yellow-brown suspension was quenched with water (50 ml). After standing for 24 h the solution turned red. Addition to the filtered solution of solid tetraphenylphosphonium bromide (1 g) gave a red precipitate. This was filtered, washed with water (30 ml), and dried under vacuum. Crystallization by dissolution in acetone (30 ml) and precipitation with 2-propanol (30 ml) afforded 2.5 g of dark red prisms of $[PPh_4]_2[Ni_6(CO)_{12}]$. Anal. Calcd: PPh_4^+, 49.62; Ni, 25.78; CO, 24.59. Found: PPh_4^+, 49.26; Ni, 25.67; CO, 24.61; PPh_4^+:Ni:CO = 1:3.01:6.05.

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Registry No. $[PPN]_2[Ni_5(CO)_{12}]$, 57108-14-2; $[NMe_4]_2[Ni_5(CO)_{12}]$, 60464-16-6; $[NEt_4]_2[Ni_5(CO)_{12}]$, 60464-15-5; $K_2[Ni_6(CO)_{12}]$, 60464-09-7; $[PPh_4]_2[Ni_6(CO)_{12}]$, 60464-17-7; $[PPN]_2[Ni_6(CO)_{12}]$, 60464-20-2; $[NMe_4]_2[Ni_6(CO)_{12}]$, 60464-19-9; $[NEt_4]_2[Ni_6(CO)_{12}]$, 60464-18-8; $[Ni_6(CO)_{12}]^{2-}$, 52261-68-4; $[Ni_5(CO)_{12}]^{2-}$, 56938-71-7; $Ni(CO)_4$, 13463-39-3.

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Carbonylnickelates. 2. Synthesis and Chemical Characterization of the Dianion $[Ni_9(CO)_{18}]^{2-}$

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The cherry red $[Ni_9(CO)_{18}]^{2-}$ dianion has been obtained both by redox condensation between $Ni(CO)_4$ and $[Ni_6(CO)_{12}]^{2-}$ and by oxidation of $[Ni_6(CO)_{12}]^{2-}$ with nickel(II) chloride. It has been isolated as $[NMe_3(CH_2Ph)]^+$, $[PPh_4]^+$, $[PPh_3Me]^+$, $[AsPh_4]^+$, and $[PPN]^+$ salts. The octadecacarbonylenneanickelate(2–) dianion is rapidly degraded by carbon monoxide (25 °C, 1 atm) to $[Ni_5(CO)_{12}]^{2-}$ and tetracarbonylnickel.

Introduction

We have recently reported the synthesis and the structural characterization of a series of carbonylplatinates having the general formula $[Pt_3(CO)_6]_n^{2-}$ (n = 1–6)[1,2] and of the dianion $[Ni_6(CO)_{12}]^{2-}$.[3] The structure of all of these dianions is based on triangular $[M_3(CO)_3(\mu\text{-}CO)_3]$ units stacked along the ternary axes. Although the stackings of these triangular units in $[Ni_6(CO)_{12}]^{2-}$ and $[Pt_6(CO)_{12}]^{2-}$ are different and give rise to approximately trigonal-antiprismatic and trigonal-prismatic geometries, respectively, it seemed possible that an enneanuclear carbonylnickelate dianion analogous to that of platinum might exist.

We now report the synthesis and the chemical characterization of the $[Ni_9(CO)_{18}]^{2-}$ dianion.

Results

The $[Ni_9(CO)_{18}]^{2-}$ dianion can be prepared in THF solution by a redox condensation reaction[4] between tetracarbonylnickel and the preformed $[Ni_6(CO)_{12}]^{2-}$ dianion. The reaction takes place according to the equilibrium

$$[Ni_6(CO)_{12}]^{2-} + 3Ni(CO)_4 \rightleftarrows [Ni_9(CO)_{18}]^{2-} + 6CO \qquad (1)$$

However this type of synthesis presents some difficulties due both to the unfavorable position of equilibrium 1 under carbon monoxide at 25 °C and to the high volatility of the tetracarbonylnickel. In practice it is necessary to carry out the reaction under vacuum using an efficient condenser at about –20 °C in order to recycle the solvent and the tetracarbonylnickel and to remove the evolved carbon monoxide. After a few hours at these conditions the transformation into $[Ni_9(CO)_{18}]^{2-}$ is practically complete.

