This article was downloaded by: On: 23 January 2011 Access details: Access Details: Free Access Publisher Taylor & Francis Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



To cite this Article Song, Li-Cheng , Dong, Yu-Bin , Hu, Qing-Mei and Huang, Xiao-Ying(1999) 'SYNTHESIS OF SINGLE AND DOUBLE μ 3-Se-CONTAINING TETRAHEDRAL MCoFe(μ_3 -Se) AND MNiFe(μ_3 -Se) (M = Mo, W) CLUSTERS *VIA* ISOLOBAL AND FUNCTIONAL REACTIONS. CRYSTAL AND MOLECULAR STRUCTURE OF η^5 -MeO₂CC₅H₄(CO)₂WCoFe(μ_3 -Se)(CO)₆', Journal of Coordination Chemistry, 47: 2, 369 – 380 To link to this Article: DOI: 10.1080/00958979908023069

URL: http://dx.doi.org/10.1080/00958979908023069

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material. J. Coord. Chem., 1999, Vol. 47, pp. 369-380 Reprints available directly from the publisher Photocopying permitted by license only © 1999 OPA (Overseas Publishers Association) N.V. Published by license under the Gordon and Breach Science Publishers imprint. Printed in Malaysia.

SYNTHESIS OF SINGLE AND DOUBLE μ_3 -Se-CONTAINING TETRAHEDRAL MCoFe(μ_3 -Se) AND MNiFe(μ_3 -Se) (M = Mo, W) CLUSTERS *VIA* ISOLOBAL AND FUNCTIONAL REACTIONS. CRYSTAL AND MOLECULAR STRUCTURE OF η^5 -MeO₂CC₅H₄(CO)₂WCoFe(μ_3 -Se)(CO)₆

LI-CHENG SONG^{a,*}, YU-BIN DONG^a, QING-MEI HU^a and XIAO-YING HUANG^b

^aDepartment of Chemistry, Nankai University, Tianjin 300071, China; ^bState Key Laboratory of Structural Chemistry, Fuzhou, Fujian 350002, China

(Received 9 March 1998; In final form 3 June 1998)

The single isolobal displacement reaction of $(\mu_3$ -Se)FeCo₂(CO)₉ with η^5 -RC₅H₄M(CO)₃Na can occur in refluxing THF to give single clusters η^5 -RC₅H₄(CO)₂MCoFe(μ_3 -Se)(CO)₆ (**1a** M = Mo, R = EtO₂C; **1b** M = W, R = MeCO; **1c** M = W, R = MeO₂C), whereas the functional transformation reaction of **1b** with 2,4-dinitrophenylhydrazine affords its phenylhydrazone derivative η^5 -2,4-(NO₂)₂C₆H₃NHNC(Me)C₅H₄(CO)₂WCoFe(μ_3 -Se)(CO)₆ (**2**). More interestingly, the double isolobal displacement reaction of two molecules of (μ_3 -Se)FeCo₂(CO)₉ with $[\eta^5$ -C₅H₄C(O)CH₂M(CO)₃Na]₂ takes place in THF at reflux, giving double clusters $[\eta^5$ -C₅H₄C(O)CH₂]₂[(CO)₂MCoFe(μ_3 -Se)(CO)₆]₂ (**3a** M = Mo; **3b** M = W), whereas **3a** reacts with Cp₂Ni in refluxing THF to afford both single and double isolobal displacement products $[\eta^5$ -C₅H₄C(O)CH₂]₂[(CO)₂MONiFe(μ_3 -Se)(CO)₃Cp][(CO)₂MoCoFe(μ_3 -Se)(CO)₆] (**4**) and $[\eta^5$ -C₅H₄C(O)CH₂]₂[(CO)₂MoNiFe(μ_3 -Se)(CO)₃Cp]₂ (**5**). All products were characterized by analysis and spectroscopy, as well as for **1c** by an X-ray diffraction analysis.

Keywords: Isolobal displacement; functional transformation; μ_3 -Se ligand; transition metal clusters; X-ray structure

^{*} Corresponding author.

INTRODUCTION

In previous papers we reported a very useful isolobal displacement method to synthesize single and double μ_3 -S-containing tetrahedral MCoFe(μ_3 -S)¹ and MNiFe(μ_3 -S) (M = Mo, W)^{2,3} clusters. Further, we also reported some interesting transformation reactions of functional groups in those cluster complexes under the action of various reagents.^{1,3} Since selenium-containing, and particularly the μ_3 -Se-containing transition metal clusters are of great interest,⁴⁻⁸ one might ask if the corresponding single and double μ_3 -Secontaining transition metal clusters could be made by similar procedures and if they would have similar structures and properties. In this paper, we answer some of these questions by describing the synthesis and characterization of a series of μ_3 -Se-containing tetrahedral MCoFe(μ_3 -Se) and MNiFe(μ_3 -Se) (M = Mo, W) cluster complexes.

EXPERIMENTAL

General Comments

All reactions were carried out under highly purified tank nitrogen using standard Schlenk or vacuum-line techniques. TLC was performed using silica gel G (10–40 µm). THF and diglyme were distilled from Na/benzophenone ketyl. The compounds (μ_3 -Se)FeCo₂(CO)₉,⁹ Cp₂Ni,¹⁰ RC₅H₄Na (R = MeO₂C, EtO₂C, MeCO)¹¹ and Na₂[η^5 -C₅H₄C(O)CH₂]₂¹² were prepared according to literature methods. Other solvents and reagents were of commercial origin and used without further purification. IR spectra were recorded on a Nicolet FT-IR 5DX infrared spectrophotometer. ¹H NMR spectra were recorded on Jeol FX 90Q and Bruker AC-P 200 spectrometers. C/H analyses and MS determinations were performed on a Perkin-Elmer 240C analyzer and an HP 5988A spectrometer, respectively. Melting points are uncorrected.

η^{5} -EtO₂CC₅H₄(CO)₂MoCoFe(μ_{3} -Se)(CO)₆ (1a)

A 100 cm³ three-necked flask fitted with a magnetic stir-bar, a rubber septum, and a reflux condenser topped with a nitrogen inlet tube, was charged with 0.784 g (4.90 mmol) of $EtO_2CC_5H_4Na$, 1.250 g (4.75 mmol) of Mo(CO)₆ and 20 cm³ of THF. The mixture was stirred at reflux for 1.5 h and upon cooling to room temperature, 1.200 g (2.38 mmol) of (μ_3 -Se)Fe-Co₂(CO)₉ was added in one portion. The mixture was refluxed for an additional 0.5 h. Solvent was removed under vacuum and the residue was extracted with CH₂Cl₂. The extracts were subjected to TLC separation using 1:1 (v/v) CH₂Cl₂/petroleum ether as eluent. From the main red-brown band was obtained 1.010 g (65%) of **1a** as a red-brown solid, m.p. 78–79°C. *Anal.* Calcd. for C₁₆H₉CoFeMoO₁₀Se(%): C, 29.52; H, 1.39; Found: C, 29.59; H, 1.94. IR (KBr, disk): $\nu_{(C=0)}$ 2072(vs), 2011(s), 1996(s), 1968(vs), 1908(vs), 1875(vs); $\nu_{(C=0)}$ 1730(s) cm⁻¹. ¹H NMR (CDCl₃, TMS, δ): 1.32 (t, 3H, J = 7.2 Hz, CH₃), 4.27 (q, 2H, J = 7.2 Hz, CH₂), 5.32, 5.48 (q, q, 2H, H³, H⁴), 5.80 (t, 2H, H², H⁵) ppm. MS (EI), m/z (⁹⁸Mo, ⁸⁰Se): 598 (M⁺ – 2CO, 11.7%), 542 (M⁺ – 4CO, 14.3), 514 (M⁺ – 5CO, 14.3), 486 (M⁺ – 6CO, 46.2), 458 (M⁺ – 7CO, 26.5), 430 (M⁺ – 8CO, 24.1), 293 (MoFe CoSe⁺, 15.6).

