

Free Radical Chemistry. Part 4.¹ Stereoelectronic Effects in the Additions of Cyclic Ethers to Fluorinated Alkenes

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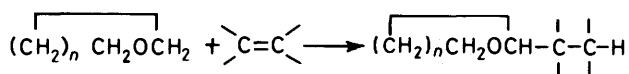
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Free-radical additions of cyclic ethers to various fluorinated alkenes have been studied and a clear stereoelectronic effect has been established. The order of reactivity is oxolane > oxepane > oxane and increasing substitution at the double bond leads to lower reactivity of the fluorinated alkene with oxolane.

In the preceding paper we explained that systematic studies of free-radical additions of ethers and other hydrocarbon derivatives to fluorinated alkenes are of interest both as a probe to the relationship of structure and reactivity in free radical reactions in general and as an approach to syntheses of new fluorocarbon compounds. In this paper we extend these studies to include reactions involving cyclic ethers and we will demonstrate by this approach that stereoelectronic effects are important in the free-radical reactions of these systems. Structural effects in the fluorinated alkene will also be described.

Additions of some cyclic ethers to various fluorinated ethenes have been studied previously²⁻⁷ and additions to hexafluoropropene⁸⁻¹¹ and perfluorocycloalkenes^{10,12-15} have also been described. More recently, additions to 3,3,3-trifluoropropene gave some novel products.¹⁶

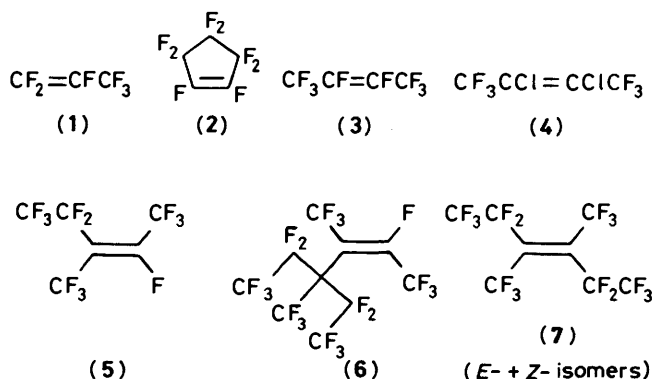
We have systematically studied γ -ray induced additions of a series of cyclic ethers to examples of various classes of fluorinated alkenes (1)–(7), using standard conditions for the reaction and isolation of products. This enables us to compare with confidence the reactivities from the percentage conversion into products. Using oxolane with each of the systems (1)–(5) we obtained very high conversion into the addition products. Only in the case of (5) was there a significant amount of a di-addition product observed (*i.e.* addition involving *both* sites adjacent to the oxygen), otherwise the reactions proceeded according to Equation (1). A trend to lower reactivity with



Equation (1)

Oxirane, $n = 0$; oxetane, $n = 1$; oxolane, $n = 2$; oxane, $n = 3$;
oxepane, $n = 4$

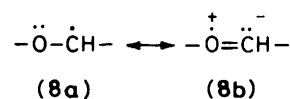
increasing substitution at the double bond is quite clear since only 10% conversion to products was observed with (6) and no significant product was obtained from (7). This change in



reactivity is most obviously attributable to steric effects, and inhibition of bimolecular hydrogen transfer is the most likely explanation for the competitive formation of di-adducts from (5), by an intramolecular process analogous to that described earlier.¹

Results of the addition of a series of cyclic ethers to (1)–(3) are contained in Table 1 and it is clear that perfluorobut-2-ene (3) is very discriminating between the cyclic ethers and in a way that cannot be attributed to a simple steric effect. The reduction in reactivity from a five to six-membered cyclic ether followed by an increase in reactivity with the seven-membered ring is quite striking and demonstrates the influence of a stereoelectronic effect on the reactivity in a free-radical process. These differences are dramatically illustrated in competition reactions involving a deficiency of the perfluoroalkene. For example, although there is no difference in reactivity between oxolane and oxane discernible from Table 1, when (1) was treated with a mixture of these ethers reaction occurred, to the limits of detection, *exclusively* with oxolane. From similar competitions (see Experimental section) we have established the following order of reactivity towards hexafluoropropene: oxolane > oxepane > oxane \approx dioxane.

