

Synthesis of bridging carbyne butterfly Fe/Se cluster complexes via reaction of complex anions $[(\mu\text{-RSe})(\mu\text{-CO})\text{Fe}_2(\text{CO})_6]^-$ Crystal structure of $(\mu\text{-EtOC})(\mu\text{-}p\text{-MeC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$

Li-Cheng Song^{a,*}, Yi Sun^a, Qing-Mei Hu^{a,b}, Yang Liu^a

^a Department of Chemistry, State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, PR China

^b Laboratory of Organometallic Chemistry, Chinese Academy of Sciences, Shanghai 20032, PR China

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Abstract

A series of bridging carbyne butterfly Fe/Se cluster complexes $(\mu\text{-EtOC})(\mu\text{-RSe})\text{Fe}_2(\text{CO})_6$ (**2–8**; $\text{R} = p\text{-MeC}_6\text{H}_4$, $m\text{-MeC}_6\text{H}_4$, $o\text{-MeC}_6\text{H}_4$, $\alpha\text{-C}_{10}\text{H}_7$, $p\text{-MeOC}_6\text{H}_4$, $p\text{-BrC}_6\text{H}_4$, $p\text{-ClC}_6\text{H}_4$) have been synthesized by in situ reaction of the $[\text{Et}_3\text{NH}]^+$ salts of anions $[(\mu\text{-RSe})(\mu\text{-CO})\text{Fe}_2(\text{CO})_6]^-$ (**1**) with electrophile Et_3OBF_4 in benzene at room temperature. Products **2–8** are new and have been characterized by elemental analysis, and IR, ^1H - and ^{13}C -NMR spectroscopy, as well as by X-ray diffraction analysis. The ^1H - and ^{13}C -NMR spectra indicated that **2–8** in solution consist of two isomers $\text{R}(a)$ and $\text{R}(e)$, whereas the X-ray diffraction analysis revealed that the single crystal of **2** ($\text{R} = p\text{-MeC}_6\text{H}_4$) is composed of one isomer $\text{R}(a)$. **2** is the first structurally characterized complex synthesized by reaction of anions **1** with Et_3OBF_4 , which strongly supports the view of anions **1** being O-centered anions in such a type of reactions.

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1. Introduction

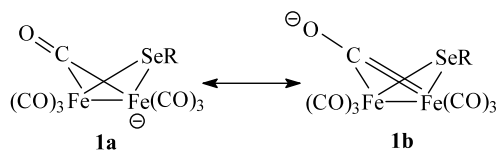
Previous studies [1–11] have indicated that anions $[(\mu\text{-RSe})(\mu\text{-CO})\text{Fe}_2(\text{CO})_6]^-$ (**1**), similar to their sulfur analogues $[(\mu\text{-RS})(\mu\text{-CO})\text{Fe}_2(\text{CO})_6]^-$, may display ambident chemical reactivity in their reactions with electrophiles, which can be readily understood upon consideration of the two resonance structures: Fe-centered anions **1a** and O-centered anions **1b** (Scheme 1).

As shown in Scheme 1, **1a** contains a normal $\mu\text{-CO}$ ligand while **1b** has a deprotonated hydroxy carbyne. So, in terms of the ambident reactivity of anions **1**, the electrophiles may attack at the negatively charged Fe

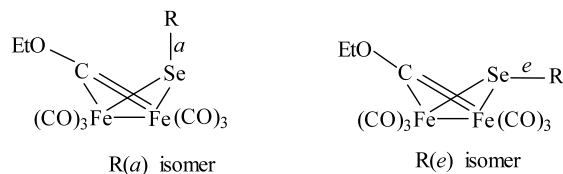
atom of **1a** followed by replacing the $\mu\text{-CO}$ ligand to give the corresponding Fe/Se cluster complexes or they may attack at the negatively charged oxygen atom of **1b** to afford O-alkylated bridging carbyne complexes. In fact, as previously indicated, the electrophiles usually attack the negatively charged Fe atom to give the expected Fe/Se cluster complexes [1–11]. Now, we wish to report the synthesis of new O-alkylated bridging carbyne complexes $(\mu\text{-EtOC})(\mu\text{-RSe})\text{Fe}_2(\text{CO})_6$ (**2–8**; $\text{R} = p$, m or $o\text{-MeC}_6\text{H}_4$, $\alpha\text{-C}_{10}\text{H}_7$, $p\text{-MeOC}_6\text{H}_4$, $p\text{-BrC}_6\text{H}_4$, $p\text{-ClC}_6\text{H}_4$), which were produced by attack of the ethyl cation derived from electrophile Et_3OBF_4 at the negatively charged oxygen of **1b**. In addition, the first crystal molecular structure for the complexes derived from reaction of anions **1** and Et_3OBF_4 is also reported, which strongly supports that anions **1** and their sulfur analogues may act as O-centered anions in the reactions with electrophile Et_3OBF_4 under given conditions.

