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Reaction of $[(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ (E = S, Se) with N_2CHCO_2Me. Synthesis and characterisation of $(\mu\text{-RE})[\mu\text{-}\eta^2\text{-N=NCH(R}')\text{CO}_2\text{Me]Fe}_2\text{(CO)}_6$ and $(\mu\text{-PhSe})[\mu\text{-}\eta^1\text{-N=NCH(Me)CO}_2\text{Me]Fe}_2\text{(CO)}_6$

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Abstract

Reaction of $[M][(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]$ (E = S, Se; M = Et_3NH, Na) with N_2CHCO_2Me give the intermediate $[M][(\mu\text{-RE})(\mu\text{-}N_2CHCO_2Me)Fe_2(CO)_6]$ (**II**). Action of **II** with counterion Et_3NH^+ yields neutral complexes $(\mu\text{-RE})(\mu\text{-}\eta^2\text{-N=NCH}_2\text{CO}_2\text{Me})\text{Fe}_2\text{(CO)}_6$ (**1**, RE = Bu^tS; **2**, RE = PhS; **3**, RE = PhSe). Treatment of **II** (M=Na) with MeI affords $(\mu\text{-RE})[\mu\text{-}\eta^2\text{-N=NCH(Me)CO}_2\text{Me]Fe}_2\text{(CO)}_6$ (**4**, RE = Bu^tS; **5**, RE = PhS). In the case of RE = PhSe, the reaction of **II** with MeI gives two isomers, $(\mu\text{-PhSe})[\mu\text{-}\eta^2\text{-N=NCH(Me)CO}_2\text{Me]Fe}_2\text{(CO)}_6$ (**6**) and $(\mu\text{-PhSe})[\mu\text{-}\eta^1\text{-N=NCH(Me)CO}_2\text{Me]Fe}_2\text{(CO)}_6$ (**7**). The structure of complex **7** has been determined by single-crystal X-ray diffraction technique. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Iron; Selenium; Sulfur; Diazenido ligand; Complex; Synthesis; Structure

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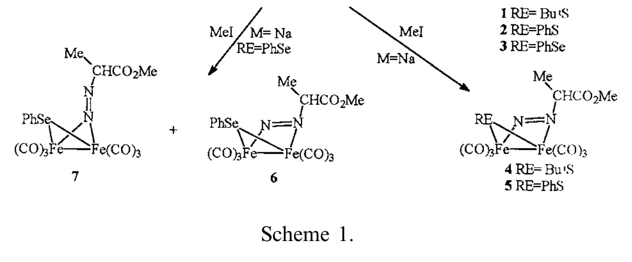
1. Introduction

We have reported the reaction of $[Et_3NH][(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]$ (E = S, Se) with aryl azides, ArN_3, forming triazenido diiron complexes (**I**) [1]. Diazo compounds bear a formal and chemical similarity to azides and can react with good nucleophiles such as RMgCl and R_3P [2]. Hence, diazo compounds are also anticipated to react with nucleophilic complexes $[(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ and the expected products would be diazenido (N_2R) diiron complexes. Although many mononuclear diazenido complexes are known, compounds in which the diazenido ligand bridges a metal–metal bond are relatively rare [3,4]. In order to prepare new multinuclear diazenido complexes and study the coordination modes of diazenido ligand at metal centres, we investigated the reaction of $[M][(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]$ (M = Et_3NH, Na) with N_2CHCO_2Me. Herein we report the results.

2. Results and discussion

The reaction of $[M][(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]$ with N_2CHCO_2Me and the synthesis of complexes $(\mu\text{-RE})[\mu\text{-}\eta^2\text{-N=NCH(R}')\text{CO}_2\text{Me]Fe}_2\text{(CO)}_6$ and $(\mu\text{-PhSe})[\mu\text{-}\eta^1\text{-N=NCH(Me)CO}_2\text{Me]Fe}_2\text{(CO)}_6$ are summarised in Scheme 1. When N_2CHCO_2Me was added to a solution



Scheme 1.