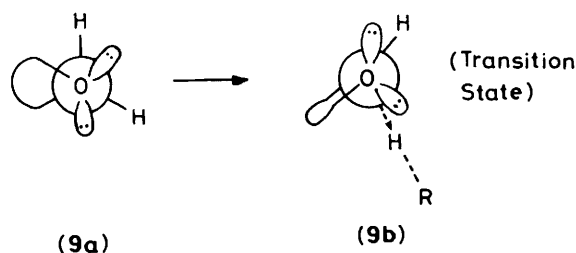
The activating influence of oxygen on the formation of a radical at an adjacent site was discussed in the previous paper¹ and may be represented by (8). Clearly, the interaction of an electron-pair on oxygen, with the orbital containing the odd electron is crucial and, therefore, subject to conformational effects.



It is well known that the flexing of a 5-membered ring is associated with a very small energy change, whereas there is a significant barrier to eclipsing in a six-membered ring.¹⁷ The stereochemistry of oxygen non-bonding pairs is less clear because results from photo-electron spectroscopy^{18,19} indicate that the electron-pairs are non-equivalent, one being more 2p-like, and a better donor than the other non-bonding pair, which is more sp^2 -like and a poorer donor. While recognising this distinction, like other authors²⁰ we have represented them as equivalent and the oxygen as tetrahedral in (9), it being understood that this is an approximation. Therefore, in the transition states leading to the radical derived from these cyclic ethers, the activating effect of adjacent oxygen will depend on the energy-requirement for each ring to produce an essentially eclipsing interaction between an electron-pair on the ether-oxygen and the breaking carbon-hydrogen bond. This is illustrated by the change from (9a) to (9b) which represents the situation in a six-membered ring. Therefore the greater reactivity of the 5-membered ring simply reflects the low energy barrier to attain the favourable conformation (9b), in comparison with that of a six-membered ring. Similarly, the

Table. γ -Ray induced additions of cyclic ethers to fluorinated alkenes and cycloalkenes

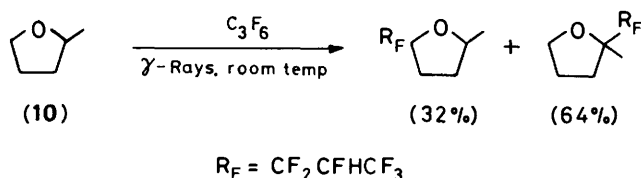
Polyfluoroalkene	Cyclic ether, % Isolated yields (% conversion) ^a				
	Oxetane	Oxolane	Oxane	Oxepane	Dioxane
(1)	65 (100)	95 (100)	95 (100)	70 (100)	80 (100)
(2)		83 (100)	76	71 (100)	49
(3)		87 (100)	21	60 (100)	34

^a Based on polyfluoroalkene consumed.

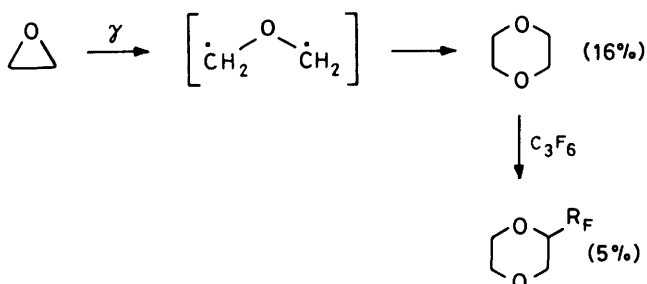
greater flexibility of a seven-membered ring accounts for the greater reactivity of oxepane over oxane.

In a previous paper we outlined the value of experiments in which acetone-butanol ratios, obtained from the decomposition of di-*t*-butyl peroxide in various solvents, correlated with reactivity of those solvents with hexafluoropropene. We have carried out similar experiments with various cyclic ethers and find no significant discrimination between the ethers shown in the Table. However, other workers²¹ have determined rate constants for reactions with *t*-butoxy radicals using flash laser photolysis techniques and observed the same relative order of reactivity for cyclic ethers, that we have observed in reactions with fluorinated alkenes. This highlights, therefore, the significance of the simple and very highly selective competition reactions described above.

