Table II. Photochemical Reaction of *trans*-Cinnamate Esters Bound to Polymer Matrices

	composition of the hydrolysate in mol %[a]		
polymer	α-truxillic acid	β-truxinic acid	δ-truxinic acid
Ⓟ$_\alpha$-**2**	100	0	0
Ⓟ$_\beta$-**2**	47	53	0
Ⓟ$_\delta$-**2**	47.3	0	52.7

[a] Note 13.

were derived from δ-truxinic acid. *In other words the polymer matrix directed the photosynthetic event to occur >50% of the time in a stereochemical direction which did not occur at all in monomeric or random polymer analogues.* This result shows that memory-containing polymers can be used to guide the subsequent stereochemical direction of a photochemical reaction. This is a new concept.

This promising observation, coupled with the previously observed memory of a generally synthesized polymer for its chemical origins, suggests many additional ways in which synthesized polymers can be constructed which are selective in subsequent chemical processes. Several of these possibilities are currently under investigation.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research. This work was also supported, in part, by the National Science Foundation (DMR78-08493). Helpful discussions with Professor Kenneth Shea and Professor Thomas Kinstle are acknowledged, with gratitude.

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(11) (a) Unpublished results, J. Damen and D. C. Neckers. The principle method reported herein was developed in several model systems. In one of these, vinylbenzyl-*tert*-Boc-L-proline was copolymerized with styrene and divinylbenzene (10:35:55). After the attached L-proline was hydrolyzed with $HBr-CF_3COOH$, the resulting bromomethyl polymer was treated with racemic *tert*-Boc-proline (Cs salt). Subsequent hydrolysis of the incorporated proline showed it to be enriched in L (L/D = 1.025). This corresponded to a 1.2% enantiomeric excess. We believe this the first example of a "single point", i.e., singly attached, chiral monomer which produced a template in a synthesized polymer. (b) α-Truxillic and β-truxinic acid also generate templates in styrene–divinylbenzene copolymers: J. Damen and D. C. Neckers, *J. Org. Chem.*, in press.

(12) As a solution in degassed benzene, *trans*-cinnamic acid isomerizes—no dimers are observed.[10]

(13) The composition of the hydrolysate was measured by gas chromatography on a 3-ft, $\frac{1}{8}$ in. column, filled with 20% Carbowax 20M on Chromosorb W. The acids from the hydrolysate were, prior to GC analysis, converted into their esters by treatment with diazomethane.

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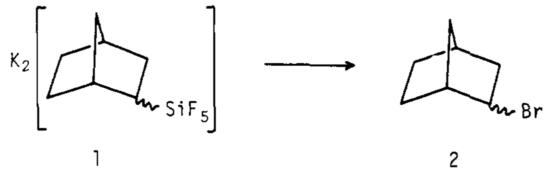
Stereochemistry at Carbon in Cleavage of the Carbon–Silicon Bond in *exo*- and *endo*-2-Norbornylpentafluorosilicates by Various Brominating Agents[1]

Sir:

Electrophilic cleavage of carbon–metal bonds is one of the most important steps in stoichiometric and catalytic organometallic reactions. Much attention has been paid to the stereochemistry of such processes not only to elucidate mechanisms but also to get the basis for many important methods of controlling stereochemistry in organic synthesis.[2] Both retention and inversion of configuration at carbon have been observed.[3,4] Attempts have recently been made to analyze the stereochemistry in terms of initial attack on a HOMO of a metal alkyl.[4b,5] Thus, for d^0 and d^{10} systems the HOMO would be a carbon–metal σ-bonding orbital, while for d^1–d^9 systems it would be either a nonbonding orbital of essentially d character or a carbon–metal σ-bonding orbital. Inversion may result from either an S_E2 (inversion) mechanism (attack on carbon) or electron transfer (attack on metal) followed by back-side attack by nucleophile, while retention of configuration might result from either an S_E2 (retention) mechanism (attack on carbon) or an oxidative addition–reductive elimination sequence (attack on metal). Evidence for the electron-transfer mechanism has been obtained for halogenolysis of organoiron[6] and -cobalt[4c] complexes. For main group organometallics, however, data as yet do not seem to be sufficient and should be accumulated to rationalize the stereochemical courses.

