Synthesis and crystal structures of the chromium(II) and chromium(III) hexafluoroacetylacetonate complexes *trans*-Cr(hfac)₂(thf)₂ and Cr(hfac)₃

Yoshiko Harada, Gregory S. Girolami *

School of Chemical Sciences, University of Illinois at Urbana-Champaign, 600 South Mathews Avenue, Urbana, IL 61801, United States

Abstract

Treatment of a tetrahydrofuran solution of $CrCl_2(thf)$ with Na(hfac), hfac = 1,1,1,5,5,5-hexafluoroacetylacetonate, followed by crystallization from diethyl ether, affords the six-coordinate chromium(II) complex $Cr(hfac)_2(thf)_2$. The crystal structures of $Cr(hfac)_2(thf)_2$ and the chromium(III) complex $Cr(hfac)_3$ have been determined by single-crystal X-ray diffraction. $Cr(hfac)_2(thf)_2$ adopts a trans octahedral geometry, in which the Cr–O(hfac) and Cr–O(thf) distances are 1.936(3) and 2.019(6) Å, respectively. $Cr(hfac)_3$ is an octahedral compound with a Cr–O distance of 1.943(5) Å. Structural comparisons with related molecules are given.
© 2006 Elsevier Ltd. All rights reserved.

1. Introduction

Transition metal β-diketonates are important compounds in inorganic chemistry and in materials science owing to their usefulness as CVD precursors [1,2] and as building blocks for molecule-based magnets [3–5]. Hexafluoroacetylacetonate (hfac) derivatives of the transition metals are especially useful because the fluorine substituents lend the complexes increased solubility, volatility, and Lewis acidity [6–9]. For example, Pd(hfac)₂ is an excellent precursor for the deposition of palladium metal by CVD [10,11], and Mn(hfac)₂ reacts with nitroxide spin radicals to make one-dimensional molecule-based magnets [3]. Owing to their importance, metal β-diketonates have been extensively investigated, but to date no structural information has been reported for chromium hexafluoroacetylacetonates. We report here the structures of a new chromium(II) hfac complex, bis(hexafluoroacetylacetonato)bis(tetrahydrofuran)-chromium(II), Cr(hfac)₂(thf)₂, as well as the previously known chromium(III) complex tris(hexafluoroacetylacetonato)chromium(III), Cr(hfac)₃.

2. Results and discussion

2.1. Synthesis of Cr(hfac)₂(thf)₂

Treatment of $CrCl_2(thf)$ with sodium 1,1,1,5,5,5-hexafluoroacetylacetonate, Na(hfac), in tetrahydrofuran at room temperature, followed by recrystallization from diethyl ether, affords dark reddish brown crystals of the new high-spin chromium(II) complex $Cr(hfac)_2(thf)_2$ (**1**). The crystals of **1** easily oxidize in air and turn green.

$$CrCl_2(thf) + 2Na(hfac) + thf \rightarrow \underset{\mathbf{1}}{Cr(hfac)_2(thf)_2} + 2NaCl$$
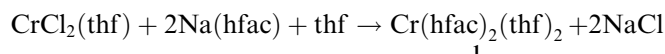
Attempts to synthesize $Cr(hfac)_2(thf)_2$ from the bis(trimethylsilyl)amido complex $Cr[N(SiMe_3)_2]_2(thf)_2$ and hexafluoroacetylacetone in thf afford instead the air-stable chromium(III) complex $Cr(hfac)_3$ (**2**) as maroon needles, after crystallization from diethyl ether or pentane. Evidently, hexafluoroacetylacetone is a strong enough acid (oxidant) to convert chromium(II) to chromium(III), probably with formation of H_2:

$$Cr[N(SiMe_3)_2]_2(thf)_2 + 3Hhfac$$
$$\rightarrow \underset{\mathbf{2}}{Cr(hfac)_3} + 2HN(SiMe_3)_2 + H_2 + 2thf$$
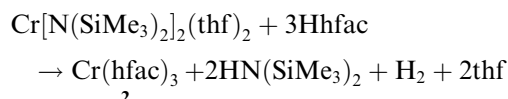
* Corresponding author. Tel.: +1 217 333 2729; fax: +1 217 244 3186.
 E-mail address: girolami@scs.uiuc.edu (G.S. Girolami).

