dentate)$_3$(unidentate)], namely a configuration between that of a pentagonal bipyramid and a capped trigonal prism.

Registry No. TeO_2, 7446-07-3; $TeCl[(HOC_2H_4)_2NCSS]_3 \cdot 2H_2O$, 86455-82-5; diethyldithiocarbamic acid, 147-84-2.

Supplementary Material Available: Listings of anisotropic thermal parameters and structure factor amplitudes and Figures 3 and 4, depicting the hydrogen bonding in the molecule and packing in the unit cell (15 pages). Ordering information is given on any current masthead page.

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Synthesis and the Crystal and Molecular Structure of a Platinum-Bridged Derivative of Roussin's Red Salt, $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$[1]

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The reaction of Roussin's red salt $Na_2[Fe_2(\mu\text{-}S)_2(NO)_4]$ and *cis*-$(Ph_3P)_2PtCl_2$ in THF yields $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$. This compound also can be synthesized through the displacement of CO by NO from $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(CO)_6$ in solution. $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$ crystallizes in the monoclinic space group $C2/c$ (C_{2h}^6; No. 15) with $a = 20.867$ (7) Å, $b = 19.616$ (6) Å, $c = 20.524$ (4) Å, $\beta = 113.18$ (2)°, $V = 7723$ (3) Å^3, and $Z = 8$. Full-matrix least-squares refinement provided a final R value of 0.060 based on 3790 independent reflections. The planar S_2Fe_2 rhombus of Roussin's red salt is folded in the platinum adduct, and both linear and substantially bent nitrosyl ligands are present on each Fe atom.

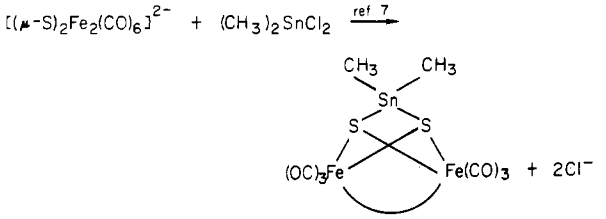
Introduction

Although Roussin's red salt was first reported in 1858,[3] its constitution and structure remained unknown for some time. In 1882, Pavel[4] correctly reported the composition of its potassium salt to be "$Fe(NO)_2SK + 2H_2O$". A dimeric constitution, "$Fe(NO)_2S$, $Fe(NO)_2$, and K_2S", was suggested.[4] Although the structure of the diethyl derivative, obtained by reaction of Roussin's red salt with iodoethane,[2] was determined in 1958 by Thomas et al.,[5] the structure of a salt of the $Fe_2S_2(NO)_4^{2-}$ anion was not reported until 1981.[6]

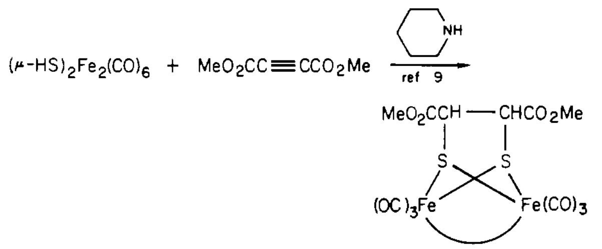
Both the diethyl derivative, $(\mu\text{-}C_2H_5S)_2Fe_2(NO)_4$, and the $S_2Fe_2(NO)_4^{2-}$ anion contain a planar Fe_2S_2 rhombus. Noteworthy is the large distance between S atoms in the Fe_2S_2 unit: 3.633 Å in the diethyl derivative[5] and 3.575 Å in anion.[6]

In earlier work, the seemingly related $Fe_2S_2(CO)_6^{2-}$ anion had been prepared by reaction of $Fe_2(\mu\text{-}S)_2(CO)_6$ with 2 molar equiv of $LiBEt_3H$ in THF at −78 °C.[7] Although the structure of a salt of this anion has not been reported to date, the crystal structure of its diethyl derivative, $(\mu\text{-}C_2H_5S)_2Fe_2(CO)_6$, showed that a puckered Fe_2S_2 core unit is present, with a S···S distance of 2.932 Å.[8] It may be expected that the S···S distance in the $S_2Fe_2(CO)_6^{2-}$ anion will only be slightly greater than that found for $(\mu\text{-}C_2H_5S)_2Fe_2(CO)_6$.

