102614-94-8; **4f**, 102614-95-9; **5a**, 93708-92-0; **5b**, 93708-93-1; **5c**, 93708-94-2; **5d**, 102614-96-0; **6**, 93709-02-5; **7**, 102614-97-1; **8a**, 93708-95-3; **8b**, 93708-96-4; **8c**, 93708-97-5; **8d**, 102614-98-2; **8e**, 102614-99-3; **8f**, 102615-00-9; **9**, 102615-01-0; **10a**, 93708-98-6; **10b**, 93708-99-7; **10c**, 93709-00-3; **10d**, 102615-02-1; **12**, 102615-03-2; **13a**, 93709-05-8; **13b**, 102615-05-4; **13c**, 102615-06-5; **13d**, 102615-07-6; **14a**, 102680-25-1; **14b**, 102680-26-2; **14c**, 102680-27-3; **14d**, 102680-28-4; **15a**, 93753-21-0; **15b**, 102680-29-5; **15c**, 93753-22-1; **15d**, 102615-09-8; **16**, 102615-08-7; **17a**, 102615-14-5; **17b**, 102615-11-2; **17c**, 93709-04-7; **17d**, 102615-13-4; **18**, 102615-10-1; **19a**, 102648-60-2; **19b**, 102615-16-7; **20**, 102615-15-6; [$Pt_2Me_8(\mu\text{-bipym})$], 102615-17-8; [$Pt_2Me_8(\mu\text{-}SMe_2)_2$], 102615-18-9.

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^{125}Te Coupling to ^{1}H, ^{13}C, and ^{125}Te Nuclei in NMR Studies of ^{125}Te-Enriched 1,3-Ditellurole

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Detailed ^{13}C, ^{1}H, and ^{125}Te NMR studies of 1,3-ditellurole enriched to 80% in the ^{125}Te isotope ($I = {}^1/_2$) gave $^1J(^{125}\text{Te}-^{13}\text{CH}_2)$ = 178.7 Hz, $^1J(^{125}\text{Te}-^{13}\text{CH})$ = 288.3 Hz, $^2J(^{125}\text{Te}-^{13}\text{CH})$ = 12.3 Hz, $^2J(^{125}\text{Te}-\text{C}^1H_2)$ = 25.9 Hz, $^2J(^{125}\text{Te}-\text{C}^1H)$ = 126.5 Hz, $^3J(^{125}\text{Te}-\text{C}^1H)$ = 33.5 Hz, $^2J(^{125}\text{Te}-^{125}\text{Te})$ = 260.3 Hz, and $^3J(^1\text{H}-^1\text{H})$ = 8.12 Hz. The ^{125}Te–^{125}Te coupling is comparable in magnitude to $^1J(^{125}\text{Te}-^{125}\text{Te})$ in diorgano ditellurides. The magnitude of the ^{125}Te–^{125}Te coupling may include contributions from two- and three-bond couplings as well as direct coupling of ^{125}Te nuclei, which are less than the sum of the van der Waals radii apart. The experimental spectra were matched by computer simulation to a root-mean-square error of 0.35. The ^{125}Te–^{125}Te coupling was determined by an iterative process to be 260.3 ± 0.3 Hz.

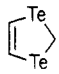
Introduction

Although tellurium-125 is a spin $^1/_2$ nucleus with ~7% natural abundance and a frequency 0.28 times that of ^{1}H, little is known about the ^{125}Te–^{125}Te coupling in organotellurium compounds.[1] In symmetric species, $^1J(^{125}\text{Te}-^{123}\text{Te})$ couplings have been measured by using ^{123}Te NMR ($I = {}^1/_2$, natural abundance 0.89%),[2] from which $^1J(^{125}\text{Te}-^{125}\text{Te})$ can be calculated by using the magnetogyric ratios of ^{125}Te ($\gamma = -8.453 \times 10^7$ rad T^{-1} s^{-1}) and ^{123}Te ($\gamma = -7.011$ rad T^{-1} s^{-1}).[3] In unsymmetrical diaryl ditellurides, $^1J(^{125}\text{Te}-^{125}\text{Te})$ couplings were measured directly by using satellites in the ^{125}Te spectrum; they were between 170 and 207 Hz and were concentration and solvent dependent.[2c] In symmetrical ditellurides, $^1J(^{125}\text{Te}-^{123}\text{Te})$ couplings were concentration dependent and in the range 213–269 Hz.[2c,3] No longer range Te–Te couplings for organotellurium compounds have been reported.