We report here the first stereochemical aspects at carbon in electrophilic intermolecular cleavage of an aliphatic carbon–silicon bond.[7] The alkyl–silicon bond in organopentafluorosilicates readily undergoes oxidative cleavage with various electrophiles which do not affect that in neutral tetracoordinate alkylsilanes.[1,8] On the basis of these novel reactivities, we recently have reported several new synthetic methods[1] including the preparation of organic halides from olefins.[9] We have now determined the stereochemistry of cleavage of the carbon–silicon bond in *exo*- and *endo*-2-norbornylpentafluorosilicates by bromine,[9a,10] N-bromosuccinimide (NBS),[9a] and copper(II) bromide.[9b] The first two reactions proceeded with predominant inversion of configuration, while essentially complete stereochemical scrambling was observed in the last.

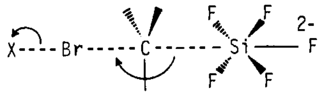


1 2

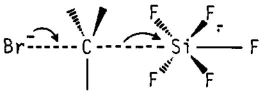
exo- and *endo*-2-norbornylpentafluorosilicates (**1**)[11] were prepared as follows. *exo*-2-Norbornyltrichlorosilane was easily prepared by the stereoselective hydrosilylation of norbornene with trichlorosilane catalyzed by chloroplatinic acid.[12] Silicate formation was achieved in the usual manner by treatment directly with potassium fluoride in water.[9a] The preparation of the endo isomer was more tricky. *endo*-2-Norbornyltrichlorosilane (endo 95%) was prepared by the palladium-black-catalyzed hydrogenation (1 atm) in THF of *endo*-norborn-5-en-2-yltrichlorosilane which was obtained by careful fractional distillation of the Diels-Alder adducts[12,13] between vinyltrichlorosilane and cyclopentadiene. The trichlorosilyl derivative was converted into the trifluorosilyl derivative by treatment with copper(II) fluoride dihydrate in ether[14] and then reacted with potassium fluoride in water to give the endo silicate. During the silicate formation no stereochemical scrambling occurred, as confirmed by comparison of the isomeric purity of 2-norbornyltrimethylsilane[13] obtained by methylation (MeMgBr in ether) of the silicate with that obtained from the trichlorosilyl precursor.

Table I summarizes the stereochemistry of the cleavage of *exo*- and *endo*-**1** with NBS, bromine, and copper(II) bromide in methanol, THF, benzene, and carbon tetrachloride. The exo/endo ratios of 2-norbornyl bromide (**2**) were determined by ^{1}H NMR analysis (2-methine proton ratio)[15] of a crude product after usual workup.[16] The bromine cleavage in polar solvents proceeds in a highly stereospecific fashion with >95% inversion of configuration at carbon. In nonpolar solvents, a low order of stereoselectivity was observed for the cleavage of the exo isomer, while the cleavage of the endo isomer proceeded with higher than 97% inversion. Predominant inversion (≥93%) has also been observed in the NBS cleavage. The reaction is very sluggish in nonpolar solvents such as benzene and carbon tetrachloride, but the stereospecificity seems to be little dependent upon the nature of the solvent. In contrast, the copper(II) bromide cleavage proceeds nonstereospecifically to form **2** in an exo/endo ratio of 70-87/30-13, the ratio being essentially independent of the stereochemistry of the starting material and of the nature of the solvent.

Inversion stereochemistry observed in the bromine and NBS cleavage reactions may result either from an S$_E$2 (open, inversion) process involving back-side attack on carbon by bromine or NBS molecule (A) or an electron-transfer mechanism leading to back-side nucleophilic attack on carbon by a bromide ion (B).



(A)



(B)

Although no distinction between S$_E$2 and electron-transfer mechanisms is possible at present, we suggest here the possibility of an electron-transfer mechanism for the inversion stereochemistry in bromine cleavage (Scheme I),[18] based on the following arguments.

