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# THE REACTION OF SCANDIUM TRIFLUORIDE WITH CYCLOPENTADIENYL SALTS AND THE CRYSTAL AND MOLECULAR STRUCTURE OF THE TRIMER OF FLUORODICYCLOPENTADIENYLSCANDIUM

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## Summary

The reaction of $ScF_3$ with $(C_5H_5)_2Mg$ gives a mixture of $(C_5H_5)_3Sc$ and $(\eta$-$C_5H_5)_2ScF$, not pure $(C_5H_5)_3Sc$ as reported previously. The same mixture is obtained on treating $ScF_3$ with $C_5H_5Na$ in tetrahydrofuran. The two products can be separated since $(\eta$-$C_5H_5)_2ScF$ is soluble in toluene whereas $(C_5H_5)_3Sc$ is not. The structure of $(\eta$-$C_5H_5)_2ScF$ shows it to be trimeric with a planar $(Sc–F)_3$ ring (average Sc–F distance 2.046(8) Å, F–Sc–F and Sc–F–Sc angles 86.5(3) and 153.4(4)°, respectively). Crystal data: monoclinic, *Cc*, *a* 17.019(8), *b* 15.273(7), *c* 10.359(8) Å, $\beta$ 92.33(5)°, $Z = 4$; Mo-$K_\alpha$ radiation, $R = 0.076$ for 1238 observed reflections and 325 variables.

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## Introduction

In continuation of our studies on transition metal clusters containing oxygen atoms [1] we wished to investigate the reactions of $(C_5H_5)_3Sc$ with nitrogen oxides. We have suggested that $((\eta$-$C_5H_5)Sc)_nO_m$ clusters should exist [2]. Several preparations of $(C_5H_5)_3Sc$ have been described in the literature [3–6]. All suffer from problems which would be of no account if it were not for the high cost of scandium. The procedure of Birmingham and Wilkinson uses anhydrous $ScCl_3$ [3]; commercial samples of this salt invariably contain intractable oxides which interfere in the reaction with $C_5H_5Na$. Conversion of $Sc_2O_3$ into pure $ScCl_3$ is possible, but the yields are not quantitative [7]. We were attracted to the procedure of Reid and Wailes, who claimed an 83% yield of $(C_5H_5)_3Sc$ using $ScF_3$ and molten $(C_5H_5)_2Mg$ [6]. Scandium trifluoride is commercially available in high purity (uncontaminated by oxide). However when we used the product prepared and purified as described by Reid and Wailes [6] it soon became apparent that we were dealing with a mixture of compounds. Further investigation revealed that reactions between $ScF_3$ and cyclo-

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pentadienyl salts gave a mixture of $(C_5H_5)_3Sc$, an interesting trimer $((\eta\text{-}C_5H_5)_2ScF)_3$ and a small quantity of another unidentified fluoride. These results and the structure of $((\eta\text{-}C_5H_5)_2ScF)_3$ are reported here.

## Results and discussion

### *The reaction between $ScF_3$ and cyclopentadienyl salts*

The preparation of $(C_5H_5)_3Sc$ by heating $ScF_3$ with $(C_5H_5)_2Mg$ in a sealed tube at 220°C, as described by Reid and Wailes, gives a mixture of $(C_5H_5)_3Sc$ and $(\eta\text{-}C_5H_5)_2ScF$ in the ratio of approximately 2/1. The same two products are obtained when $ScF_3$ is treated with $C_5H_5Na$ in tetrahydrofuran; in this case the $(C_5H_5)_3Sc/(\eta\text{-}C_5H_5)_2ScF$ ratio is approximately 1/1. Reid and Wailes purified their material by sublimation and identified it as $(C_5H_5)_3Sc$ by infrared spectroscopy and X-ray powder pattern. In fact both $((\eta\text{-}C_5H_5)_2ScF)_3$ and $(C_5H_5)_3Sc$ sublime together, have very similar infrared spectra and similar X-ray powder patterns (though their crystal parameters are different); $(C_5H_5)_3Sc$ is yellow, $((\eta\text{-}C_5H_5)_2ScF)_3$ yellow-orange.