η^{5} -MeCOC₅H₄(CO)₂WCoFe(μ_{3} -Se)(CO)₆ (1b)

The flask described above was charged with 0.660 g (5.08 mmol) of MeCOC₅H₄Na, 1.670 g (4.75 mmol) of W(CO)₆ and 20 cm³ of diglyme. The mixture was stirred at reflux for 6 h. After the solvent was removed under vacuum, 20 cm³ of THF and 1.200 g (2.38 mmol) of (μ_3 -Se)FeCo₂(CO)₉ were added and the mixture was refluxed for an additional 0.5 h. After the same workup as for **1a**, 1.060 g (63%) of **1b** was obtained as a red-brown solid, m.p. 104–105°C. *Anal.* Calcd. for C₁₅H₇CoFeO₉SeW(%): C, 25.42; H, 1.00; Found: C, 25.49; H, 0.75. IR (KBr, disk): $\nu_{(C=O)}$ 2073(vs), 2032(vs), 2016(vs), 1991(vs), 1967(vs), 1901(s), 1868(s); $\nu_{(C=O)}$ 1688(s) cm⁻¹. ¹H NMR (CDCl₃, TMS, δ): 2.44 (s, 3H, CH₃), 5.54, 5.72 (q, q, 2H, H³, H⁴), 5.80–6.04 (m, 2H, H², H⁵) ppm. MS (EI), *m/z* (¹⁸⁴W, ⁸⁰Se): 710 (M⁺, 0.1%), 682 (M⁺ – CO, 0.1), 654 (M⁺ – 2CO, 0.3), 626 (M⁺ – 3CO, 0.3), 598 (M⁺ – 4CO, 0.4), 570 (M⁺ – 5CO, 1.3), 542 (M⁺ – 6CO, 1.9), 514 (M⁺ – 7CO, 1.3), 486 (M⁺ – 8CO, 2.3), 379 (WFeSeCo⁺, 0.2).

η^{5} -MeO₂CC₅H₄(CO)₂WCoFe(μ_{3} -Se)(CO)₆ (1c)

The same procedure as for **1b** was followed, except using 0.720 g (4.90 mmol) of MeO₂CC₅H₄Na instead of MeCOC₅H₄Na; 1.250 g (73%) of **1c** was obtained as a red-brown solid, m.p. 85–86°C. *Anal.* Calcd. for C₁₅H₇-CoFeO₁₀SeW(%): C, 24.86; H, 0.97; Found: C, 24.93; H, 0.95. IR (KBr, disk): $\nu_{(C=O)}$ 2065(vs), 2024(vs), 2008(vs), 1967(vs), 1918(s); $\nu_{(C=O)}$ 1729 cm⁻¹. ¹H NMR (CDCl₃, TMS, δ): 3.94 (s, 3H, CH₃), 5.54, 5.72 (q, q, 2H, H³, H⁴), 5.96 (t, 2H, H², H⁵). MS (EI), *m/z* (¹⁸⁴W, ⁸⁰Se): 726 (M⁺, 0.1%), 698 (M⁺ - CO,

0.1), 670 (M⁺ – 2CO, 0.5) 642 (M⁺ – 3CO, 0.4) 614 (M⁺ – 4CO, 0.7), 586 (M⁺ – 5CO, 1.9), 558 (M⁺ – 6CO, 2.8), 530 (M⁺ – 7CO, 1.8), 502 (M⁺ – 8CO, 1.8), 379 (WFeSeCo⁺, 0.3).

η^{5} -2,4-(NO₂)₂C₆H₃NHNC(Me)C₅H₄(CO)₂WCoFe(μ_{3} -Se)(CO)₆ (2)

A solution of 2,4-dinitrophenylhydrazine (2.000 g, 10 mmol) in 10 cm³ of 98% H_2SO_4 was added dropwise to a stirred solution prepared from 14 cm^3 of H₂O and 46 cm³ of 95% EtOH. The resulting red-orange solution of 2,4dinitrophenylhydrazine was filtered out and kept under nitrogen for use. The flask described above was charged with 0.280 g (0.39 mmol) of 1b, 3 cm³ of 2,4-dinitrophenylhydrazine solution and 20 cm³ of EtOH. The mixture was stirred for 7h at room temperature, filtered and washed with EtOH $(10 \text{ cm}^3 \times 2)$. The solid was redissolved in methylene chloride and purified by TLC using 2:1 (v/v) CH_2Cl_2 /petroleum ether as eluent. The main yelloworange band was collected to afford 0.200 g (58%) of 2 as a brown solid, m.p. 150°C (dec). Anal. Calcd. for C₂₁H₁₁CoFeN₄O₁₂SeW(%): C, 28.38; H, 1.25; N, 6.30; Found: C, 27.99; H, 1.18; N, 6.14. IR (KBr, disk): $\nu_{(C=O)}$ 2065(s), 2016(vs), 1991(vs), 1920(s), 1852(m); $\nu_{(C=N)}$ 1614(s) cm⁻¹. ¹H NMR $(CDCl_3, TMS, \delta)$: 2.24 (s, 3H, CH₃), 5.52, 5.64 (q, q, 2H, H³, H⁴), 5.88 (q, 2H, H², H⁵), 7.96 (d, 1H, J = 6.4 Hz, H⁶ of benzene ring), 8.40 (d, 1H, J = 6.4 Hz, H⁵ of benzene ring), 9.16 (s, 1H, H³ of benzene ring), 11.36 (s, 1H, NH) ppm.

$[\eta^{5}-C_{5}H_{4}C(O)CH_{2}]_{2}|(CO)_{2}MoCoFe(\mu_{3}-Se)(CO)_{6}]_{2}$ (3a)

The flask described above was charged with 0.387 g (1.50 mmol) of Na₂[η^{5} -C₅H₄C(O)CH₂]₂, 0.792 g (3.00 mmol) of Mo(CO)₆ and 20 cm³ of THF. The mixture was stirred at reflux for 15h to give an intermediate [η^{5} -C₅H₄C(O)CH₂]₂[(CO)₃MoNa]₂. Upon cooling to room temperature, 1.514 g (3.00 mmol) of (μ_{3} -Se)FeCo₂(CO)₉ was added in one portion and the mixture was refluxed for an additional 0.5h. Solvent was removed under vacuum and the residue was extracted with methylene chloride. The extracts were subjected to TLC separation using 3:2 (v/v) CH₂Cl₂/petroleum ether as eluent. From the main red-brown band was obtained 1.200 g (65%) of **3a** as a brown solid, m.p. 144°C (dec). *Anal.* Calcd. for C₃₀H₁₂Co₂Fe₂-Mo₂O₁₈Se₂(%): C, 29.06; H, 0.97; Found: C, 29.25; H, 0.91. IR (KBr, disk): $\nu_{(C=O)}$ 2073(vs), 2016(vs), 1959(vs), 1885(s); $\nu_{(C=O)}$ 1688(s) cm⁻¹. ¹H NMR (CDCl₃, TMS, δ): 3.14 (s, 4H, CH₂CH₂), 5.48, 5.60 (q, q, 4H, H³, H³', H⁴, H^{4'}), 5.92 (t, 4H, H², H^{2'}, H⁵, H^{5'}) ppm.