The redox condensation equilibrium (1) is also significant during the initial step of the reduction of tetracarbonylnickel with alkali metals in THF,[5] when $Ni(CO)_4$ is still present in large excess. Thus formation of the enneanuclear dianion has been monitored by infrared spectroscopy during the first hours of reaction, although it then disappears probably due to the progressive lowering of the $Ni(CO)_4$ concentration. The decrease in $Ni(CO)_4$ concentration not only has a negative effect on equilibrium 1 but also makes easier further reduction of the $[Ni_9(CO)_{18}]^{2-}$ dianion itself.

The synthesis of the $[Ni_9(CO)_{18}]^{2-}$ dianion is more conveniently carried out by reaction of the preformed $[Ni_6(CO)_{12}]^{2-}$ dianion with oxidizing agents such as nickel(II) chloride. Progressive addition, from a microburet, of an ethanolic solution of $NiCl_2$ and monitoring the reaction by ir spectroscopy give the stoichiometry

$$3[Ni_6(CO)_{12}]^{2-} + NiCl_2 \xrightarrow{\text{EtOH}} 2[Ni_9(CO)_{18}]^{2-} + Ni + 2Cl \qquad (2)$$

Although the formation of nickel metal has only been verified in a qualitative way, addition of a large excess of nickel(II) chloride results in complete decomposition to nickel metal and tetracarbonylnickel. With proper use of this oxidizing agent the $[Ni_9(CO)_{18}]^{2-}$ dianion is obtained in nearly quantitative

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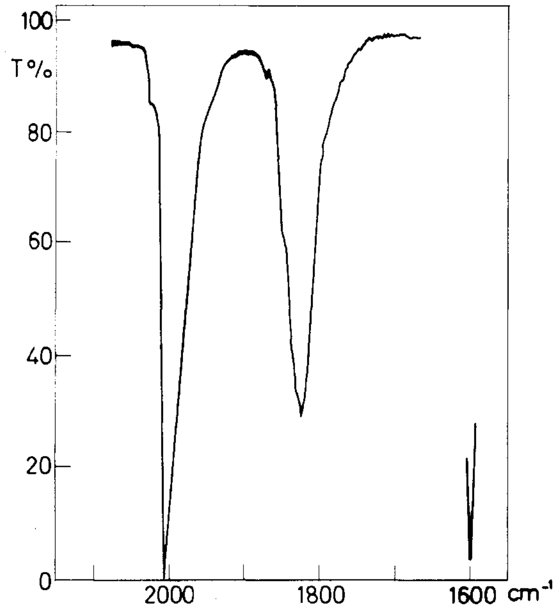

Figure 1. Infrared spectrum of $[PPh_4]_2[Ni_9(CO)_{18}]$ in THF solution.

yield and has been isolated as tetrasubstituted ammonium, phosphonium, and arsonium salts.

These salts are generally soluble in THF, acetone, and acetonitrile and have been crystallized from acetone–2-propanol or THF–toluene by a slow-diffusion technique. The analytical data (see Experimental Section) have been confirmed by x-ray studies on the $[PPh_4]^+$ and $[AsPh_4]^+$ salts. The structure of the dianion consists of three triangular $[Ni(CO)_3(\mu\text{-}CO)_3]$ units stacked along the ternary axes,[6] and, although the relative stacking of the triangles is different, it is strictly related to the structure previously found for the analogous $[Pt_9(CO)_{18}]^{2-}$ dianion.[1]

In agreement with the structural data the infrared spectrum of the $[Ni_9(CO)_{18}]^{2-}$ dianion presents absorptions in both the terminal (2005 cm^{-1}) and edge-bridging (1825 cm^{-1}) carbonyl stretching regions as shown in Figure 1. As found for $[Ni_6(CO)_{12}]^{2-}$ and $[Pt_6(CO)_{12}]^{2-}$,[2,5] the infrared spectrum of $[Ni_9(CO)_{18}]^{2-}$ shows slightly lower frequencies (ca. 20 cm^{-1}) than those of the analogous $[Pt_9(CO)_{18}]^{2-}$, indicating better back-donation in the nickel case.

