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[HFe(CO)₄]⁻ as a reagent for the synthesis of tin / iron clusters. Partial crystal structure of (NEt₄)₂[SnCl₂{Fe(CO)₄}₂] · SnCl₄

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Abstract

The reaction of the NEt₄⁺ and PPN⁺ (bis(triphenylphosphine)nitrogen(+)) salts of [HFe(CO)₄]⁻ with several tin halides has been investigated. Reaction of (NEt₄)[HFe(CO)₄] with ClSnR₃ (R = C₆H₅, *p*-C₆H₄(CH₃)) and Cl₂Sn(C₆H₅)₂ gives the bimetallic species (NEt₄)[R₃SnFe(CO)₄] and (PPN)[Cl(C₆H₅)₂SnFe(CO)₄], respectively. Reaction of SnCl₄ with (PPN)[HFe(CO)₄] gives the orange trimetallic complex (PPN)₂[Cl₂Sn{Fe(CO)₄}₂]. Reaction of [NEt₄][HFe(CO)₄] with SnCl₄ in toluene gives the SnCl₄ solvate of the corresponding NEt₄⁺ salt, an X-ray diffraction study of which has enabled approximate location of the Sn, Fe, and Cl atoms. The Sn atom in the anion is tetrahedrally surrounded by two chlorides and two Fe(CO)₄ units with an average Sn–Fe bond distance of 2.58(3) Å. Addition of an excess of the NEt₄⁺ salt to SnCl₄ gives the red (NEt₄)₃[ClSn{Fe(CO)₄}₃], which reverts to (NEt₄)₂[Cl₂Sn{Fe(CO)₄}₂] up on treatment with 1 equivalent of SnCl₄. The use of (PPN)₂[Cl₂Sn{Fe(CO)₄}₂] as a possible precursor for larger nuclearity clusters has also been examined.

Introduction

Mixed main-group/transition metal clusters are of particular interest because of the possibility of using such clusters as molecular precursors in catalytic processes and as models for studies of cooperative effects [1]. In the course of our continuing investigations on complexes containing metal–metal bonds, we have shown that the

carbonylmetalate anion $[\text{HFe}(\text{CO})_4]^-$ is useful starting material for the synthesis of this class of derivatives. Thus, the reaction of the iron anion with SbCl_3 in THF solutions gives high yields of the tetrahedral cluster $(\text{PPN})_2[\text{ClSb}\{\text{Fe}(\text{CO})_4\}_3]$ through a process involving hydrogen chloride elimination [2]. Similar results are obtained with BiCl_3 . On the other hand, the reaction of the $(\text{NEt}_4)[\text{HFe}(\text{CO})_4]$ with EX_2 ($\text{E} = \text{Zn}, \text{Cd}, \text{Hg}$; $\text{X} = \text{halides}$), gives after ligand replacement, the di-hydrides $[(\text{OC})_4\text{HFeEFeH}(\text{CO})_4]$, which are in turn excellent precursors for the trimetallic anions $[(\text{OC})_4\text{FeEFe}(\text{CO})_4]^{2-}$ [3].

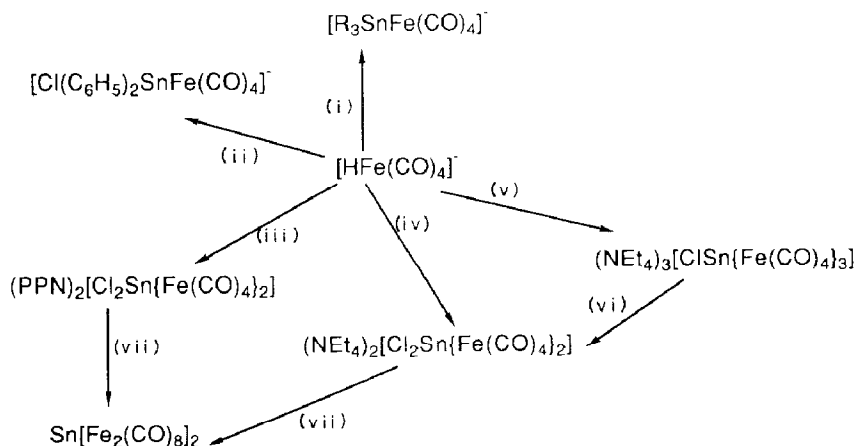
With the object of extending knowledge in the expanding field of iron-tin complexes [4], we have carried out a study of the synthesis and characterization of new mixed metal clusters, and the results are described below.

