituent on the arene ring is an electron-releasing group which would exert the opposite effect, and (3) packing forces. The difference in the dihedral angles of (I) and (II) must be due to the influence of the methyl group which counteracts the FeCp complexation effect.

The method of synthesis of (I) resulted in a 1:1 ratio of two conformers, whereas the 2-methylthianthrene complex, prepared by a different synthetic route, yielded only the in-fold conformer. To ascertain whether both conformers were formed in the latter synthesis, a dynamic ^{1}H NMR study in the range of 175–370 K was carried out. The spectrum remained unchanged at all temperatures, indicating only one conformer or, perhaps, ring flipping. However, the co-crystallization of the two conformers of (I) seems to indicate that both of them produced in the reaction remain stable at and below room temperature (both synthetic routes are completed at room temperature) with no ring flipping.

The PF_6^- groups are reasonably well behaved with the P—F bond distances ranging between 1·598 (4) and 1·610 (3) Å in *A* and 1·585 (3) and 1·601 (3) Å in *B*. No F—P—F angle deviates from the octahedral geometry by more than 0·6° in *A* and 0·7° in *B*.

Currently, we are involved in further work which should clarify the stereochemistry of formation of heterocycle complexes in a double nucleophilic sub-

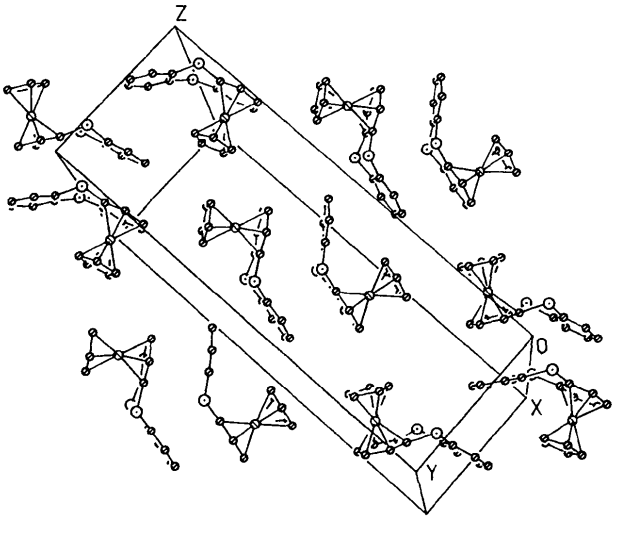

Fig. 3. Packing diagram of (I).

stitution reaction and the influence of substituents on the molecule folding.

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Relationship Between X-ray Structure and Phosphodiesterase Activity of Co^{III} Complexes: Structure of Dinitro[tris(2-aminoethyl)amine]cobalt(III) Chloride

BY JIK CHIN* AND MARC DROUIN

Department of Chemistry, McGill University, Montreal, Canada, H3A 2K6

AND ANDRÉ G. MICHEL*

Laboratoire de chimie structurale, Département de chimie, Faculté des Sciences, Université de Sherbrooke, Sherbrooke, Québec, Canada, J1K 2R1

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Abstract. $[Co(NO_2)_2(C_6H_{18}N_4)]Cl$, $M_r = 332{\cdot}6$, orthorhombic, *Pcmb*, $a = 8{\cdot}452\ (6)$, $b = 12{\cdot}0735\ (4)$, $c = 12{\cdot}1733\ (8)$ Å, $V = 1242{\cdot}23$ Å^3, $D_x = 1{\cdot}778$ Mg m^{-3}, $Z = 4$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}61$ mm^{-1}, $F(000) = 687{\cdot}80$, room temperature, final $R = 0{\cdot}024$ for 749 reflections in the range $00{\cdot}00 < 2\theta < 47{\cdot}90°$. All of the *cis* N—Co—N bond angles in the parent compound are essentially the same as the corresponding angles in $[(tren)Co(CO_3)]^+$ [tren: tris(2-aminoethyl)amine]. In sharp contrast, one of the *cis* N—Co—N bond angles increases dramatically on going from $[(cyclen)Co(NO_2)_2]^+$ to $[(cyclen)Co(CO_3)]^+$ (cyclen: 1,4,7,11-tetraazacyclododecane). Relative reactivity of $[(tren)Co(OH_2)(OH)]^{2+}$ and $[(cyclen)Co(OH_2)(OH)]^{2+}$ in hydrolyzing phosphate diesters is explained in terms of the *cis* N—Co—N bond angle.

Introduction. Currently there is much interest in designing catalysts that hydrolyze the phosphate diester backbone of DNA (Dervan & Moser, 1987). We recently showed that *cis* aquahydroxo(tetraamine)Co^{III} complexes are highly efficient at hydrolyzing phosphate diesters (Chin & Zou, 1988). Furthermore, the reactivity of Co^{III} complexes is extremely sensitive to the tetraamine ligand structure (Chin, Banaszczyk, Jubian & Zou, 1989; Chin & Banaszczyk, 1989*a*,*b*). For example, the phosphate diester bond in (1) is hydrolyzed about 50 times more rapidly than that in (2). In order to find out why (1) is so much more reactive than (2), we determined the crystal structure of (2′) (see Fig. 1).

Experimental. $[(tren)Co(NO_2)_2]Cl$ was prepared from $[(tren)CoCl_2]Cl$ following the procedure described for the preparation of $[(en)_2Co(NO_2)_2]Cl$ (Bernal, 1985). Anal. calc. for $[Co(C_6H_{18}N_4)(NO_2)_2]Cl$: C 21·68, H 5·46, N 25·39%. Found: C 22·06, H 5·25, N 25·66%.

The X-ray diffraction was performed on an orange rectangular crystal, 0·20 × 0·20 × 0·25 mm; Enraf–Nonius CAD-4 diffractometer with graphite monochromator and Mo $K\alpha$ radiation. The cell dimen-

* Authors to whom correspondence should be addressed.

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