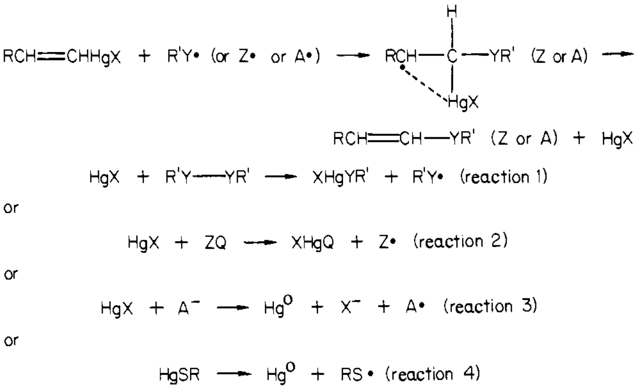
Table I. Photostimulated Reactions of Vinylmercurials

vinylmercurial	reactant	conditions[a]	products	% yield[b]
(*E*)-$Me_3CCH=CHHgCl$	*n*-BuSSBu-*n*	PhH, 17 h	(*E*)-$Me_3CCH=CHSBu$-*n*	100
(*E*)-$Me_3CCH=CHHgCl$	PhSSPh	PhH, 6 h	(*E*)-$Me_3CCH=CHSPh$	100
(*E*)-$Me_3CCH=CHHgCl$	PhSeSePh	PhH, 2 h	(*E*)-$Me_3CCH=CHSePh$	95
(*E*)-$Me_3CCH=CHHgCl$	PhTeTePh	PhH, 18 h	(*E*)-$Me_3CCH=CHTePh$	89
(*E*)-$Me_3CCH=CHHgCl$	$PhSO_2Cl$	PhH, 4 h[c]	(*E*)-$Me_3CCH=CHSO_2Ph$	84
(*E*)-$Me_3CCH=CHHgCl$	$(EtO)_2POK$	Me_2SO, 29 h	(*E*)-$Me_3CCH=CHP(O)(OEt)_2$	76
(*E*)-$Me_3CCH=CHHgCl$	PhP(OBu)OK	Me_2SO, 3.5 h	(*E*)-$Me_3CCH=CHP(O)(OBu)Ph$	84
(*E*)-$Me_3CCH=CHHgCl$	*p*-$MePhSO_2Na$	*t*-$BuOH/H_2O$, 31 h	(*E*)-$Me_3CCH=CHSO_2PhMe$-*p*	81[d]
(*E*)-$Me_3CCH=CHHgCl$	*n*-$PrSO_2Na$	Me_2SO/*t*-$BuOH/H_2O$, 30 h	(*E*)-$Me_3CCH=CHSO_2Pr$-*n*	75[d]
(*E*)-$Me_3CCH=CHHgCl$	PhSH	PhH, $NaHCO_3$, 17 h	(*E*)-$Me_3CCH=CHSPh$	99
(*E*)-$Me_3CCH=CHHgCl$	*t*-BuSH	PhH, $NaHCO_3$, 15 h	(*E*)-$Me_3CCH=CHSCMe_3$	100
(*E*)-$Me_3CCH=CHHgCl$	$PhCH_2SH$	PhH, $NaHCO_3$, 5 h	(*E*)-$Me_3CCH=CHSCH_2Ph$	64
(*Z*)-$HOCH_2C(Cl)=CHHgCl$	PhSH	PhH, $NaHCO_3$, 5.25 h	$HOCH_2C(Cl)=CHSPh$	61
$CH_2=CHHgCl$	PhSH	PhH, $NaHCO_3$, 3 h	$CH_2=CHSPh$	100
(*E*)-$EtC(OAc)=C(Et)HgCl$	PhSH	PhH, $NaHCO_3$, 11 h	$EtC(OAc)=C(Et)SPh$	92
(*E*)-$Me_3CCH=CHHgOAc$	PhSH	PhH, 17 h	(*E*)-$Me_3CCH=CHSPh$	100
(*E*)-$Me_3CCH=CHHgOAc$	$PhCH_2SH$	PhH, 18 h	(*E*)-$Me_3CCH=CHSCH_2Ph$	97
$(Ph)_2CH=CHHgBr$	MeSSMe	PhH, 8 h	$(Ph)_2CH=CHSMe$	100
$(Ph)_2CH=CHHgBr$	*p*-$MePhSO_2Na$	DMSO, 40 h	$(Ph)_2CH=CHSO_2PhMe$-*p*	61
$(Ph)_2CH=CHHgBr$	Me_2CHI	PhH, 2 h[c]	$(Ph)_2CH=CHCH(Me)_2$	50
(*E*)-$Me_3CCH=CHHgSPh$		PhH, 3 h	(*E*)-$Me_3CCH=CHSPh$	99
(*E*)-$Me_3CCH=CHHgSBu$-*n*		PhH, 6 h	(*E*)-$Me_3CCH=CHSBu$-*n*	100
$(Ph)_2C=CHHgSPh$		PhH, 15 h	$(Ph)_2C=CHSPh$	100
$(Ph)_2C=C(CH_3)HgSPh$		PhH, 6 h	$(Ph)_2C=C(CH_3)SPh$	100
(*E*)-$PhCH=CHHgSPh$		PhH, 6 h	$PhCH=CHSPh$, $E{:}Z = 10{:}1$	100
(*E*)-*n*-$PrCH=CHHgSPh$		PhH, 4 h	*n*-$PrCH=CHSPh$, $E{:}Z = 1.2{:}1$	100
(*E*)-*n*-$PrCH=CHHgCl$	$(EtO)_2POK$	Me_2SO, 11 h	*n*-$PrCH=CHP(=O)(OEt)_2$, $E{:}Z = 10{:}1$	56
(*E*)-*n*-$PrCH=CHHgCl$	*p*-$MePhSO_2Na$	Me_2SO/*t*-$BuOH/H_2O$, 22 h	*n*-$PrCH=CHSO_2PhMe$-*p*, $E{:}Z = 6{:}1$	71[d]
(*E*)-$PhCH=CHHgCl$	*p*-$MePhSO_2Na$	Me_2SO/*t*-BuOH, 19 h	(*E*)-$PhCH=CHSO_2PhMe$-*p*, $E{:}Z > 20{:}1$	81[d]
(*E*)-$PhCH=CHHgCl$	*n*-BuSH	PhH, $NaHCO_3$, 3 h	(*E*)-$PhCH=CHSBu$-*n*	100
(*E*)-$PhCH=CHHgCl$	PhSeSePh	PhH, 2h	(*E*)-$PhCH=CHSePh$	90
$CH_2=CHHgCl$	PhSeSePh	PhH, 2h	$CH_2=CHSePh$	91
$CH_2=CHSn(Bu\text{-}n)_3$	PhSSPh	PhH, 4h	$CH_2=CHSPh$	~100