The proximity of the two sulfur atoms in these sulfur-ligand-bridged $Fe_2(CO)_6$ complexes has allowed the preparation of many $Fe_2(CO)_6$ species that contain diverse organic group, metalloid, and metal bridges between the sulfur atoms. The reactions in eq 1 and 2 are illustrative. Transition-metal derivatives such as the bis(triphenylphosphine)platinum(II) compound **1** were also prepared, by the reaction of either $(\mu\text{-}S)_2Fe_2(CO)_6^{2-}$ with $(Ph_3P)_2PtCl_2$[7] or $(\mu\text{-}S)_2Fe_2(CO)_6$ with $(Ph_3P)_4Pt$.[10]



(1)



(2)

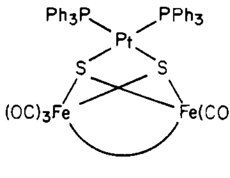


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Table I. Geometric Aspects of Some Iron Nitrosyl Dimers

	$(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$ (B = S)[d]	$(\mu\text{-}C_2H_5S)_2Fe_2\text{-}(NO)_4$[a] (B = S)	$(Me_4N)_2Fe_2S_2\text{-}(NO)_4$[b] (B = S)	$(\mu\text{-}I)_2Fe_2(NO)_4$[c] (B = I)
	Distances, Å			
Fe-Fe	2.802 (5)	2.720 (3)	2.715, 2.713	3.05
Fe-B	2.297 (7) (Fe1-S1) 2.274 (6) (Fe1-S2) 2.258 (6) (Fe2-S1) 2.269 (6) (Fe2-S2)	2.262 (4) 2.277 (4)	2.242-2.250	2.58
B···B	3.241 (7)	3.63	3.565, 3.575	4.15
Fe-N	1.69 (2) (Fe1-N1A) 1.65 (2) (Fe1-N1B) 1.69 (2) (Fe2-N2A) 1.61 (2) (Fe2-N2B)	1.67 (1)	1.660-1.677	1.67
	Angles, deg			
Fe-B-Fe	75.9 (3) (B = S1) 76.2 (3) (B = S2)	74	74.2, 74.5	72.6 (1)
B-Fe-B	90.3 (3) (Fe1) 91.4 (3) (Fe2)	106.0 (1)	105.8, 105.5	107.4 (1)
N-Fe-N	112.9 (8) (N1A-Fe1-N1B) 114.0 (9) (N2A-Fe2-N2B)	117.4 (2)		116
O-Fe-O	97.3 (9) (O1A-Fe1-O1B) 102.2 (8) (O2A-Fe2-O2B)	106.6 (2)		98
Fe-N-O	149.9 (13) (Fe1-N1A-O1A) 166.6 (14) (Fe1-N1B-O1B) 150.7 (14) (Fe2-N2A-O2A) 177.6 (14) (Fe2-N2B-O2B)	167.7 (35) 167.2 (35)		161 ± 4
Fe_2S_1-Fe_2S_2 dihedral angle	129.4			

[a] Reference 5. [b] Reference 6 (information given is very incomplete). [c] Reference 23. Mean values are presented. [d] B = bridging atom(s).

