hexenylmagnesium chloride to 1 equiv of tetrakis[bromo(tri-*n*-butylphosphine)silver(I)][4] in THF at -78 °C. This solution of 7 was warmed to 0 °C for 45 min, cooled to -78 °C, and quenched with a THF solution of I_2. Cyclization of the 5-hexenyl group bound to silver was found to increase from 11% to 48% as detected by GLC analysis of 1-iodo-5-hexene and cyclopentylmethyl iodide.

[Ag(2.2.2)]I. [Ag(2.2.2)]NO_3 (0.546 g, 1.00 mmol), which was prepared by following a procedure reported by Lehn[24] in 88% yield, mp 200 °C (lit.[24] mp 200 °C), was dissolved in 70 mL of dry ethanol under N_2. Sodium iodide (0.150 g, 1.00 mmol) was dissolved in 10 mL of dry ethanol and added by syringe to the stirred cryptate solution, precipitating $NaNO_3$ immediately. The mixture was filtered, the solvent was removed on a rotary evaporator and the solid residue was dried under vacuum (1 torr, 24 h). A 74% yield was realized: mp 245-249 °C dec; ^1H NMR ($CDCl_3$) δ 2.70 (t, 12, J = 6 Hz), 3.69 (br s, 24).

***n*-Butyl[Ag(2.2.2)] (8).** [Ag(2.2.2)]I (0.0918 g, 0.150 mmol) was mixed with THF (5 mL) in a N_2-flushed centrifuge tube. The cryptate did not dissolve. The resulting suspension was then cooled to -78 °C for 0.5 h at which point 1 mequiv of *n*-butyllithium was added. The original suspension did not dissolve and thermal decompositions consequently had to be carried out by using the resulting suspension.

Tetrakis[iodo(tri-*n*-butylphosphine)]copper(I) was synthesized by the method previously described[25] in 66% yield, mp 72 °C (lit.[25] mp 75 °C).

***n*-Butyl(tri-*n*-butylphosphine)copper(I) (10)** was synthesized as previously described[5a] and **lithio(di-*n*-butyl)(tri-*n*-butylphosphine)copper(I) (9)** was prepared by treating **10** with an additional 1 equiv of *n*-butyllithium.

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Registry No. 1, 52543-55-2; 2, 76011-13-7; 3, 76011-14-8; 5, 76010-99-6; 7, 76011-01-3; 8, 76095-41-5; 9, 24743-93-9; 10, 26679-41-4; tetrakis[iodo(tri-*n*-butylphosphine)silver(I)], 59448-71-4; 1-bromobutane, 109-65-9; bromomagnesium[di-*n*-butyl(tri-*n*-butylphosphine)silver(I)], 76011-03-5; lithium[(*n*-butyl)(*n*-pentyl)(tri-*n*-butylphosphine)silver(I)], 76011-04-6; 5-hexenylmagnesium chloride, 52669-93-9; butylmagnesium bromide, 693-03-8; tetrakis[bromo(tri-*n*-butylphosphine)silver(I)], 76011-05-7; [Ag(2.2.2)]I, 76095-42-6; tetrakis[iodo(tri-*n*-butylphosphine)copper(I)], 59245-99-7; methylsilver, 75993-65-6; lithium[dimethylsilver(I)], 76011-10-4; dilithium[trimethylsilver(I)], 76011-11-5; trilithium[tetramethylsilver(I)], 76011-12-6; butane, 106-97-8; 1-butene, 106-98-9; octane, 111-65-9; methyl iodide, 74-88-4; pentane, 109-66-0.

(24) Dietrich, B.; Lehn, J. M.; Sauvage, J. P. *Tetrahedron* **1973**, *29*, 1647-58.

(25) Mann, F. G.; Purdie, D.; Wells, A. F. *J. Chem. Soc.* **1936**, 1503-13.