$[\eta^{5}-C_{5}H_{4}C(O)CH_{2}]_{2}|(CO)_{2}WCoFe(\mu_{3}-Se)(CO)_{6}]_{2}$ (3b)

The flask described above was charged with 0.387 g (1.50 mmol) of Na₂[η^{5} -C₅H₄C(O)CH₂]₂, 1.056 g (3.00 mmol) of W(CO)₆ and 20 cm³ of diglyme. The mixture was stirred at reflux for 6h to give an intermediate [η^{5} -C₅H₄C(O)CH₂]₂[(CO)₃WNa]₂. After removal of the solvent under vacuum, 20 cm³ of THF and 1.514 g (3.00 mmol) of (μ_{3} -Se)FeCo₂(CO)₉ were added and the mixture was refluxed for an additional 0.5 h. The same workup as **3a** gave 0.318 g (15%) of **3b** as a brown solid, m.p. 154–155°C. *Anal.* Calcd. for C₃₀H₁₂Co₂Fe₂O₁₈Se₂W₂(%): C, 25.45; H, 0.85; Found: C, 25.43; H, 0.85. IR (KBr, disk): $\nu_{(C=O)}$ 2073(vs), 2008(vs), 1967(vs), 1877(s); $\nu_{(C=O)}$ 1680(s) cm⁻¹. ¹H NMR (CDCl₃, TMS, δ), 3.14 (s, 4H, CH₂CH₂), 5.54, 5.71 (q, q, 4H, H³, H³', H⁴, H⁴'), 5.80–6.00 (m, 4H, H², H²', H⁵, H⁵') ppm.

$[\eta^{5}-C_{5}H_{4}C(O)CH_{2}]_{2}[(CO)_{2}MoNiFe(\mu_{3}-Se)(CO)_{3}Cp]-$ [(CO)₂MoCoFe(\mu_{3}-Se)(CO)_{6}] (4) and $[\eta^{5}-C_{5}H_{4}C(O)CH_{2}]_{2}[(CO)_{2}MoNiFe(\mu_{3}-Se)(CO)_{3}Cp]_{2}$ (5)

The flask described above was charged with 1.265 g (1.02 mmol) of 3a, 0.578 g (3.06 mmol) of Cp₂Ni and 30 cm³ of THF. The mixture was stirred at reflux for 12 h. Solvent was removed under vacuum and the residue was extracted with methylene chloride. The extracts were subjected to TLC separation using 4:1 (v/v) CH_2Cl_2 /petroleum ether as eluent. From the first brown-green band was obtained 0.115 g (9%) of 4 as a brown-green solid, m.p. 151°C (dec). Anal. Calcd. for C₃₂H₁₇CoFe₂Mo₂NiO₁₅Se₂(%): C, 31.49; H, 1.40. Found: C, 31.91; H, 1.53. IR (KBr, disk): $\nu_{(C=O)}$ 2073(vs), 2024(vs), 1999(vs), 1967(vs), 1951(vs), 1877(s); $\nu_{(C=O)}$ 1680(s) cm⁻¹. ¹H NMR (CDCl₃, TMS, δ): 3.08 (s, 4H, CH₂CH₂), 5.32 (s, 5H, C₅H₅), 5.40-5.54; 5.58-5.74 (m, m, 4H, H³, H³', H⁴, H⁴'), 5.84-6.12 (m, 4H, H², H²', H⁵, $H^{5'}$) ppm. From the second deep-green band was obtained 0.225 g (18%) of 5 as a black-green solid, m.p. 202°C (dec). Anal. Calcd. for C₃₄H₂₂Fe₂Mo₂₋ Ni₂O₁₂Se₂(%): C, 33.99; H, 1.85; Found: C, 33.73; H, 1.91. IR (KBr, disk). $\nu_{(C=O)}$ 2073(w), 2032(vs), 1983(vs), 1950(vs), 1877(vs); $\nu_{(C=O)}$ 1680(s) cm⁻¹. ¹H NMR (CDCl₃, TMS, δ): 3.04 (s, 4H, CH₂CH₂), 5.30 (s, 10H, 2C₅H₅), 5.40-5.52, 5.58-5.70 (m, m, 4H, H³, H³', H⁴, H⁴'), 5.96-6.12 (m, 4H, H², $H^{2'}, H^{5}, H^{5'}$) ppm.

Crystal Structure Determination of 1c

Suitable crystals of 1c for X-ray diffraction analysis were grown by slow evaporation of a CH_2Cl_2 /hexane solution at $-20^{\circ}C$. Details of crystal

L.-C. SONG et al.

Formula	C ₁₅ H ₇ CoFeO ₁₀ SeW
M	724.80
Crystal size (mm)	$0.85 \times 0.55 \times 0.35$
Crystal system	Monoclinic
Space group	P2/n
$a(\mathbf{A})$	17.606(6)
$b(\mathbf{A})$	11.844(6)
c(Å)	19.033(6)
β (°)	95.85(3)
$V(Å^3)$	3948(5)
Ζ	8
$Dc (g \text{ cm}^{-3})$	2.44
<i>F</i> (000)	2704
$\mu(\text{Mo-K}\alpha)(\text{cm}^{-1})$	93.69
$T(\mathbf{K})$	296
Diffractometer	Enraf-nonius CAD4
$2\theta_{\max}$ (°)	49.9
Scan width (°)	$0.60 + 0.35 \tan \theta$
Transmission factors	0.9531-1.000
Reflections collected	7527
Unique reflections	7298
Observed reflections $[I > 3.00\sigma(I)]$	4815
R	0.049
R _w	0.059
Max., residuals (eų)	1.54

TABLE 1 Details of crystal parameters, data collection and structure refinement for 1c

parameters, data collections and structure refinements are given in Table I. A brown crystal measuring $0.85 \times 0.55 \times 0.35$ mm was mounted on a glass fibre and placed on an Enraf-Nonius CAD4 diffractometer with a graphite monochromator. A total of 7298 independent reflections were collected at 23°C with MoK α radiation ($\lambda = 0.71069$ Å) by a $\omega/2\theta$ scan mode. Of the total reflections, 4815 were considered to be observed with $I > 3\sigma(I)$ and used in subsequent refinement. Data were corrected for Lp factors.

The structure was solved by direct methods and Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included in final calculations but not refined. All calculations were performed on a micro-VAX II computer using the TEXSAN program system.

RESULTS AND DISCUSSION

Synthesis and Characterization of Single and Double Clusters 1a-c, 2, 3a, b, 4 and 5

Analogous to the preparation of single MCoFe(μ_3 -S) (M = Mo, W) cluster complexes,¹ we found that the mononuclear transition metal isolobal

reagents η^5 -RC₅H₄(CO)₃MNa (M = Mo, W; R = MeCO, MeO₂C, EtO₂C) reacted with (μ_3 -Se)FeCo₂(CO)₉ in THF at reflux, *via* a single isolobal displacement reaction,^{1,3} to afford three new single MCoFe(μ_3 -Se) clusters **1a-c**, as red-brown solids in 63–73% yields (1). We also found that cluster complex **1b** could undergo a functional transformation reaction with an aqueous H₂SO₄/EtOH solution of 2,4-dinitrophenylhydrazine to give its phenyl hydrazone derivative **2**, a new single WCoFe(μ_3 -Se) cluster, as a deep-brown solid in 58% yield (2).



On the basis of preparation of single MCoFe(μ_3 -Se) clusters mentioned above, we further found that the dinuclear transition metal isolobal reagents $[\eta^5$ -C₅H₄C(O)CH₂]₂[(CO)₃MNa]₂ (M = Mo, W) reacted with two molecules of (μ_3 -Se)FeCO₂(CO)₉ in THF at reflux, *via* a double isolobal displacement reaction, ^{1,13} to produce two new double MCoFe(μ_3 -Se) clusters **3a,b** in 68% and 15% yields, respectively (3).



L.-C. SONG et al.