Inorganic Te cations have yielded values for longer range Te–Te couplings. The square-planar Te^{2+} cation gave $^2J(^{125}\text{Te}-^{123}\text{Te})$ of 602–604 Hz,[2a,b] comparable to $^1J(^{125}\text{Te}-^{123}\text{Te})$ of 608–676 Hz[2a,b] in the same system. In the Te^{4+} cation, a distorted trigonal prism, $^2J(^{125}\text{Te}-^{123}\text{Te})$ was determined to be 701 Hz, again comparable to $^1J(^{125}\text{Te}-^{123}\text{Te})$ in this cation of 791 and 1196 Hz.[2b] In the cubic $\text{Te}_2\text{Se}^{2+}$ cation, $^3J(^{125}\text{Te}-^{123}\text{Te})$ was determined to be 120 Hz from a sample of $\text{Te}_2\text{Se}^{2+}$ enriched to 77.3% in ^{125}Te.[4]

The large values of $^2J(^{125}\text{Te}-^{123}\text{Te})$ relative to $^1J(^{125}\text{Te}-^{123}\text{Te})$ suggest that three-center bonding may be important in the compounds described above. Three-center bonds would, in effect, lead to a partial bond between the tellurium nuclei. Additional measurements of tellurium coupling constants are needed to establish whether two-bond couplings are generally comparable to one-bond couplings.

During studies of the 1,3-ditellurole radical cation, we prepared 1,3-ditellurole (**1**) that was enriched in the ^{125}Te isotope.[5] Herein we report a detailed analysis of ^{125}Te–^{1}H and ^{125}Te–^{13}C couplings in this system as well as the first multibond ^{125}Te–^{125}Te coupling in an organotellurium compound.



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Results and Discussion

1,3-Ditellurole (**1**) was prepared from tellurium powder that was enriched to 80% in the ^{125}Te isotope. This isotope enrichment produced a 2:1 mixture of the doubly and singly ^{125}Te-labeled ditelluroles, respectively.

The ^{1}H NMR spectrum of **1** prepared from unenriched tellurium consists of two singlets centered at δ 8.86 and 4.73 for the olefinic and methylene protons, respectively. In the enriched sample, the methylene protons display a triplet from the doubly ^{125}Te-labeled ditellurole and a doublet from the singly ^{125}Te-labeled ditellurole with $^2J(^{125}\text{Te}-\text{C}^1H_2)$ of 25.9 Hz (Table I).

The olefinic protons display a more complicated splitting pattern, as shown in Figure 1. The doubly ^{125}Te-labeled ditellurole shows four lines of similar intensity, whereas the singly ^{125}Te-labeled ditellurole has an eight-line pattern that appears to be composed of two AB quartets, one to

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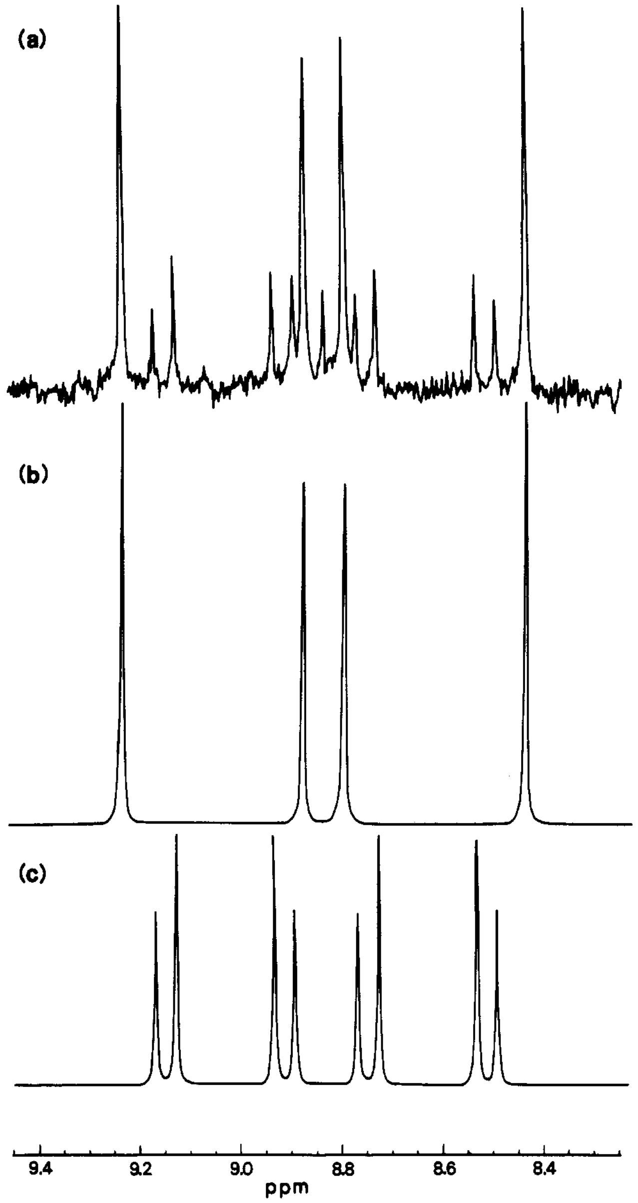