Electrophilic Attack of Elemental Fluorine on Organic Halogens. Synthesis of Fluoroadamantanes

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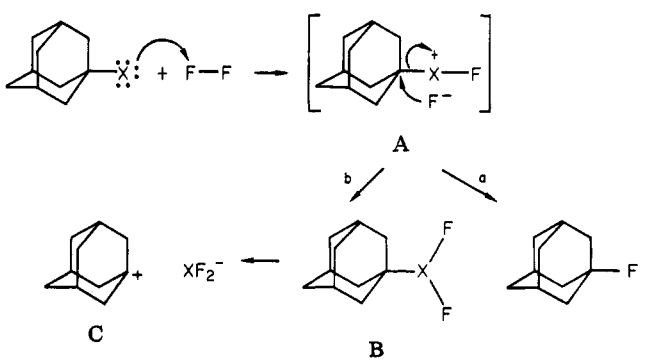
Elemental fluorine acts on bromo- and iodoadamantanes in an electrophilic mode to produce the corresponding fluoroadamantanes. The course of the reaction was investigated in several solvents. It was found that the best yields of the fluoroadamantanes were obtained when Freon ($CFCl_3$) or Freon-chloroform was used. Using methylene chloride as a solvent with iodoadamantanes—but not with the bromo derivatives—resulted in considerable amounts of the corresponding chloro compounds.

Nucleophilic substitution by halogen anion on a carbon bonded to another halogen is a well-established and known procedure. As a matter of fact, a large part of the "classic" fluorine chemistry deals with such nucleophilic substitutions.[1] There are, however, very few examples of replacement of halogen by another halogen atom at a saturated center in which an electrophilic attack is involved. Most of this work concentrates on exchange of iodine with organic iodides.[2] Other examples which do not deal with replacement of the halogen but rather with oxidations due to electrophilic attack of IF_5[3] or its "tamed" aromatic analogue[4] on organic iodides and bromides are also known,

Scheme I



but again such examples are limited.

We describe here the reaction of elemental fluorine with some bromo- and iodoadamantanes at -70 °C in various solvents. The results of these experiments are summarized

(1) See, for example: Chambers, R. D. "Fluorine in Organic Chemistry"; John Wiley and Sons: New York, 1973; Chapters 2 and 3.
(2) de la Mare, P. B. D.; Swedlund, B. E. In "The Chemistry of Functional Groups"; Patai, S., Ed.; Wiley: New York, 1973; Part 1, Chapter 7, p407.
(3) Olah, G. A.; Welch, J. *Synthesis* **1977**, 419.
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(10) Tabushi, I.; Aoyama, Y. *J. Org. Chem.* **1973**, *38*, 3447.

0022-3263/81/1946-0733$01.00/0

Table I

substrate	product (yield, %)[a]	reaction[b] solvent	ref[c]
1-AdBr	1-AdF (99)	$CHCl_3$, CH_2Cl_2, or $CFCl_3$	5
3,5-dimethyl-1-AdBr	3,5-dimethyl-1-AdF (97)[d]	$CHCl_3$, CH_2Cl_2, or $CFCl_3$	
methyl (3-bromo-1-adamantyl)-acetate	methyl (3-fluoro-1-adamantyl)-acetate (90)[e]	$CHCl_3$, $CFCl_3$	
2-AdBr	2-AdF (85)	$CHCl_3$, $CFCl_3$	6
1-AdI	1-AdX		
	X = F (74), X = OEt (21)	$CHCl_3$ (containing EtOH)	7
	X = F (96)	$CHCl_3$ (EtOH-free) or $CFCl_3$	
	X = F (17), X = Cl (83)	CH_2Cl_2	8
	X = F (55), X = Cl (25)	*t*-BuCl at −20 °C	
	X = F (80), X = Cl (10)	*t*-BuCl/$CFCl_3$ (1:1) at −70 °C	
	X = F (91), X = Cl (2)	*n*-BuCl	
2-AdI	2-AdX		
	X = F (51), X = OEt (25)	$CHCl_3$ (containing EtOH)	
	X = F (85)	$CHCl_3$ (EtOH-free) or $CFCl_3$	
	X = F (50), X = Cl (47)	CH_2Cl_2	
1-bromoadamantan-4-one	1-haloadamantan-4-one		
	X = F (50), X = Cl (45)	$CHCl_3$	9, 10
	X = F (95)	$CFCl_3$	9