More interestingly, it was found that the reaction of double cluster **3a** with an isolobal reagent Cp₂Ni in refluxing THF, *via* both single and double isolobal reactions,^{2,13} afforded an unsymmetrical double cluster **4** and a symmetrical double cluster **5** in 9% and 18% yields, respectively (4). The former contains two different tetrahedral cluster cores MoNiFe(μ_3 -Se) and MoCoFe(μ_3 -Se), while the latter possesses two identical tetrahedral cluster cores of MoNiFe(μ_3 -Se). It follows that the functional transformation reaction and the isolobal displacement reactions involving isolobal fragments^{1,2,13} such as Co(CO)₃(d⁹ML₃), η^5 -RC₅H₄(CO)₂M (a single d⁵ML₅ fragment generated *in situ* from η^5 -RC₅H₄(CO)₃MNa), [η^5 -C₅H₄C(O)CH₂]₂[(CO)₃MNa]₂) and CpNi(d⁹ML₃) generated from nickelocene have proved to be very effective in preparation of μ_3 -Se containing tetrahedral transition metal clusters.



All the new single and double cluster complexes have been characterized by analysis and spectroscopy. For single clusters 1a-c and 2, the IR spectra showed terminal carbonyl bands in the range $1868-2073 \text{ cm}^{-1}$. The IR spectra of 1a-c also showed ester and acetyl carbonyl bands at $1688-1730 \text{ cm}^{-1}$, while the IR spectrum of 2 exhibited an band for C=N at 1614 cm^{-1} . ¹H NMR spectra of the monosubstituted cyclopentadienyl ligands in single clusters 1a-c and 2 showed corresponding proton resonance signals. For instance, the four protons on the substituted cyclopentadienyl ring, similar to those of corresponding μ_3 -S cluster complexes,¹ belong to an AA'BB' pattern and usually exhibited, due to overlap, two quartets at higher field assigned to H³ and H⁴, and one triplet (one multiplet for **1b**) at lower field assigned to H² and H⁵ of the cyclopentadienyl ring. Although the mass spectrum of **1a** displayed only the corresponding fragment ion peaks, the mass spectra of **1b** and **1c** showed their molecular ion peaks. It is worth pointing out that the peak of fragment ion [MFeCoSe]⁺ existed in all

376

mass spectra of 1a-c, which indicated that the tetrahedral cluster core MCoFe(μ_3 -Se) is quite stable.

For double clusters 3a,b, 4 and 5, the IR spectra contained four to six absorption bands in the range $1877-2073 \,\mathrm{cm}^{-1}$ for terminal carbonyls, besides one absorption band at around 1680 cm⁻¹ for ketonic carbonyls. ¹H NMR spectra showed one singlet at about 3.1 ppm for the bridged ethylene group, whereas double clusters 4 and 5 exhibited another singlet at about 5.3 ppm for the parent Cp ring coordinated to the Ni atom. In addition, the four protons for each substituted cyclopentadienyl ring in 3a,b, 4 and 5 are also in an AA'BB' pattern and showed three sets of resonance signals due to overlap, namely, the upfield two sets of signals, usually two multiplets, were assigned to H³ and H⁴ remote from the succinoyl bridge and the downfield signal was attributed to H^2 and H^5 close to the bridge. It is worth noting that for 3a,b and 5 there must be a pair of enantiomers and meso form and for 4 two pairs of enantiomers, due to the presence of tetrahedral cluster cores of chirality. However, they could not be separated into single optical isomers by TLC and might exist as a mixture of several optical isomers. This is consistent with the four protons for each substituted cyclopentadienyl ring of **3a,b**, **4** and **5** having such complicated ¹H NMR spectra.

Single-Crystal Structure of 1c

In order to further confirm the structures of the tetrahedral MCoFe(μ_3 -Se) cluster complexes, an X-ray diffraction analysis of 1c was undertaken. Final atomic coordinates with equivalent isotropic thermal factors are listed in Table II. Although there are two crystallographically independent molecules (i.e., A and B) in the asymmetric unit (see Figure 1), only selected bond lengths and bond angles of A are listed in Table III. This is because the structural features of two independent molecules A and B are essentially the same. As seen from Figure 1, the molecule contains a chiral tetrahedral cluster core WCoFeSe, eight carbonyls and a MeO₂C-substituted cyclopentadienyl ring. The Se atom is coordinated to the W, Fe and Co atoms in a μ_3 -type of arrangement. The substituted cyclopentadienyl ring is coordinated to the W atom and the distance of the W atom to cyclopentadienyl ring centroid is 1.99(1) A. All eight carbonyls attached to W, Fe and Co atoms in the molecule are terminal, which is consistent with IR bands in the range $1918-2065 \text{ cm}^{-1}$. The substituent MeO₂C is conjugated very well with the π -system of the cyclopentadienyl ring, since the dihedral angle between the plane of the cyclopentadienyl ring and that of O(27)-C(26)-O(28) is quite small (4.71°) and the bond length C(21)-C(26) (1.47(2)Å) is much