Results and discussion

Preparation of the complexes

In spite of the fact that the $[\text{HFe}(\text{CO})_4]^-$ anion is an excellent reagent for ligand replacement reactions, we have observed that when it is allowed to react with organometallic tin halides, condensation reactions with elimination of hydrogen chloride predominate. For example, the action of $(\text{NEt}_4)[\text{HFe}(\text{CO})_4]$ on THF solutions of ClSnR_3 ($\text{R} = \text{C}_6\text{H}_5, p\text{-C}_6\text{H}_4(\text{CH}_3)$) at room temperature gave the bimetallic $(\text{NEt}_4)[\text{R}_3\text{SnFe}(\text{CO})_4]$ compounds in high yields (Scheme 1). Although the $[\text{R}_3\text{SnFe}(\text{CO})_4]^-$ ($\text{R} = \text{Cl}$ [5]; C_6H_5 [6]) anion had been obtained earlier, the method described represents a substantial improvement in other synthetic routes. The analytical and IR spectral data for both complexes are listed in Table 1. The easy and clean formation of these bimetallic iron/tin compounds prompted us to investigate the possible synthesis of species of higher nuclearity.

Attempts to obtain a trimetallic Fe_2Sn system from $\text{Cl}_2\text{Sn}(\text{C}_6\text{H}_5)_2$ and $(\text{PPN})[\text{HFe}(\text{CO})_4]$ were unsuccessful. Addition of a large excess of the anion to THF solutions of the tin halide (molar ratio 3/1) invariably afforded the bimetallic $(\text{PPN})[\text{Cl}(\text{C}_6\text{H}_5)_2\text{SnFe}(\text{CO})_4]$. It seemed to us that the failure to attach a



Scheme 1. Reactions of $[\text{HFe}(\text{CO})_4]^-$ with tin compounds. (i) R_3SnCl ($\text{R} = \text{C}_6\text{H}_5, p\text{-C}_6\text{H}_4(\text{CH}_3)$); (ii) $\text{R} = \text{Cl}_2\text{Sn}(\text{C}_6\text{H}_5)_2$; (iii) $\text{R} = \text{SnCl}_4$; (iv) $\text{R} = \text{SnCl}_4$ (molar ratio 2Fe/1Sn); (v) $\text{R} = \text{SnCl}_4$ (molar ratio 3Fe/1Sn); (vi) $\text{R} = \text{SnCl}_4$; (vii) $\text{R} = \text{Cu}^+$ (molar ratio 1Sn/2Cu).

Table 1
Analytical and IR spectra data of complexes

Complexes	IR spectra (KBr) $\nu(\text{CO}) (\text{cm}^{-1})$	Analyses (found (calc.) (%))			
		C	H	N	Cl
$(\text{NEt}_4)[(\text{C}_6\text{H}_5)_3\text{SnFe}(\text{CO})_4]$	1990 (s)	55.29	5.70	1.92	
	1905 (s)	(55.24)	(5.40)	(2.15)	
	1865 (vs, br)				
$(\text{NEt}_4)[(p\text{-C}_6\text{H}_4(\text{CH}_3))_3\text{SnFe}(\text{CO})_4]$	1995 (s)	57.50	6.34	1.86	
	1907 (s)	(57.45)	(5.94)	(2.02)	
	1865 (vs, br)				
$(\text{PPN})[\text{Cl}(\text{C}_6\text{H}_5)_2\text{SnFe}(\text{CO})_4]$	2000 (s)	61.58	4.20	1.48	4.03
	1940 (s)	(61.54)	(3.97)	(1.38)	(3.49)
	1925 (s)				
	1900 (vs)				
	1885 (vs)				
$(\text{NEt}_4)_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$	2009 (m)	36.31	4.91	3.66	9.20
	1978 (s)	(36.68)	(5.13)	(3.56)	(9.02)
	1928 (s, sh)				
	1890 (vs, br)				
	1878 (vs, br)				
$(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$	2005 (w)	59.72	3.53	1.61	3.81
	1974 (s)	(59.95)	(3.77)	(1.74)	(4.42)
	1904 (vs)				
	1892 (vs)				
	1873 (s)				
$(\text{NEt}_4)_3[\text{ClSn}\{\text{Fe}(\text{CO})_4\}_3]$	2005 (w)	41.33	6.11	3.88	3.18
	1975 (m)	(41.24)	(5.77)	(4.00)	(3.38)
	1955 (s)				
	1860 (vs, br)				