[a] Indicated yields are determined by GC and isolated yields are practically the same. [b] Unless otherwise stated, the solvents used are of commercial grade, so the EtOH present in $CHCl_3$ was not removed. [c] The referenced compounds are known and their spectral and physical data match those described in the literature. [d] A new compound (oil): NMR δ 2.3–1.15 (m, 13 H), 0.903 and 0.897 (2 s, 3 H each); ^{19}F NMR ϕ^* 134.2 (s). Anal. Calcd for $C_{12}H_{19}F$: C, 79.12; H, 10.44; mol wt − Me, 167. Found: C, 79.35; H, 10.31; mol wt − Me, 167 (mass spectrum). [e] A new compound (oil): IR 1730 cm^{-1} (C=O); NMR δ 3.66 (s, 3 H), 2.20 (s, 2 H), 2.2–1.54 (m, 14 H); ^{19}F NMR ϕ^* 132 (s). Anal. Calcd for $C_{13}H_{19}FO_2$: C, 69.03; H, 8.41; mol wt 226. Found: C, 68.97; H, 8.37; mol wt 226 (mass spectrum).

in Table I. In addition to the results given in Table I, it should be mentioned that when moisture is not strictly excluded, a small percent (2–5%) of the corresponding alcohols is also formed when the bromo derivatives are reacted and up to 20% when the iodo compounds are involved. It should be noted here that neither the bromo nor the iodo compounds react with ethanol, water, or methylene chloride, even on heating.

The incorporation of the ethoxy or the hydroxy group and the better yields of the tertiary fluorine products compared to the secondary ones indicate that the reaction mechanism involves the stable adamantyl cation. Measurements reveal that when chloroform or a mixture of $CHCl_3$–$CFCl_3$ (1:1) is used, only 1 mol equiv of fluorine is required for the disappearance of the reactants containing a tertiary bromo or iodo atom. Still, as can be seen from Table I, there are differences in the products. While considerable amounts of ethoxy and hydroxy adamantanes can be isolated from the reaction of the 1-iodo derivative, all the 1-bromo compounds are practically fully converted to the corresponding fluoroadamantanes. These differences can be explained by Scheme I. It is well established now that at low temperatures, in the absence of radical initiators and in the presence of electron donors, elemental fluorine acts as an electrophile,[11,12] thus forming the intermediate A. When X = Br, reaction path a takes place rapidly because bromine is a strong electronegative atom and the positive charge is concentrated mainly on the tertiary carbon of the adamantane. In this case intermediate A collapses through the attack of the nucleophilic fluorine anion on the carbocationic center, resulting in 1-fluoroadamantane.

When, however, X = I intermediate A can proceed apart from path a also through path b. Species with positive iodine are much more stable than those with positive bromine and what is more, the iodine being a larger atom can easily accommodate several fluorine atoms. Stable compounds of type B are known mainly in the aromatic field[13] but in this case it decomposes readily to the stable carbocation C. This cation will, of course, react with a fluoride anion, ($IF_2^- \rightleftarrows IF + F^-$), with water, or with ethanol, producing fluoro-, hydroxy-, or ethoxyadamantanes, respectively.

One of the features of the reaction of bromo or iodo compounds with fluorine is the red to brown color which is developed a short time after the reaction starts and intensifies as it proceeds. While we are not sure what this color is, we have noticed that it disappears when treated with thiosulfate solution. When this color results from the reaction of bromoadamantane with fluorine and then 1-dodecene is added, the only material that can be isolated from the olefinic parent compound is the 1,2-dibromo derivative. This olefin, however, does not react and does not decolorize the red purple color resulting from the reaction of iodoadamantane and fluorine. Under similar conditions iodine does not react with 1-dodecene. It seems therefore, that what is responsible for the color is elemental bromine or iodine which may be formed from the decomposition of the respective halogen fluorides.

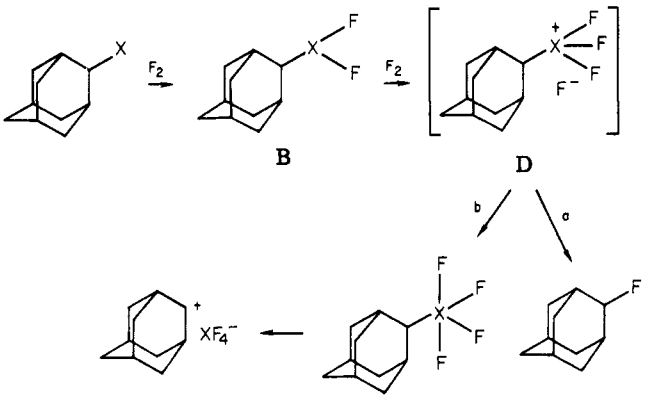
We believe that with the 2-haloadamantanes the reaction takes a slightly different course. When a mixture of

(11) Barton, D. H. R.; Hesse, R. H.; Markwell, R. E.; Pechet, M. M.; Rozen, S. *J. Am. Chem. Soc.* **1976**, *98*, 3036. Alker, D.; Barton, D. H. R.; Hesse, R. H.; James J. L.; Markwell, R. E.; Pechet, M. M.; Rozen, S.; Takashita, T.; Toh, H. T. *Nouv. J. Chim.* **1980**, *4*, 239.