Mechanical separation of the two products is possible since on sublimation $(C_5H_5)_3Sc$ forms large crystals whereas $((\eta\text{-}C_5H_5)_2ScF)_3$ remains amorphous. This explains why Atwood and Smith [8] obtained single crystals of $(C_5H_5)_3Sc$ from material prepared by the method of Reid and Wailes [6]. However the most convenient separation is extraction with toluene, in which $((\eta\text{-}C_5H_5)_2ScF)_3$ is highly soluble but $(C_5H_5)_3Sc$ completely insoluble. The toluene solution repeatedly deposited crystals which analysed as $(C_5H_5)_2ScF$ and which were structurally characterised as $((\eta\text{-}C_5H_5)_2ScF)_3$ (see below), but the infrared and NMR spectra of the product clearly indicated the presence of a species which was neither $(C_5H_5)_3Sc$ nor $((\eta\text{-}C_5H_5)_2ScF)_3$. This third product showed resonances at 6.10 ($C_5H_5$), 1.32 and 0.90 ppm in the $^1H$ and at $-45.25$ ppm in the $^{19}F$ NMR spectrum (toluene-$d_8$ solution). The infrared spectrum showed a triplet of bands of descending but strong intensity at 2960, 2910 and 2845 $cm^{-1}$. The product may also have a peak at $m/e$ 511 in the mass spectrum (see below). The third product could be separated by extraction with hexane, in which it, but not $((\eta\text{-}C_5H_5)_2ScF)_3$, was slightly soluble. However we have been unable to obtain sufficient of this third product to characterise it.

Attempts to increase the yield of $(C_5H_5)_3Sc$ at the expense of $(\eta\text{-}C_5H_5)_2ScF$ by increasing the amount of cyclopentadienyl salt in the preparations were not successful. The yield of purified $(C_5H_5)_3Sc$ obtained from the reaction between $ScF_3$ and $(C_5H_5)_2Mg$ by us was 18%. Hence of the preparations described that of Birmingham and Wilkinson [3] gives the highest yield of pure $(C_5H_5)_3Sc$.

### *Properties and structure of $((\eta\text{-}C_5H_5)_2ScF)_3$*

The trimer $((\eta\text{-}C_5H_5)_2ScF)_3$ is very sensitive to traces of water. This sensitivity involves the $(\eta\text{-}C_5H_5)$ rings since the Alizarin test for fluoride in aqueous medium [9] is initially negative. Only over a period of time does the pink colour develop. This indicates a strong Sc–F bond. A further indication of this strength is the appearance of $(C_5H_5)_5Sc_3F_3^+$ ($m/e$ 517) as the peak of highest $m/e$ in the mass spectrum, rather than $(C_5H_5)_6Sc_3F_2^+$ (563). A peak at $m/e$ 511 which was always observed in the mass spectrum cannot be assigned to a fragment of $((C_5H_5)_2ScF)_3$

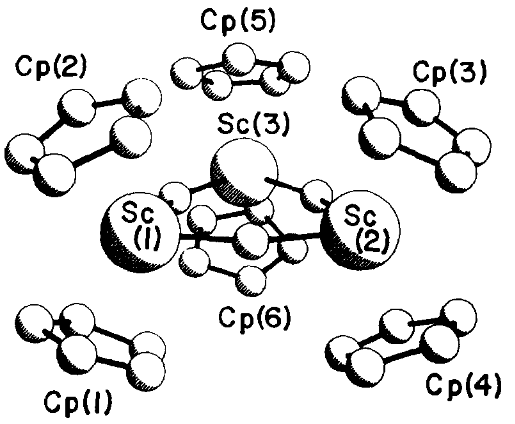

Fig. 1. The molecular structure of $((\eta\text{-}C_5H_5)_2ScF)_3$.

or $(C_5H_5)_3Sc$ and may be due to the third component of the mixture of products which is described above. The resonance at $-66.76$ ppm (relative to $CCl_3F$) in the $^{19}F$ NMR spectrum of $((\eta\text{-}C_5H_5)_2ScF)_3$ is also in the normal region for a covalent fluoride. This resonance is sharp and intense at $-75°C$ but collapses to a broad, weakly intense envelope at room temperature. This indicates that the trimer is in equilibrium with the monomer and/or other oligomers at this temperature.

