Table I. Data for Bimolecular H/D Exchange and Termolecular Addition of D_2 to $(\eta^4\text{-}C_4H_6)Fe(CO)^-\cdot$ $(m/z\ 138)^a$

% decay of m/z 138	(C$_4$H$_5$D)-Fe(CO)⁻· m/z 139	(C$_4$H$_4$D$_2$)-Fe(CO)⁻· m/z 140	(C$_4$H$_3$D$_3$)-Fe(CO)⁻· m/z 141	(C$_4$H$_2$D$_4$)-Fe(CO)⁻· + (C$_4$H$_6$)-Fe(CO)(D)$_2$⁻· m/z 142	(C$_4$H$_5$D)-Fe(CO)(D)$_2$⁻· m/z 143	(C$_4$H$_4$D$_2$)-Fe(CO)(D)$_2$⁻· m/z 144	(C$_4$H$_3$D$_3$)-Fe(CO)(D)$_2$⁻· m/z 145	(C$_4$H$_2$D$_4$)-Fe(CO)(D)$_2$⁻· m/z 146
5^{b,c}	22	28		20	15	15		
28^d	23	39	2	26	3	7		
90^e	7	16	11	28	8	16	8	6

^{a} These data are derived from the integrals of the ion signals for each mass. Small signals (2–3% of m/z 138) for unknown ions at m/z 139, 140 ((OC)$_3$Fe⁻·?), and 141 in the starting negative ion spectrum and their possible reactions with D_2 impede a quantitative interpretation of the data. ^{b} The errors in this data set are the largest since the ion product signals are the smallest of the three points given. ^{c} 2.33 × 10^{11} molecules/cm^3 of D_2 added. ^{d} 1.83 × 10^{12} molecules/cm^3 of D_2 added. ^{e} 1.63 × 10^{13} molecules/cm^3 of D_2 added.

Our observation that up to four H/D exchanges in the diene ligand of $(C_4H_6)Fe(CO)^-\cdot$ (1) occurred with D_2 strongly suggests that only the two terminal methylene groups are involved.[11] We interpret these results by reversible oxidative-addition of D_2 to 1 giving dideuteride 2 followed by rearrangment to the cis-CH_2D-π-allyl complex 3;[12] 2–4 are excited adducts. Kinetic barriers must exist for one or both of these steps since the bimolecular rate constant for decay of 1 is much smaller than that of the collision limit (k_{col} = 1.5 × 10^{-9} cm^3/molecule/s).[13] Rotation of the C-CH_2D bond in 3 would place a hydrogen of the CH_2D group in proximity to Fe and rearrangement would yield 4. Reductive-elimination of HD from 4 giving 5 would be irre-

versible due to the short ion–neutral reaction time (≤7 ms) and high dilution of product ions and neutrals (≤10^8 ions/cm^3) in these experiments. Repetition of these steps in bimolecular ion–D_2 collisions would then produce the observed additional three H/D exchanged ions terminating at 6. Competitive with these H/D
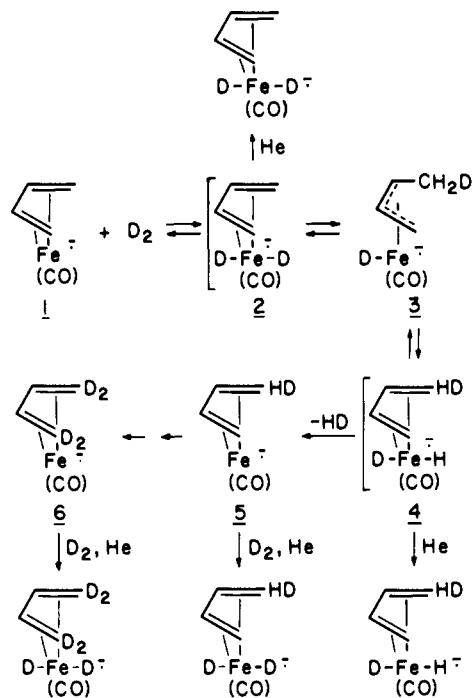
exchanges is termolecular formation of the five total adducts (m/z 142–146) which requires collisional stabilization with the He/CH_4 buffer gas to remove excess vibrational energy in the initially formed excited adducts.