The salts of the $[Ni_9(CO)_{18}]^{2-}$ dianion are air sensitive both in solution and in the solid state, and air oxidation results in the formation of tetracarbonylnickel and decomposition byproducts. Oxidation with excess nickel(II) or copper(II) chloride in methanol, as well as with copper or silver tetrafluoroborate in THF or acetonitrile, results in decomposition to tetracarbonylnickel and nickel metal even at low temperature. No evidence for the formation of the hypothetical species $[Ni_{12}(CO)_{24}]^{2-}$, related to the green $[Pt_{12}(CO)_{24}]^{2-}$, has been so far obtained. However, in the oxidation of $[AsPh_4][Ni_9(CO)_{18}]$ with $AgBF_4$ in THF we have observed the precipitation of a red-orange intermediate which analyzes as $[AsPh_4][AgNi_9(CO)_{18}]$ (see Experimental Section). Its ir spectrum shows absorptions at higher frequencies than those of the starting material (ν_{CO} in acetonitrile at 2040 (s) and 1830 (ms) cm^{-1}) and suggests that the silver atom interacts with the metal skeleton of the cluster. Reaction of this compound with sodium iodide in THF results in the precipitation of silver iodide and regenerates the $[Ni_9(CO)_{18}]^{2-}$ dianion.

Reaction of $[Ni_9(CO)_{18}]^{2-}$ with acetic or phosphoric acid in organic solvents results in decomposition with intermediate formation of tetracarbonylnickel and a red-violet anion apparently identical with that obtained by reaction of the $[Ni_6(CO)_{12}]^{2-}$ dianion with acids at pH 4 in aqueous solution (ν_{CO} in THF at 2020 and 1860 cm^{-1}; ^1H NMR signal in acetone at ca. τ 28).[5]

Reaction with carbon monoxide (1 atm, 25 °C) is rapid

$$[Ni_9(CO)_{18}]^{2-} + 10CO \rightleftarrows [Ni_5(CO)_{12}]^{2-} + 4Ni(CO)_4 \qquad (3)$$

This reaction takes place with intermediate formation of $[Ni_6(CO)_{12}]^{2-}$ corresponding to the reversion of equilibrium 1, and this is followed by the known reaction of $[Ni_6(CO)_{12}]^{2-}$ with the excess carbon monoxide.[5] Evidence for the intermediate formation of $[Ni_6(CO)_{12}]^{2-}$ has been obtained by carrying out the reaction with a deficiency of carbon monoxide.

The relatively low stability of the $[Ni_9(CO)_{18}]^{2-}$ dianion is also illustrated by reaction with a large excess of iodide ions which results in the demolition of the cluster according to

$$[Ni_9(CO)_{18}]^{2-} + 2I^- \rightarrow [Ni_6(CO)_{12}]^{2-} + 2[Ni(CO)_3I]^- + Ni \qquad (4)$$

Discussion

The isolation of the $[Ni_9(CO)_{18}]^{2-}$ dianion is related to the existence of the $[Pt_3(CO)_6]_n{}^{2-}$ (n = 1–6) dianions.[1,2] The stacking of three triangular units along the ternary axes becomes possible even at the shorter Ni–Ni distances because of the outward deformation of the carbonyl groups in the two external triangular units. However, the hypothetical tetramer $[Ni_{12}(CO)_{24}]^{2-}$ would be expected to have a much lower stability, because there is no easy way of relieving the strain imposed by the nonbonding interactions between the two internal triangular units.

The reaction of the $[Ni_9(CO)_{18}]^{2-}$ dianion with carbon monoxide contrasts with the stability of the isostructural $[Pt_9(CO)_{18}]^{2-}$ toward carbon monoxide and is similar to the behavior found for the analogous $[Ni_6(CO)_{12}]^{2-}$ and $[Pt_6(CO)_{12}]^{2-}$ dianions.[2,5] This observation provides further evidence that the difference in the averaged energies $\bar{D}_{M-CO}$ and $\bar{D}_{M-M}$ for group 8 metals generally decreases on descending a subgroup.[4]

Experimental Section

All the operations were carried out under nitrogen or carbon monoxide. All the solvents were carefully purified and dried before use. Infrared spectra were recorded on a Perkin-Elmer 457 grating spectrophotometer using calcium fluoride cells. The analytical data were obtained as described elsewhere.[5] The solution of $Ni(EtOH)_xCl_2$ in ethanol was prepared according to the literature.[7]