Compound **2** has been reported previously by Sievers and Morris [12–14]. It is usually described as a green crystalline material [12,14,15], but is evidently dichroic and the crystals also can appear as maroon-colored.

The infrared spectrum of $Cr(hfac)_3$ matches that reported previously [13]. The spectrum of $Cr(hfac)_2(thf)_2$ is very similar to that of $Cr(hfac)_3$ except that it exhibits additional bands at 1029, 916, 872, and 467 cm^{-1} due to the thf ligands.

2.2. Crystal structures of Cr(hfac)₂(thf)₂ and Cr(hfac)₃

Compound **1** crystallizes in the tetragonal space group $P4_2/mnm$, with two molecules per unit cell. The crystal data are summarized in Table 1, selected distances and angles are given in Table 2, and the molecular geometry of **1** is shown in Fig. 1. The chromium center, which resides on a site of *mmm* symmetry, is trans octahedral, with the two bidentate hfac ligands in the equatorial plane and the two thf molecules in the axial positions. The Cr–O(hfac) distance of 1.936(3) Å is slightly shorter than the two Cr–O distances of 1.984(3) and 1.979(3) Å present in the square-planar acetylacetonate complex $Cr(acac)_2$ [16]. The 92.7° O–Cr–O bite angle of $Cr(hfac)_2(thf)_2$, on the other hand, is greater than the corresponding 90.3(1)° bite angle of $Cr(acac)_2$ [16]. The Cr–O(thf) distance in **1** of 2.019(6) Å is only slightly longer than the Cr–O(hfac) distances. A large tetragonal distortion is not expected in this complex because no Jahn–Teller effect is operative: the axial ligands are chemically different from the equatorial ligands, and, perforce, the electronic symmetry is already broken.

The structure of $Cr(hfac)_2(thf)_2$ is very similar to those of several reported $Mn(hfac)_2L_2$ compounds, the principal difference being that the M–O(hfac) distances are about 0.15–0.2 Å shorter in **1**, as expected for high-spin Cr(II) and Mn(II) species. For example, the Mn–O(hfac) lengths are 2.120(3)–2.145(4) Å in $Mn(hfac)_2(proxyl)_2$ (proxyl =

Table 1
Crystallographic data for Cr(hfac)₂(thf)₂ and Cr(hfac)₃

	CrC₁₈H₁₈F₁₂O₆	CrC₁₅H₃F₁₈O₆
Space group	$P4_2/mnm$	$P\bar{3}c1$
Unit cell dimensions		
a (Å)	8.5111(7)	18.0862(11)
b (Å)	8.5111(7)	18.0862(11)
c (Å)	16.585(2)	12.0790(11)
α (°)	90	90
β (°)	90	90
γ (°)	90	120
V (Å³)	1201.4(2)	3421.8(4)
Z	2	6
M_w	610.32	673.17
ρ_{calc} (g cm⁻³)	1.687	1.960
λ (Å)	0.71073	0.71073
μ_{calc} (cm⁻¹)	6.01	6.80
R_1	0.1325	0.1388
wR_2	0.1856	0.1610
T (°C)	−75	−75

$R_1 = \sum(||F_o| - |F_c||) / \sum |F_o|$, $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$.

Table 2
Selected bond distances (Å) and bond angles (°) for Cr(hfac)₂(thf)₂[a]

Distances			
Cr(1)–O(1)	1.936(3)	C(1)–C(2)	1.510(8)
Cr(1)–O(2)	2.019(6)	C(2)–C(3)	1.381(6)
O(1)–C(2)	1.293(6)	O(2)–C(4)	1.438(9)
C(1)–F(1)	1.312(5)	C(4)–C(5)	1.44(2)
C(1)–F(2)	1.327(7)		
Angles			
O(1)–Cr(1)–O(1)′	180.0	F(1)–C(1)–C(2)	111.8(3)
O(1)–Cr(1)–O(1)″	92.7(2)	F(2)–C(1)–C(2)	113.6(6)
O(1)–Cr(1)–O(1)‴	87.3(2)	O(1)–C(2)–C(3)	127.1(5)
O(1)–Cr(1)–O(2)	90.0	O(1)–C(2)–C(1)	111.7(5)
O(2)–Cr(1)–O(2)	180.0	C(3)–C(2)–C(1)	121.1(5)
C(2)–O(1)–Cr(1)	125.2(3)	C(4)–O(2)–Cr(1)	125.4(5)
F(1)–C(1)–F(2)	106.0(4)	C(5)–C(4)–O(2)	107.6(9)