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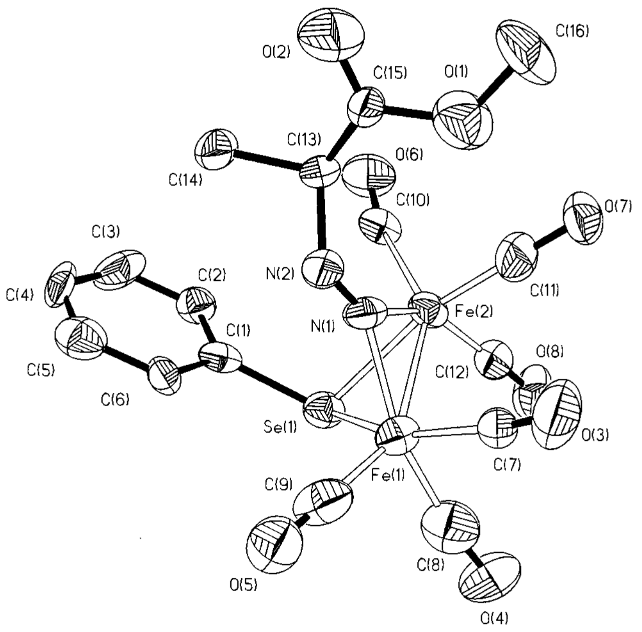
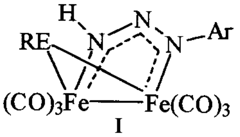


Fig. 1. ORTEP representation of the molecular structure of complex **7**.

Table 1
Selected bond distances (Å) and angles (°) for complex **7**

Bond distances			
Fe(1)–N(1)	1.915(10)	Fe(2)–N(1)	1.986(13)
Fe(1)–Se(1)	2.397(3)	Fe(2)–Se(1)	2.395(2)
Fe(1)–Fe(2)	2.458(3)	N(1)–N(2)	1.193(15)
Bond angles			
N(1)–Fe(1)–Se(1)	82.5(4)	N(1)–Fe(2)–Se(1)	81.2(4)
N(1)–Fe(1)–Fe(2)	52.2(4)	N(1)–Fe(2)–Fe(1)	49.7(3)
Se(1)–Fe(1)–Fe(2)	59.10(8)	Se(1)–Fe(2)–Fe(1)	59.19(8)
Fe(1)–Se(1)–Fe(2)	61.71(8)	N(1)–N(2)–C(13)	112.5(13)
N(2)–N(1)–Fe(1)	136.6(11)	N(2)–N(1)–Fe(2)	145.3(10)
C(1)–Se(1)–Fe(1)	109.1(3)	Fe(2)–N(1)–Fe(1)	78.1(4)

of [M][(μ-RE)(μ-CO)Fe$_2$(CO)$_6$] (E = S, Se) at room temperature, a reaction occurred, with gas evolution and colour change from brown–red to red–orange in a few minutes. In the case of M = Et$_3$NH, complexes (μ-RE)(μ-η^2-N=NCH$_2$CO$_2$Me)Fe$_2$(CO)$_6$ (**1**–**3**) were isolated after stirring overnight. When M = Na, the reaction mixture of [M][(μ-RE)(μ-CO)Fe$_2$(CO)$_6$] and N$_2$CHCO$_2$Me was further treated with MeI to give (μ-RE)[μ-η^2-N=NCH(Me)CO$_2$Me]Fe$_2$(CO)$_6$ (**4**–**6**) and (μ-RE)[μ-η^1-N=NCH(Me)CO$_2$Me]Fe$_2$(CO)$_6$ (**7**, RE = PhSe). In addition, (μ-RE)$_2$Fe$_2$(CO)$_6$ was also produced in each reaction. The formation of complexes **1**–**6** and complex **7** could be through anionic intermediates **IIa** (for **1**–**6**) and **IIb** (for **7**), respectively, which formed by nucleophilic attack of the iron atoms of [(μ-RE)(μ-CO)Fe$_2$(CO)$_6$]$^-$ at the terminal nitrogen of N$_2$CHCO$_2$Me, followed by coordination of the lone pair of α-nitrogen (for **IIa**) or β-nitrogen (for **IIb**) and concomitant loss of the μ-CO ligand.