Figure 1. (a) Experimental ^{1}H NMR spectrum of the olefinic protons in 80% ^{125}Te-enriched 1,3-ditellurole (1) at 200 MHz. (b) PANIC-simulated ^{1}H NMR spectrum of the olefinic protons in doubly ^{125}Te-labeled 1 using coupling constants in Table I. (c) PANIC-simulated ^{1}H NMR spectrum of the olefinic protons in singly ^{125}Te-labeled 1 using coupling constants in Table I.

either side of the total pattern. In the singly labeled species the ^{125}Te nucleus sets up a small magnetic field at each of the proton sites as a result of the spin–spin coupling interaction between the tellurium and the protons. The small field either adds to or subtracts from the external magnetic field depending upon the particular spin state of the tellurium nucleus. The magnitude of the small fields at the two protons are unequal because the protons couple to the tellurium with different coupling constants. Thus in a molecule containing tellurium in a specified spin state the two protons act as if they had different chemical shifts. The coupling between the two protons is reflected in the NMR spectrum by the presence of an AB quartet corresponding to the particular spin state of the tellurium nucleus. Because there are two possible spin states for the tellurium, there are two such AB quartets. The magnitudes of the couplings of the protons to each other and to the tellurium were determined from the spectrum computer analysis and are shown in Table I.

We assume that the identity of the tellurium isotope has no effect on the magnitude of the chemical shifts or coupling constants. The parameters found for the singly labeled species may then be used as a starting point for analysis of the spectrum of the doubly labeled species.

Table I. ^{1}H, ^{13}C, and ^{125}Te Couplings in 1,3-Ditellurole (1)

coupling[a]	coupling const,[b] Hz
$\lvert{}^1J({}^{125}\mathrm{Te}{-}{}^{13}C\mathrm{H}_2)\rvert$	178.7
$\lvert{}^1J({}^{125}\mathrm{Te}{-}{}^{13}C\mathrm{H})\rvert$	288.3
$\lvert{}^2J({}^{125}\mathrm{Te}{-}{}^{13}C\mathrm{H})\rvert$	12.3
$\lvert{}^2J({}^{125}\mathrm{Te}{-}\mathrm{C}{}^1H_2)\rvert$	25.9
$\lvert{}^2J({}^{125}\mathrm{Te}{-}\mathrm{C}{}^1H)\rvert$	126.5
$\lvert{}^3J({}^{125}\mathrm{Te}{-}\mathrm{C}{}^1H)\rvert$	33.5
$\lvert{}^2J({}^{125}\mathrm{Te}{-}{}^{125}\mathrm{Te})\rvert$	260.3
$\lvert{}^3J({}^1\mathrm{H}{-}{}^1\mathrm{H})\rvert$	8.12

[a] Changing the signs of the coupling constants did not interfere with the spectra simulation. [b] The expected errors in the coupling constants predicted by the computer program are 0.3 Hz in all cases.