[a] Primed atoms are related to the unprimed atoms by the transformation $(-x, -y, -z)$, doubly primed atoms by $(-x, -y, z)$, and triply primed atoms by $(x, y, -z)$.
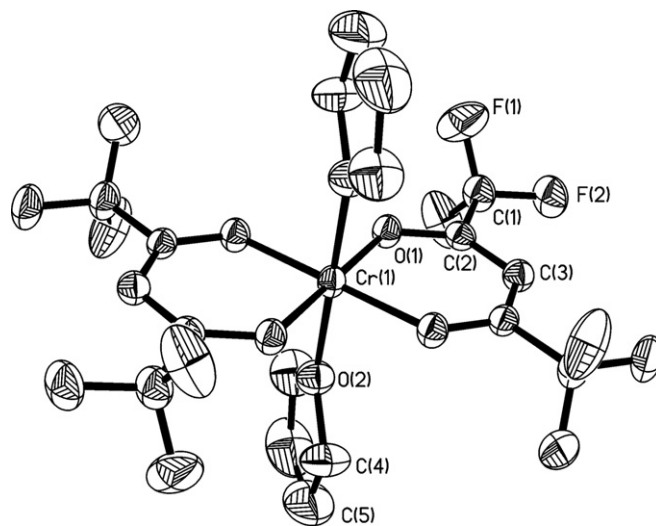

Fig. 1. X-ray crystal structure of Cr(hfac)₂(thf)₂ (**1**). The 30% probability density surfaces are shown. Hydrogen atoms have been removed for clarity.

2,2,5,5-tetramethylpyrrolidinyl-1-oxy) [17], 2.120(4)–2.160(3) Å in $Mn(hfac)_2(tempo)_2$ (tempo = 2,2,6,6-tetramethylpiperidinyl-1-oxy) [17], 2.169(3)–2.212(3) Å in $Mn(hfac)_2(Him)_2$ (Him = imidazole) [18], and 2.171(2)–2.143(3) Å in $Mn(hfac)_2(NOPy)_2$ (NOPy = 4-(N-oxy-N-tert-butylamino)pyridine) [19].

Crystals of the chromium(III) complex **2**, grown by sublimation, conform to the trigonal space group $P\bar{3}c1$ with six molecules per unit cell, but are twinned by merohedry. Because the volumes of the two twin individuals were very nearly equal (volume fractions of 0.547 and 0.453), the diffraction pattern conforms very closely to that expected for hexagonal symmetry under the space group $P6_3/mcm$. Interestingly, the X-ray diffraction pattern of **2** has previously been indexed to the space group $P6_3/mmc$ but with different cell axes (a = 21.2 and c = 12.4 Å vs. our finding of a = 18.1 and c = 12.1 Å) [20]. The different apparent space group and cell dimensions probably are consequences

of polymorphism. That polymorphism is likely shown by the fact that crystals of $Cr(hfac)_3$ are not isomorphous with those of $Fe(hfac)_3$ [21] and $Mn(hfac)_3$ [22]. The latter two complexes crystallize in the monoclinic crystal system, space group $P2_1/c$.