The structure of $((\eta\text{-}C_5H_5)_2ScF)_3$ is shown in Fig. 1 and the important bond distances and angles in Tables 1 and 2 respectively. A complete list of bond distances and angles is provided in the Supplementary material *. The trimeric structure is the third of its type after $((\eta\text{-}C_5H_5)_2ZrO)_3$ [10] and $((\eta\text{-}C_5(CH_3)_5)_2UCl)_3$ [11]; all these trimers involve highly electropositive metals attached to highly electronegative ligands. In fact $((\eta\text{-}C_5H_5)_2ScF)_3$ is remarkably similar to $((\eta\text{-}C_5(CH_3)_5)_2UCl)_3$ [11]; the average F–Sc–F angle in the planar six-membered $(ScF)_3$ ring is 86.5(3)° compared to the analogous Cl–U–Cl angle of 84.5(3)°, and

TABLE 1

BOND DISTANCES IN $((\eta\text{-}C_5H_5)_2ScF)_3$ (Å). STANDARD DEVIATIONS ARE GIVEN IN BRACKETS

| | | | |
|---|---|---|---|
| Sc(1)–F(1) | 2.051(8) | Sc(1)–Cp(1) | 2.151 [a] |
| Sc(1)–F(3) | 2.063(8) | Sc(1)–Cp(2) | 2.225 |
| Sc(2)–F(1) | 2.040(9) | Sc(2)–Cp(3) | 2.201 |
| Sc(2)–F(2) | 2.063(8) | Sc(2)–Cp(4) | 2.175 |
| Sc(3)–F(2) | 2.026(8) | Sc(3)–Cp(5) | 2.199 |
| Sc(3)–F(3) | 2.034(9) | Sc(3)–Cp(6) | 2.196 |
| Av. | 2.046(8) | Av. | 2.191 |

[a] Cp is the centroid of the $(\eta\text{-}C_5H_5)$ ring. The numbering scheme is shown in Fig. 1.

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TABLE 2

ANGLES IN $((\eta\text{-}C_5H_5)_2ScF)_3$ (°). STANDARD DEVIATIONS ARE GIVEN IN BRACKETS

| | | | |
|---|---|---|---|
| F(1)–Sc(1)–F(3) | 86.6(3) | Sc(1)–F(3)–Sc(3) | 152.9(4) |
| F(1)–Sc(2)–F(2) | 86.2(3) | Cp(1)–Sc(1)–Cp(2) | 128.3 [a] |
| F(2)–Sc(3)–F(3) | 86.7(3) | Cp(3)–Sc(2)–Cp(4) | 129.7 |
| Sc(1)–F(1)–Sc(2) | 153.5(4) | Cp(5)–Sc(3)–Cp(6) | 130.7 |
| Sc(2)–F(2)–Sc(3) | 153.9(5) | | |

[a] Cp is the centroid of the $(\eta\text{-}C_5H_5)$ ring. The numbering scheme is shown in Fig. 1.