If we assume that rearrangement of a deuterium in 2 could also occur to C_2 (or C_3) of the diene ligand, an excited, 15-electron $(\eta^3\text{-}CH_2=CHCHDCH_2)Fe(CO)(D)^-\cdot$ (7) complex would result. The absence of a fifth or sixth H/D exchange in the reaction of 1 with D_2 means that 7 cannot detach the π-bond of the η^3-homoallyl ligand to effect rotation about the C_1–C_2 bond (required for H/D exchange) via the higher energy, 13-electron $(\eta^1\text{-}1\text{-}$but-3-enyl-$d_1)Fe(CO)(D)^-\cdot$ intermediate. Thus, the deuterium at C_2 in 7 must return to Fe reforming 2.

Ultrasound-Induced Electrochemical Synthesis of the Anions Se$_2{}^{2-}$, Se^{2-}, Te$_2{}^{2-}$, and Te^{2-}

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Organoselenium and -tellurium compounds have become increasingly important as reagents and intermediates in organic synthesis.[1-6] Furthermore, they are promising donor molecules for conductive and photoconductive organic materials.[7-12] Re-

(11) Wrighton and Schroeder (Wrighton, M. S.; Schroeder, M. A. *J. Am. Chem. Soc.* **1973**, *95*, 5764–5765) observed that photolysis of a benzene solution of Cr(CO)$_6$, D_2, and a 1,3-diene that could achieve the s-cis configuration produced the cis olefin by 1,4-addition of D_2 to the diene.
(12) For examples of reversible hydride transfer between transition-metal centers and hydrocarbon ligands, in the condensed phase, see: Howarth, O. W.; McAteer, C. H.; Moore, P.; Morris, G. E. *J. Chem. Soc., Chem. Commun.* **1981**, 506–507 and references therein. Transfer in the gas phase see: Jacobsen, D. B.; Freiser, B. S. *J. Am. Chem. Soc.* **1985**, *107*, 72–80 and references therein.
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Table I. Electroreduction of Se or Te (2×10^{-3} F are consumed) Followed by Addition of an Electrophile (15 min of Contact)

electrolysis no.	Se or Te (mmol)	electrophile (mmol)	solvent	Se or Te derivatives (mmol)	current efficiency, %
1	Se (5)	*p*-cyanobenzyl chloride (2)	DMF	**1** (0.90)	90
2	Se (1)	benzyl chloride (2)	CH₃CN	**2** (traces)	
				4 (0.50)	50
3	Te (5)	benzyl chloride (2)	CH₃CN	**3** (0.525)	70[a]
				5 (0.175)	
4	Te (1)	benzyl chloride (2)	CH₃CN	**3** (0.125)	6
				5 (0.375)	37.5
5	Se (5)	**6**[b] (1)	THF	**7** (0.82)	82

[a] See the text. [b] See ref 26.

search has moved from organoselenium to organotellurium chemistry. A large number of synthetic methods have been developed to prepare organoselenium derivatives. A convenient and widely used technique is the reduction of selenium by trialkylborohydrides[13] which, however, remain relatively expensive materials. In organotellurium chemistry, the number of synthetic reactions is rather small[6,11] and the commercially available tellurium is known to exhibit a low chemical reactivity, due to its surface oxidation.[12]

We report here the use of ultrasound for the convenient and selective electrochemical synthesis of the Se_2^{2-}, Se^{2-}, Te_2^{2-}, and Te^{2-} anions. Prior to these results, a method for the electrochemical synthesis of dialkyl and diaryl chalcogenides was described which involved sacrificial Se[14] and Te[15] electrodes. To overcome the total insolubility in *N,N*-dimethylformamide (DMF) of Te and Se, a Te stick or a mixture of Se and graphite was fused upon a Pt net, and the electrode thus obtained was cathodically polarized in DMF. An alkyl or aryl halide was present during electrolysis or added after. Very recently, we have observed that gray selenium can be conveniently reduced to Se_2^{2-} on mercury and solid cathodes (Pt, carbon cloth)[16] in aprotic media (DMF, acetonitrile, tetrahydrofuran (THF)) under mechanical stirring. The electrochemical reduction occurs beyond –0.8 V vs. SCE on Pt and carbon electrodes and –1.1 V on mercury. In our group, we have observed that this technique fails in the case of Te powder.