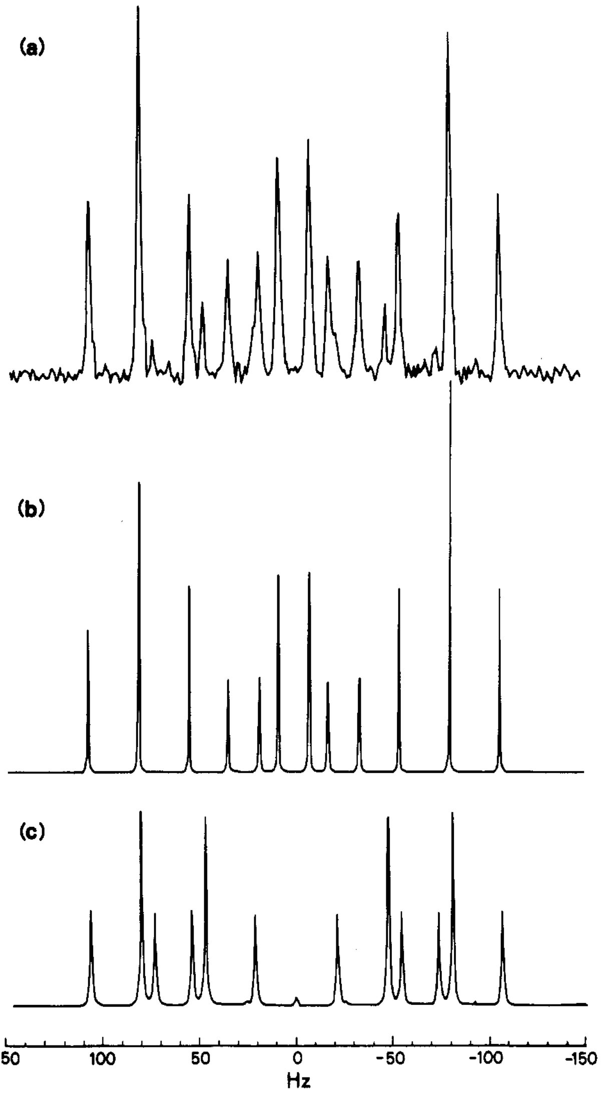


Figure 2. (a) Experimental ^{125}Te NMR spectrum of 80% ^{125}Te-enriched 1,3-ditellurole (1) at 63.22 MHz. (b) PANIC-simulated ^{125}Te NMR spectrum of doubly ^{125}Te-labeled 1 using the coupling constants in Table I. (c) PANIC-simulated ^{125}Te NMR spectrum of singly ^{125}Te-labeled 1 using the coupling constants in Table I.

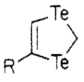
The coupling constants measured for the singly ^{125}Te-labeled ditellurole (1) accurately reproduce the experimental spectrum when used in computer simulation, as shown in Figure 1c. In the doubly ^{125}Te-labeled system, the experimental spectrum could be reproduced as the A portion of an AA′XX′ pattern by including a large ^{125}Te–^{125}Te coupling constant of 200–300 Hz. The ^{1}H NMR spectrum was insensitive to variation of the magnitude of the ^{125}Te–^{125}Te coupling over this range.

The ^{125}Te–^{125}Te coupling constant could be accurately extracted from the ^{125}Te NMR spectrum of the ^{125}Te-enriched sample of ditellurole 1. The ^{125}Te NMR spectrum of enriched 1 was complicated, as shown in Figure 2. Broad-band proton decoupling collapsed the spectrum to a singlet centered at −53.5 ppm (upfield) from bis(diethyldithiocarbamato)tellurium(II).[6] The ^{125}Te–^{1}H and ^{1}H–^{1}H couplings determined from the olefinic region of the ^{1}H NMR spectrum of the singly ^{125}Te-labeled 1 as well as the ^{125}Te–C$^{1}H_2$ coupling were used to reproduce accurately the experimental ^{125}Te NMR spectrum of singly and doubly ^{125}Te-labeled 1, as shown in Figure 2. For the doubly ^{125}Te-labeled 1, ^{2}J(^{125}Te–^{125}Te) was determined to be 260.3 ± 0.3 Hz by an iterative process to give a root-mean-square error of 0.35.