second $\text{Fe}(\text{CO})_4$ fragment to tin might be due to the high steric hindrance at the tin atom, so we tried to minimize this effect by using the less crowded SnCl_4 as starting material. Reaction of $(\text{PPN})[\text{HFe}(\text{CO})_4]$ with SnCl_4 in toluene gave an insoluble orange compound, which after recrystallization from acetone yielded air-stable orange microcrystals of $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$. The formulation of this di-an-

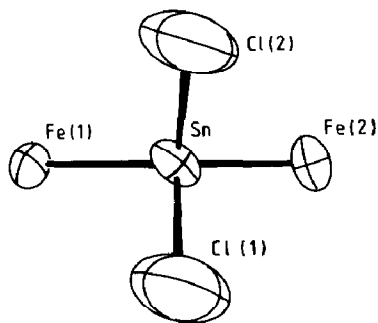


Fig. 1. Skeleton of the anion $[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]^{2-}$. Selected dimensions are as follows: Sn-Fe(1) 2.58(3), Sn-Fe(2) 2.57(3), Sn-Cl(1) 2.35(5), Sn-Cl(2) 2.35(4) Å; Fe(1)-Sn-Fe(2) 126(1), Fe(1)-Sn-Cl(1) 108(1), Fe(1)-Sn-Cl(2) 111(2), Fe(2)-Sn-Cl(1) 110(2), Fe(2)-Sn-Cl(2) 107(2), Cl(1)-Sn-Cl(2) 89(2)°.

Table 2

Some bond lengths (Å) and angles (°) in $[\text{NEt}_4][\text{SnCl}_2\{\text{Fe}(\text{CO})_4\}_2]\cdot\text{SnCl}_4$

Cl(11)–Sn(1)	2.52(4)	Cl(12)–Sn(1)–Cl(11)	134(1)
Cl(12)–Sn(1)	2.50(4)	Cl(13)–Sn(1)–Cl(11)	107(1)
Cl(13)–Sn(1)	2.55(4)	Cl(13)–Sn(1)–Cl(12)	106(1)
Cl(14)–Sn(1)	2.45(4)	Cl(14)–Sn(1)–Cl(11)	104(1)
Fe(1)–Sn(2)	2.58(3)	Cl(14)–Sn(1)–Cl(12)	104(1)
Fe(2)–Sn(2)	2.57(3)	Cl(14)–Sn(1)–Cl(13)	96(1)
Cl(21)–Sn(2)	2.35(5)	Fe(2)–Sn(2)–Fe(1)	126(1)
Cl(22)–Sn(2)	2.35(4)	Cl(21)–Sn(2)–Fe(1)	108(1)
		Cl(21)–Sn(2)–Fe(2)	110(2)
		Cl(22)–Sn(2)–Fe(1)	111(2)
		Cl(22)–Sn(2)–Fe(2)	107(2)
		Cl(22)–Sn(2)–Cl(21)	89(2)

ion was based on the elemental analyses and IR spectrum (Table 1). It is noteworthy that $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ did not react with an excess of $(\text{PPN})[\text{HFe}(\text{CO})_4]$.