[a] Reactants were irradiated with a 275-W sunlamp approximately 6 in. from the Pyrex reaction flask. The sunlamp maintained a temperature of approximately 35–45 °C. The mercurials (3 mmol) in 10–50 mL of solvent were reacted with a stoichiometric amount of the disulfide or $PhSO_2Cl$, a 10–20% excess of the anions or free thiols, and a 10-fold excess of Me_2CHI. The exclusion of molecular oxygen is essential. [b] Appropriate ^1H NMR, IR, and high-resolution mass spectra were obtained for all products. Satisfactory elemental analyses were obtained for all new compounds except for $Me_3CCH=CHTePh$ (oxidatively reactive), $HOCH_2C(Cl)=CHSPh$ (unstable), and *n*-$PrCH=CHP(O)\cdot(OEt)_2$, for which an NMR yield is reported. The only previously reported 1-alkenylmercury mercaptide is $CH_2=CHHgSR$.[5] [c] Irradiated in Pyrex in a RPR-100 Rayonet reactor at 350 nm. [d] Previously reported in ref 2.

Scheme I



disulfides to participate in reaction 5, although they react readily in reaction 1, may indicate that in the presence of the alkyl disulfide the vinylmercury intermediate ($(Ph)_2C=CHHg$) at least partially decomposes to yield vinyl radicals which do not lead to chain propagation, possibly because of abstraction of the α-hydrogen atoms of the disulfide.