Surprisingly, 2-methylloxolane (10), with hexafluoropropene,



gave addition at both sites α - to the oxygen showing discrimination in favour of the tertiary site. This reaction is especially interesting in comparison with the fact that di-isopropyl ether did not react under comparable conditions.¹ It would be too facile to attribute this difference entirely to a favourable stereoelectronic effect arising from the conformation of the cyclic system because we also concluded earlier¹ that steric effects, unfavourable to the propagation step, can be important in additions to acyclic ethers.



Oxetane gave a reasonable yield of adduct with (1) but oxirane gave only products arising from ring-opening. We have also attempted, unsuccessfully so far, to bring about reaction of 12-crown-4 and 18-crown-6 polyethers (the latter at elevated temperatures) with hexafluoropropene and this is surprising in comparison with the reactivity of dioxane (see the Table). We can only conclude, therefore, that these are examples of stereoelectronic effects acting very unfavourably against their reactions with radicals. Crown ethers adopt conformations²² that are very favourable to co-ordination to atoms located centrally and models indicate that these same conformations would be very unfavourable to interaction of electron-pairs on oxygen with the adjacent radical site (see 9b).

Experimental

Apparatus and techniques used have been described in the preceding paper and the isolation procedures are also identical with those described therein.¹ Some of the compounds described in this paper did not respond satisfactorily to normal procedures for elemental analysis and gave values outside what would normally be regarded as satisfactory. In all cases, however, molecular weights were confirmed by mass spectrometry and purity was established by g.l.c., using different column packings.

Additions with Oxolane.—Oxolane (10.4 g, 144 mmol) and hexafluoropropene (1) (5.5 g, 37 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)oxolane⁸ (8.1 g, 95%), m.p. 136–137 °C (760 mmHg). Similarly, oxolane (7.5 g, 104 mmol) and octafluorocyclopentene (2) (4.9 g, 23 mmol) gave 2-(1,2,3,3,4,4,5,5-octafluorocyclopentyl)oxolane (5.4 g, 83%) (mixture of diastereoisomers), b.p. 110 °C (70 mmHg), δ_{H} 1.90 (4 H, m, 2 \times CH₂), 3.73 (3 H, br, CH₂O, CHO), and 4.6 (1 H, br, CHF); δ_{F} 115 to 141 (6 F, overlapping br, 3CF₂), 187.8, 194.8, 198.7, 200.5 (1 F, m, CF), 209.3, 314.3, 222.3, and 228.5 (1 F, m, CFH); m/z 283 ($M^+ - 1$, 0.7%), and 71 (100).

Oxolane (5.8 g, 81 mmol) and octafluorobut-2-ene (3) (4.2 g, 21 mmol) gave 2-(1,2,3,3,3-pentafluoro-1-trifluoromethylpropyl)oxolane (5.1 g, 89%) (mixture of diastereoisomers), b.p. 149–150 °C (760 mmHg) (Found: C, 36.2; H, 3.2; F, 55.0. C₈H₈F₈O requires C, 35.3; H, 2.9; F, 55.9%); δ_{H} 1.87 (4 H, m, 2 \times CH₂), 3.63 (3 H, m, CH₂O, CHO), and 5.10 (1 H, br, CHF); δ_{F} 75.7 (6 F, m, 2 \times CF₃), 180.0, 191.8, 196.0 (1 F, m, CF) and 212.2, and 217.3 (1 F, m, CFH); m/z 271 ($M^+ - 1$, 0.6%) and 71 (100).