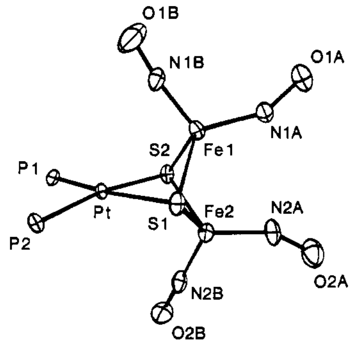
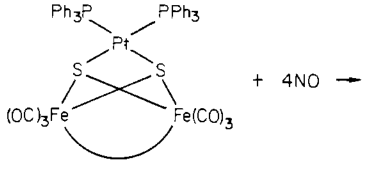


Figure 1. Molecular structure of $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$ (phenyl rings omitted for clarity, 50% probability thermal ellipsoids). Bond lengths: Fe1-Fe2 = 2.802 (5) Å, Fe1-Pt = 2.887 (3) Å, Fe2-Pt = 2.973 (3) Å, Pt-S1 = 2.426 (4) Å, Pt-S2 = 2.433 (4) Å, Pt-P1 = 2.279 (5) Å, Pt-P2 = 2.298 (4) Å. Bond angles: S1-Pt-S2 = 83.7 (3)°, P1-Pt-P2 = 99.2 (3)°, S1-Pt-P2 = 85.9 (3)°, S2-Pt-P1 = 91.4 (3)°.

A study of the reaction of Roussin's red salt with organometallic and organometalloidal electrophiles had shown that disubstituted derivatives, e.g., $(\mu\text{-}R_3SnS)_2Fe_2(NO)_4$ (R = CH_3, Ph), $(\mu\text{-}Ph_3PbS)_2Fe_2(NO)_4$, $(\mu\text{-}PhHgS)_2Fe_2(NO)_4$, and $[(\eta^5\text{-}C_5H_5)Fe(CO)_2S]_2Fe_2(NO)_4$, could be readily prepared.[1] In view of the long S···S nonbonded distance in the $(\mu\text{-}S)_2Fe_2(NO)_4^{2-}$ anion and the derived ethyl ester, it was by no means clear that bridged derivatives analogous to the known $Me_2Sn(\mu_3\text{-}S)_2Fe_2(CO)_6$, $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(CO)_6$, etc. could be prepared with the $S_2Fe_2(NO)_4$ system. For instance, if the S_2Fe_2-core geometry were to remain unchanged, the formation stable Me_2Sn or $(Ph_3P)_2Pt$ derivatives would only be possible if dimers or higher oligomers were formed. On the other hand, monomeric derivatives could only be expected if the Fe_2S_2-core geometry changed from the planar to a puckered configuration. This question, it was felt, was an interesting one that merited study, and we report here the results of our synthetic, spectroscopic, and structural studies of $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$.

Results and Discussion

The reaction of Roussin's red salt, $Na_2[S_2Fe_2(NO)_4]\cdot 8H_2O$, in THF with an equimolar quantity of *cis*-$(Ph_3P)_2PtCl_2$ (2 h at room temperature) gave a black, air-sensitive crystalline solid in 95% yield. Its C/H/N analysis was in agreement with its formulation as a $(Ph_3P)_2Pt$ derivative of Roussin's red salt, and determination of its molecular weight by field desorption mass spectroscopy and, in CH_2Cl_2 solution, by vapor pressure lowering indicated that it was a monomer, $(Ph_3P)_2PtS_2Fe_2(NO)_4$. An alternate, equally successful synthesis used the reaction of $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(CO)_6$[7] with NO in refluxing benzene (eq 3). The latter procedure has been used in other cases to convert ligand-bridged $Fe_2(CO)_6$ to $Fe_2(NO)_4$ complexes.[11]



$(Ph_2P)_2PtS_2Fe_2(NO)_4 + 6CO$ (3)
(90%)

The spectroscopy (IR, ^1H and ^{31}P NMR) of $(Ph_3P)_2PtS_2Fe_2(NO)_4$ (see Experimental Section) was not informative with regard to the structure, and therefore an X-ray crystal structure determination was undertaken.