	Atom	x/a	y/b	z/c	B (eq)
	W(1)	0.02972(3)	0.03351(5)	0.19102(3)	2.42(2)
$\begin{array}{cccc} Sc(1) & -0.05310(9) & 0.147(1) & 0.26346(9) & 4.61(8) \\ Sc(2) & 0.15742(7) & 0.4115(1) & 0.50346(8) & 3.18(6) \\ Co(1) & -0.0686(1) & -0.0472(2) & 0.2827(1) & 3.8(1) \\ Co(2) & 0.1361(1) & 0.5978(2) & 0.3330(1) & 3.20(8) \\ Fc(1) & 0.0464(41) & 0.0692(2) & 0.3366(1) & 3.11(9) \\ Fc(2) & 0.2426(1) & 0.4743(2) & 0.5957(1) & 3.34(9) \\ O(1) & 0.1528(7) & 0.221(1) & 0.1959(7) & 6.77) \\ O(2) & 0.1622(5) & -0.129(1) & 0.2453(6) & 4.9(5) \\ O(3) & -0.2080(6) & -0.065(2) & 0.1878(8) & 11(1) \\ O(4) & -0.1263(7) & -0.082(2) & 0.2407(7) & 8.6(9) \\ O(5) & 0.0055(7) & -0.264(1) & 0.2675(7) & 6.3(7) \\ O(6) & -0.0157(8) & 0.135(1) & 0.4660(7) & 7.7(8) \\ O(7) & 0.1288(6) & -0.122(1) & 0.4030(7) & 5.6(6) \\ O(8) & 0.1678(9) & 0.236(1) & 0.3536(8) & 9(1) \\ O(9) & 0.3744(6) & 0.691(1) & 0.3566(7) & 5.9(6) \\ O(10) & 0.3819(6) & 0.539(1) & 0.4740(7) & 6.2(7) \\ O(11) & 0.018(6) & 0.659(1) & 0.4214(7) & 6.0(7) \\ O(12) & 0.2122(7) & 0.818(1) & 0.6625(8) & 6.7(7) \\ O(13) & 0.038(8) & 0.588(1) & 0.6453(8) & 8.0(8) \\ O(14) & 0.3612(7) & -0.302(1) & 0.6287(8) & 8.0(8) \\ O(15) & 0.1561(8) & 0.382(2) & 0.7062(8) & 9(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.6287(8) & 8.0(8) \\ O(15) & 0.151(8) & 0.382(2) & 0.7062(8) & 9(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.6287(8) & 8.1(8) \\ O(15) & 0.151(8) & 0.363(1) & 0.4643(8) & 8.0(8) \\ O(15) & 0.151(8) & 0.363(1) & 0.4643(8) & 8.0(8) \\ O(14) & -0.1033(8) & -0.067(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.230(1) & 3.2(7) \\ C(3) & -0.1544(9) & -0.060(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.359(1) & 3.3(9) \\ C(7) & 0.981(7) & -0.067(1) & 0.3398(8) & 3.2(6) \\ C(3) & -0.1544(9) & -0.069(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.359(1) & 3.3(7) \\ C(5) & -0.0222(8) & -0.179(2) & 0.355(4) & 9.458(1) \\ C(10) & 0.330(8) & 0.412(1) & 0.4699(9) & 3.8(7) \\ C(2) & 0.0320(7) & -0.090(1) & 0.0905(8) & 3.4(7) \\ C(3) & 0.0328(8) & 0.635(1) & 0.357(9) & 3.3(9) \\ C(1) & 0.0348(8) & 0.035(1) & 0.357(9) & 3.3(7) \\ C(3) & 0.0328(8) & 0.635(1) & 0.357(9) & 3.3(7) \\ C(3) & 0.0348($	W(2)	0.25285(3)	0.53384(5)	0.45348(3)	2.62(2)
$\begin{array}{cccc} Sc(2) & 0.15742(7) & 0.4115(1) & 0.50346(8) & 3.18(6) \\ Co(1) & -0.0686(1) & -0.0472(2) & 0.2827(1) & 3.8(1) \\ Co(2) & 0.1361(1) & 0.5978(2) & 0.5330(1) & 3.12(9) \\ Fe(1) & 0.0464(1) & 0.0692(2) & 0.3366(1) & 3.11(9) \\ Fe(2) & 0.2426(1) & 0.4743(2) & 0.5957(1) & 3.34(9) \\ O(1) & 0.1528(7) & 0.221(1) & 0.1959(7) & 6.7(7) \\ O(2) & 0.1622(5) & -0.129(1) & 0.2453(6) & 4.9(5) \\ O(3) & -0.2080(6) & -0.065(2) & 0.1878(8) & 11(1) \\ O(4) & -0.1263(7) & -0.284(1) & 0.2675(7) & 6.3(7) \\ O(5) & 0.0055(7) & -0.264(1) & 0.2675(7) & 6.3(7) \\ O(6) & -0.0157(8) & 0.135(1) & 0.4660(7) & 7.7(8) \\ O(7) & 0.1288(6) & -0.122(1) & 0.4030(7) & 5.6(6) \\ O(8) & 0.1678(9) & 0.236(1) & 0.3536(7) & 5.9(6) \\ O(10) & 0.3819(6) & 0.691(1) & 0.3566(7) & 5.9(6) \\ O(10) & 0.3819(6) & 0.659(1) & 0.4214(7) & 6.0(7) \\ O(12) & 0.0153(8) & 0.588(1) & 0.6453(8) & 8.0(8) \\ O(14) & 0.0538(8) & 0.588(1) & 0.6453(8) & 8.0(8) \\ O(15) & 0.151(8) & 0.352(2) & 0.7062(8) & 9(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.6829(8) & 8.1(8) \\ O(15) & 0.151(8) & 0.382(2) & 0.7062(8) & 9(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.2308(8) & 3.2(6) \\ O(10) & 0.1387(8) & 0.651(1) & 0.6829(8) & 8.1(8) \\ O(15) & 0.151(8) & 0.0657(2) & 0.230(1) & 6(1) \\ C(3) & -0.052(8) & -0.077(2) & 0.3732(9) & 4.2(8) \\ C(4) & -0.0133(8) & -0.067(2) & 0.230(1) & 6(1) \\ C(5) & -0.0222(8) & -0.179(2) & 0.3732(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.110(2) & 0.664(1) & 5.1(9) \\ C(10) & 0.3330(8) & 0.412(1) & 0.6699(9) & 3.9(7) \\ C(10) & 0.3320(8) & 0.412(1) & 0.6699(9) & 3.9(7) \\ C(10) & 0.3320(8) & 0.432(1) & 0.6699(9) & 3.9(7) \\ C(10) & 0.3320(8) & 0.412(1) & 0.6448(8) & 4.0(7) \\ C(23) & -0.0530(8) & -0.059(1) & 0.064(1) & 5.1(9) \\ C(4) & -0.058(8) & 0.033(1) & 0.0624(8) & 3.2(7) \\ C(24) & -0.058(8) & 0.033(1) & 0.0624(8) & 3.2(7) \\ C(25) & 0.0135(8) & 0.077(1) & 0.3381(7) & 3.3(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.0338(7) & 3.5(7) \\ C(24) & -0.058(8) & 0.065(1) & 0.3557(9) & 3.9(7) \\ C(35) & 0.$	Se(1)	-0.05310(9)	0.1447(1)	0.26346(9)	4.61(8)
$\begin{array}{cccc} Co(2) & -0.0686(1) & -0.0472(2) & 0.2827(1) & 3.8(1) \\ Co(2) & 0.136(1) & 0.5978(2) & 0.5330(1) & 3.20(8) \\ Fe(1) & 0.0464(1) & 0.0692(2) & 0.3366(1) & 3.11(9) \\ Fe(2) & 0.2426(1) & 0.4743(2) & 0.5957(1) & 3.34(9) \\ O(1) & 0.1528(7) & 0.221(1) & 0.1959(7) & 6.7(7) \\ O(2) & 0.1622(5) & -0.129(1) & 0.2453(6) & 4.9(5) \\ O(3) & -0.2080(6) & -0.065(2) & 0.1878(8) & 11(1) \\ O(4) & -0.1263(7) & -0.264(1) & 0.2675(7) & 6.3(7) \\ O(5) & 0.0055(7) & -0.264(1) & 0.2675(7) & 6.3(7) \\ O(6) & -0.0157(8) & 0.135(1) & 0.4660(7) & 7.7(8) \\ O(7) & 0.1288(6) & -0.122(1) & 0.4300(7) & 5.6(6) \\ O(8) & 0.1678(9) & 0.236(1) & 0.3536(8) & 9(1) \\ O(9) & 0.3744(6) & 0.691(1) & 0.5366(7) & 5.9(6) \\ O(10) & 0.3819(6) & 0.350(1) & 0.4740(7) & 6.2(7) \\ O(12) & 0.2122(7) & 0.818(1) & 0.5625(8) & 6.7(7) \\ O(12) & 0.2122(7) & 0.818(1) & 0.5625(8) & 6.7(7) \\ O(12) & 0.2122(7) & 0.818(1) & 0.5625(8) & 8.0(8) \\ O(14) & 0.3612(7) & 0.302(1) & 0.6453(8) & 8.0(8) \\ O(14) & 0.3612(7) & 0.302(1) & 0.6287(8) & 8.0(8) \\ O(14) & 0.3612(7) & 0.302(1) & 0.6287(8) & 8.0(8) \\ O(15) & 0.1561(8) & 0.382(2) & 0.7062(8) & 9(1) \\ O(16) & 0.1387(8) & 0.067(2) & 0.236(1) & 0.1977(8) & 4.2(7) \\ C(2) & 0.113(7) & -0.067(1) & 0.2308(8) & 3.2(6) \\ C(3) & -0.1544(9) & -0.060(2) & 0.223(1) & 7(1) \\ C(4) & -0.103(8) & -0.067(2) & 0.369(1) & 6(1) \\ C(5) & -0.0222(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(6) & 0.010(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.199 \\ C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4669(9) & 4.3(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.0634(1) & 5.199 \\ C(14) & 0.337(1) & 0.5184(2) & 0.375(7) & 3.0(6) \\ C(23) & -0.058(7) & -0.097(1) & 0.0375(7) & 3.0(6) \\ C(34) & -0.058(7) & -0.097(1) & 0.3752(7) & 3.0(6) \\ C(35) & 0.138(8) & 0.053(1) & 0.359(7) & 5.4(6) \\ C(24) & -0.058(7) & -0.097(1) & 0.338(7) & 2.5(5) \\ C(25) & 0.0138(8) & 0.059(1) & 0.3395(7) & 5.3(6) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3395(7) & 5.3(6) \\ C(36) & 0.$	Se(2)	0.15742(7)	0.4115(1)	0.50346(8)	3.18(6)