We were not able to carry out an X-ray crystal structure determination $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{O})_4\}_2]$ because of extensive disorder in the crystals. Examination of a crystal of the NEt_4 salt (containing one molecule of solvation of SnCl_4 per anion) enabled us to locate the Sn, Fe, and Cl atoms in the core of the anion and the Sn and Cl atoms of the SnCl_4 . Because of considerable disorder no other atoms could be located.

A view of the core of the anion is shown in Fig. 1*; relevant bond angles and distances are given in Table 2 and atomic coordinates in Table 3. Further details are available from the authors.

The tin atom in the anion displays a distorted tetrahedral geometry with no Fe–Fe bonds. To the best of our knowledge this is the first example of a structurally authenticated tin complex with two independent $\text{Fe}(\text{CO})_4$ ligands. The mean tin–iron bond length, 2.58(3) Å, is intermediate between these previously found. $\text{Sn}[\text{Fe}_2(\text{CO})_8]_2$ (2.54 Å) [9], $[(\text{CH}_3)_3\text{SnFe}(\text{CO})_4]_2$ (2.65 Å) [10], $[(\text{C}_5\text{H}_5)_2\text{SnFe}(\text{CO})_4]_2$ (2.66 Å) [11], and $[\text{W}(\text{CO})_5\text{SnClFe}(\text{CO})_4]_2$ [$\text{Na}((\text{C}_2\text{H}_5)_2\text{O})_4(\text{C}_4\text{H}_8\text{O}_2)$] (2.694 Å) [12]. The complex can be also regarded as an opened trimetallic triangle Fe_2Sn , which is the expected geometry for a trimetallic system containing 50 electrons [13].

* Crystal data: Monoclinic, space group $\text{P}2_1/a$, a 22.207(8), b 16.822(4), c 21.086(8) Å, β 120.60(4)°, U 6780(7) Å³, Z = 4, $\lambda(\text{Mo-}K_\alpha)$ 0.71069 Å. A poor quality prismatic crystal (0.05 × 0.05 × 0.08 mm) was selected and mounted on a Philips PW-1100 four-circle diffractometer. Unit-cell parameters were determined from automatic centring of 25 reflections ($4 \leq \theta \leq 12^\circ$) and refined by least-squares method. Intensities were collected with graphite monochromatized Mo- K_α radiation, using the ω -scan technique with scan width 1.0° and scan speed 0.03 s⁻¹. 5643 reflections were measured in the range $2 \leq \theta \leq 25^\circ$ and 1794 of were taken as observed under the criterion $I \geq 2.5 \sigma(I)$. Three reflections were measured every two hours as orientation and intensity control. No significant intensity decay was observed. Lorentz-polarization but no absorption corrections were made. The structure was solved from a Patterson synthesis. The structure was refined by full-matrix least squares method, using the SHELX 76 computer program [7]. The function minimized was $\sum w\{|F_o| - |F_c|\}|^2$, where $w = (\sigma^2(F_o) + 0.007|F_o|^2)^{-1}$. f , f' , f'' were taken from [8]. The R factor at this point was 0.203 for all observed reflections.

Table 3

Atomic coordinates ($\times 10^4$) for Sn, Fe and Cl atoms in $[\text{NEt}_4][\text{SnCl}_2\{\text{Fe}(\text{CO})_4\}_2]\cdot\text{SnCl}_4$ (e.s.d. in parentheses)

	x/a	y/b	z/c	B_{eq}
Sn(1)	944(8)	2747(7)	4044(8)	4.59(96)
Sn(2)	6082(7)	2502(6)	3922(7)	3.53(78)
Fe(1)	5280(14)	3706(16)	3765(15)	5.13(202)
Fe(2)	6259(15)	1299(16)	4721(15)	5.10(215)
Cl(11)	1875(18)	2214(22)	5208(18)	1.34(231)
Cl(12)	209(20)	2219(22)	3116(19)	1.13(239)
Cl(13)	716(28)	4186(27)	4247(23)	6.48(325)
Cl(14)	1581(25)	3059(30)	3393(27)	5.53(373)
Cl(21)	7178(27)	2984(35)	4110(29)	9.15(411)
Cl(22)	5882(29)	2004(35)	2827(28)	9.04(414)