Ultrasound has been shown to increase the rate of electrolysis.[17] The ultrasonically promoted electrochemical reduction of insoluble Se or Te powder is performed in a H-type cell whose compartments are separated by glass frits and filled with an aprotic solvent (DMF, acetonitrile, THF) containing 0.1 M Bu₄NBF₄ or Bu₄NPF₆ (0.2 M in the case of THF). Ultrasound (vat Bransonic B 32) and cooling by an ice bath of the cell are maintained during the electrolysis which is performed with an Amel 522 potentiostat and a Tacussel IG5-N integrator. An inert atmosphere of argon is carefully maintained. The cathode, anode, and reference electrode are a carbon cloth, a Pt grid, and a saturated calomel electrode (SCE), respectively.

In acetonitrile as solvent, the electrochemical reduction of Se with ultrasound is observed beyond –0.8 V, and both the cathodic and central compartments of the cell turn brown as long as less than one electrode per Se unit is involved in the reduction process. This color corresponds to the formation of the Se_2^{2-} anion (vide infra). To maintain a reasonable faradaic current (about 200 mA), the working potential has to be progressively moved to more negative values. As the electrolysis proceeds beyond one electron per Se atom, the cathodic and central compartments turn dark

green and Se^{2-} anions are generated. A similar behavior is observed in the case of Te whose reduction occurs beyond –1.1 V in acetonitrile. The Te_2^{2-} and Te^{2-} anions are successively electrogenerated (vide infra) with corresponding dark red and blue color of both the cathodic and central compartments. In THF and DMF, electroreduction of Se powder is slightly easier than in acetonitrile since it starts at $–0.65 \pm 0.05$ V. The dianions Se^{2-} and Te^{2-} are extremely air sensitive, the dianions Se_2^{2-} and Te_2^{2-} being regenerated.

After electrolysis, addition of an electrophile such as an alkyl halide RX leads to the synthesis of dialkyl diselenides, selenides, ditellurides, and tellurides according to (1) and (2). Examples

$$2E \xrightarrow{+2e} E_2^{2-} \xrightarrow{+2RX} REER + 2X^- \qquad (1)$$

$$E \xrightarrow{+2e} E^{2-} \xrightarrow{+2RX} RER + 2X^- \qquad (2)$$

$$E = Se \text{ or } Te$$

are given in Table I (entries 1–4).

In Table I, the amounts of isolated organoselenium derivatives **1**,[16] **2**,[18] and **4**[18] and organotellurium derivatives **3**[19] and **5**[20] and
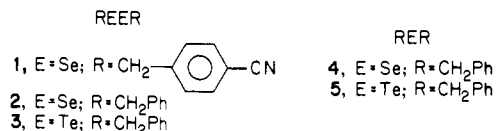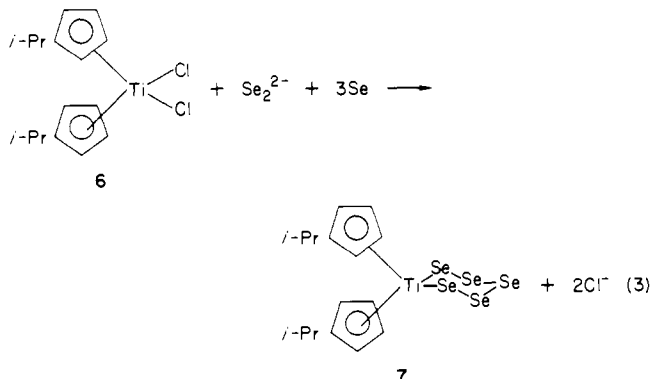
their corresponding current efficiencies have been calculated from ¹H NMR data after treatment of the solution contained in the cathodic compartment of the cell (dilution with water, extraction with diethyl ether, and removal of ether). Organotellurium compounds[21] especially dibenzyl ditelluride (**3**)[22-24] are known to be extremely light sensitive. The decomposition of **3** leads to black Te and dibenzyl telluride (**5**). In electrolysis no. 3, Table I, decomposition of **3** to **5** has obviously occurred during treatment of the electrolysis products so that it can be reasonably assumed that only **3** was initially obtained (0.70 mmol) in a 70% current efficiency.