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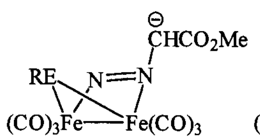
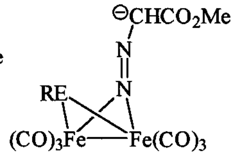


IIa



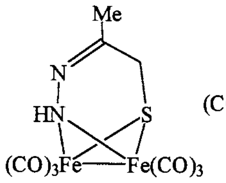
IIb

Attempts to isomerise the η^1- and η^2-bridged isomers were unsuccessful although a related reversible photochemical/thermal interconversion of η^1- and η^2-bridged (aryldiazo)triosmium clusters, (μ-H)(μ-N$_2$Ar)Os$_3$(CO)$_{10}$, has been reported [5]. Heating complexes (μ-RE)[μ-η^2-N=NCH(R′)CO$_2$Me]Fe$_2$(CO)$_6$ in benzene gave (μ-RE)$_2$Fe$_2$(CO)$_6$ and decomposed species.

Complexes (μ-RE)$_2$Fe$_2$(CO)$_6$ (RE = Bu^{t}S, PhS and PhSe) are known and have been identified by comparison of their melting points, IR and ^{1}H-NMR spectra with those of authentic samples [6–8].

Complexes **1**–**7** are deep red or brown red solids (**1**, **4** and **7**) or viscous oil (**2**, **3**, **5** and **6**) and have been characterised by elemental analyses, ^{1}H-NMR and IR spectra. The ^{1}H-NMR spectrum of each of complexes **1**–**3** displayed two inequivalent proton signals for CH$_2$ group and signals for RE and OMe groups. The ^{1}H-NMR spectra of complexes **4**–**7** exhibited the respective signals for CHMe group and RE and OMe groups. In addition, the ^{1}H-NMR spectrum of complex **7** showed that the chemical shifts of CHMe group appeared at higher field than corresponding those of complexes **4**–**6**. The IR spectra of these complexes all gave N=N, carboxylato group and terminal carbonyl absorption bands. The N=N absorption for η^1-diazenido ligand appeared at 1557 cm^{-1}, and the bands for η^2-diazenido ligands were in the range of 1567–1578 cm^{-1}. Although the N=N absorptions of complexes **1** and **4** are between that of complex **7** and those of the η^2-complexes (**2**, **3**, **5** and **6**), their ^{1}H-NMR spectra are consistent with those of η^2-complexes. For example, the chemical shifts of CHMe group at δ 1.45 ppm and δ 4.20–4.55 ppm for complex **4** were close to those of complex **6** (δ 1.35–1.60 and δ 3.75–4.10 ppm) rather than those of complex **7** (δ 0.75 and δ 3.10–3.40 ppm). Attempts to grow the single crystals of complexes **1**–**6** for X-ray analyses were unsuccessful. However, complex **7** has been characterised by single-crystal X-ray diffraction. The structure is presented in Fig. 1, and the selected bond lengths and angles are in Table 1. The molecule is a diiron hexacarbonyl complex with both

PhSe ligand and η^1-N=NCH(Me)CO_2Me ligand bridging the two metals. The two metal atoms [Fe(1)Fe(2)] and the two nitrogen atoms [N(1)N(2)] are coplanar [the sum of the angles around N(1) is 360°]. The orientation of phenyl group is axial. The N(1) binds two iron atoms in a little asymmetrical manner [N(1)–Fe distances 1.915(10) and 1.986(13) Å, respectively]. While the Se atom is bound almost symmetrically across the metals and the Fe–Se bond lengths [2.395(2) and 2.397(3) Å, respectively] are comparable to those of (μ-PhSe)(μ-PhCH_2SC=S)$Fe_2(CO)_6$ (av. 2.374 Å) [9] and (μ-Se_2)$Fe_2(CO)_6$ (av. 2.364 Å) [10]. The Fe(1)–Fe(2) distance of 2.458(3) Å is very close to that of complex **8** [2.459(1) Å] [11]. The Fe–N distances (av. 1.95 Å) is also close to those of complex **8** (av. 1.969 Å), but longer than those found in complex **9** (av. 1.863 Å) [12]. The N–N bond length of 1.193(15) Å is shorter than those of related η^1-diazenido transition metal complexes such as complex **9** [1.238(3) Å], [(μ-η^1-N=NPh)Mn$(CO)_4$]$_2$ [1.234(3) Å] [13] and (μ-H)[μ-η^1-N=N(*p*-tol)]$Os_3(CO)_{10}$ [1.238(18) Å] [14], close to those of η^2-diazenido complexes (μ-H)(μ-η^2-N=NPh)-$Os_3(CO)_{10}$ [1.20(4) Å] [5] and [$(C_5Me_5)_2Ir_2(CO)_2$(μ-η^2-N=NC_6H_4OMe)][BF_4] [1.205(9) Å] [3].