* Corresponding author. Tel.: +86-22-23504452; fax: +86-22-23504853.

E-mail address: lsong@public.tpt.tj.cn (L.-C. Song).



Scheme 1.



Scheme 3.

2. Results and discussion

2.1. Reaction of $[(\mu-RSe)(\mu-CO)Fe_2(CO)_6]^-$ (1**) with Et_3OBF_4 leading to $(\mu-EtOC)(\mu-RSe)Fe_2(CO)_6$ (**2–8**; $R = p, m$ or $o-MeC_6H_4$, $\alpha-C_{10}H_7$, $p-MeOC_6H_4$, $p-BrC_6H_4$, $p-ClC_6H_4$)**

We found that the $[Et_3NH]^+$ salts of anions **1**, prepared from $RSeH$, $Fe_3(CO)_{12}$ and Et_3N in benzene [5], reacted in situ with ca. an equivalent of Et_3OBF_4 at room temperature to give a series of bridging carbonyl butterfly Fe/Se cluster complexes **2–8** (Scheme 2).

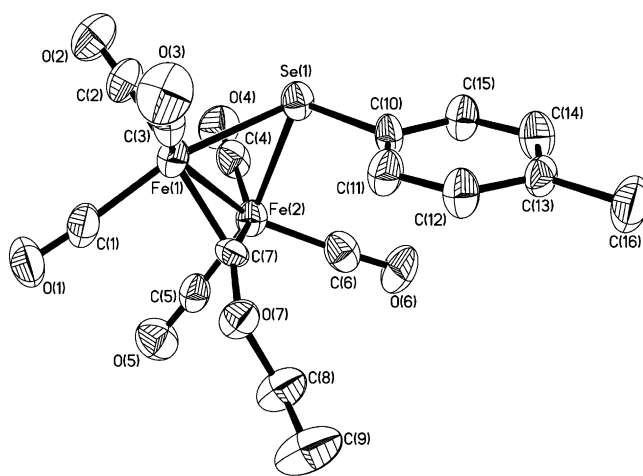
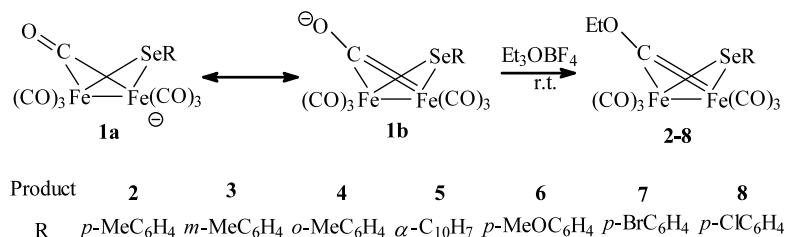
Products **2–8** can be simply regarded as produced by alkylation of the negatively charged oxygen atom of the resonance hybrid **1b** with ethyl cation Et^+ generated in situ from electrophile Et_3OBF_4 . These products have been fully characterized by elemental analysis, and IR, 1H - and ^{13}C -NMR spectroscopy, as well as by X-ray diffraction analysis for one representative product. For example, the IR spectra of **2–8** showed several absorption bands in the range 2068 – 1950 cm^{-1} for their terminal carbonyls and one absorption band in the region 1300 – 1296 cm^{-1} for their ether functionalities in carbonyl ligands, whereas the 1H -NMR spectra of **2–8** exhibited signals assignable to all the hydrogen-containing organic groups. However, it is worth pointing out that the ethyl group showed two sets of signals: one set of signals includes one broad singlet (for **4**, a triplet) at ca. 1.3 ppm assigned to CH_3 of the ethyl group and one broad singlet (for **4**, a quartet) at ca. 4.5 ppm assigned to CH_2 of the ethyl group, whereas the other set of signals contains one broad singlet (for **4**, a triplet) at ca. 1.6 ppm attributed to CH_3 and one broad singlet (for **4**, a quartet) at ca. 4.9 ppm attributed to CH_2 . This is consistent with that products **2–8** each consist of two isomers in which the substituent R is attached to the bridged Se atom by an axial or equatorial bond [12] (Scheme 3). It is also worth pointing out that the ^{13}C -NMR spectra of μ -carbonyl carbon of **2–8** was demon-