$\begin{array}{cccccc} Co(2) & 0.1361(1) & 0.978(2) & 0.5330(1) & 3.20(8) \\ Fe(1) & 0.0464(1) & 0.0692(2) & 0.3366(1) & 3.11(9) \\ Fe(2) & 0.2425(1) & 0.271(1) & 0.1959(7) & 6.7(7) \\ O(2) & 0.1528(7) & 0.221(1) & 0.2453(6) & 4.9(5) \\ O(3) & -0.2080(6) & -0.065(2) & 0.1878(8) & 11(1) \\ O(4) & -0.1263(7) & -0.082(2) & 0.4207(7) & 8.6(9) \\ O(5) & 0.0055(7) & -0.264(1) & 0.2675(7) & 6.3(7) \\ O(6) & -0.0157(8) & 0.135(1) & 0.4600(7) & 7.7(8) \\ O(7) & 0.1288(6) & -0.122(1) & 0.4030(7) & 5.6(6) \\ O(8) & 0.1678(9) & 0.236(1) & 0.3536(8) & 9(1) \\ O(9) & 0.3744(6) & 0.691(1) & 0.5366(7) & 5.9(6) \\ O(10) & 0.3819(6) & 0.350(1) & 0.4740(7) & 6.2(7) \\ O(11) & 0.0163(6) & 0.659(1) & 0.4740(7) & 6.2(7) \\ O(12) & 0.212(7) & 0.818(1) & 0.5625(8) & 6.7(7) \\ O(13) & 0.0358(8) & 0.588(1) & 0.6453(8) & 8.0(8) \\ O(14) & 0.3612(7) & 0.302(1) & 0.6287(8) & 8.0(8) \\ O(14) & 0.3612(7) & 0.302(1) & 0.6287(8) & 8.0(8) \\ O(14) & 0.3612(7) & -0.067(1) & 0.2308(8) & 3.2(6) \\ C(3) & -0.1544(9) & -0.066(2) & 0.223(1) & 7(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.7352(7) & 3.0(6) \\ C(3) & -0.1544(9) & -0.066(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.336(8) & 3.2(6) \\ C(3) & -0.1544(9) & -0.066(2) & 0.223(1) & 7(1) \\ C(5) & -0.022(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(3) & -0.1544(9) & -0.066(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.369(1) & 6.11 \\ C(5) & -0.022(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(6) & 0.100(1) & 0.110(2) & 0.446(1) & 5.1(9) \\ C(7) & 0.998(17) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.199(9) & 3.9(7) \\ C(10) & 0.333(8) & 0.412(1) & 0.499(9) & 3.4(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.0080(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.099(1) & 0.0077(8) & 3.6(7) \\ C(23) & 0.038(7) & 0.059(1) & 0.0381(7) & 2.5(5) \\ C(24) & -0.058(7) & 0.059(1) & 0.3381(7) & 2.3(6) \\ C(33) & 0.186(18) & 0.574(1) & 0.3381(7) & 3.3(6) \\ C(34) & -0.058(7) & 0.069(1) & 0.3395(7) & 3.8(7) \\ C(55) & 0.0138(8) & 0.057(1) & 0.3395(7) & 3.6(7) \\ C(24) & -0.058$	Co(1)	-0.0686(1)	-0.0472(2)	0.2827(1)	3.8(1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Co(2)	0.1361(1)	0.5978(2)	0.5330(1)	3.20(8)
$\begin{array}{ccccccc} Fe(2) & 0.2426(1) & 0.4743(2) & 0.5957(1) & 3.34(9) \\ O(1) & 0.1528(7) & 0.221(1) & 0.1959(7) & 6.7(7) \\ O(2) & 0.1622(5) & -0.129(1) & 0.2453(6) & 4.9(5) \\ O(3) & -0.2080(6) & -0.065(2) & 0.1878(8) & 11(1) \\ O(4) & -0.1263(7) & -0.082(2) & 0.4207(7) & 8.6(9) \\ O(5) & 0.0055(7) & -0.264(1) & 0.2675(7) & 6.3(7) \\ O(6) & -0.0157(8) & 0.135(1) & 0.4660(7) & 7.7(8) \\ O(7) & 0.1288(6) & -0.122(1) & 0.4030(7) & 5.6(6) \\ O(8) & 0.1678(9) & 0.236(1) & 0.3536(8) & 9(1) \\ O(9) & 0.3744(6) & 0.691(1) & 0.5356(7) & 5.9(6) \\ O(10) & 0.3819(6) & 0.550(1) & 0.4214(7) & 6.2(7) \\ O(11) & 0.0163(6) & 0.659(1) & 0.4214(7) & 6.2(7) \\ O(12) & 0.212(7) & 0.818(1) & 0.5625(8) & 6.7(7) \\ O(13) & 0.0358(8) & 0.588(1) & 0.6453(8) & 8.0(8) \\ O(15) & 0.1561(8) & 0.382(2) & 0.7662(8) & 9(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.6287(8) & 8.0(8) \\ O(15) & 0.1561(8) & 0.382(2) & 0.7662(8) & 9(1) \\ O(16) & 0.3187(8) & 0.651(1) & 0.2308(8) & 3.2(6) \\ C(3) & -0.1544(9) & -0.060(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.369(1) & 6(1) \\ C(5) & -0.0222(8) & -0.179(2) & 0.2732(9) & 4.2(7) \\ C(3) & -0.122(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(6) & 0.010(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.399 \\ C(14) & 0.0333(8) & 0.412(1) & 0.4694(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.337(1) & 0.510(1) & 0.731(2) & 0.6478(9) & 4.5(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.0603(1) & 4.7(8) \\ C(14) & -0.0538(7) & 0.059(1) & 0.063(1) & 5.109(9) & 3.9(7) \\ C(10) & 0.338(8) & 0.053(1) & 0.053(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.338(8) & 0.053(1) & 0.053(1) & 0.732(2) & 0.514(9) & 4.8(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.063(1) & 4.7(8) \\ C(14) & 0.073(8) & 0.035(1) & 0.035(8) & 3.2(6) \\ C(24) & -0.058(7) & 0.059(1) & 0.063(1) & 4.7(8) \\ C(15) & 0.188(1) & 0.059(1) & 0.0632(1) & 5.109(9) & 3.9(7) \\ C(25) & 0.0138(8) & 0.035(1) & 0.3398(7) & 5.0(6) \\ C(34) & -0.058(7) &$	Fe(1)	0.0464(1)	0.0692(2)	0.3366(1)	3.11(9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Fe(2)	0.2426(1)	0.4743(2)	0.5957(1)	3.34(9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(1)	0.1528(7)	0.221(1)	0.1959(7)	6.7(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(2)	0.1622(5)	-0.129(1)	0.2453(6)	4.9(5)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(3)	-0.2080(6)	-0.065(2)	0.1878(8)	11(1)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(4)	-0.1263(7)	-0.082(2)	0.4207(7)	8.6(9)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(5)	0.0055(7)	-0.264(1)	0.2675(7)	6.3(7)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(6)	-0.0157(8)	0.135(1)	0.4660(7)	7.7(8)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(7)	0.1288(6)	-0.122(1)	0.4030(7)	5.6(6)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(8)	0.1678(9)	0.236(1)	0.3536(8)	9(1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(9)	0.3744(6)	0.691(1)	0.5366(7)	5.9(6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(10)	0.3819(6)	0.350(1)	0.4740(7)	6.2(7)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(11)	0.0163(6)	0.659(1)	0.4214(7)	6.0(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(12)	0.2122(7)	0.818(1)	0.5625(8)	6.7(7)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(13)	0.0358(8)	0.588(1)	0.6453(8)	8.0(8)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(14)	0.3612(7)	0.302(1)	0.6287(8)	8.0(8)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	O(15)	0.1561(8)	0.382(2)	0.7062(8)	9(1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(16)	0.3187(8)	0.651(1)	0.6829(8)	8.1(8)
$\begin{array}{cccccc} C(2) & 0.1135(7) & -0.067(1) & 0.2308(8) & 3.2(6) \\ C(3) & -0.1544(9) & -0.060(2) & 0.223(1) & 7(1) \\ C(4) & -0.1033(8) & -0.067(2) & 0.369(1) & 6(1) \\ C(5) & -0.0222(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(6) & 0.010(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.3(9) \\ C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4699(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.664(1) & 5.1(9) \\ C(16) & 0.2884(8) & 0.584(2) & 0.6478(9) & 4.5(8) \\ C(21) & 0.0645(7) & 0.006(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.058(7) & 0.059(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.0072(8) & 3.6(7) \\ C(24) & -0.058(6) & 0.099(1) & 0.0352(6) & 3.9(5) \\ C(25) & 0.0135(8) & 0.097(1) & 0.072(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0354(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2587(7) & 0.479(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(38) & 0.4348(5) & 0.649(1) & 0.3728(7) & 5.5(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array} \right)$	C(1)	0.1063(9)	0.152(1)	0.1977(8)	4.2(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(2)	0.1135(7)	-0.067(1)	0.2308(8)	3.2(6)
$\begin{array}{cccccc} C(4) & -0.1033(8) & -0.067(2) & 0.369(1) & 6(1) \\ C(5) & -0.0222(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(6) & 0.010(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.3(9) \\ C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4699(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.664(1) & 5(1) \\ C(16) & 0.2884(8) & 0.584(2) & 0.6478(9) & 4.5(8) \\ C(21) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.058(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.3390(7) & 5.0(6) \\ C(38) & 0.4348(5) & 0.649(1) & 0.3728(7) & 5.5(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.4788(2) & 0.331(1) & 7(1) \\ C(1) & 0.500(1) & $	C(3)	-0.1544(9)	-0.060(2)	0.223(1)	7(1)