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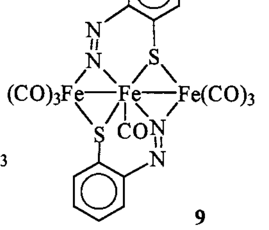

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3. Experimental

All reactions were carried out under nitrogen using standard Schlenk techniques. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl. PhSeH [15], $Fe_3(CO)_{12}$ [16] and N_2CHCO_2Me [17] were obtained by published procedures. The solutions of [M][(μ-RS)(μ-CO)$Fe_2(CO)_6$] and [M][(μ-PhSe)(μ-CO)$Fe_2(CO)_6$] were prepared by the methods described in literatures [9,18]. Infrared spectra were obtained by using a VECTOR22 spectrometer. ^{1}H-NMR spectra were recorded on either a Varian EM360L or a JEOL FX-90Q spectrometer. Elemental analyses were performed with a 240C analyser.

*3.1. Synthesis of (μ-RE)(μ-η^2-N=NCH_2CO_2Me)-$Fe_2(CO)_6$ (**1**, RE = Bu^tS; **2**, RE = PhS; **3**, RE = PhSe)*

A solution of the triethylammonium salt of [(μ-Bu^tS)(μ-CO)$Fe_2(CO)_6$]$^-$ was generated by the reacting 1.16 g (2.30 mmol) of $Fe_3(CO)_{12}$, 0.26 ml (2.30 mmol) of Bu^tSH and 0.33 ml (2.36 mmol) of Et_3N in 30 ml of THF at room temperature (r.t.). To the solution was added 0.23 g (2.30 mmol) of N_2CHCO_2Me with stirring. After stirring overnight, the solvent was removed at reduced pressure and the residue extracted with 2:8 (v/v) CH_2Cl_2–petroleum ether. After removal of the solvent, the material remaining was subjected to filtration chromatography (silica gel). Petroleum ether eluted a first band which gave, after recrystallisation from petroleum ether, 0.04 g (8%) of (μ-Bu^tS)$_2Fe_2(CO)_6$ [6] as red crystals. Further elution with 2:3 (v/v) CH_2Cl_2–petroleum ether followed by evaporation of the solvent gave 0.86 g (80%) of red solid **1**, m.p. 50–52°C. Anal. Found: C, 33.46; H, 3.05; N, 6.06. Calc. for $C_{13}H_{14}Fe_2N_2O_8S$: C, 33.22; H, 3.00; N, 5.96%. ^{1}H-NMR ($CDCl_3$): δ (ppm) 1.02 (s, 9H, Bu^t), 3.75 (s, 3H, Me), 4.33, 4.77 (s, s, 2H, CH_2). IR (KBr disc): ν (cm^{-1}) 2074vs, 2036vs, 2012vs, 1987vs, 1965vs (Fe–CO); 1753s (CO); 1567s (N=N).

Compound **2** was prepared according to the same procedure as for **1**, but PhSH was used instead of Bu^tSH. After similar work-up, (μ-PhS)$_2Fe_2(CO)_6$ (37%) [7] and **2** (61%) were obtained. **2**, deep red oil. Anal. Found: C, 37.08; H, 2.20; N, 5.64. Calc. for $C_{15}H_{10}Fe_2N_2O_8S$: C, 36.77; H, 2.06; N, 5.72%. ^{1}H-NMR ($CDCl_3$): δ (ppm) 3.70 (s, 3H, Me), 3.96, 4.10 (s, s, 2H, CH_2), 7.10 (s, 5H, Ph). IR (CCl_4): ν (cm^{-1}) 2078vs, 2048vs, 1990vs (Fe–CO); 1748s (CO); 1576s (N=N).