the average Sc–F–Sc angle is 153.4(4)° compared to the U–Cl–U angle of 155.5(4)°. On the other hand $((\eta\text{-}C_5H_5)_2ZrO)_3$ has an O–Zr–O angle of 97.4(2)° and a Zr–O–Zr angle of 142.5(3)°. All three trimers have M–X distances which do not vary significantly around the ring: Sc–F 2.046(17); U–Cl 2.901(16) [11]; Zr–O 1.958(8) Å [10], where the figure in brackets represents the maximum deviation from the average of the six distances in each case. The A–M–A angle for a $Cp_2MA_2$ complex with zero *d* electrons usually lies in the range 94–97° [12]. One reason for the difference between $((\eta\text{-}C_5H_5)_2ZrO)_3$ on the one-hand and $((\eta\text{-}C_5H_5)_2ScF)_3$ and $((\eta\text{-}C_5(CH_3)_5UCl)_3$ on the other is that in the former $\pi$-donation of electrons from oxygen to the empty $1a_1$ orbital on the $(\eta\text{-}C_5H_5)_2Zr$ fragment is possible. The Zr–O distance is in fact much shorter than the sum of the covalent radii, indicating such $\pi$-bonding to be present. Hence the in-plane angles of the $(ZrO)_3$ unit are arranged to maximise in-plane $\pi$-bonding. In $((\eta\text{-}C_5H_5)_2ScF)_3$ and $((\eta\text{-}C_5(CH_3)_5)_2UCl)_3$, $\pi$-donation by the univalent but bridging halides is unlikely to be important, and the M–X distances are close to or greater than the sum of the covalent radii. The in-plane angles are therefore arranged to maximise the bridging capabilities of the halide. The trimeric nature of $((\eta\text{-}C_5H_5)_2ScF)_3$ as compared to the dimeric $((\eta\text{-}C_5H_5)_2ScCl)_2$ [13] is in accord with the observation that fluorine bridged compounds strongly prefer to form trimers but seldom dimers, in contrast to other bridging atoms [14–17].

The average Sc–F distance (2.046(8) Å) is similar to the distances in $ScF_3$ (2.012(5) Å [18]) and in ScOF (2.106(4) Å [19,20]), these being the only other accurately determined Sc–F distances. Both the average Sc–Cp distance (the distance to the centroid of the $(\eta\text{-}C_5H_5)$ ring) of 2.191 Å and the average Cp–Sc–Cp angle (129.6°) are similar to those in $((\eta\text{-}C_5H_5)_2ScCl)_2$ (2.18 Å and 131.8° [13]). The average Cp–Zr–Cp angle in $((\eta\text{-}C_5H_5)_2ZrO)_3$ is 123.8° [10].

## Experimental

All operations were conducted under argon or vacuum using standard vacuum line and Schlenk tube techniques. Solvents were pre-dried, stored under vacuum over methyllithium and distilled in vacuum. Scandium trifluoride was purchased from Alfa Inorganics, all other chemicals were reagent grade. Instruments used were a Perkin–Elmer 683 infrared spectrophotometer, Varian XL-200 NMR spectrometer and Picker FACS 1 diffractometer. Microanalyses were by the Analytische Laboratorien, Engelskirchen, W. Germany.