The reaction scheme attributes a surprisingly high reactivity to the species HgX which may be much more reactive than Hg_2Cl_2 (as, for example, R_3Sn and R_3SnSnR_3). Thus, in DMF Hg_2Cl_2 is disproportionated to Hg^0 and $HgCl_2$ but not otherwise reduced by anions such as $PhSO_2^-$. Furthermore, Hg_2Cl_2 undergoes no reaction with $PhSO_2Cl$ in PhH (heterogeneous).

Preliminary results indicate that reactions 1–3 or 5 may occur with other 1-alkenylmetals. Thus, we have found that a photostimulated free-radical chain reaction occurs between $(n\text{-}Bu)_3SnCH=CH_2$ and PhSSPh to afford $CH_2=CHSPh$ in essentially a quantitative yield.

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Reduction of CO_2 to CO by Transition-Metal Dianions[1]

Sir:

We wish to report an unusual CO_2 reduction using electron transfer from transition-metal dianions, in which the oxygen sink is an alkali metal carbonate (eq 1). Since alkali metals provide the ultimate reducing power in the preparations of the transition-metal dianions and the carbon monoxide formed can be photochemically released from the product carbonyl complexes, the overall reaction can be summarized as in eq 2.

$$M_2[M'(CO)_n] + 2CO_2 \rightarrow M_2CO_3 + [M'(CO)_{n+1}] \quad (1)$$

$$n = 5,\ M = Na \text{ or } Li,\ M' = W$$

$$n = 4,\ M = Na,\ M' = Ru \text{ or } Fe$$

$$2M + 2CO_2 \rightarrow M_2CO_3 + CO \quad (2)$$

Treatment of a solution of $Li_2[W(CO)_5]$ in tetrahydrofuran (thf)[2] with gaseous CO_2 at −78 °C resulted in immediate bleaching

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(1) This paper is dedicated to the memory of Professor R. B. Woodward.

of the characteristic orange of the $[W(CO)_5]^{2-}$ dianion. After a few seconds Li_2CO_3 began to precipitate. When the mixture was warmed to 25 °C, solution IR spectra showed that $[W(CO)_6]$ was the only carbonyl-containing product produced in significant quantities. After removal of the solvent in vacuo the mixture was extracted with diethyl ether, to leave a 74% yield[5] of Li_2CO_3.[4] Removal of the ether in vacuo left an 82% yield[5] of $[W(CO)_6]$[6] (eq 3). When the reaction was repeated with $^{13}CO_2$, the sole tungsten-containing product was $[W(^{12}CO)_5(^{13}CO)]$,[7] confirming that the added CO_2 was the source of one of the carbonyl ligands in the product $[W(CO)_6]$. The reaction is equally facile and clean when the dianion is introduced as the sodium salt—the reaction of $Na_2[W(CO)_5]$[2] with CO_2 gives an 83% yield of $[W(CO)_6]$, suggesting that the reaction is insensitive to the nature of the counterion.

$$M_2[W(CO)_5] + 2CO_2 \rightarrow [W(CO)_6] + M_2CO_3 \quad (3)$$

$$M = \text{Li or Na}$$

Since the dianion precursor, $[W(CO)_5(NMe_3)]$, is prepared by photolytic substitution of a carbonyl in $[W(CO)_6]$ the overall reaction constitutes reduction of CO_2 to CO (eq 2) and is in principle cyclic.