(1) The cleavage reaction occurs smoothly irrespective of the nature of the solvent. (2) The stereochemical scrambling observed with *exo*-**1** in nonpolar solvents suggests the presence of a competing path which proceeds through the formation of an alkyl radical[19] or, less likely, a carbonium ion.[20] Nucleophilic attack on the exo isomer from the more crowded endo side may be less favorable than that on the endo isomer from the exo side, particularly in nonpolar solvents. Partial diffusion of a norbornyl radical might occur from the "cage" arising from the exo silicate, giving rise to the formation of a mixture of exo and endo bromide. (3) We have recently obtained some experimental results indicating that the reaction of organopentafluorosilicates with TCNE proceeds through an initial one-electron transfer.[21]

Scheme I

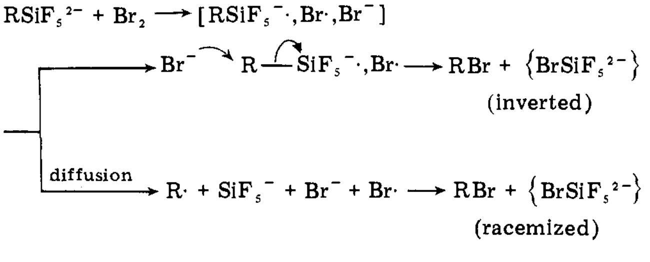


Table I. Cleavage Reactions of *exo*- and *endo*-**1** with Bromine, NBS, and CuBr$_2$ to Form 2-Norbornyl Bromide (**2**)

reagent	solvent	exo/endo ratio[a] (yield, %)[b] of bromide **2**: from *exo*-**1**	exo/endo ratio[a] (yield, %)[b] of bromide **2**: from *endo*-**1**[c]
Br$_2$[d]	MeOH	5/95 (57)	96/4 (56)
	THF	5/95 (31)	96/4 (43)
	benzene	42/58 (67)	98/2 (58)
	CCl$_4$	27/73 (63)	97/3 (57)
NBS[e]	MeOH	2/98 (71)	98/2 (70)
	THF	10/90[f] (33)	96/4[f] (42)
	benzene	7/93 (8)	94/6 (3)
	CCl$_4$	2/98 (6)	98/2 (3)
CuBr$_2$[e]	MeOH	71/29 (39)	78/22 (20)
	THF	73/27 (51)	76/24 (48)
	benzene	70/30 (46)	84/16 (38)
	CCl$_4$	80/20 (35)	87/13 (18)

[a] Determined by ^{1}H NMR (≤±5%). [b] Determined by GLC. [c] Endo 95%. Ratios are corrected based on this purity. [d] 0 °C-room temperature, 4 h. [e] 50 °C, 4 h. [f] The bromide was contaminated with unknown impurities.

Scheme II

$$RSiF_5^{2-} + CuBr_2 \longrightarrow R\cdot + CuBr + BrSiF_5^{2-}$$

$$R\cdot + CuBr_2 \longrightarrow RBr + CuBr$$

The NBS-cleavage reaction seems to be mechanistically different from bromine cleavage. Retardation of the cleavage reaction itself in nonpolar solvents implies that the transition state might involve a charge-separated species and might be sensitive to the steric bulk. An S$_E$2 (inversion) mechanism would be more plausible, but further studies are required to elucidate the mechanism.

Complete loss of stereospecificity in the CuBr$_2$ cleavage clearly indicates that the norbornyl radical is involved in the stereochemistry-determining step. The exo/endo ratios observed in our case are essentially the same as those shown in the reactions of norbornyl and substituted norbornyl radicals with halogenating agents.[19] The present stereochemical result is consistent with the mechanism shown in Scheme II, similar to that proposed for the copper(II) cleavage of various organometallic compounds.[2b,9b,22]

From the synthetic point of view, we have presented the most efficient method for the stereoselective synthesis of *endo*-2-norbornyl bromide (endo ≥98%) from norbornene via the stereoselective exo hydrosilylation and subsequent treatment of the silicate, particularly with NBS in MeOH.[23]

Stereoselective organic synthesis on the basis of the present observations and further mechanistic studies are now in progress in our laboratories.

Acknowledgment. We thank the Ministry of Education for the Grant-in-Aid for Scientific Research (No. 303523) and Shin-etsu Chemical Co., Ltd., for support of the work.