The crystal data for **2** are summarized in Table 1 and the structure is shown in Fig. 2. There are two independent chromium centers in the asymmetric unit, one residing on a site of $\bar{3}$ symmetry and the other on a site of 3 symmetry. We will present the results for the latter molecule, which had the smaller atomic displacement parameters. $Cr(hfac)_3$ is, as expected, an octahedral complex with three equivalent hfac ligands. The two Cr–O distances of 1.943(5) and 1.955(5) Å are very similar to the 1.936(3) Å Cr–O(hfac) distance in the chromium(II) complex **1**; the bite angles of the hfac ligands in **2** are 90.8(2)° (Table 3).
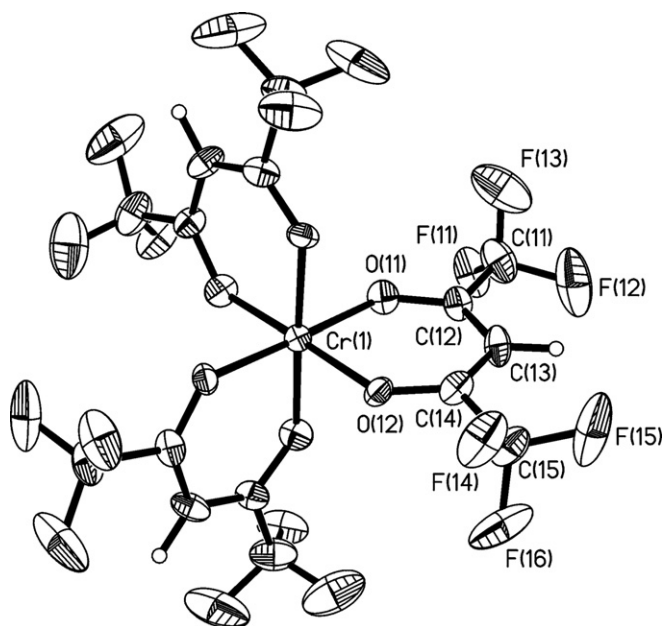

Fig. 2. X-ray crystal structure of $Cr(hfac)_3$ (**2**). The 30% probability density surfaces are shown, except for the hydrogen atoms, which are represented by arbitrarily-sized spheres.

Table 3
Selected bond distances (Å) and bond angles (°) for $Cr(hfac)_3$[a]

Distances

Cr(1)–O(11)	1.943(5)	C(11)–C(12)	1.483(13)
Cr(1)–O(12)	1.955(5)	C(12)–C(13)	1.373(10)
O(11)–C(12)	1.278(8)	C(13)–C(14)	1.381(10)
O(12)–C(14)	1.249(9)	C(14)–C(15)	1.556(12)
C(11)–F(13)	1.267(12)	C(15)–F(15)	1.295(10)
C(11)–F(11)	1.287(13)	C(15)–F(16)	1.305(10)
C(11)–F(12)	1.347(13)	C(15)–F(14)	1.326(11)

Angles

O(11)–Cr(1)–O(11)′	90.5(2)	C(12)–C(13)–C(14)	122.2(7)
O(11)–Cr(1)–O(12)	90.8(2)	O(12)–C(14)–C(13)	128.8(7)
O(12)–Cr(1)–O(11)″	178.5(2)	O(12)–C(14)–C(15)	113.2(8)
O(12)–Cr(1)–O(12)′	90.0(2)		

[a] Primed atoms are related to the unprimed atoms by the transformation $(-x + y + 1, -x + 1, z)$, and doubly primed atoms by $(-y + 1, x - y, z)$.

The structure of **2** deduced crystallographically is very similar to that determined previously by electron diffraction. In the gas phase, $Cr(hfac)_3$ adopts a slightly distorted octahedral structure with a Cr–O distance of 1.957(8) Å and an O–Cr–O angle of 87.0° [23]. In the analogous chromium(III) acetylacetonate compound $Cr(acac)_3$ [24], the Cr–O distances lie between 1.942(7) and 1.959(7) Å, and the average bite angle is 91.1°.

The structure is also similar to that of tris(β-diketonate) complexes of other first-row metals. The structure of $Fe(hfac)_3$ exhibits Fe–O lengths of 1.969–2.021(10) Å, and O–Fe–O angles of 86.5–87.6(4)° [21]. In the acetylacetonate derivative $Fe(acac)_3$, the Fe–O distances lie between 1.986 and 2.004(6) Å and the O–Fe–O angles are 86.7–87.3(3)° [25].

Unlike $Cr(hfac)_3$, the manganese complex $Mn(hfac)_3$ is a tetragonally-elongated octahedron owing to a Jahn–Teller effect [22]. The long Mn–O distances are 2.141(3) and 2.147(3) Å whereas the short Mn–O distances are between 1.906(2) and 1.937(3) Å. The average O–Mn–O bite angle is 88.8°. At least two polymorphs of the acetylacetonate analog $Mn(acac)_3$ are known: one in which the molecules are axially compressed [26], and one in which they are axially elongated [27].