The Se_2^{2-} and Te_2^{2-} anions as well as the Se^{2-} and Te^{2-} anions tend to migrate toward the anodic compartment since the central compartment of the cell becomes colored during electrolysis. Furthermore, some benzyl chloride has been observed to be left in electrolyses no. 2–4, Table I. In the case of the Se_2^{2-} and Te_2^{2-} anions, this migration phenomena does not decrease significantly the current efficiencies since **1** and **3** can be obtained in 90% and 70% yield. However, this effect is more dramatic in the case of the Se^{2-} and Te^{2-} dianions since the current efficiency drops to 50% in the best case (electrolysis no. 2, Table I). The addition

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of alkyl halide in the central compartment of the cell would improve the current efficiency.[25]

Electrochemical reduction of Se and Te with ultrasound can provide an elegant technique to prepare organotransition-metal chalcogenides. An example corresponds to electrolysis no. 5 of Table I. The formation of the pentaselenide 7[27] corresponds to the overall process (3).

(25) The influence of the nature of the supporting electrolyte has not been investigated. However, it may be suggested that use of alkali salts would modified the current efficiency. Furthermore, as suggested by a reviewer, use of a cation-exchange membrane such as Nafion should prevent the loss of anions and thus improve the yields of alkylated products.

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the discovery of the complex *trans*-[Fe(η^2-H$_2$)(H)(dppe)$_2$]BF$_4$ (**1**) (dppe = PPh$_2$CH$_2$CH$_2$PPh$_2$), which is stable to H$_2$ loss up to 50 °C and undergoes a unique intramolecular exchange of terminal hydride with the hydrogens of the η^2-H$_2$ ligand.[5] The ruthenium analogue *trans*-[Ru(η^2-H$_2$)(H)(dppe)$_2$]BF$_4$ (**2**) contains a more labile dihydrogen ligand.

The title complexes are prepared by reaction of a THF or benzene solution of the corresponding dihydride complexes MH$_2$(dppe)$_2$[6] under hydrogen at 22 °C with approximately 1 equiv of HBF$_4$·Et$_2$O.[7] They can also be prepared by reaction of H$_2$ with monohydrides [MH(dppe)$_2$]BF$_4$ (see below).[8] A similar reaction using HClO$_4$ was reported to yield [FeH$_3$(dppe)$_2$]ClO$_4$ although no physical properties of the complex were reported.[9] Reactions with weaker acids give monohydride complexes.[10]

Complex **1** is a pale yellow solid that must be stored under hydrogen or argon since it reacts slowly with nitrogen to give *trans*-[FeH(N$_2$)(dppe)$_2$]BF$_4$.[11] This dihydrogen adduct dissolves to give stable solutions under argon at 22 °C in THF or CH$_2$Cl$_2$ but THF solutions slowly evolve hydrogen when heated under vacuum to 66 °C ($t_{1/2}$ ~2.5 h). In the presence of the more strongly coordinating ligands acetonitrile or carbon monoxide (L) 1 mol of H$_2$ per Fe is rapidly evolved at 25 °C and the complexes *trans*-[FeH(L)(dppe)$_2$]BF$_4$[11–13] are obtained. White complex **2** loses up to 1 mol of H$_2$ in the solid state at 25 °C under vacuum in 10 min to give orange-yellow [RuH(dppe)$_2$]BF$_4$.[14] The reverse reaction with H$_2$ or D$_2$ is complete in 30 s. Reversible binding of dihydrogen by **2** is also observed in oxygen-donor solvents whereas reaction with CH$_3$CN and CO but not N$_2$ rapidly and irreversibly yields [RuH(L)(dppe)$_2$]BF$_4$.[13,15]

The η^2-dihydrogen ligand in **1** is symmetrically coordinated with Fe–H distances of 1.53 (8) and 1.55 (7) Å, slightly longer than the terminal Fe–H distance of 1.28 (8) Å (Figure 1).[16] The H–H

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Two Molecular Hydrogen Complexes: *trans*-[M(η^2-H$_2$)(H)(PPh$_2$CH$_2$CH$_2$PPh$_2$)$_2$]BF$_4$ (M = Fe, Ru). The Crystal Structure Determination of the Iron Complex

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Recently the complexes M(CO)$_3$(PR$_3$)$_2$(H$_2$) (M = Mo, W; R = Cy, *i*-Pr),[1] Cr(CO)$_5$(H$_2$),[2–4] and Cr(CO)$_4$(H$_2$)$_2$[4] have been reported to contain novel η^2-dihydrogen ligands which retain an H–H bond. Hydrogen is readily lost from these complexes and only the tricyclohexylphosphine molybdenum complex[1b] and the two tungsten complexes are isolable. By contrast we report here

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