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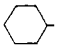
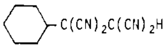
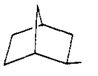
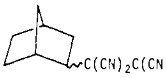
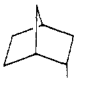
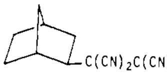
Alkylation of Tetracyanoethylene with Organopentafluorosilicates: Implication of One-Electron-Transfer Mechanism[1]

Sir:

Remarkable attention has been given to the elucidation of the mechanism of cleavage of carbon-metal bonds and the importance of an electron-transfer process has been suggested not only for transition metal alkyls but also for main group organometallics.[2] Alkyl carbon-silicon bonds in hexacoordinate organopentafluorosilicates, $K_2[RSiF_5]$, are now known to be cleaved readily by the action of various electrophiles or oxidizing agents which do not affect those in neutral tetracoordinate silanes.[3] However, a fundamental insight into how carbon-silicon bonds in organosilicates are cleaved has not yet been obtained. To test whether an electron-transfer mechanism is possible for carbon–silicon bond cleavage reactions, we have investigated the reaction of alkylpentafluorosilicates with tetracyanoethylene (TCNE)[4] which is considered to be a typical one-electron acceptor. We describe here the first case of alkylation of TCNE with alkylpentafluorosilicates and several experimental results consistent with a one-electron-transfer mechanism.[5]

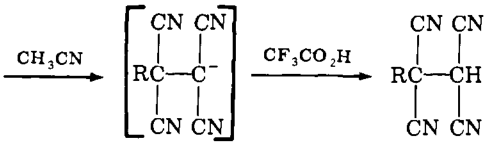
Table I. Reaction of Alkylpentafluorosilicates with TCNE in CH_3CN[a]

R in $K_2[RSiF_5]$	conditions	product[b]	yield (%)[c]
CH_3-	rt, 1 h		0[d]
C_2H_5-	rt, 1 h	$C_2H_5C(CN)_2C(CN)_2H$	11
n-C_8H_{17}-	3 °C, 0.5 h	*n*-$C_8H_{17}C(CN)_2C(CN)_2H$	64
	3 °C, 8 h		34
 (*exo* 100%)	3 °C, 8 h	 (*exo*/*endo* = 1/1)[e]	19
 (*endo* 95%)	3 °C, 8 h		5

[a] $K_2[RSiF_5]$ (5.0 mmol) was allowed to react with 1.0 mmol of TCNE in 10 mL of CH_3CN and decomposed with trifluoroacetic acid. [b] Identified by ^1H NMR, IR, and MS spectra and elemental analyses. [c] Isolated yield based on TCNE. [d] Methylpentafluorosilicate and TCNE were recovered unchanged. [e] Determined by ^1H NMR. [f] Only exo isomer was observed by ^1H NMR.

The addition of TCNE to a suspension of *n*-octylpentafluorosilicate in acetonitrile at −40 °C under an atmosphere of nitrogen resulted in the formation of a deep blue mixture. On warming to 3 °C it turned to grayish green and gradually became dark brown in 0.5 h. After treatment with trifluoroacetic acid, 1,1,2,2-tetracyanodecane (**1**, R = C_8H_{17}),[6] the alkylation product, was isolated in 64% yield. The results obtained with several alkylpentafluorosilicates are summarized in Table I.[7]

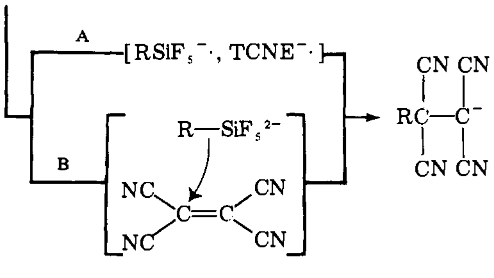
$K_2[RSiF_5]$ + TCNE



2 **1**

While no electronic spectral evidence for participation of charge-transfer complexes in the present reaction was obtained owing to the insolubility of potassium organopentafluorosilicates in organic solvents, electron spin resonance (ESR) studies have proven the formation of the TCNE anion radical. Thus, shaking a degassed acetonitrile solution of TCNE with powder of octylpentafluorosilicate gave intense ESR absorptions due to the TCNE anion radical ($a_N = 0.157_1$ mT, $g = 2.0027_7$).[8]

$RSiF_5^{2-}$ + TCNE



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