The generality of the reaction is indicated by preliminary results[9] which show that $Na_2[Fe(CO)_4]$[10] and $Na_2[Ru(CO)_4]$[11] will also reduce CO_2, giving solutions of the corresponding pentacarbonyls[12] (eq 4). It has been reported[13] that $[Ru(CO)_5]$ decomposes

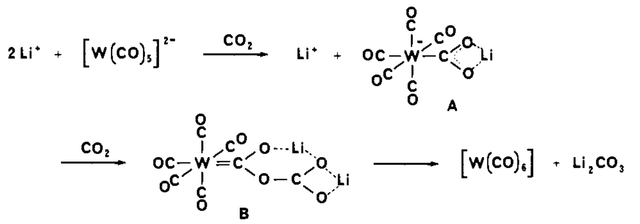
$$Na_2[M(CO)_4] + 2CO_2 \rightarrow [M(CO)_5] + Na_2CO_3 \quad (4)$$

$$M = \text{Fe or Ru}$$

photochemically, with evolution of CO, to give $[Ru_3(CO)_{12}]$, from which $Na_2[Ru(CO)_4]$ is prepared. In principle these reactions again allow a cyclic conversion of CO_2 to CO, but in practice $[Ru_3(CO)_{12}]$ recovery after the photolysis is inefficient.[14]

The literature contains several earlier reports of reactions of transition-metal ions with CO_2 to give carbonyl complexes or carbonates, but in none of these cases was both carbonyl and carbonate formation demonstrated. Green has reported[15] that Mg, Li, and Al derivatives of $[(\eta\text{-}C_5H_5)_2MH_2]$ (M = Mo, W), which behave as if they contain anionic transition-metal moieties, react with CO_2 to give $[(\eta\text{-}C_5H_5)_2M(CO)]$ (M = Mo, W) or, in one case,[15c] $[(\eta\text{-}C_5H_5)_2Mo(CO)Br]PF_6$. Evans has observed[16] Na_2CO_3 formation during the reaction of $Na[(\eta\text{-}C_5H_5)Fe(CO)_2]$ with CO_2 to give $[\{(\eta\text{-}C_5H_5)Fe(CO)_2\}_2]$. Although the use of $^{13}CO_2$ gave dimer containing labeled carbonyls, no evidence was presented for the net formation of CO during this reaction.

Scheme I



It has also been shown that CO_2 will react with neutral complexes of the group 4 metals to give products resulting from deoxygenation and disproportionation of the CO_2, as illustrated in eq 5,[17] and it has been suggested that this reaction is driven by the strength of the metal-to-oxygen bond formed between the $(\eta\text{-}C_5H_5)_2Ti$ moieties and the unusual μ^3-carbonato ligand in the product. It is clear from our results that the formation of

$$4[(\eta\text{-}C_5H_5)_2Ti(CO)_2] + 4\,^{13}CO_2 \rightarrow [\{(\eta\text{-}C_5H_5)_2Ti\}_2(^{13}CO_3)]_2 + 8CO + 2\,^{13}CO \quad (5)$$

high-energy transition-metal–oxygen bonds is not a prerequisite for carbonate-forming reactions. Finally, we note an interesting report by Meshitsuka et al.,[18] who have claimed that nickel and cobalt phthalocyanines are active catalysts for the electrode reduction of CO_2. The authors have pointed out that the metal phthalocyanines would be reduced to their dinegative states at the potentials involved.

The mechanism of the reaction presents an interesting puzzle. We would tentatively suggest a two-intermediate sequence (Scheme I) in which initial nucleophilic attack on a CO_2 molecule forms a carboxylate dianion, intermediate A, probably stabilized by interaction of at least one of the alkali metal cations as shown. Reaction of A with a second molecule of CO_2 could give intermediate B, containing a C_2O_4 ligand, which again would probably be stabilized by counterion interaction as shown. Collapse of B would give $[W(CO)_6]$ and Li_2CO_3. There are precedents in the literature for both proposed intermediates. Reaction of the nucleophilic cobalt complex K[Co(pr-salen)] with CO_2 gives $[Co(\text{pr-salen})KCO_2(\text{thf})]$, from which the CO_2 can be removed in vacuo.[21] This species has been shown by single-crystal X-ray diffraction studies[21] to contain a $CoCO_2K$ unit similar to that proposed in intermediate A. It is also known that reaction of the electron-rich iridium complex $[IrCl(C_8H_{14})(PMe_3)_3]$ with CO_2 gives $[IrCl(C_2O_4)(PMe_3)_3]$ containing a C_2O_4 moiety similar to that suggested in B[19] and that this species itself decomposes to otherwise uncharacterized species containing complexed CO and CO_2. The mechanisms of this reaction, of the reaction in eq 5, and of related reactions[20] have been discussed in terms of the further reactions of intermediates containing C_2O_4 ligands.[17,19] It should be noted that the C_2O_4 ligand acts as a bis-chelate ligand in the iridium complex and that there is no suggestion of a metal–carbon double bond in this molecule. This feature of B is reminiscent of the metal–carbon multiple bonding postulated in intermediates such as $[W(CO)_5COLiPh]$, formed during the preparation of $[W(CO)_5COMePh]$ from $[W(CO)_6]$.[22] A number of other mechanisms might also be suggested for the reaction, and