The molecular structure of $(Ph_3P)_2PtS_2Fe_2(NO)_4$ that was determined in this study is shown in Figure 1. Selected bond lengths and angles are given in Table I and positional and isotropic thermal parameters are given in Table II. Two features of the $PtFe_2S_2$ cluster are especially noteworthy. First, the S_2Fe_2 rhombus is no longer planar: it has, as expected,

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Table II. Positional and Isotropic Thermal Parameters for $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$

atom	x	y	z	B, Å^2
Pt	0.29637 (4)	0.15220 (4)	0.28791 (4)	*a*
Fe1	0.41199 (14)	0.24205 (13)	0.30725 (15)	*a*
Fe2	0.35779 (15)	0.24422 (14)	0.41226 (14)	*a*
P1	0.3019 (3)	0.0365 (2)	0.2825 (3)	*a*
P2	0.1840 (2)	0.1686 (2)	0.2083 (3)	*a*
S1	0.3029 (3)	0.2755 (2)	0.2974 (3)	*a*
S2	0.4122 (3)	0.1569 (3)	0.3824 (2)	*a*
N1A	0.4745 (8)	0.3011 (7)	0.3429 (8)	*a*
O1A	0.5204 (9)	0.3331 (8)	0.3402 (9)	*a*
N1B	0.4082 (7)	0.2122 (9)	0.2306 (8)	*a*
O1B	0.4194 (12)	0.1892 (9)	0.1817 (8)	*a*
N2A	0.4111 (9)	0.3066 (9)	0.4604 (9)	*a*
O2A	0.4303 (9)	0.3426 (9)	0.5102 (8)	*a*
N2B	0.3077 (10)	0.2110 (8)	0.4484 (8)	*a*
O2B	0.2737 (9)	0.1847 (8)	0.4764 (8)	*a*
C1A	0.2374 (9)	−0.0035 (9)	0.3102 (9)	2.8 (4)
C1B	0.3892 (11)	−0.0449 (11)	0.3987 (11)	3.7 (4)
C1C	0.2944 (8)	0.0063 (8)	0.1963 (8)	3.0 (3)
C2A	0.1411 (10)	0.2179 (9)	0.2560 (10)	3.4 (4)
C2B	0.1324 (9)	0.0606 (10)	0.1156 (9)	3.7 (4)
C2C	0.1723 (10)	−0.2170 (9)	0.1280 (9)	3.7 (4)
C1AA	0.1439 (11)	−0.0828 (10)	0.3046 (11)	5.4 (5)
C1AB	0.1933 (10)	−0.0571 (10)	0.2765 (9)	4.7 (4)
C1AC	0.2291 (10)	0.0268 (10)	0.3697 (10)	5.1 (5)
C1AD	0.1447 (11)	−0.0568 (11)	0.3658 (11)	5.5 (5)
C1AE	0.1835 (11)	0.0004 (11)	0.3974 (11)	6.1 (5)
C1BA	0.4407 (11)	0.0100 (10)	0.3219 (10)	5.1 (5)
C1BB	0.5088 (13)	−0.0195 (12)	0.3669 (12)	7.1 (6)
C1BC	0.5134 (13)	−0.0592 (12)	0.4203 (12)	7.2 (6)
C1BD	0.4589 (13)	−0.0729 (11)	0.4390 (12)	7.0 (6)
C1BE	0.3815 (10)	−0.0038 (9)	0.3396 (9)	5.8 (5)
C1CA	0.2867 (10)	−0.0633 (10)	0.1799 (10)	4.9 (4)
C1CB	0.3013 (10)	0.0534 (10)	0.1496 (10)	5.2 (5)
C1CC	0.2843 (12)	−0.0854 (11)	0.1131 (12)	6.5 (6)
C1CD	0.3046 (11)	0.0303 (11)	0.0837 (11)	6.2 (5)
C1CE	0.2953 (12)	−0.0386 (12)	0.0676 (12)	6.8 (6)
C2AA	0.1117 (11)	0.2837 (11)	0.2280 (10)	5.2 (5)
C2AB	0.0798 (14)	0.3224 (14)	0.2697 (15)	9.2 (8)
C2AC	0.1091 (13)	0.2320 (13)	0.3567 (13)	8.0 (7)
C2AD	0.0838 (12)	0.2944 (13)	0.3292 (13)	7.4 (6)
C2AE	0.1441 (13)	0.1926 (12)	0.3190 (13)	7.2 (6)
C2BA	0.0808 (11)	0.0785 (10)	0.1966 (10)	5.4 (5)
C2BB	0.1247 (9)	0.0949 (9)	0.1703 (9)	4.9 (4)
C2BC	0.0884 (12)	0.0034 (11)	0.0824 (11)	7.4 (6)
C2BD	0.0247 (13)	0.0229 (13)	0.1639 (13)	8.6 (7)
C2BE	0.0394 (12)	−0.0127 (12)	0.1104 (12)	7.5 (6)
C2CA	0.2301 (11)	0.2481 (10)	0.1195 (11)	5.9 (5)
C2CB	0.1045 (11)	0.2225 (10)	0.0741 (11)	5.3 (5)
C2CC	0.2197 (13)	0.2857 (12)	0.0576 (13)	7.4 (6)
C2CD	0.0959 (12)	0.2606 (12)	0.0123 (12)	6.7 (6)
C2CE	0.1492 (12)	0.2901 (11)	0.0047 (12)	6.4 (6)