### *Tris(fluorodicyclopentadienylscandium)*

To a solution of $(C_5H_5)Na$ (0.57 mol) in tetrahydrofuran (150 $cm^3$) was added $ScF_3$ (10 g, 0.1 mol). The mixture was refluxed for 12 h, giving a pink solution and a pink precipitate. The solvent was removed in vacuum and the product dried by heating at 110°C under dynamic vacuum for 7 h. The residue showed resonances at 1.00 (weak, broad), 1.40, 2.71 (w), 3.48 (multiplet), 3.89 (very weak) and 6.08, 6.13 ppm (intense singlets on a very broad intense envelope) in its NMR spectrum (tetrahydrofuran-$d_8$ as solvent). A mixture of $(C_5H_5)_3Sc$ and $((\eta\text{-}C_5H_5)_2ScF)_3$ was then sublimed out of the residue at 225°C under vacuum. The NMR of the sublimate (measured in tetrahydrofuran-$d_8$) was a composite of the spectra of $((\eta\text{-}C_5H_5)_2ScF)_3$, the unknown product described in the Results and discussion above, and $(C_5H_5)_3Sc$; for the latter compound a resonance was found at 6.33 ppm (singlet) in tetrahydrofuran-$d_8$ as solvent. The mixture was extracted twice with 130 $cm^3$ portions of toluene giving bright yellow $(C_5H_5)_3Sc$ (2.0 g, 0.086 mol, 9%) as the residue and a yellow-orange solution. On reduction of the volume of this solution to approx. 30 $cm^3$ crystals of $((\eta\text{-}C_5H_5)_2ScF)_3$ (2.6 g, 0.044 mol, 14%) formed. This material was suitable for further chemical investigation, but for the analytical and spectroscopic investigations it was freed from impurity by further recrystallisation from toluene/hexane (2/1). Anal. Found: C, 61.4; H, 5.6; F, 9.0%. $C_{30}H_{30}F_3Sc_3$ calcd.: C, 61.9; H, 5.2; F, 9.8%. NMR (in toluene-$d_8$) $^1H$:6.16 ppm, singlet, $(\eta\text{-}C_5H_5)$; $^{13}C$:113.66 ppm, singlet $(\eta\text{-}C_5H_5)$; $^{19}F$: − 66.76 ppm, singlet. Mass spectrum: $m/e$ 517 $((C_5H_5)_5Sc_3F_3^+)$; 369 $((C_5H_5)_4Sc_2F^+)$; 323 $((C_5H_5)_3Sc_2F_2^+)$; 194 $((C_5H_5)_2ScF^+)$; 175 $((C_5H_5)_2Sc^+)$; 129 $((C_5H_5)ScF^+)$. Infrared spectrum: two very weak absorptions at 3095 and 2945 $cm^{-1}$ ($\nu$(C–H)), 1455 $cm^{-1}$ (weak, ring deform.), 1020 $cm^{-1}$ (weak, broad ($\delta$(CCH)) and 790 $cm^{-1}$ (medium, broad, $\pi$(CH)).

### *Determination of the crystal and molecular structure of $((\eta\text{-}C_5H_5)_2ScF)_3$*

Crystals obtained as described above were coated with Apiezon grease, mounted in sealed tubes and examined by photographic and diffractometric methods. Crystal data: $C_{30}H_{30}F_3Sc_3$, $M = 582.5$; monoclinic, $Cc$, $a$ 17.019(8), $b$ 15.273(7), $c$ 10.359(8) Å, $\beta$ 92.33(5)°, $Z = 4$; $D_c = 1.44$; crystal dimensions $0.4 \times 0.4 \times 0.2$ mm; Mo-$K_\alpha$ radiation, $\lambda$ 0.71073 Å; $\mu$ 0.75 $mm^{-1}$. The intensities of 1760 unique reflections ($2\theta < 45°$) were measured by the $\omega$–$2\theta$ scan technique at room temperature; 1238 reflections were judged as observed ($I \geqslant 2\sigma(I)$) and used for the structure determination. No absorption correction was applied. The final $R$ $[= \Sigma\Delta|F|/\Sigma|F|]$ was 0.076, $R_w$ $[= (\Sigma w(\Delta F)^2/\Sigma w F^2)^{\frac{1}{2}}]$ 0.071 for 325 variables. The maximum residual intensity in the final difference map was 1.1 e $Å^{-3}$ and the minimum was 0.5 e $Å^{-3}$.

Preliminary positions for the scandium atoms were found using the MULTAN-80 program [21]. The position of these atoms were used in the phasing of a Fourier synthesis and the positions of all non-hydrogen atoms were determined by successive Fourier, difference Fourier and partial refinement techniques. The structure was refined by a standard least-squares technique, minimizing the function $\Sigma w(\Delta F)^2$ with a weighting scheme of the form $w = 1/(\sigma(F)^2 + kF^2)$ based on counting statistics. Scattering factors were taken from International Tables for X-ray Crystallography [22] and were corrected for both the real and imaginary parts of the anomalous dispersion where appropriate. Final refinement was with anisotropic thermal parameters for all atoms except hydrogen. The hydrogen atoms were allowed to ride on the carbon atoms with a fixed C–H distance of 1.08 Å, $sp^2$ hybridisation at the C atom and a single, variable isotropic thermal parameters for all H atoms. During the final refinement it was noticed that the carbon atoms of rings (1) and (6) had unusually large thermal parameters and the C–C distances and angles in these rings are abnormal. It appears that these rings are disordered. No simple disorder was evident from the difference map and after several attempts to model a disorder it was concluded that the introduction of additional parameters could not be justified by the data. The somewhat high final *R* value is partly due to