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(2) $Li_2[W(CO)_5]$ and $Na_2[W(CO)_5]$[3] can be obtained in high yield and purity in thf solution by reduction of $[W(CO)_5(NMe_3)]$ or $[W(CO)_6]$ with the appropriate alkali naphthalide. Details of the preparation of these and other transition-metal dianions will be published shortly: J. M. Maher, R. P. Beatty, and N. J. Cooper, manuscript in preparation.

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(4) Identified by comparison of the IR spectrum of a Nujol mull with that of an authentic sample.

(5) Isolated yields are based on the amount of $[W(CO)_5(NMe_3)]$ used to prepare the dianion. Solution IR spectra suggest that the CO_2 reduction is essentially quantitative.

(6) Identified by solution IR spectroscopy and mass spectrometry.

(7) Identified by comparison of the IR spectrum of a pentane solution with that reported in the literature[8] and by isotopic analysis of the parent and fragmentation peaks in the mass spectrum. The high isotopic purity of the parent ion indicated that no intermolecular scrambling of carbonyl ligand had occurred.

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it is perfectly possible, for example, that elimination of Li_2O from A is followed by reaction with CO_2 to give Li_2CO_3.

Further experiments are in progress to elucidate the mechanisms of these reactions, to explore their generality, and to develop catalytic variants. We are also investigating the electrochemical implications.

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Silicon-29 Nuclear Magnetic Resonance Study of the Surface of Silica Gel by Cross Polarization and Magic-Angle Spinning

Sir:

The properties of silica gels, especially their surface characteristics, have been the subject of much research and interest for several years.[1-3] A variety of physical and spectroscopic techniques have been applied to the study of this interesting system.[2-10] One of the more important aspects of chemical research on silica gels recently has been the development of stationary phases for catalysis and for chromatography by covalent attachment of appropriate moieties to the silica gel surface. As part of a program of study of such systems by NMR,[11] we have addressed the structure of the silica gel surface by ^{29}Si NMR, using cross polarization and magic-angle spinning (CP/MAS).[12-16] Lippmaa and co-workers have very recently reported a ^{29}Si NMR study of solid silicates.[12]

By using CP/MAS techniques, one can obtain ^{29}Si NMR spectra on silica gel in reasonable times (minutes to hours) and with a highly useful level of resolution. The $^{29}Si-^1H$ cross polarization restricts detection to silicon nuclei that are near protons, i.e., at or near the surface. Figure 1 shows a typical ^{29}Si CP/MAS spectrum, obtained on a JEOL FX-60QS spectrometer, of SG-2 silica gel, prepared by the method of Peri and Hensley (without the autoclave step).[3] The spectrum shows peaks at −109.3, −99.8, and −90.6 ppm (relative to liquid Me_4Si), which can be identified with silicon sites in a reasonable surface model.