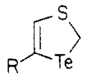
In the simulation, the protons were placed 826 Hz apart and ~100 000 Hz upfield from the Te nuclei. The ^{125}Te NMR spectrum for doubly ^{125}Te-labeled ditellurole 1 is accurately simulated in Figure 2b, and the olefinic region for the ^{1}H NMR spectrum of doubly ^{125}Te-labeled 1 is accurately reproduced in the same simulation (Figure 1b).

Decoupling of ^{125}Te by irradiating at the frequency of the ^{125}Te signal of 1 collapsed the ^{1}H NMR spectrum to two singlets centered at δ 8.86 and 4.73.

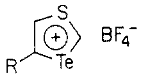
The observed coupling constants were assigned by comparison with suitable models. The ^{125}Te satellite peaks in the ^{1}H NMR spectra of the olefinic protons of unsymmetrical ditelluroles 2[7] showed a large ^{125}Te–^{1}H coupling (126.0–130.5 Hz) and a smaller ^{125}Te–^{1}H coupling (24.0–33.5 Hz). The smaller values were assigned to ^{3}J(^{125}Te–^{1}H) upon comparison with the splitting of the ^{125}Te satellites of the olefinic protons in thiatelluroles **3a**[8] and **3b**[8] of 19.3 and 21.0 Hz, respectively. The larger coupling constants are thus assigned to ^{2}J(^{125}Te–^{1}H).



2a, R = D
b, R = Ph
c, R = Me
d, R = Bu



3a, R = Ph
b, R = Me



4a, R = Ph
b, R = Me

^{2}J(^{125}Te–^{1}H) couplings are larger than ^{3}J(^{125}Te–^{1}H) couplings through sp^{2}-hybridized bonds in other systems as well. Oxidation of thiatelluroles 3 with trityl fluoroborate gave the thiatellurolylium cations 4.[9] In these cations, ^{125}Te satellites gave ^{2}J(^{125}Te–^{1}H) couplings through sp^{2}-hybridized bonds of 69.0 and 67.5 Hz for **4a** and **4b**, respectively, and ^{3}J(^{125}Te–^{1}H) couplings were 30 Hz in each case.

The ^{125}Te–^{125}Te coupling in ditellurole 1 is comparable in magnitude to ^{1}J(^{125}Te–^{125}Te) couplings determined for diorgano ditellurides.[2,3] Although this value seems large, a combination of two- and three-bond couplings as well as direct coupling could contribute to the large value in 1. An X-ray crystal structure of 1 places the Te nuclei 3.55 A apart, well under twice the sum of the van der Waals radius of Te (2.06 A).[5] CNDO/2 calculations[10] on 1 using the experimental geometry give a nonzero Wiberg index between the two Te atoms.[5] Such bonding between the Te atoms could contribute to the magnitude of the ^{125}Te–^{125}Te coupling as well.

The proton-decoupled ^{13}C NMR spectrum of unenriched 1 consisted of two signals at δ 119.96 and −39.02. The ^{125}Te satellites in this spectrum gave ^{1}J(^{125}Te–^{13}CH) of 288.3 Hz, ^{1}J(^{125}Te–^{13}CH$_2$) of 178.7 Hz, and ^{2}J(^{125}Te–^{13}CH) of 12.3 Hz (Table I). The proton-decoupled ^{13}C NMR spectrum of the ^{125}Te-enriched sample gave five lines for the olefinic carbons of the doubly ^{125}Te-labeled 1 as the X portion of an AA′X spectrum as well as two doublets for the olefinic carbons of the singly ^{125}Te-labeled 1. The coupling constants were identical with those determined from the ^{125}Te satellites in the ^{13}C NMR spectrum of the unenriched sample of 1. As expected, the methylene carbon in the proton-decoupled ^{13}C NMR spectrum appeared as a triplet for the doubly ^{125}Te-labeled 1 and as a doublet for the singly ^{125}Te-labeled 1 with ^{1}J(^{125}Te–^{13}CH$_2$) of 178.7 Hz.

Experimental Section

Ditelluroles 2[7] and thiatelluroles 3[8] were prepared by the literature procedures. A 95.67% enriched sample of ^{125}Te powder was obtained from Oakridge National Laboratories. This material was diluted with natural-abundance Te powder to give a sample 80% enriched in the ^{125}Te isotope. Deuteriochloroform was passed through an activity I column of alumina before use.