1. Synthesis of $[Ni_9(CO)_{18}]^{2-}$ salts by Oxidation of $[Ni_6(CO)_{12}]^{2-}$ with $Ni(EtOH)_xCl_2$. A 2.12-g sample of $K_2[Ni_6(CO)_{12}]\cdot xH_2O$ (x = ca. 4) was dissolved in ethanol (30 ml) under nitrogen and reacted drop by drop with increasing amounts of a 0.161 M ethanolic solution of $Ni(EtOH)_xCl_2$. The reaction was monitored by infrared. After addition of ca. 5.5 ml of reagent, the infrared spectrum of the solution showed only carbonyl absorptions at 2005 (s) and 1825 (s) cm^{-1} due to the $[Ni_9(CO)_{18}]^{2-}$ dianion. The solution was filtered and solid tetraphenylphosphonium chloride (ca. 1 g) was added. The resulting brown precipitate was filtered, washed with methanol (10 ml), and dried under vacuum. $[PPh_4]_2[Ni_9(CO)_{18}]$ is soluble in acetone, acetonitrile, and THF, sparingly soluble in alcohol, and insoluble in water and apolar solvents such as toluene. It was crystallized by dissolution in acetone (20 ml) and precipitation by diffusion of 2-propanol (30 ml) to give 1.8 g of dark prisms. Anal. Calcd for $[PPh_4]_2[Ni_9(CO)_{18}]$: PPh_4^+, 39.66; Ni, 30.88; CO, 29.46. Found: PPh_4^+, 39.14; Ni, 30.85; CO, 29.39; PPh_4^+:Ni:CO = 1:4.55:9.07.

The trimethylbenzylammonium, triphenylmethylphosphonium, tetraphenylarsonium, and bis(triphenylphosphine)imminium salts of $[Ni_9(CO)_{18}]^{2-}$ were prepared analogously with similar yields.

2. Synthesis of $[NMe_3(CH_2Ph)]_2[Ni_9(CO)_{18}]$ by Redox Condensation of $K_2[Ni_6(CO)_{12}]\cdot xH_2O$ with $Ni(CO)_4$. $Ni(CO)_4$ (2 ml), $K_2[Ni_6(CO)_{12}]\cdot xH_2O$ (1.34 g), and THF (30 ml) were placed in a 100-ml, two-necked, round-bottomed flask equipped with a condenser cooled at −20 °C. The mixture was stirred 8 h at 45 °C under static vacuum to favor CO evolution. The resulting dark red solution was evaporated to dryness and the residue was dissolved in methanol (20 ml). Addition of solid trimethylbenzylammonium chloride in excess gave a red-brown precipitate of $[NMe_3(CH_2Ph)]_2[Ni_9(CO)_{18}]$; yield 1.2 g.

3. Synthesis of $[AsPh_4][AgNi_9(CO)_{18}]$. $[AsPh_4]_2[Ni_9(CO)_{18}]$ (1.2 g) was dissolved under nitrogen in THF (20 ml) and cooled to −70 °C. After dropwise addition during stirring of a solution of $AgBF_4$ (0.13 g) in THF (10 ml), the initial dark red solution became colorless and a dark precipitate separated out. This was filtered, washed with THF (10 ml), and dried under vacuum. Extraction with acetonitrile (30 ml) gave a red-orange solution which gave an orange microcrystalline precipitate of $[AsPh_4][AgNi_9(CO)_{18}]$ by addition of diisopropyl ether (30 ml); yield 0.72 g. The compound is soluble in acetone and acetonitrile and sparingly soluble or insoluble in methanol, 2-propanol, and THF. Anal. Calcd for $[AsPh_4][AgNi_9(CO)_{18}]$: $AsPh_4^+$, 25.0; Ag, 7.05; Ni, 34.5. Found: $AsPh_4^+$, 24.4; Ag, 7.3; Ni, 32.3.

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Registry No. $[PPh_4]_2[Ni_9(CO)_{18}]$, 60512-63-2; $[NMe_3(CH_2Ph)]_2[Ni_9(CO)_{18}]$, 60512-62-1; $[PPh_3Me]_2[Ni_9(CO)_{18}]$, 60512-61-0; $[AsPh_4]_2[Ni_9(CO)_{18}]$, 60512-60-9; $[PPN]_2[Ni_9(CO)_{18}]$, 60512-59-6; $[AsPh_4][AgNi_9(CO)_{18}]$, 60512-58-5; $K_2[Ni_6(CO)_{12}]$, 60464-08-6; $Ni(CO)_4$, 13463-39-3.

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High-Pressure Phase Transformations of the Systems $Ni_{1-x}Co_xTe_2$ and $Ni_{1-x}Fe_xTe_2$

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The solid solutions $Ni_{1-x}Co_xTe_2$ ($x \leq 0.6$) and $Ni_{1-x}Fe_xTe_2$ ($x \leq 0.1$) were found to crystallize at ambient pressure with the cadmium iodide structure. Transformations to the pyrite structure at 1000–1300 °C and under 60 kbars pressure in a high-pressure belt apparatus were found to be a function of the cobalt or iron substitution.