strated by the low field resonance of 388 – 377 ppm , which is in agreement with δC of the bridging carbonyl ligand of other carbonyl complexes reported previously by other workers [13–20]. In addition, the two singlets appeared in the low field for the carbonyl C of **2–8** are consistent with the fact that each of the complexes has two isomers of $R(a)$ and $R(e)$ (Scheme 3).

2.2. Crystal structure of **2**

To confirm the structures of the bridging carbonyl butterfly Fe/Se cluster complexes **2–8** generated from the resonance structure of **1b**, we carried out a single-crystal X-ray diffraction study on molecular structure of **2**. The ORTEP drawing of this molecular structure is shown in Fig. 1, whereas Table 1 lists its selected bond lengths and angles.

As can be seen in Fig. 1, complex **2** consists of a butterfly cluster core $Fe(1)Fe(2)C(7)Se(1)$, two sets of three CO's attached to $Fe(1)$ and $Fe(2)$, and two substituents EtO and $p-MeC_6H_4$ bound to $C(7)$ and

Fig. 1. Molecular structure of **2** showing the atom labeling scheme.

Scheme 2.

Table 1
Selected bond lengths (Å) and angles (°) for **2**

<i>Bond lengths</i>			
Fe(1)–C(7)	1.843(10)	Fe(1)–Se(1)	2.390(3)
Fe(1)–Fe(2)	2.513(4)	Fe(2)–Se(1)	2.438(3)
C(7)–O(7)	1.307(11)	O(7)–C(8)	1.489(13)
<i>Bond angles</i>			
C(7)–Fe(1)–Fe(2)	46.2(3)	Se(1)–Fe(1)–Fe(2)	59.58(6)
C(7)–Fe(2)–Fe(1)	47.1(3)	Se(1)–Fe(2)–Fe(1)	57.70(9)
Fe(1)–Se(1)–Fe(2)	62.72(10)	O(7)–C(7)–Fe(2)	141.4(8)
O(7)–C(7)–Fe(1)	131.2(8)	Fe(2)–C(7)–Fe(1)	86.7(4)
C(7)–O(7)–C(8)	118.0(8)	C(7)–Fe(1)–Se(1)	82.1(3)

Se(1) atoms, respectively. It is noteworthy that the selenolate and carbyne ligands are bridged to Fe(1) and Fe(2) unsymmetrically (the bond lengths Se(1)–Fe(1) = 2.390(3), Se(1)–Fe(2) = 2.438(3), C(7)–Fe(1) = 1.843(10) and C(7)–Fe(2) = 1.818(10) Å). The observed slight asymmetry would appear to be generated by nonbonding interactions between the two bridges, which are bent towards Fe(2) atom.

So, this structure is somewhat similar to that of μ -RS-containing carbyne complex $(\mu\text{-MeOCH=CHC})(\mu\text{-}t\text{-BuS})\text{Fe}_2(\text{CO})_6$ [13] prepared by another method. The average bond length (1.831 Å) of Fe(1)–C(7) and Fe(2)–C(7) in **2** is very close to the average Fe–C_{carbyne} bond length (1.866 Å) in $(\mu\text{-MeOCH=CHC})(\mu\text{-}t\text{-BuS})\text{Fe}_2(\text{CO})_6$ and both are much shorter than the bond length of the corresponding Fe–C_{carbene} bond length (1.976 Å) in complex $(\mu\text{-EtOC=CHCO}_2\text{CMe})(\mu\text{-}t\text{-BuS})\text{Fe}_2(\text{CO})_6$ [21]. In addition, it is worth noting that the angle of C(10)–Se(1)–C(7) (84.5°) reveals that the substituent *p*-MeC₆H₄ is attached to Se(1) by an axial bond, i.e. the crystal molecule of **2** belongs to R(*a*) isomer.