3. Experimental

All synthetic procedures were carried out in vacuum or under argon. Tetrahydrofuran, toluene, diethyl ether, and pentane were distilled from sodium/benzophenone. Sodium hydride (Aldrich) was used without further purification. Hexafluoroacetylacetone (Oakwood) was degassed by a freeze-pump-thaw method. The starting materials $CrCl_2(thf)$ [28] and $Cr[N(SiMe_3)_2]_2(thf)_2$ [29] were prepared as previously described. The IR spectra were recorded as Nujol mulls between KBr plates on a Nicolet Impact 410 FTIR instrument. Elemental analyses were performed by the University of Illinois Microanalytical Laboratory. The crystallographic data were collected by the George L. Clark X-ray Laboratory at the University of Illinois. Magnetic moments were determined by the Evans method [30].

3.1. Sodium hexafluoroacetylacetonate

A 60% (w/w) suspension of sodium hydride in mineral oil (5.51 g, 0.138 mol) was washed with toluene (60 mL), and the resulting solid was suspended in tetrahydrofuran (50 mL). To this suspension was added hexafluoroacetylacetone (8.00 mL, 0.0565 mol), and the reaction mixture was stirred at room temperature for 40 min. The yellow-brown solution was filtered, and the solid residue was washed with additional tetrahydrofuran (22 mL). The filtrate and the washings were combined and taken to dryness under vacuum. The yellow-white solid was washed with pentane (100 mL) and dried under vacuum to afford a white solid. Yield: 12.0 g (84%). *Anal.* Calc. for Na(hfac) · 0.33thf: C, 29.9; H, 1.46. Found: C, 29.8; H, 1.50%. IR (cm^{-1}): 1672

m, 1556 m, 1533 m, 1514 m, 1488 m, 1318 w, 1261 s, 1202 s, 1144, s, 1079 w, 1046 w, 893 w, 800 m, 795 m, 767 w, 740 w, 665 m, 580 m, 525 w.

3.2. Bis(hexafluoroacetylacetonato)bis(tetrahydrofuran) chromium(II)

To a solution of $CrCl_2(thf)$ (2.18 g, 11.2 mmol) in tetrahydrofuran (30 mL) was added a solution of Na(hfac) · 0.33thf (5.55 g, 21.8 mmol) in tetrahydrofuran (30 mL). The initially pale blue solution changed to dark brown. The reaction mixture was stirred overnight at room temperature. The solvent was removed under vacuum, and the resulting residue was extracted with diethyl ether (150 mL). The extract was filtered, and the filtrate was cooled to $-20\,°C$ to afford dark brown crystals. Yield: 1.67 g (24%). M.p.: 117–118 °C. *Anal.* Calc. for $CrC_{18}H_{18}F_{12}O_6$: C, 35.4; H, 2.97; Cr, 8.52. Found: C, 35.3; H, 3.60; Cr, 8.46%. IR (cm^{-1}): 1641 m, 1615 m, 1564 w, 1511 w, 1442 s, 1366 w, 1348 w, 1255 s, 1225 s, 1173 s, 1136 s, 1029 m, 916 m, 872 m, 810 m, 742 m, 688 m, 613 w, 580 m, 534 w, 478 sh, 467 m. μ_{eff} (thf): 4.6 μ_B.

3.3. Tris(hexafluoroacetylacetonato)chromium(III)

To a solution of $Cr[N(SiMe_3)_2]_2(thf)_2$ (2.19 g, 4.23 mmol) in diethyl ether (120 mL) at $-78\,°C$ was added hexafluoroacetylacetone (1.2 mL, 8.48 mmol) dropwise over 10 min. The initially blue-grey solution turned red-brown. The solution was warmed to room temperature, filtered, concentrated to ∼40 mL, and cooled to $-20\,°C$. The resulting red needles were isolated; an additional crop of crystals could be obtained by concentrating and cooling the supernatant. Yield: 0.51 g (26%). M.p.: 92–93 °C. The identity of the product was determined by IR and crystallography. IR (cm^{-1}): 3156 w, 1635 m, 1614 m, 1567 w, 1541 w, 1442 s, 1376 w, 1365 w, 1259 s, 1224 s, 1164 s, 1148 s, 1108 m, 916 m, 872 w, 814 m, 748 m, 678 m, 598 m, 534 w.