Compound **3** was prepared according to the same procedure as for **1**, but PhSeH was used instead of Bu^tSH. After similar work-up, (μ-PhSe)$_2Fe_2(CO)_6$ (33%) [8] and complex **3** (63%) were obtained. **3**, deep red oil. Anal. Found: C, 33.82; H, 2.04; N, 5.63. Calc. for $C_{15}H_{10}Fe_2N_2O_8Se$: C, 33.56; H, 1.88; N, 5.22%. ^{1}H-NMR (CCl_4): δ (ppm) 3.65 (s, 3H, Me), 3.95, 4.20 (s, s, 2H, CH_2), 7.05 (s, 5H, Ph). IR (CCl_4): ν (cm^{-1}) 2073vs, 2031vs, 1988vs (Fe–CO); 1748s (CO); 1575s (N=N).

*3.2. Synthesis of (μ-RS)[μ-η^2-N=NCH(Me)CO_2Me]-$Fe_2(CO)_6$ (**4**, R = Bu^t; **5**, R = Ph)*

To a solution of [Na][(μ-RS)(μ-CO)$Fe_2(CO)_6$] generated from 1.0 g (1.98 mmol) of $Fe_3(CO)_{12}$ and an equimolar amount of Bu^tSNa (from Bu^tSH and NaH) in 30 ml of THF was added 0.2 g (2.0 mmol) of N_2CHCO_2Me and stirred for 30 min at r.t. To the mixture 0.3 ml (4.81 mmol) of MeI was added and stirred overnight. The solvent was removed at reduced pressure and the residue extracted with 2:8 (v/v) CH_2Cl_2–petroleum ether. After removal of the solvent, the material remaining was subjected to filtration chromatography (silica gel). Elution with petroleum ether yielded, after recrystallisation from petroleum ether, 0.06 g (13%) of red crystalline (μ-Bu^{t}S)$_2$Fe$_2$(CO)$_6$ [6]. Further elution with 2:3 (v/v) CH$_2$Cl$_2$–petroleum ether followed by evaporation of the solvent and recrystallisation from CH$_2$Cl$_2$–petroleum ether gave 0.66 g (69%) of brown red crystalline **4**, m.p. 50–52°C. Anal. Found: C, 34.64; H, 3.25; N, 5.88. Calc. for $C_{14}H_{16}Fe_2N_2O_8S$: C, 34.74; H, 3.33; N, 5.79%. ^{1}H-NMR (CDCl$_3$): δ (ppm) 1.05 (s, 9H, Bu^t), 1.45 (d, $J = 6$ Hz, 3H, Me), 3.75 (s, 3H, Me), 4.20–4.55 (m, 1H, CH). IR (CCl$_4$): ν (cm^{-1}) 2075vs, 2032vs, 1989vs (Fe–CO); 1750s (CO); 1568s (N=N).

Compound **5** was prepared according to the same procedure as for **4**, but PhSH was used instead of Bu^{t}SH. After similar work-up, (μ-PhS)$_2$Fe$_2$(CO)$_6$ (26%) [7] and **5** (30%) were obtained. **5**, deep red oil. Anal. Found: C, 38.30; H, 2.35; N, 5.70. Calc. for $C_{16}H_{12}Fe_2N_2O_8S$: C, 38.13; H, 2.40; N, 5.56%. ^{1}H-NMR (CDCl$_3$): δ (ppm) 1.22–1.45 (b, 3H, Me), 3.65 (s, 3H, Me), 3.90–4.40 (m, 1H, CH), 7.10 (s, 5H, Ph). IR (CCl4): ν (cm^{-1}) 2078vs, 2037vs, 2000vs (Fe–CO); 1749s (CO); 1578s (N=N).