Introduction

It has been reported[1] that several 3d transition metal ditellurides transform to the pyrite structure under pressure. Bither[1] studied both iron and cobalt ditelluride which crystallize with the marcasite structure and transform to the pyrite structure at 1200 °C and 65 kbars pressure. He also observed the partial transformation of nickel ditelluride from the cadmium iodide to the pyrite structure at 1000 °C and 89 kbars pressure.

Since the disulfides of iron, cobalt, and nickel have been reported[2] to form solid-solution series of the type $Fe_xCo_{1-x}S_2$ and $Ni_xCo_{1-x}S_2$ ($0 \leq x \leq 1$), it would be of interest to determine the extent of solid solution formation of the series $Ni_{1-x}Co_xTe_2$ and $Ni_{1-x}Fe_xTe_2$. In addition, the degree of transformation from the cadmium iodide to the pyrite structure can be investigated in this series as a function of the cobalt or iron substitution.

Experimental Section

1. Preparation. The metals (Gallard–Schlesinger, 99.999%) were reduced prior to use by heating under a 15% hydrogen/85% argon mixture (nickel, 3 h at 600 °C; cobalt, 8 h at 850 °C; iron, 20 h at 950 °C). The tellurium (Gallard–Schlesinger, 99.999%) was sublimed under vacuum from 600 to 400 °C.

Polycrystalline samples of the systems $Ni_{1-x}Co_xTe_2$ and $Ni_{1-x}Fe_xTe_2$ were synthesized by reacting stoichiometric quantities of the elements in sealed, evacuated silica tubes. All samples were reacted for a period of 2–3 weeks with several intermittent grindings in a dry nitrogen atmosphere. At the end of each heat treatment the samples were cooled to room temperature at the rate of 15 °C/h.

The reacted polycrystalline powders were subjected to high pressures and temperatures in a belt apparatus of the type described by Hall.[3] Pyrex capsules served as reaction containers. The experimental conditions are given in Table I. At the end of each experiment the temperature was lowered to 600 °C over a period of several hours and then reduced to room temperature in less than 20 min. Subsequently the pressure was reduced to 1 atm.

Table I. High-Pressure Results

Compn	Exptl conditions	Results
$NiTe_2$	1300 °C, 60 kbars	No transformn
$CoTe_2$	1300 °C, 60 kbars	Complete transformn to pyrite
$FeTe_2$	750 °C, 60 kbars	No transformn
	1000 °C, 60 kbars	No transformn
	1400 °C, 60 kbars	No transformn; decompn
$Ni_{1-x}Co_xTe_2$ ($0.1 \leqslant x \leqslant 0.8$)	1300 °C, 60 kbars	Partial transformn to pyrite plus cadmium iodide
$Ni_{1-x}Fe_xTe_2$ ($0.1 \leqslant x \leqslant 0.3$)	1000 °C, 60 kbars	Partial transformn to pyrite plus marcasite
$Ni_{1-x}Fe_xTe_2$ ($0.4 \leqslant x \leqslant 0.8$)	1000 °C, 60 kbars	Complete transformn to pyrite
$Ni_{0.1}Fe_{0.9}Te_2$	1000 °C, 60 kbars	Partial transformn to pyrite plus marcasite

2. X-Ray Measurements. Powder diffraction patterns were obtained with a Norelco diffractometer using monochromatic high-intensity Cu Kα radiation (λ 1.5405 Å). Fast scans of 1° (2θ) min^{-1} were taken to determine the phases present in each sample. Lattice parameters were determined from slow scans of $^1/_4$ (2θ) min^{-1} in the interval $12° \leq 2\theta \leq 130°$. The parameters were refined by a least-squares fit of the data obtained from high-angle reflections ($2\theta > 50°$) corrected relative to an internal MgO standard.

3. Density Measurements. The densities of the single-phase samples were determined using the hydrostatic technique described by Adams.[4] A Mettler H54 analytical balance was employed and the density fluid, perfluoro(1-methyldecalin), was calibrated with a high-purity silicon crystal ($\rho = 2.328$ g/cm^3 at 22 °C). Prior to any density measurements, the samples were ground and outgassed thoroughly to ensure reproducible results.

Results

1. Ambient Pressure. Compounds crystallizing with the cadmium iodide structure were obtained for $x \leq 0.6$ in the $Ni_{1-x}Co_xTe_2$ system. The lattice parameters and densities of these compounds are listed in Table II. Figure 1 shows the variation in the a_0 and c_0 values as a function of composition. In the $Ni_{1-x}Fe_xTe_2$ system, the cadmium iodide structure was found only for $x \leq 0.1$. Mixtures of both cadmium iodide and