Preparation of ^{125}Te-Enriched 1,3-Ditellurole (1). *n*-Butyllithium (1.0 mL, 2.4 M) was added dropwise to a stirred solution of (trimethylsilyl)acetylene (0.23 g, 2.4 mmol) in 5 mL of dry tetrahydrofuran (THF) cooled to −70 °C under an argon atmosphere. The 80% ^{125}Te-enriched tellurium powder (0.20 g, 1.6 mmol) was added. The mixture was warmed to ambient temperature. After 2 h, the reaction mixture was cooled to −70 °C, and a solution of iodochloromethane (0.35 g, 2.0 mmol) in 1 mL of dry THF was added. The mixture was stirred for 15 min and quenched with 50 mL of water. The product was extracted with dichloromethane (3 × 15 mL). The combined extracts were washed with brine, dried over sodium sulfate, and concentrated to give 0.27 g (63%) of 80% ^{125}Te-enriched (chloromethyltelluro)(trimethylsilyl)acetylene as a pale yellow oil: ^{1}H NMR (CDCl$_3$) δ 4.77 (d, J = 30.8 Hz, and s, 2 H), 0.20 (s, 9 H); field-desorption mass spectrum (FDMS), *m/e* 271 (weak, $C_6H_{11}ClSi^{125}Te$).

Tellurium powder (0.125 g, 1.00 mmol) 80% enriched in ^{125}Te was added to 2 mL (2.0 mmol) of a 1 M solution of lithium triethylborohydride in THF with stirring under an argon atmosphere. After 2 h at ambient temperature, 2 mL of 1 M sodium ethoxide in ethanol was added, followed by 0.27 g (1.0 mmol) of [^{125}Te](chloromethyltelluro)(trimethylsilyl)acetylene in 2 mL of dimethylformamide. The mixture was stirred for 15 h at ambient temperature. The reaction mixture was diluted with water (25 mL) and extracted with dichloromethane (3 × 15 mL). The combined extracts were dried over Na$_2$SO$_4$ and concentrated. The residue was purified by chromatography on silica gel eluted with 1/1 (v/v) hexane/dichloromethane. The product fraction was concentrated and recrystallized from methanol to give 0.063 g (21%) of the 80% [^{125}Te]ditellurole: mp 84–85 °C; FDMS, *m/e* 290 (major peak in parent ion cluster, $C_3H_4{}^{125}Te_2$).

Nuclear Magnetic Resonance Spectroscopy. ^{125}Te NMR spectra were obtained with a Nicolet NT 200 widebore multinuclear spectrometer at 63.22 MHz in 16K of memory zero-filled to 32K with a spectral width of 20 kHz (2.4 Hz/data point; pulse repetition time, 1.00 s) and a pulse width of 7 μs. Samples were run deuterium locked and were referenced to external bis(diethyldithiocarbamato)tellurium(II).[6] The ^{125}Te NMR spectra were resolution enhanced by a double-exponential procedure that is part of the Nicolet software package. The ^{125}Te NMR samples were placed in a 12-mm broad-band probe. Proton decoupling

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was achieved with broad-band modulated decoupling.

^1H NMR spectra were recorded on the same instrument as above with a 5-mm ^1H observe probe. A broad-band decoupling coil tuned to ^{125}Te and set on the frequency of ^{125}Te in 1,3-ditellurole at 4 W of power was used to decouple ^{125}Te. Proton spectra were referenced to internal Me_4Si. ^{13}C NMR spectra were recorded on a JEOL FX270 spectrometer at 27.14 MHz in 16K of memory with a spectral width of 15 kHz (1.8 Hz/data point; pulse repetition time, 0.5 s) and a pulse width of 4 μs. Samples were run deuterium locked and were referenced to internal Me_4Si. Proton decoupling was broad band.

Spectra were simulated with a Bruker Aspect 2000 computer in the program PANIC, part of the Bruker software package. Line broadening of 1 Hz was added to the simulated spectra.

Registry No. 1, 73772-13-1; ^{125}Te, 14390-73-9.