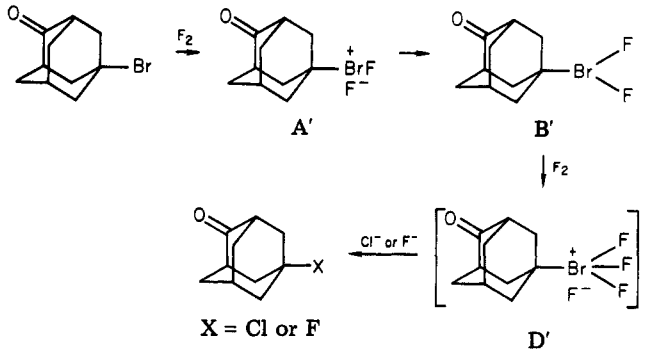
(12) Rozen, S.; Gal, Ch.; Faust, Y. *J. Am. Chem. Soc.* **1980**, *102*, 6860.

(13) For $ArIF_2$ see, for example: Ruppert, I. *J. Fluorine Chem.* **1980**, *15*, 173. For a description of CH_3IF_2 see: Gibson, J. A.; Jansen, A. F. *J. Chem. Soc., Chem. Commun.* **1973**, 739.

Scheme II



Scheme III



$CHCl_3$–$CFCl_3$ (1:1) is used as solvent, nearly 2 mol equiv of fluorine are needed to produce 2-fluoroadamantane either from the bromo derivative or from the iodo one. The proposed mechanism is summarized in Scheme II.

In these cases, where the secondary carbon–halogen bond is stronger than that in the tertiary halogen compounds, intermediate A (Scheme I) is also largely converted to B when X = Br.[14] An additional mole of fluorine forms intermediate D. If X = Br, the positive charge on the bromine atom weakens the C–Br bond, causing the resulting ion pair to easily collapse to give 2-fluoroadamantane (path a). With X = I, the reaction follows path b as in Scheme I and, again, a relatively high proportion of 2-ethoxyadamantane or 2-hydroxyadamantane can be formed if EtOH or H_2O is present.[15] It must be noted that most stages of the suggested mechanism are highly polar in both Schemes I and II and therefore will probably be solvent dependent. Thus, when the reaction solvent is Freon ($CFCl_3$) alone and not the usual medium of $CHCl_3$–$CFCl_3$, the formation of one or all intermediates is slowed down. Since we are maintaining a constant stream of fluorine during the reaction, various amounts of fluorine which are passed through the reaction mixture, in the case of Freon alone, are unconsumed.

Another important point is the chlorination reaction which is observed with the iodo compounds but is absent when the corresponding bromo derivatives are reacted (see Table I). It should be noted that this chlorination occurs most readily in CH_2Cl_2, is almost absent in $CHCl_3$, and is not detectable when the solvent is $CFCl_3$.

In a series of experiments we have found that elemental chlorine reacts rapidly with both 1-iodo- and 2-iodoadamantane to give the corresponding chloro derivatives but does not react at all with the bromo compounds. When passing a stream of nitrogen–diluted fluorine, however, through CH_2Cl_2 at −70 °C, we could not detect any chlorine.[16] We believe that after the initial attack of the fluorine molecule on the iodoadamantanes some of the unsolvated fluoride anions in the intermediate A (Scheme I) leak out of the cage when X = I. These fluoride anions, which are unsolvated and hence strong nucleophiles, react with the methylene chloride in a nucleophilic mode, thus producing chloride anions which in their turn react with any adamantyl cation available. The carbon in Freon ($CFCl_3$) is too hindered for such a nucleophilic attack and the case with chloroform, a carbon which is also quite shielded by the three chlorine atoms, reacts too slowly to produce considerable amounts of chloride anions. Apparently the same steric factors are also responsible for the low yields of chlorination at −70 °C with *t*-BuCl–$CFCl_3$ as solvent and even with *t*-BuCl alone at −20 °C. No such steric hindrance exists when *n*-BuCl serves as a solvent, and the reason for the low yield of chlorination here is of course the slow nucleophilic substitution at the carbon attached to the primary chlorine.