3. Experimental

All reactions were carried out under an atmosphere of highly purified nitrogen using standard Schlenk and vacuum-line techniques. Benzene was distilled under nitrogen from sodium/benzophenone ketyl, while triethylamine from potassium hydroxide. Fe₃(CO)₁₂ [22], Et₃OBf₄ [23] and the substituted aryl selenols [24] were prepared according to the literature procedures. The products were separated by TLC (20 × 25 × 0.25 cm, silica gel G) and further purified by recrystallization from a mixed diethyl ether–hexane solvent. IR spectra were recorded on a Bio–Rad FTS 135 or Bruker Vector 22 infrared spectrophotometer. ¹H-NMR spectra were recorded on a Bruker AC-P200 NMR spectrometer. ¹³C-NMR spectra were recorded on a Unity plus 400 or Mercury VX 300 spectrometer. C/H analyses were performed on an Elementar Vario EL analyzer.

Melting points were determined on a Yanaco MP-500 apparatus and were uncorrected.

3.1. Preparation of $(\mu\text{-EtOC})(\mu\text{-}p\text{-MeC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$ (**2**)

A 100 ml two-necked flask equipped with a magnetic stir bar, a N₂ inlet tube, and a serum cap was charged with 0.504 g (1.0 mmol) of Fe₃(CO)₁₂, 0.188 g (1.1 mmol) of *p*-MeC₆H₄SeH, 30 ml of benzene, and 0.17 ml (1.2 mmol) of Et₃N. The mixture was stirred at room temperature (r.t.) for ca. 20 min to give a red–brown solution containing [Et₃NH][$(\mu\text{-}p\text{-MeC}_6\text{H}_4\text{Se})(\mu\text{-CO})\text{-Fe}_2(\text{CO})_6$]. To the solution was added 0.19 g (1.0 mmol) of Et₃OBf₄ and then the mixture was stirred at r.t. for 4 h to give a deep red solution with some black precipitates. The resulting mixture was filtered and the filtrate was condensed under reduced pressure. The residue was subjected to TLC separation using acetone–petroleum ether (v/v, 1/20) as eluent. From the second major red band was obtained 0.232 g (46%) of **2** as a red solid. M.p. 126 °C (dec.). Anal. Found: C, 37.89; H, 2.62. C₁₆H₁₂Fe₂O₇Se. Calc.: C, 37.91; H, 2.39%. IR (KBr disk): $\nu(\text{C–O–C}_{\text{carbyne}})$, 1296s; terminal C≡O, 2066s, 2026vs, 1993vs, 1970vs cm^{–1}. ¹H-NMR(CDCl₃): δ 1.62, 1.24 (2br.s, 3H, CH₂CH₃), 2.33, 2.23 (2s, 3H, *e-p*-CH₃C₆H₄, *a-p*-CH₃C₆H₄), 4.87, 4.55 (2br.s, 2H, CH₂), 7.50–6.90 (m, 4H, C₆H₄) ppm. ¹³C-NMR(CDCl₃): δ 15.27, 14.70 (2s, CH₂CH₃), 21.34 (s, *p*-CH₃), 87.39 (s, CH₂), 138.25–125.60 (more than 6 s, C₆H₄), 209.64, 210.99, 210.94 (3s, Fe–CO), 387.23, 379.16 (2 s, carbyne C) ppm.

3.2. Preparation of $(\mu\text{-EtOC})(\mu\text{-}m\text{-MeC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$ (**3**)

The same procedure as that for **2** was followed, but using 0.188 g (1.1 mmol) of *m*-MeC₆H₄SeH instead of *p*-MeC₆H₄SeH to give 0.179 g (35%) of **3** as a red solid. M.p. 54–55 °C. Anal. Found: C, 37.74; H, 2.41. C₁₆H₁₂Fe₂O₇Se. Calc.: C, 37.91; H, 2.39%. IR (KBr disk): $\nu(\text{C–O–C}_{\text{carbyne}})$, 1296s; terminal C≡O, 2066s, 2026vs, 1997vs, 1977vs, 1950s cm^{–1}. ¹H-NMR(CDCl₃): δ 1.56, 1.23 (2br.s, 3H, CH₂CH₃), 2.34, 2.22 (2s, 3H, *e-m*-CH₃C₆H₄, *a-m*-CH₃C₆H₄), 4.84, 4.54 (2br.s, 2H, CH₂), 7.41–6.96 (m, 4H, C₆H₄) ppm. ¹³C-NMR(CDCl₃): δ 14.42 (s, CH₂CH₃), 21.29 (s, *m*-CH₃), 87.23 (s, CH₂), 138.60–128.81 (more than 6s, C₆H₄), 210.52 (br.s, Fe–CO), 386.96, 379.03 (2s, carbyne C) ppm.