$\begin{array}{ccccc} C(5) & -0.0222(8) & -0.179(2) & 0.2732(9) & 4.2(8) \\ C(6) & 0.010(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.3(9) \\ C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4669(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.6644(1) & 5(1) \\ C(16) & 0.2884(8) & 0.584(2) & 0.664(1) & 5(1) \\ C(12) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.0588(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0975(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0624(8) & 3.2(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3549(8) & 3.6(7) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.3381(7) & 5.6(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(38) & 0.4348(5) & 0.649(1) & 0.3728(7) & 5.5(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.330(1) & 7(1) \\ \end{array}$	C(4)	-0.1033(8)	-0.067(2)	0.369(1)	6(1)
$\begin{array}{ccccc} C(6) & 0.010(1) & 0.110(2) & 0.416(1) & 5.2(9) \\ C(7) & 0.0981(7) & -0.047(1) & 0.3752(7) & 3.0(6) \\ C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.3(9) \\ C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4699(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.664(1) & 5(1) \\ C(16) & 0.2884(8) & 0.584(2) & 0.6478(9) & 4.5(8) \\ C(21) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.0588(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(38) & 0.4348(5) & 0.649(1) & 0.3728(7) & 5.5(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(5)	-0.0222(8)	-0.179(2)	0.2732(9)	4.2(8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(6)	0.010(1)	0.110(2)	0.416(1)	5.2(9)
$\begin{array}{cccccc} C(8) & 0.120(1) & 0.171(2) & 0.345(1) & 5.3(9) \\ C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4699(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.6648(1) & 5(1) \\ C(16) & 0.2884(8) & 0.584(2) & 0.6478(9) & 4.5(8) \\ C(21) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.0588(7) & 0.059(1) & 0.0972(8) & 3.8(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2587(7) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(7)	0.0981(7)	-0.047(1)	0.3752(7)	3.0(6)
$\begin{array}{cccccc} C(9) & 0.3267(8) & 0.633(1) & 0.5109(9) & 3.9(7) \\ C(10) & 0.3330(8) & 0.412(1) & 0.4699(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.664(1) & 5(1) \\ C(16) & 0.2884(8) & 0.584(2) & 0.6478(9) & 4.5(8) \\ C(21) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.058(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.3549(8) & 3.6(7) \\ C(32) & 0.2387(7) & 0.479(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.6(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(8)	0.120(1)	0.171(2)	0.345(1)	5.3(9)
$\begin{array}{ccccccc} C(10) & 0.330(8) & 0.412(1) & 0.4699(9) & 4.3(8) \\ C(11) & 0.0638(8) & 0.635(1) & 0.4644(8) & 4.0(7) \\ C(12) & 0.1849(8) & 0.732(2) & 0.5514(9) & 4.4(8) \\ C(13) & 0.0748(9) & 0.590(1) & 0.603(1) & 4.7(8) \\ C(14) & 0.317(1) & 0.371(1) & 0.614(1) & 5.1(9) \\ C(15) & 0.188(1) & 0.419(2) & 0.664(1) & 5(1) \\ C(16) & 0.2884(8) & 0.584(2) & 0.6478(9) & 4.5(8) \\ C(21) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.0588(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0905(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3395(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.356(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.3395(7) & 3.8(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3576(9) & 3.9(7) \\ C(36) & 0.3949(8) & 0.574(1) & 0.3395(7) & 5.6(6) \\ C(39) & 0.500(1) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(9)	0.3267(8)	0.633(1)	0.5109(9)	3.9(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(10)	0.3330(8)	0.412(1)	0.4699(9)	4.3(8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$C(\Pi)$	0.0638(8)	0.635(1)	0.4644(8)	4.0(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(12)	0.1849(8)	0.732(2)	0.5514(9)	4.4(8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(13)	0.0748(9)	0.390(1)	0.603(1)	4./(8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(14)	0.31/(1)	0.3/1(1)	0.614(1)	5.1(9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(15)	0.188(1)	0.419(2)	0.664(1)	5(1)
$\begin{array}{cccccc} C(21) & 0.0645(7) & 0.006(1) & 0.0804(7) & 2.5(5) \\ C(22) & 0.0230(7) & -0.090(1) & 0.0975(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.058(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3549(8) & 3.6(7) \\ C(32) & 0.2587(7) & 0.479(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(10)	0.2884(8)	0.584(2)	0.6478(9)	4.5(8)
$\begin{array}{cccccc} C(22) & 0.0250(7) & -0.050(1) & 0.0973(7) & 3.0(6) \\ C(23) & -0.0530(8) & -0.059(1) & 0.1024(8) & 3.5(7) \\ C(24) & -0.0588(7) & 0.059(1) & 0.0905(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3549(8) & 3.6(7) \\ C(32) & 0.2587(7) & 0.479(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(21)	0.0043(7)	0.000(1)	0.0804(7)	2.5(5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(22)	0.0230(7)	-0.090(1)	0.0975(7)	3.0(0)
$\begin{array}{ccccccc} C(24) & -0.038(7) & 0.039(1) & 0.090(8) & 3.6(7) \\ C(25) & 0.0135(8) & 0.097(1) & 0.0772(8) & 3.8(7) \\ C(26) & 0.1438(8) & 0.003(1) & 0.0624(8) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3549(8) & 3.6(7) \\ C(32) & 0.2587(7) & 0.479(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(23)	-0.0330(8)	-0.039(1)	0.1024(8)	3.3(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(24)	-0.0388(7)	0.039(1)	0.0903(8)	3.0(7)
$\begin{array}{ccccccc} C(25) & 0.1436(5) & 0.003(1) & 0.0024(6) & 3.2(6) \\ O(27) & 0.1646(5) & 0.0999(9) & 0.0362(6) & 3.9(5) \\ O(28) & 0.1838(6) & -0.081(1) & 0.0682(7) & 5.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3549(8) & 3.6(7) \\ C(32) & 0.2587(7) & 0.479(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(38) & 0.4348(5) & 0.649(1) & 0.3728(7) & 5.5(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	C(25)	0.0133(8)	0.097(1) 0.003(1)	0.0772(8)	3.8(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	O(27)	0.1436(8)	0.003(1)	0.0024(8)	3.2(0)
$\begin{array}{ccccccc} C(23) & 0.1836(3) & -0.081(1) & 0.0662(7) & 3.4(6) \\ C(29) & 0.2386(8) & 0.096(2) & 0.008(1) & 6(1) \\ C(31) & 0.3094(7) & 0.571(1) & 0.3549(8) & 3.6(7) \\ C(32) & 0.2587(7) & 0.479(1) & 0.3381(7) & 3.3(6) \\ C(33) & 0.1861(8) & 0.521(1) & 0.3395(7) & 3.8(7) \\ C(34) & 0.1890(8) & 0.635(1) & 0.3576(9) & 3.9(7) \\ C(35) & 0.2659(7) & 0.669(1) & 0.3668(8) & 3.4(6) \\ C(36) & 0.3949(8) & 0.574(1) & 0.355(1) & 4.1(7) \\ C(37) & 0.4187(5) & 0.4730(9) & 0.3309(7) & 5.0(6) \\ C(38) & 0.4348(5) & 0.649(1) & 0.3728(7) & 5.5(6) \\ C(39) & 0.500(1) & 0.458(2) & 0.331(1) & 7(1) \\ \end{array}$	O(27)	0.1040(5)	0.0999(9)	0.0302(0)	5.9(5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(20)	0.1336(0)	-0.081(1)	0.0062(7)	5.4(0)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(23)	0.2000(8)	0.090(2)	0.006(1)	36(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(32)	0.3594(7)	0.371(1) 0.479(1)	0.3347(0) 0.3381(7)	3.0(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(32)	0 1861(8)	0.772(1)	0.3301(7)	3.3(0)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(34)	0.1890(8)	0.635(1)	0.3576(9)	3 9(7)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(35)	0.2659(7)	0.669(1)	0.3668(8)	3 4(6)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(36)	0.3949(8)	0.574(1)	0.355(1)	4 1(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(37)	0.4187(5)	0.4730(9)	0.3309(7)	5 0(6)
C(39) 0.500(1) 0.458(2) 0.331(1) 7(1)	C(38)	0.4348(5)	0.649(1)	0.3728(7)	5 5(6)
	C(39)	0.500(1)	0.458(2)	0.331(1)	7(1)