[a] Anisotropic thermal parameters–Table S1 (supplementary material).

folded across the Fe$\cdots$Fe axis upon coordination to $(Ph_3P)_2Pt$. Second, both linear and bent nitrosyl ligands are present on each Fe atom. Examples of structurally characterized complexes containing both linear and bent nitrosyls are rare[12,13] and have not been found previously for iron complexes.

The $(Ph_3P)_2PtS_2Fe_2(NO)_4$ cluster may be viewed as a square-planar Pt complex (root-mean square deviation of the fitted atoms from the least-squares plane is 0.0719 Å) joined to two tetrahedral iron atoms through mutual sulfur atoms. The Fe$\cdots$Pt and Fe$\cdots$Fe distances observed are less than 3 Å, while the Fe$\cdots$Pt distances in the structurally similar $(Ph_3P)_2Pt(\mu_3\text{-}Se)_2Fe_2(CO)_6$, a complex in which the iron atoms are octahedrally coordinated, are greater than 3.45 Å.[14b]

The nonlinear FeNO group may arise, in part, from the tendency of NO ligands to geniculate in tetrahedral dinitrosyl complexes.[12,13,15,16] However, the particular asymmetry of this bending as observed in $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$ implicates other factors. The puckering of the S_2Fe_2 ring removes the C_{2v} symmetry of the $Fe(NO)_2$ moiety and any degeneracy of the NO orbitals. Local symmetry about the metal atom and in the vicinity of the MNO bond presumably dictates whether or not the MNO unit is linear. If the local symmetry about the nitrosyl axis is C_{3v} or higher, the nitrosyl is generally linear and considered a 3-electron donor. Lower symmetry usually produces a bending of the MNO group.

The M–M bonding appears to dictate the local symmetry that describes the structure of $(Ph_3P)_2PtS_2Fe_2(NO)_4$. If no important Pt–Fe interaction is assumed (the distance of ~2.95 Å is rather long and the PtS_2P_2 geometry is typical), the bonding symmetry that influences the π orbitals of the "B" nitrosyls is that of three similar "soft" π-acid ligands. However, an Fe–Fe interaction (~2.8 Å), with iron orbitals protruding toward the nitrosyl on the opposing iron atom, can influence the local symmetry of the nitrosyls. The metal orbitals can overlap with N π orbitals to cause bending and the $Fe\text{–}N_A\text{–}O_A$ angle of ~150°.