3.3. Preparation of $(\mu\text{-EtOC})(\mu\text{-}o\text{-MeC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$ (**4**)

The same procedure as that for **2** was followed, but using 0.188 g (1.1 mmol) of *o*-MeC₆H₄SeH instead of *p*-

MeC₆H₄SeH to give 0.301 g (59%) of **4** as a red solid. M.p. 61–62 °C. Anal. Found: C, 37.92; H, 2.51. C₁₆H₁₂Fe₂O₇Se. Calc.: C, 37.91; H, 2.39%. IR (KBr disk): $\nu(\text{C}-\text{O}-\text{C}_{\text{carbyne}})$, 1300s; terminal C≡O, 2066s, 2026vs, 1997vs, 1970vs, 1950s cm⁻¹. ¹H-NMR(CDCl₃): δ 1.63, 1.38 (2t, 3H, CH₂CH₃), 2.58, 2.51 (2s, 3H, *e*-o-CH₃C₆H₄, *a*-o-CH₃C₆H₄), 4.86, 4.58 (2q, 2H, CH₂), 7.70–6.82 (m, 4H, C₆H₄) ppm. ¹³C-NMR (CDCl₃): δ 15.30, 14.79 (2s, CH₂CH₃), 23.98, 22.92 (2s, *o*-CH₃), 87.61, 87.33 (2s, CH₂), 143.16–125.05 (more than 6s, C₆H₄), 210.91, 210.34, 209.58 (3s, FeCO), 386.92, 379.91 (2s, carbyne C) ppm.

3.4. Preparation of $(\mu\text{-EtOC})(\mu\text{-}\alpha\text{-C}_{10}\text{H}_7\text{Se})\text{Fe}_2(\text{CO})_6$ (**5**)

The same procedure as that for **2** was followed, but using 0.228 g (1.1 mmol) of $\alpha\text{-C}_{10}\text{H}_7\text{SeH}$ instead of *p*-MeC₆H₄SeH and using CH₂Cl₂–petroleum ether (v/v, 1/20) in place of acetone–petroleum ether as eluent to afford 0.280 g (52%) of **5** as a red solid. M.p. 142 °C (dec). Anal. Found: C, 42.05; H, 2.21. C₁₉H₁₂Fe₂O₇Se. Calc.: C, 42.03; H, 2.23%. IR (KBr disk): $\nu(\text{C}-\text{O}-\text{C}_{\text{carbyne}})$, 1299s; terminal C≡O, 2065s, 2024vs, 2003vs, 1972vs, 1953s cm⁻¹. ¹H-NMR(CDCl₃): δ 1.66, 1.20 (2br.s, 3H, CH₂CH₃), 4.91, 4.49 (2br.s, 2H, CH₂), 8.46–7.17 (m, 7H, *el* α -C₁₀H₇) ppm. ¹³C-NMR (CDCl₃): δ 14.48, 13.93 (2s, CH₂CH₃), 86.91(s, CH₂), 137.98–124.64 (more than 5 s, C₁₀H₇), 209.98, 209.45 (2s, FeCO), 385.53, 377.62 (2s, carbyne C) ppm.

3.5. Preparation of $(\mu\text{-EtOC})(\mu\text{-p-MeOC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$ (**6**)

The same procedure as that for **2** was followed, but using 0.200 g (1.1 mmol) of *p*-MeOC₆H₄SeH instead of *p*-MeC₆H₄SeH to produce 0.216 g (54%) of **6** as a red oil. Anal. Found: C, 36.64; H, 2.45. C₁₆H₁₂Fe₂O₈Se. Calc.: C, 36.75; H, 2.31%. IR (KBr disk): $\nu(\text{C}-\text{O}-\text{C}_{\text{carbyne}})$, 1298s; terminal C≡O, 2066s, 2024vs, 1997vs, 1980vs cm⁻¹. ¹H-NMR(CDCl₃): δ 1.61, 1.26 (2br.s, 3H, CH₂CH₃), 3.71 (br.s, 3H, *el* α -p-CH₃OC₆H₄), 4.82, 4.54 (2br.s, 2H, CH₂), 7.49–6.61(m, 4H, C₆H₄) ppm. ¹³C-NMR (CDCl₃): δ 14.46 (s, CH₂CH₃), 55.24 (s, *p*-CH₃O), 87.12 (s, CH₂), 135.13–114.36 (more than 6s, C₆H₄), 210.69, 210.25 (2s, FeCO), 387.14, 378.89 (2s, carbyne C) ppm.