3.4. Crystallographic studies [31]

Single crystals of $Cr(hfac)_2(thf)_2$, (**1**), grown from diethyl ether, were mounted on glass fibers with Paratone-N oil (Exxon) and immediately cooled to $-75\,°C$ in a cold nitrogen gas stream on the diffractometer. (Single crystals of $Cr(hfac)_3$, (**2**), grown by sublimation in vacuum, were treated similarly. Subsequent comments in parentheses will refer to this compound.) Standard peak search and indexing procedures, followed by least squares refinement, yielded the cell dimensions given in Table 1. The measured intensities were reduced to structure factor amplitudes and their esd's by correction for background and Lorentz and polarization effects. Systematic absences for $0kl$ ($k+l \neq 2n$) were consistent with space groups $P4_2/mnm$, $P4_2nm$, and $P\bar{4}n2$; the centrosymmetric space group $P4_2/mnm$ was chosen and was eventually proven correct upon successful refinement of the proposed model. Systemati-

cally absent reflections were deleted and symmetry-equivalent reflections were averaged to yield the data set used to solve the structure. (For **2**, systematic absences for $h\bar{h}0l$ ($l \neq 2n$) were consistent with the trigonal groups $P3c1$ and $P\bar{3}c1$, and the hexagonal groups $P6_3cm$, $P\bar{6}c2$, and $P6_3/mcm$. The diffraction pattern conformed closely to the Laue group $6/mmm$, but no satisfactory solution could be found in any of the hexagonal space groups. Eventually, a satisfactory solution was found in the space group $P\bar{3}c1$.)

The structure of **1** was solved by direct methods, and positions for all the non-hydrogen atoms were deduced from an E-map (SHELXTL). (For **2**, Patterson methods led to a refinable model consisting of two independent chromium atoms. Subsequent least squares refinement and difference Fourier calculations revealed the positions of many of the remaining non-hydrogen atoms. Some of the expected non-hydrogen atoms, however, did not surface in the difference maps, and suitable atoms were added, with bond distance restraints, to complete the molecules.)

The analytical approximations to the scattering factors were used, and all structure factors were corrected for both real and imaginary components of anomalous dispersion. All 832 (2022) unique data were used in the least squares refinement. The quantity minimized by the least squares program was $\sum w(F_o^2 - F_c^2)^2$, where $w = [(\sigma(F_o^2))^2 + (0.0852P)^2 + 0.6505P]^{-1}$ and $P = (F_o^2 + 2F_c^2)/3$. (For **2**, $w = [(\sigma(F_o^2))^2 + (0.0749P)^2]^{-1}$.)

(For **2**, despite the fact that all of the non-hydrogen atoms had been located, wR_2 remained above 0.65 and the "most disagreeable reflections" list was composed entirely of reflections for which $|F_c/F_c(max)| < 0.03$ and $F_o^2 \gg F_c^2$. This result suggested that the crystal was twinned by merohedry. An investigation of possible twin laws eventually revealed one that led to a substantial reduction in the weighted residual. The twin law relates hkl to $hk\bar{l}$, which corresponds to reflection through the $\mathbf{a}*\mathbf{b}*$ plane. The intensities were calculated from the expression $F_c^2 = vF_c(hkl)^2 + (1-v)F_c(hk\bar{l})^2$, where v is the volume fraction of one of the twin individuals. The volume fraction of the major twin individual refined to 0.547(3). At this point, all restraints on atom positions were lifted).

In the final cycle of least squares, independent anisotropic displacement factors were refined for the non-hydrogen atoms. Hydrogen atoms were readily apparent among the largest peaks in the difference maps, and their locations were refined independently. (For **2**, hydrogen atoms were readily apparent among the largest peaks in the difference maps, but they tended to refine to give unreasonable distances, so they were fixed in "idealized" positions with C–H = 0.95 Å.) The isotropic displacement parameters for the hydrogen atoms were set equal to 1.2 times U_{eq} for the attached carbon atom. Although corrections for crystal decay were unnecessary, an empirical absorption correction was applied (SADABS), the maximum and minimum transmission factors being 0.970 and 0.931 (0.998 and 0.937). Successful convergence was indicated by the maximum shift/error of 0.002 (0.001) for the last cycle.

Final refinement parameters are given in Table 1. The largest peak in the final Fourier difference map (0.35 e Å^{-3}) was located 0.97 Å from Cr1. (For **2**, the largest peak in the final Fourier difference map (0.39 e Å^{-3}) was located 2.1 Å from O21, F23, and C22.) A final analysis of variance between observed and calculated structure factors showed no apparent errors.