Although the nitrosyl ligands are asymmetrically geniculated, the O–Fe–O and N–Fe–N angles agree with the linear correlation of O–M–O and N–M–N angles found in other tetrahedral dinitrosyl complexes.[17] Structural features observed in iron nitrosyl dimers are compared in Table I. The Fe$\cdots$Fe distance in $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$ is slightly longer than the Fe–Fe bond in either $(\mu\text{-}C_2H_5S)_2Fe_2(NO)_4$ or the $(\mu\text{-}S)_2Fe_2(NO)_4{}^{2-}$ ion, whereas the S$\cdots$S distance has been shortened. The fold in the S_2Fe ring is indicated by the 129.4° dihedral angle between the Fe_2S planes.

In conclusion, we note that $(Ph_3P)_3Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$ has been sought, without success, by other workers. Beck et al.[18] reported that they were unable to prepare this complex by the reaction of *cis*-$(Ph_3P)_2PtCl_2$ with $[Ph_4As]_2[S_2Fe_2(NO)_4]$. Rauchfuss and Weatherill[19] claimed the isolation of $(Ph_3P)_2PtS_2Fe_2(NO)_4$ from the reaction of Roussin's red potassium salt with *cis*-$(Ph_3P)_2PtCl_2$ as an "unstable solid" and cited a ν(NO) of 1725 cm^{-1}.

Experimental Section

General Comments. All reactions were carried out under an atmosphere of nitrogen.

IR spectra were recorded with a Perkin-Elmer 457A grating infrared spectrophotometer; NMR spectra were measured with Varian Associates T60 (proton) and JEOL FX90Q (^{31}P at 32.6 MHz) spectrometers. Field desorption (FD) mass spectra were obtained at the MIT Mass Spectrometry Facility with a MAT 731 instrument that has a range of 0–2000 μm at ±8 kV and is equipped for photoplate detection. (For a note on the FD mass spectrum of $(Ph_3P)_2Pt(\mu\text{-}S)_2Fe_2(NO)_4$, as well as of other metal complexes, see ref 20.)

Roussin's red sodium salt was prepared by the method of Pavel[4] (see also ref 21) from Roussin's black ammonium salt[3,4,21] and isolated as a hydrate, $Na_2[(\mu\text{-}S)_2Fe_2(NO)_4]\cdot 8H_2O$. It dissolves in THF to give a deep red solution.

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Preparation of $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$. A. From Roussin's Red Sodium Salt. A 250-mL one-necked round-bottomed flask with a nitrogen inlet side arm equipped with a serum cap and a magnetic stir bar was charged with 0.31 g (0.63 mmol) of $Na_2S_2Fe_2(NO)_4{\cdot}8H_2O$ and then was flushed with nitrogen. THF (25 mL) was added to give a deep red solution of Roussin's red salt and subsequently 0.50 g (0.63 mmol) of *cis*-$(Ph_3P)_2PtCl_2$ was added against a counterflow of nitrogen. The resulting solution was stirred under nitrogen at room temperature. After 2 h the solution had turned to a deep black cherry color. The solvent was removed at reduced pressure, and the residue was chromatographed (under nitrogen) on a silicic acid column. Dichloromethane eluted 0.61 g (95% yield) of $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$, a black, crystalline solid, mp 198–201 °C. The product is very air sensitive in solution and moderately so in the solid state.

Anal. Calcd for $C_{36}H_{30}O_4S_2N_4P_2Fe_2Pt$: C, 42.58; H, 2.98; N, 5.52. Found: C, 42.43; H, 2.76; N, 5.45. Molecular weight (vapor pressure lowering in CH_2Cl_2 at 0 °C): 1010 ± 50 (calcd 1015.5). IR (CD_2Cl_2): 3065 (w), 1482 (m), 1100 (m), ν(NO) 1735 (vs) and 1690 (vs) cm^{-1}. ^1H NMR ($CDCl_3$): δ 7.10–7.35. ^{31}P NMR (CH_2Cl_2): δ 21.06 (J(Pt–P) = 3394 Hz). FD mass spectrum (20 eV): m/z 1014 (^{194}Pt, 32.9%), 1015 (^{195}Pt, 33.8%), 1016 (^{196}Pt, 25.3%); no fragment ions.