*3.3. Synthesis of (μ-PhSe)[μ-η^2-N=NCH(Me)CO$_2$Me]Fe$_2$(CO)$_6$ (**6**) and (μ-PhSe)[μ-η^1-N=NCH(Me)CO$_2$Me]Fe$_2$(CO)$_6$ (**7**)*

To a solution of [Na][(μ-PhSe)(μ-CO)Fe$_2$(CO)$_6$] generated from 1.0 g (1.98 mmol) of Fe$_3$(CO)$_{12}$ and an equimolar amount of PhSeNa (from PhSeH and NaH) in 30 ml of THF was added 0.2 g (2.0 mmol) of N$_2$CHCO$_2$Me and stirred for 40 min at r.t. To the mixture 0.3 ml (4.81 mmol) of MeI was added and stirred overnight. The solvent was removed at reduced pressure and the residue extracted with 2:8 (v/v) CH$_2$Cl$_2$–petroleum ether. After removal of the solvent, the material remaining was subjected to filtration chromatography (silica gel). Elution with petroleum ether yielded, after recrystallisation from petroleum ether, 0.11 g (19%) of brown red crystalline (μ-PhSe)$_2$Fe$_2$(CO)$_6$ [8]. Further elution with 2:3 (v/v) CH$_2$Cl$_2$–petroleum ether developed two red bands successively. The two bands gave, after evaporation of the solvent and recrystallisation from CH$_2$Cl$_2$–petroleum ether, 0.13 g (12%) of **7** and 0.19 g (18%) **6**, respectively. **7**, brown red crystals, m.p. 84–86°C. Anal. Found: C, 34.91; H, 2.14; N, 4.82. Calc. for $C_{16}H_{12}Fe_2N_2O_8Se$: C, 34.88; H, 2.20; N, 5.08%. ^{1}H-NMR (CDCl$_3$): δ (ppm) 0.75 (d, $J = 7$ Hz, 3H, Me), 3.10–3.40 (m, 1H, CH), 3.65 (s, 3H, Me), 7.20 (s, 5H, Ph). IR (KBr disc): ν (cm^{-1}) 2070vs, 2032vs, 1998vs, 1981vs (Fe–CO); 1745s (CO); 1557m (N=N). **6**, deep red oil. Anal. Found: C, 34.67; H, 2.27; N, 4.97. Calc. for $C_{16}H_{12}Fe_2N_2O_8Se$: C, 34.88; H, 2.20; N, 5.08%. ^{1}H-NMR (CDCl$_3$): δ (ppm) 1.35–1.60 (b, 3H, Me), 3.68 (s, 3H, Me), 3.75–4.10 (m, 1H, CH), 7.20 (s, 5H, Ph). IR (CCl$_4$): ν (cm^{-1}) 2073vs, 2032vs, 1995vs (Fe–CO); 1748s (CO); 1575s (N=N).

Table 2
Crystal data and refinement for complex **7**

Formula	$C_{16}H_{12}Fe_2N_2O_8Se$
Formula weight	550.94
Crystal system	Triclinic
Space group	$P\bar{1}$
a (Å)	7.7488(15)
b (Å)	16.761(3)
c (Å)	18.373(4)
α (°)	116.27(3)
β (°)	95.83(3)
γ (°)	97.83(3)
V (Å^3)	2084.3(7)
Z	4
Crystal size (mm)	0.40 × 0.40 × 0.05
Radiation λ (Å)	0.71073
Temperature (K)	293(2)
$2\theta_{\max}$ (°)	51.26
Total reflections	2858
Independent reflections	2858
Reflections with $[I>2\sigma(I)]$	2368
Goodness-of-fit on F^2	1.299
Final R indices $[I>2\sigma(I)]$	
R_1 [a]	0.0969
wR_2 [b]	0.2696
Largest differences peak and hole (e Å^{-3})	1.292 and −1.498

[a] $R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$.
[b] $wR_2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma wF_o^4]^{1/2}$.

3.4. Crystal data and structure determination of complex 7

Suitable crystals of complex **7** were grown from petroleum ether–CH$_2$Cl$_2$ solution at r.t. Data were collected on an IP Rigaku diffractometer using graphite-monochromated Mo–K$_\alpha$ radiation. Structure was solved by direct method and refined by full-matrix least-squares on F^2 with anisotropic thermal parameters for non-hydrogen atoms on a PC using SHELXS-97 [19] and SHELXL-97 [20], respectively. A summary of crystal data and refinement parameters is given in Table 2. The high value of R is attributed to the relatively poor quality of the crystal resulting in a weak set of data.

4. Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 140437. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2

1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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