Acknowledgements

Appendix A. Supplementary material

CCDC 626676 and 626677 contain the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2006.12.007.

References

[1] T. Maruyama, H. Akagi, J. Electrochem. Soc. 143 (1996) 1955.
[2] S. Gu, P. Atanasova, M.J. Hampden-Smith, T.T. Kodas, Thin Solid Films 340 (1999) 45.
[3] A. Caneschi, D. Gatteschi, P. Rey, R. Sessoli, Inorg. Chem. 27 (1988) 1756.
[4] A. Caneschi, D. Gatteschi, J.P. Renard, P. Rey, R. Sessoli, Inorg. Chem. 28 (1989) 2940.
[5] D. Luneau, P. Rey, J. Laugier, E. Belorizky, A. Cogne, Inorg. Chem. 31 (1992) 3578.
[6] R.E. Sievers, B.W. Ponders, M.L. Morris, R.W. Moshier, Inorg. Chem. 2 (1963) 693.
[7] W.R. Walker, N.C. Li, J. Inorg. Nucl. Chem. 27 (1965) 2255.
[8] B.D. Fahlman, A.R. Barron, Adv. Mater. Opt. Electron. 10 (2000) 223.
[9] W. Partenheimer, R.S. Drago, Inorg. Chem. 9 (1970) 47.
[10] W. Lin, B.C. Wiegand, R.G. Nuzzo, G.S. Girolami, J. Am. Chem. Soc. 118 (1996) 5977.
[11] W. Lin, R.G. Nuzzo, G.S. Girolami, J. Am. Chem. Soc. 118 (1996) 5988.
[12] R.E. Sievers, R.W. Moshier, M.L. Morris, Inorg. Chem. 1 (1962) 966.
[13] M.L. Morris, R.W. Moshier, R.E. Sievers, Inorg. Chem. 2 (1963) 411.
[14] M.L. Morris, D.A. Aikens, Nature 207 (1965) 631.
[15] P. Mushak, M.T. Glenn, J. Savory, Fluorine Chem. Rev. 6 (1973) 43.
[16] F.A. Cotton, C.E. Rice, G.W. Rice, Inorg. Chim. Acta 24 (1977) 231.
[17] M.H. Dickman, L.C. Porter, R.J. Doedens, Inorg. Chem. 25 (1986) 2595.
[18] G. Mago, M. Hinago, H. Miyasaka, N. Matsumoto, H. Okawa, Inorg. Chim. Acta 254 (1997) 145.
[19] M. Kitano, Y. Ishimaru, K. Inoue, N. Koga, H. Iwamura, Inorg. Chem. 33 (1994) 6012.
[20] H.S. Jarret, J. Chem. Phys. 27 (1957) 1298.
[21] C.E. Pfluger, P.S. Haradem, Inorg. Chim. Acta 69 (1983) 141.
[22] E. Bouwman, K.G. Caulton, G. Christou, K. Folting, C. Gasser, D.N. Hendrickson, J.C. Huffman, E.B. Lobkovsky, J.D. Martin, P. Michel, H.-L. Tsai, Z. Xue, Inorg. Chem. 32 (1993) 3463.
[23] B.G. Thomas, M.L. Morris, R.L. Hilderbrandt, Inorg. Chem. 17 (1978) 2901.
[24] B. Morosin, Acta Crystallogr. 19 (1965) 131.
[25] J. Iball, C.H. Morgan, Acta. Crystallogr. 23 (1967) 239.
[26] J.P. Fackler, A. Avdeef, Inorg. Chem. 13 (1974) 1864.
[27] B.R. Stults, R.S. Marianelli, V.W. Day, Inorg. Chem. 18 (1979) 1853.
[28] L.F. Larkworthy, M.H. Nelson-Richardson, Chem. Ind. (1974) 164.
[29] D.C. Bradley, M.B. Hursthouse, C.W. Newing, A.J. Welch, J. Chem. Soc., Chem. Commun. (1972) 567.
[30] D.F. Evans, G.V. Fazakerley, R.F. Phillips, J. Chem. Soc. A (1971) 1931.
[31] Crystallographic programs and procedures used are described in J.L. Brumaghim, J.G. Priepot, G.S. Girolami, Organometallics 18 (1999) 2139.