B. From $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(CO)_6$. A 300-mL, three-necked round-bottomed flask equipped with a magnetic stir bar, two serum stoppers, and a reflux condenser topped with a gas-inlet tube was charged with 0.37 g (0.35 mmol) of $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(CO)_6$[7] and flushed with nitrogen. Benzene (150 mL) was added by syringe, and then nitric oxide was bubbled into the resulting orange-red solution through a gas dispersion tube inserted through one of the serum stoppers. The solution was stirred and heated at reflux for 3 h while nitric oxide was bubbled in at the rate of 3.6 L/h. The course of the reaction was monitored by taking IR spectra of aliquots. Heating was stopped when the carbonyl bands of the starting material had disappeared. Benzene was removed at reduced pressure, leaving a black, crystalline solid. The latter was redissolved in benzene, the solution was filtered under nitrogen, and the solvent was again removed at reduced pressure to leave 0.321 g (90%) of a black, air-sensitive, crystalline solid, mp 198–200 °C. IR ($CDCl_3$): ν(NO) 1740 (vs), 1690 (vs) cm^{-1}. ^{31}P NMR (CH_2Cl_2): δ 21.08 (J(Pt–P) = 3392 Hz).

Structure Determination

A crystal of the title compound was obtained by crystallization from toluene/pentane. A crystal was mounted under mineral oil in a quartz capillary. Crystal data: $C_{36}H_{30}O_4N_4S_2P_2Fe_2Pt$, mol wt 1015.5, crystal dimensions ca. 0.25-mm diameter (irregular sphere), monoclinic, space group $C2/c$ (No. 15), $a = 20.867$ (7) Å, $b = 19.616$ (6) Å, $c = 20.524$ (4) Å, $\beta = 113.18$ (2)°, $V = 7723$ (3) $Å^3$, $\rho_{calcd} = 1.69$ g/cm^3, $Z = 8$, μ(Mo Kα) = 46.2 cm^{-1}. Intensity data (9768 reflections, $2.0° \leq 2\theta \leq 55.0°$, $h,k,\pm l$) were recorded on a Syntex P2_1 four-circle diffractometer at ambient temperature (ca. 22 °C) with graphite-monochromated Mo Kα radiation. The first 1000 reflections indicated systematic absences ($h + k = 2n + 1$) and a C lattice. The remaining data were collected for $h + k = 2n$ only. The 3790 independent reflections with $I > 3.0\,\sigma(I)$ were corrected for Lorentz, polarization, and absorption effects.[22] The platinum atom was located by heavy-atom methods, and 11 remaining non-hydrogen atoms were found by Fourier syntheses. $R = 0.060$ and $R_w = 0.067$.

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Note Added in Proof: The crystal and molecular structure of $CoI(NO)_2(P(C_6H_5)_3)$ has been determined at low temperature (−170 °C).[24] The complex contains only linear nitrosyl ligands (Co–N–O = 165.2 (2), 163.3 (2)°) in contrast to the related disordered complex, $CoI(NO)_2(P(C_6H_5)_2R)$ ($R = CH_2CH_2P(O)(C_6H_5)_2$), which is reported to possess both bent and linear nitrosyl groups (Co–N–O = 121 (2), 149 (6), 155 (4), 166 (3)°).[13c]

Registry No. $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(NO)_4$, 86310-22-7; $Na_2S_2Fe_2(NO)_4$, 58204-17-4; *cis*-$(Ph_3P)_2PtCl_2$, 15604-36-1; $(Ph_3P)_2Pt(\mu_3\text{-}S)_2Fe_2(CO)_6$, 75249-93-3.

Supplementary Material Available: Anisotropic thermal parameters (Table S1) and a listing of observed and calculated structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

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