During the synthesis of $(\text{NEt}_4)_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ from $(\text{NEt}_4)[\text{HFe}(\text{CO})_4]$ and SnCl_4 we observed that, in contrast to the observations on the $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$, an excess of the anion gave a deep red solution, from which we isolated an air-unstable red product that analyzed as $(\text{NEt}_4)_3[\text{ClSn}\{\text{Fe}(\text{CO})_4\}_3]$. Moreover, its IR spectrum both in the solid and in solution was close to that of $(\text{PPN})_2[\text{ClSb}\{\text{Fe}(\text{CO})_4\}_3]$. Interestingly, treatment of the red compound with a stoichiometric amount of SnCl_4 gave the trimetallic $(\text{NEt}_4)_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ as indicated by its IR spectrum. This supports the formulation proposed for the tetrametallic cluster, and along with recent results of Whitmire [14] and ourselves [2] relating to the formation of antimony-iron clusters, indicates that the reactivity of the $[\text{HFe}(\text{CO})_4]^-$ salts towards halides of main group elements increases as the cation is varied in the sequence PPN^+ , NEt_4^+ , and K^+ .

Reactivity

We investigated the reactions of the tin/iron species prepared in this work in order to explore their potential in forming long metallic chains. We were especially interested in the complexes $(\text{PPN})[\text{ClR}_2\text{SnFe}(\text{CO})_4]$ and $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ because they display two potentially active centres, the chloride ligands and the negative iron; replacement of chloride in these species by metal fragments would increase their nuclearity, and on the other hand the nucleophilic character of the iron atoms could probably be used to advantage in metathetical reactions. These expectations were not fulfilled. Thus, addition of metal carbonylate anions, such as $[\text{M}(\text{CO})_3(\eta\text{-C}_5\text{H}_5)]^-$ ($\text{M} = \text{Mo}$ or W) to solutions of our complexes did not lead to any change, and the starting materials were recovered unaltered. Moreover, when the compounds $(\text{PPN})[\text{ClR}_2\text{SnFe}(\text{CO})_4]$ and $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ were treated with the electrophiles ClAuPPh_3 and HgCl_2 , the known $(\text{OC})_4\text{Fe}\{\text{AuPPh}_3\}_2$ and the insoluble, polymeric $[\text{HgFe}(\text{CO})_4]_x$, respectively were obtained, as indicated by the IR spectra, together with other minor products that were not investigated. These results are in good agreement with the view that long metallic chains break easily to give mixtures of compounds with fewer metal atoms [15].

The failure to replace the chloride of $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ by $[\text{M}(\text{CO})_3(\eta\text{-C}_5\text{H}_5)]^-$ fragments could be due to the negative charges on the trimetallic complex, which inhibits the approach of the nucleophiles. For this reason we thought that

oxidation of the tin-iron compound to $[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ would facilitate the metathetical reaction. However, addition of $[\text{Cu}(\text{NCCH}_3)_4]\text{BF}_4$ solutions of $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$ gave the spiro complex $\text{Sn}[\text{Fe}_2(\text{CO})_8]_2$ [16], as indicated by the IR spectrum. This suggests that alongside the oxidation of the iron atoms, there is another process, involving the abstraction of the chlorides by the cuprous cation to give a precipitate of CuCl .

Experimental

Solvents were dried by standard methods and all manipulations and reactions were performed in Schlenk-type vessels under dry nitrogen. Elemental analyses were carried out at the Institut de Bio-Orgànica de Barcelona. Infrared spectra were recorded on a Perkin-Elmer 1330 spectrophotometer.