Surface Organometallic Chemistry: Formation of the Grafted Anionic Clusters $[HFeOs_3(CO)_{13}]^-$, $[HFeRu_3(CO)_{13}]^-$, and $[FeCo_3(CO)_{12}]^-$ on a Hydroxylated Magnesia

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Received January 24, 1986

The reaction of the mixed hydrido carbonyl $H_2FeM_3(CO)_{13}$ (M = Ru, Os) and $HFeCo_3(CO)_{12}$ with the surface of partially hydroxylated magnesia has been studied in situ by IR spectroscopy, evolution of gases, and extraction of the adsorbed complexes. At room temperature, $H_2FeM_3(CO)_{13}$ (M = Ru, Os) and $HFeCo_3(CO)_{12}$ are deprotonated at the surface of magnesia to give the anions $[HFeM_3(CO)_{13}]^-$ (M = Ru, Os) and $[FeCo_3(CO)_{12}]^-$ with virtually no disproportionation to other cluster species. The anions $[HFeM_3(CO)_{13}]^-$ (M = Ru, Os) were also synthesized by proton abstraction by KOH and by KH. It is suggested that on the surface of the magnesia the deprotonation occurs via surface OH groups. From IR spectra apparently there is no tight ion pair between the anions on the surface and the Lewis centers which is in contrast with that which is observed for $[HFe_3(CO)_{11}]^-$ (M = Fe). A mechanistic explanation is proposed. Thermal decomposition of the surface-bound anions has been studied.

Introduction

Earlier studies have shown that $Fe_3(CO)_{12}$ reacts with surface OH groups of alumina,[1] magnesia,[1] zinc oxide,[1] or NaY zeolite[2] to give the corresponding anionic hydride $[HFe_3(CO)_{11}]^-$ linked to the surface cation, M^{n+} (M^{n+} = Al^{3+}, Mg^{2+}, Zn^{2+}) via a tight ion pair. The formation of this anionic hydride corresponds to a nucleophilic attack of surface OH groups at coordinated CO followed by β-H elimination and formation of CO_2 adsorbed as carbonate. We report here the results of a study which shows that certain anionic mixed-metal clusters linked to basic surfaces can be obtained through proton abstraction by surface OH groups.[17] The OH groups of a magnesia [surface] deprotonate the mixed-metal hydrido clusters $H_2FeM_3(CO)_{13}$ (M = Ru, Os) or $HFeCo_3(CO)_{12}$, to the corresponding anions $[HFeM_3(CO)_{13}]^-\cdot{}^1/_2Mg^{2+}$ [surface] (M = Ru, Os) and $[FeCo_3(CO)_{12}]^-\cdot{}^1/_2Mg^{2+}$ [surface] without significant formation of secondary products. These conclusions are based upon the absence of gas evolution in placing the cluster on the magnesia support, IR spectra of adsorbed species, and IR spectra of species extracted from the surface. The relative absence of secondary products is of importance in delineating thermal stabilities of bound species and products from thermal decomposition, and in the ultimate production of metal particles which has also been explored in this investigation.

Table I. Infrared Spectra (CO Stretching Region, cm^{-1})

Surface-Bound Species	Extracted Anion ($[PPN]^+$ Salt)
$[HFeOs_3(CO)_{13}]^-$	$[HFeOs_3(CO)_{13}]^-$
2078 w, 2036 ms, 2016 s, 1997 s, 1975 sh, 1941 sh, 1788 w (br)[a]	2078 w, 2037 s, 2013 s, 1997 s, 1964 m, 1940 m, 1794 w
2077 w, 2035 s, 2013 s (br), 1998 s (br), 1937 sh, 1792 w (br)[b]	
$[HFeRu_3(CO)_{13}]^-$	$[HFeRu_3(CO)_{13}]^-$
2070 w, 2030 s (br), 2007 s (sh), 1990 s, 1972 sh[a]	2070 w, 2032 s, 2013 s, 1998 s, 1970 sh, 1940 sh, 1844 w, 1809 w (br)
$[FeCo_3(CO)_{12}]^-$	$[FeCo_3(CO)_{12}]^-$
2065 w, 2004 s, 1968 m (br), 1928 w, 1888 m (br), 1814 w (br)[a]	2062 w, 2003 vs, 1967 w, 1928 w, 1888 m (br), 1811 m (br)

[a] Before extraction. [b] Material on surface after extraction.

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[‡] Université Paul Sabatier.

[§] The Ohio State University.

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