TABLE 3

POSITIONAL PARAMETERS OF THE NON-HYDROGEN ATOMS IN $((\eta\text{-}C_5H_5)_2ScF)_3$ (×1000)

| Atom [a] | *x/a* | *y/b* | *z/c* |
|---|---|---|---|
| Sc(1) | 417.2 | 339.3(2) | 542.6 |
| Sc(2) | 183.1(2) | 335.7(2) | 519.9(3) |
| Sc(3) | 299.3(2) | 185.4(1) | 777.5(3) |
| F(1) | 299.9(5) | 359.2(5) | 499.4(7) |
| F(2) | 218.2(5) | 249.1(5) | 663.9(8) |
| F(3) | 382.1(5) | 253.3(5) | 682.6(7) |
| C(11) | 481(1) | 328(2) | 339(2) |
| C(12) | 416(1) | 289(3) | 317(2) |
| C(13) | 422(2) | 216(2) | 392(4) |
| C(14) | 497(3) | 219(3) | 467(3) |
| C(15) | 530(1) | 292(2) | 425(4) |
| C(21) | 500(1) | 475(1) | 557(2) |
| C(22) | 423(1) | 502(1) | 580(2) |
| C(23) | 405(1) | 464(1) | 698(2) |
| C(24) | 467(1) | 416(1) | 746(2) |
| C(25) | 525(1) | 424(1) | 663(2) |
| C(31) | 304(1) | 310(1) | 930(2) |
| C(32) | 232(1) | 267(1) | 950(2) |
| C(33) | 245(1) | 187(1) | 998(2) |
| C(34) | 327(1) | 176(1) | 1018(2) |
| C(35) | 366(1) | 250(1) | 978(2) |
| C(41) | 306(3) | 73(1) | 608(2) |
| C(42) | 367(2) | 55(1) | 689(3) |
| C(43) | 341(2) | 30(1) | 807(3) |
| C(44) | 262(2) | 28(1) | 800(2) |
| C(45) | 241(1) | 54(1) | 677(2) |
| C(51) | 182(1) | 444(1) | 699(2) |
| C(52) | 108(1) | 400(1) | 699(2) |
| C(53) | 69(1) | 424(2) | 587(2) |
| C(54) | 113(2) | 480(2) | 515(2) |
| C(55) | 184(1) | 492(1) | 591(2) |
| C(61) | 197(2) | 218(2) | 358(4) |
| C(62) | 121(3) | 208(2) | 418(3) |
| C(63) | 78(1) | 281(2) | 375(2) |
| C(64) | 126(3) | 330(2) | 296(3) |
| C(65) | 187(3) | 292(3) | 289(3) |

[a] Standard deviations are given in brackets. The numbering scheme for the Sc and F atoms is shown in Fig. 1. The atoms C(11)–C(15) define Cp(1), C(21)–C(25) Cp(2), C(51)–C(55) Cp(3), C(61)–C(65) Cp(4), C(31)–C(35) Cp(5) and C(41)–C(45) Cp(6).

this disorder. The SHELX-76 program package [23] was used for the refinement; the diagrams were by PLUTO [24].

The positional parameters for the non-hydrogen atoms of $((\eta\text{-}C_5H_5)_2ScF)_3$ are given in Table 3. Tables of hydrogen atom positions, thermal parameters, equations of some mean planes, comprehensive lists of bond distances and angles and a table of $|F_0|$ and $|F_c|$ are available as supplementary material.

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