It may be worth noting that when 1-chloroadamantane is treated with fluorine no reaction takes place. Comparing the various carbon–halogen bond strengths and considering the high electronegativity and electron affinity of fluorine and chlorine and the relatively small size of the chlorine atom explain why electrophilic fluorine does not react directly with organic chlorides.[11] This further supports the idea that the origin of the chlorine in the reaction of iodoadamantanes with F_2 is from the nucleophilic attack of F^- on the carbon of the chlorinated solvent and not from any initial attack of F_2 on the chlorine atoms of the solvents.[17]

Unlike the other tertiary bromoadamantanes, 1-bromoadamantan-4-one, when dissolved in $CHCl_3$ or $CHCl_3$–$CFCl_3$, consumes 2 mol equiv of fluorine. Also unlike the other bromo derivatives we find here a high proportion of the 1-chloroadamantane-4-one. These two phenomena are probably related. The oxoadamantyl cation should be less readily formed than the adamantyl cation itself. Hence, the intermediate A′ (Scheme III) will not be converted immediately to the oxoadamantyl cation in the same manner as intermediate A in Scheme I. B′ will then have a chance to react with another molecule of fluorine, producing the ion pair D′. The fluoride anion in the two relatively long-lived intermediates A′ and D′ has enough time to react even with the chloroform, producing enough chloride anions for the final stage of the reaction. That the reaction of the 1-bromoadamantan-4-one with F_2 is considerably slower than the parallel reaction of 1-bromoadamantane can be seen from the reaction of fluorine with a mixture of these two bromo derivatives. The 1-bromoadamantane reacts much more readily and only after its complete conversion to 1-fluoroadamantane does the bromo ketone begin to react. Here again, like in all the previous reactions, changing the solvent to $CFCl_3$ alone causes the reaction to slow down considerably, but on the other hand no source for Cl^- is present so the yield of the

(14) Bromine is capable, under various conditions, of attaining oxidation states of +1, +3, and +5 with fluorine while iodine can form, in addition, the IF_7 compound. See, for example: Emeleus, H. J. "The Chemistry of Fluorine and Its Compounds"; Academic Press: New York, 1969; p 11.

(15) The fact that a little less than 2 mol equiv of fluorine is needed for the reaction (1.7–1.9 mol equiv) can indicate that a small portion of the reaction of 2-haloadamantanes still proceeds through the mechanism described in Scheme I.

(16) Passing F_2 through CH_2Cl_2 does not produce any oxidizing material. Adding an olefin to CH_2Cl_2 which was previously treated with F_2 results in no reaction at all.

(17) For incorporation of chlorine from the solvent into a fluorination reaction using XeF_2, see: Shakelford, S. A.; McGuire, R. R.; Pflug, J. L. *Tetrahedron Lett.* **1977**, 363.

1-fluoroadamantane-4-one is higher than 95%.

Experimental Section

^1H NMR spectra were measured with Bruker WH-90 spectrometer at 90 MHz and with tetramethylsilane as internal standard. ^{19}F spectra were recorded with the same instrument at 84.67 MHz and were reported in parts per million upfield from $CFCl_3$ as internal standard (ϕ^*). Mass spectra were measured with a Du Pont 21-491B spectrometer.

General Procedure. Caution: Fluorine is, of course, a powerful oxidant, a strong poison, and very corrosive material. An appropriate vacuum line in a well-ventilated place should be constructed when working with fluorine. Such a vacuum line containing a 0.5-lb fluorine cylinder and double valve system in a commercial barricade can be obtained from Matheson Corporation. A detailed description of the working system can be obtained from Matheson report no. G-115B. The reaction itself can be carried out in glass vessels.

A mixture of about 1.5% fluorine in nitrogen was slowly bubbled through a cold solution (-75 °C) of about 1.5 g of an adamantane derivative. An efficient vibromixer (Chempec Inc., Hoboken, NJ) was used in order to ensure a good suspension of the gas bubbles in the solution. Dry ethanol-free chloroform and methylene chloride were obtained by refluxing and then distilling these solvents over P_2O_5. The same procedure was employed for $CFCl_3$ although it usually does not contain ethanol.