The studies of Marsmann[17] on silicate–water glass solutions provide the following ^{29}Si chemical shifts for the relevant structural types of interest here: −107.2 ppm, $Si^*(OSi<)_4$; −94.6 ppm, $HOSi^*(OSi<)_3$; −86.6 ppm, $(HO)_2Si^*(OSi<)_2$. Considering bulk susceptibility differences (for which corrections were not made),

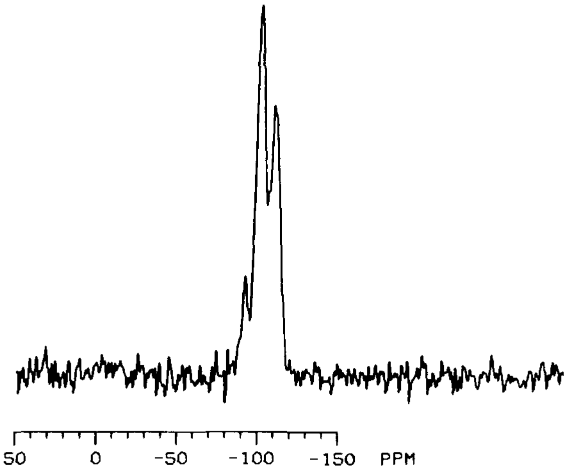

Figure 1. ^{29}Si CP/MAS spectrum of silica gel (SG-2) obtained at 11.88 MHz (1014 scans, 20-ms contact times, 1-s repetitions).

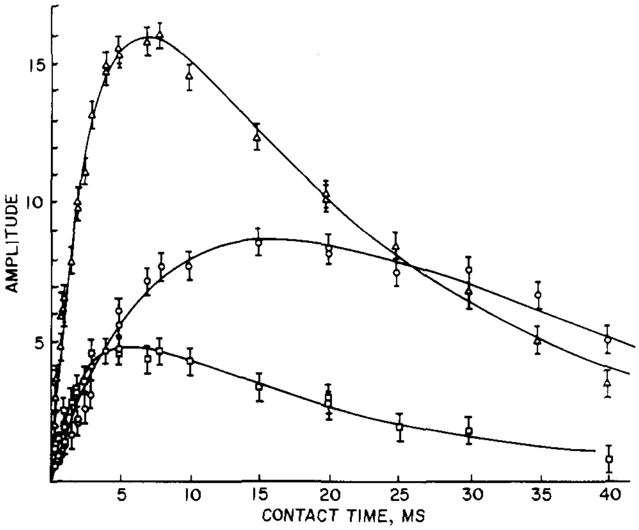

Figure 2. Plot of ^{29}Si CP/MAS amplitudes (relative scale) as a function of Hartmann–Hahn contact time. Similar results were obtained from integrated areas. (△) Data for −99.8-ppm resonance; (○) data for −109.3-ppm resonance; (□) data for −90.6-ppm resonance.

Table I. Summary of Cross Polarization Relaxation Results on Silica Gel

structural type[a]	δ_{Si}[b]	T_{SiH},[c] ms	$T_{1\rho H}$,[d] ms	$M_{\alpha e}$[e]
$(HO)_2Si^*(OSi\leqslant)_2$	−90.6	2.3 ± 0.2	20.3 ± 1.4	6.3 ± 0.2
$HOSi^*(OSi\leqslant)_3$	−99.8	2.9 ± 0.1	22.1 ± 0.8	21.7 ± 0.4
$Si^*(OSi\leqslant)_4$	−109.3	12.7 ± 1.7[f]	21[g]	18.8 ± 0.5

[a] The silicon nucleus of interest is indicated by an asterisk. The structural assignments are based on chemical shift arguments given in the text. [b] Chemical shifts in ppm with respect to liquid Me_4Si; higher numbers correspond to lower shieldings. [c] $^{29}Si-^1H$ cross polarization relaxation time in milliseconds. [d] Proton spin-lattice relaxation time in the rotating frame in milliseconds. [e] Magnitude of the maximum available magnetization in arbitrary, relative units. [f] As the cross polarization time for $^*Si(OSi\leqslant)_4$ is rather large, an accurate measure requires that the radio-frequency fields be extremely well matched over the entire sample. Hence, the value 12.7 ms should be considered qualitative. [g] This value was chosen as an average of the other two $T_{1\rho H}$ values and used as a constraint on the least-squares analysis for this peak. In an unconstrained analysis the value 33 ± 9 was obtained for $T_{1\rho H}$; the corresponding T_{SiH} and $M_{\alpha e}$ values were 9.0 ± 2.1 ms and 13.9 ± 1.7, respectively.

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