TABLE II Atomic coordinates and equivalent isotropic temperature factors for 1c

TABLE	III Selected bond le	Selected bond lengths (Å) and angles (°) for 1c		
W(1)-Se(1)	2.484(2)	W(1)-Co(1)	2.751(2)	
W(1) - Fe(1)	2.789(2)	Se(1)-Fe(1)	2.305(3)	
Se(1) - Co(1)	2.323(3)	Co(1) - Fe(1)	2.574(3)	
W(1) - C(1)	1.94(1)	C(21) - C(26)	1.47(2)	
C(21) - C(22)	1.41(2)	C(26) - O(28)	1.21(2)	
C(26)-O(27)	1.32(2)			
C(1)-W(1)-C(2)	85.7(6)	Se(1)-W(1)-Co(1)	52.38(7)	
Se(1)-W(1)-Fe(1)	51.44(6)	CO(1) - W(1) - Fe(1)	55.37(7)	
Fe(1)-Se(1)-Co(1)	67.59(9)	Fe(1) - Se(1) - W(1)	71.13(7)	
Co(1)-Se(1)-W(1)	69.72(7)	C(3) - Co(1) - C(4)	102.5(7)	
Se(1)-Co(1)-Fe(1)	55.87(8)	Se(1)-Co(1)-W(1)	57.90(7)	
Fe(1)-Co(1)-W(1)	63.07(7)	C(6) - Fe(1) - C(7)	94.5(7)	
C(25)-C(21)-C(22)	106(1)	Se(1) - Fe(1) - Co(1)	56.53(9)	
Se(1) - Fe(1) - W(1)	57.43(7)	CO(1) - Fe(1) - W(1)	61.55(7)	
O(28)-C(26)-O(27)	124(1)	O(28)-C(26)-C(21)	124(Ì)	



FIGURE 1 Molecular structure of 1c, showing atom labeling scheme.

shorter than a normal C–C single bond. Bond lengths in the WCoFeSe cluster core are Se(1)–Fe(1)=2.305(3) Å,¹⁴ Fe(1)–Co(1)=2.574(3) Å,¹ Co(1)–W(1)=2.751(2) Å,¹ W(1)–Fe(1)=2.789(2) Å¹ Se(1)–W(1)=2.484(2) Å,¹⁵ and Se(1)–Co(1)=2.323(3) Å,¹⁶ which are similar to those corresponding bond lengths reported in the literature, respectively.

Supplementary Material

Tables of fractional atomic coordinates, thermal parameters, interatomic parameters, observed and calculated factors are available on request from the authors.

Acknowledgments

We are grateful to the National Natural Science Foundation of China, the State Key Laboratory of Structural Chemistry and the State Key Laboratory of Elemento-Organic Chemistry for financial support of this work.

References

- [1] L.-C. Song, J.-Y. Shen, Q.-M. Hu and X.-Y. Huang, Organometallics 14, 98 (1995).
- [2] L.-C. Song, Y.-B. Dong, Q.-M. Hu, X.-Y. Huang and J. Sun, Organometallics 16, 4540 (1997).
- [3] L.-C. Song, Y.-B. Dong, Q.-M. Hu, Y.-K. Li and J. Sun, Polyhedron 17, 1579 (1998).
- [4] L. Linford and H.G. Raubenheimer, Adv. Organomet. Chem. 32, 1 (1991).
- [5] L.C. Roof and J.W. Kolis, Chem. Rev. 93, 1037 (1993).
- [6] P. Mathur, S. Ghosh, A. Sarkar, C.V.V. Satyanarayana and V.G. Puranik, Organometallics 16, 4392 (1997).
- [7] L.-C. Song, C.-G. Yan, Q.-M. Hu, R.-J. Wang and T.C.W. Mak, Organometallics 14, 5513 (1995).
- [8] L.-C. Song, C.-G. Yan, Q.-M. Hu, R.-J. Wang, T.C.W. Mak and X.-Y. Huang, Organometallics 15, 1535 (1996).
- [9] M. Cowie, R.L. Dekock, T.R. Wagenmaker, D. Seyferth, R.S. Henderson and M.K. Gallagher, Organometallics 8, 119 (1989).
- [10] M.L. Jolly and D.J. Chazan, Inorg. Synth. 11, 22 (1968).
- [11] (a) W.P. Hart, D.W. Macomber and M.D. Rausch, J. Am. Chem. Soc. 102, 1196 (1980);
 (b) W.P. Hart, S. Dong and M.D. Rausch, J. Organomet. Chem. 282, 111 (1985).
- [12] T.E. Bitterwolf, J. Organomet. Chem. 386, 9 (1990).
- [13] (a) R. Hoffman, Angew. Chem., Int. Ed. Engl. 21, 711 (1982); (b) H. Vahrenkamp, Comments Inorg. Chem. 4, 253 (1985).
- [14] R.E. Bachman, K.H. Whitmire and J. van Hal, Organometallics 14, 1792 (1995).
- [15] M. Gorzellik, H. Bock, L. Gang, B. Nuber and M.L. Ziegler, J. Organomet. Chem. 412, 95 (1991).
- [16] P. Mathur, P. Sekar, C.V.V. Satyanarayana and M.F. Mahon, Organometallics 14, 2115 (1995).

380