Shortly after the fluorine had been introduced a red-brown color (probably Br_2 or I_2—see text) was observed. The intensity of this color increased as the reaction progressed. The disappearance of the starting material and the appearance of the products as a function of the amount of fluorine passed through the reaction mixture were monitored by GC on a 3% SE-30 column. A trap of slightly acidic aqueous KI solution was usually placed after the rection mixture vessel so that the excess gas had to pass through it. At the beginning of the reaction no iodine was liberated from this KI solution, thus ensuring that all the fluorine had been consumed. The amount of the fluorine used during the reaction could easily be calculated from knowing its partial pressure with the aid of a gauge which is part of the system mentioned above and from the known volume of the F_2/N_2 mixture. Only when the substrate no longer reacts with fluorine or consumes it very slowly is excess of this element able to reach the KI solution: I_2 is liberated and titrated with thiosulfate. Since fluorine is practically insoluble in the solvents we worked with, it is easy to find out exactly how much fluorine reacts with the substrate and how much is leaving the reaction mixture unreacted.

After the reaction was complete (usually 1-3 h) it was poured into thiosulfate solution, washed with water, dried, and separated. Wherever a purification was required, it was achieved by chromatography on a silica open column or by a high-performance LC 10-μm silica column, using petroleum ether or cyclohexane as eluant. Usually the adamantane derivatives were eluted in the initial fractions. In the case of the known compounds their physical and spectral properties completely match the reported ones. A full characterization of the new compounds is presented in Table I.

Registry No. 1-AdBr, 768-90-1; 2-AdBr, 7314-85-4; 1-AdI, 768-93-4; 2-AdI, 18971-91-0; 1-AdF, 768-92-3; 2-AdF, 16668-83-0; 1-AdOEt, 6221-75-6; 1-AdCl, 935-56-8; 2-AdOEt, 29542-65-2; 2-AdCl, 7346-41-0; 3,5-dimethyl-1-AdBr, 941-37-7; 3,5-dimethyl-1-AdF, 30934-81-7; methyl (3-bromo-1-adamantyl)acetate, 14575-01-0; 5-bromo-2-adamantanone, 20098-20-8; methyl (3-fluoro-1-adamantyl)acetate, 75751-22-3; 5-fluoro-2-adamantanone, 41171-83-9; 5-chloro-2-adamantanone, 20098-17-3.

Light-Induced Free-Radical Reactions of 2-Methoxy-6-methyltetrahydropyran: Irreversible Ring Opening and Multisite Hydrogen Abstraction

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Acetophenone-initiated photodegradation of *cis*-2-methoxy-4-methyltetrahydropyran (**7**) in benzene solvent produced six products. The structures of the products, and studies using optically active **7**, showed that hydrogen abstraction occurs at both C-2 and C-6 of the tetrahydropyran ring. The products derived from the C-2 radical indicated two pathways for this radical: ring opening and loss of methyl, which is consistent with previous work on other similar systems. Results with optically active **7** showed that the ring-opening pathway is irreversible. Generation of an open-chain radical independently also showed that cyclization to a six-membered ring does not occur. The products from the photodegradation were *trans*-2-methoxy-6-methyltetrahydropyran (**8**), methyl hexanoate (**10**), 6-methyltetrahydropyran-2-one (**9**), methyl 5-phenylhexanoate (**11**), 1,7-dimethyl-7-phenyl-6,8-dioxobicyclo[3.2.1]octane (**13**), and 2-methoxy-2-(1-phenyl-1-hydroxyethyl)-6-methyltetrahydropyran (**12**). The yield of lactone **9** was found to be sensitive to the amount of residual oxygen present during the photolysis.

The photodegradation of polysaccharides like cellulose is quite complex.[1-4] Consequently, studies on the photodegradation of carbohydrate models like 2-methoxytetrahydropyrans have evolved.[5-7] The models contain an acetal functional group which is a common linkage in polymeric carbohydrates and probably a site quite reactive toward photodegradation.[8] Hydrogen abstraction by a photoexcited ketone at the 2-position, resulting in the generation of radical **2**, appears to be the initial step in the

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