Starting materials. Compounds $(\text{NEt}_4)[\text{HFe}(\text{CO})_4]$ and $(\text{PPN})[\text{HFe}(\text{CO})_4]$ ($\text{PPN} = \text{bis}(\text{triphenylphosphine})\text{nitrogen}(1 +)$) were prepared [17] as previously described. All other reagents were used as purchased.

Preparation of $Q[\text{R}_3\text{SnFe}(\text{CO})_4]$ ($\text{R} = \text{C}_6\text{H}_5, p\text{-C}_6\text{H}_4(\text{CH}_3)$) ($Q = \text{NEt}_4^+, \text{PPN}^+$). To a suspension of $(\text{NEt}_4)[\text{HFe}(\text{CO})_4]$ (0.67 g, 2 mmol) in THF (50 ml) was added solid Ph_3SnCl (0.78 g, 2 mmol) or $(p\text{-tolyl})_3\text{-SnCl}$ (0.85 g, 2 mmol). The mixture was stirred for 1 h at room temperature until the solid dissolved, and the solution was then concentrated to half volume and hexane was slowly added to precipitate a white solid, which was recrystallized from acetone/methanol. Yield 90%. The PPN salt was prepared similarly.

Preparation of $(\text{PPN})[\text{Cl}(\text{C}_6\text{H}_5)_2\text{SnFe}(\text{CO})_4]$. To a solution of $(\text{PPN})[\text{HFe}(\text{CO})_4]$ (1.58 g, 2.2 mmol) in THF (40 ml) precooled to -40°C was added dropwise a solution of $\text{Cl}_2\text{Sn}(\text{C}_6\text{H}_5)_2$ (0.61 g, 1.8 mmol) in THF (50 ml). The mixture solution was stirred for 1 h, filtered and evaporated to dryness in vacuo. The residual solid was extracted three times with acetone (3×20 ml), to give a colourless solution which was concentrated to 20 ml. An equal volume of ether was added and the mixture kept in a freezer overnight. The cream solid that separated was filtered off and dried in vacuo. Yield 70%.

Preparation of $(\text{NEt}_4)_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$. To a suspension of $(\text{NEt}_4)[\text{HFe}(\text{CO})_4]$ (3 g, 10 mmol) in toluene (40 ml) precooled to -20°C was added dropwise a solution of SnCl_4 (1.3 g, 5 mmol) in toluene (50 ml). During the addition a deep red solid formed. After 1 h stirring the red solid was filtered off and dried in vacuo. It was then extracted twice with acetone (2×20 ml), to give an orange-yellow solution, which was evaporated to dryness. The residual solid was dissolved in methanol and ether was added to precipitate an orange solid, which was filtered off and dried in vacuo. Yield 47%.

Preparation of $(\text{PPN})_2[\text{Cl}_2\text{Sn}\{\text{Fe}(\text{CO})_4\}_2]$. To a suspension of $(\text{PPN})[\text{HFe}(\text{CO})_4]$ (2 g, 2.8 mmol) in toluene (40 ml) precooled to -20°C was added dropwise a solution of SnCl_4 (0.3 g, 1.15 mmol) in toluene (10 ml). During the addition a red solid formed. After 1 h stirring the solid was filtered off, washed with THF, dried in vacuo and extracted twice with acetone (2×20 ml), to give a yellow solution. This was concentrated to 20 ml and hexane was slowly added with agitation to give a yellow powder, which was filtered off and dried in vacuo. Yield 40%.

Preparation of $(NEt_4)_3[ClSn\{Fe(CO)_4\}_3]$. To a suspension of $(NEt_4)[HFe(CO)_4]$ (3 g, 10 mmol) in toluene (40 ml) precooled to $-20^\circ C$ was added dropwise a solution of $SnCl_4$ (0.6 g, 2.5 mmol) in toluene (25 ml). During the addition a deep red solid formed. After 1 h stirring the solid was filtered off, washed several times with THF, and dried in vacuo, then extracted twice with acetone (2×20 ml) to give a deep red solution. This was concentrated to 20 ml and hexane was slowly added with agitation to precipitate a deep red solid, which was filtered off and dried in vacuo. Yield 35%.

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