3.6. Preparation of $(\mu\text{-EtOC})(\mu\text{-p-BrC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$ (**7**)

The same procedure as that for **2** was followed, but using 0.259 g (1.1 mmol) of *p*-BrC₆H₄SeH instead of *p*-MeC₆H₄SeH to afford 0.258 g (45%) of **7** as a red solid. M.p. 130 °C (dec). Anal. Found: C, 31.47; H, 1.68. C₁₅H₉BrFe₂O₇Se. Calc.: C, 31.51; H, 1.59%. IR (KBr

disk): $\nu(\text{C}-\text{O}-\text{C}_{\text{carbyne}})$, 1298m; terminal C≡O, 2068s, 2031vs, 1995vs cm⁻¹. ¹H-NMR(CDCl₃): δ 1.61, 1.29 (2br.s, 3H, CH₂CH₃), 4.84, 4.58 (2br.s, 2H, CH₂), 7.23 (br.s, 4H, *el* α -p-BrC₆H₄) ppm. ¹³C-NMR (CDCl₃): δ 15.13, 14.61 (2s, CH₂CH₃), 87.48 (s, CH₂), 137.06–123.42 (more than 6 s, C₆H₄), 210.45, 209.95, 208.88 (3s, FeCO), 386.48, 379.01 (2s, carbyne C) ppm.

3.7. Preparation of $(\mu\text{-EtOC})(\mu\text{-p-ClC}_6\text{H}_4\text{Se})\text{Fe}_2(\text{CO})_6$ (**8**)

The same procedure as that for **2** was followed, but using 0.211 g (1.1 mmol) of *p*-ClC₆H₄SeH instead of *p*-MeC₆H₄SeH to afford 0.68 g (9%) of **8** as a red oil. Anal. Found: C, 33.96; H, 1.75. C₁₅H₉ClFe₂O₇Se. Calc.: C, 34.16; H, 1.72%. IR (KBr disk): $\nu(\text{C}-\text{O}-\text{C}_{\text{carbyne}})$, 1296m; terminal C≡O, 2067s, 2026vs, 2007vs, 1988vs, 1968s cm⁻¹. ¹H-NMR(CDCl₃): δ 1.63, 1.29 (2br.s, 3H, CH₂CH₃), 4.87, 4.56 (2br.s, 2H, CH₂), 7.24 (br.s, 4H, C₆H₄) ppm.

3.8. Single crystal structure determination of **2**

Single crystals of **2** suitable for X-ray diffraction analysis was grown by slow evaporation of its acetone–hexane solution at about 4 °C. This crystal was mounted on a Bruker SMART 1000 automated diffractometer equipped with a graphite monochromator with Mo–K α radiation ($\lambda = 0.71073$ Å). Details of the crystal data, data collection, and structure refinements are summarized in Table 2. The structure was solved by direct methods and expanded by Fourier techniques.

Table 2
Crystal data and structural refinements details for **2**

Empirical Formula	C ₁₆ H ₁₂ Fe ₂ O ₇ Se
Formula weight	506.92
Temperature (K)	293(2)
Crystal system	Triclinic
Space group	<i>P</i> $\bar{1}$
<i>a</i> (Å)	9.530(12)
<i>b</i> (Å)	9.530(12)
<i>c</i> (Å)	11.988(15)
α (°)	73.88(3)
β (°)	73.88(3)
γ (°)	69.167(6)
<i>V</i> (Å ³)	958(2)
<i>Z</i>	2
<i>D</i> _{calc} (g cm ⁻³)	1.757
<i>F</i> (000)	500
μ (Mo–K α) (mm ⁻¹)	3.446
Scan type	ω –2 θ
2 θ _{max} (°)	50.04
Data/restraints/parameters	3241/0/235
<i>R</i>	0.0716
<i>R</i> _w	0.1343
Goodness-of-fit indicator	0.909
Largest difference peak and hole (e Å ⁻³)	0.805 and –0.608

The final refinements were accomplished by the full-matrix least-squares method with anisotropic thermal parameters for non-hydrogen atoms. The calculations were performed using the TEXSAN crystallographic software package of Molecular Structure Corporation.

4. Supplementary material

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center, CCDC no. 197383. 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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