Oxolane (8.8 g, 122 mmol) and 2,3-dichlorohexafluorobut-2-ene (4) (8.7 g, 37 mmol) gave 2-(1,2-dichloro-1-trifluoromethyl-3,3,3-trifluoropropyl)oxolane (89%) (mixture of diastereoisomers), b.p. 98–100 °C (25 mmHg) (Found: C, 31.7; H, 3.0; F, 41.5; Cl, 25.0. C₈H₈Cl₂F₆O requires C, 31.5; H, 2.6; F, 37.4; Cl, 23.3%); δ_{H} 2.10 (4 H, m, 2 \times CH₂), 3.87 (2 H, m, CH₂O), and 4.67 (2 H, m, CHO, CHCl); δ_{F} 66.3, 66.8, 67.4, 68.2, and 69.2 (m, 2 \times CF₃); m/z 233 ($M^+ - \text{HCl}_2$, 0.5%) and 71 (100).

Oxolane (5.9 g, 81 mmol) and perfluoro-3-methylpent-2-ene (5) (3.4 g, 11 mmol) gave 2-[1,3,3,4,4,4-hexafluoro-1,2-di(trifluoromethyl)butyl]oxolane (70%) (mixture of diastereoisomers) (Found: C, 32.5; H, 1.8. C₁₀H₈F₁₂O requires C, 32.3; H, 2.1%); δ_{H} 1.78 (4 H, m, 2 \times CH₂), 3.57 (2 H, m, CH₂O), and 4.08 (2 H, br, 2CH); δ_{F} 60.0, 60.8 (3 F, m, CF₃CH), 72.0, 73.5 (3 F, m, CF₃CF), 86.3, 88.0 (3 F, m, CF₃CF₂), 109.9, 115.9 (AB, J 282 Hz), and 114.0 (m) (2 F, CF₂), and 180.0 (1 F, m, CF); m/z 371 ($M^+ - 1$, 1%), 71 (100); and 2,5-bis[1,3,3,4,4,4-hexafluoro-1,2-bis(trifluoromethyl)butyl]oxolane (22%) (mixture of diastereoisomers), b.p. 232 °C (760 mmHg) (Siwoloboff) (Found: C, 28.8; H, 0.5; F, 65.9. C₁₆H₈F₂₄O requires C, 28.6; H, 1.2; F, 67.9%); δ_{H} 2.13 (4 H, m, 2 \times CH₂), and 4.0 (4 H, br, 2 \times CHO, 2 \times CHF); δ_{F} 61.5 (6 F, m, 2 \times CF₃CH), 73.5, 74.7 (6 F, m, 2 \times CF₃CF), 87.2, 89.0 (6 F, m, 2 \times CF₃CF₂), 111.6, 115.9 (AB, J 310 Hz) and

114.8 (4 F, m, $2 \times \text{CF}_2$) and 180.0 (2 F, m, $2 \times \text{CF}$); m/z 613 (1%), 371 (86), and 47 (100).

Oxolane (9.7 g, 135 mmol) and perfluoro-4-ethyl-3,4-dimethylhex-2-ene (6) (13.9 g, 28 mmol) gave mainly starting materials with a small amount of product (0.5 g) tentatively identified by g.l.c.-m.s. as 2-[1,4,4,5,5,5-hexafluoropentyl-3-pentafluoroethyl-1,2,3-tris(trifluoromethyl)]oxolane (4%); [m/z 553 ($M^+ - \text{F}$, 0.5%) and 71 (100)].

Oxolane (8.7 g, 121 mmol) and perfluoro-3,4-dimethylhex-3-ene (7) (11.5 g, 29 mmol) gave quantitative recovery of the starting materials.

Additions with Oxetane.—Oxetane (4.0 g, 69 mmol) and hexafluoropropene (1) (5.4 g, 36 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)oxetane (7.1 g, 65%) (mixture of diastereoisomers), b.p. 47–52 °C (45 mmHg) (Found: C, 35.1; H, 2.9. $\text{C}_6\text{H}_6\text{F}_6\text{O}$ requires C, 34.6; H, 2.9%); δ_{H} 2.60 (2 H, q, J 7.5 Hz, CH_2), 4.40 (2 H, t, J 7.5 Hz, CH_2O), and 5.0 (2 H, br, CHO, CHF); δ_{F} 75.9 (dq, J 6, 11 Hz) and 76.8 (p, J 8 Hz) (3 F, CF_3), 127.9 (sextet, J 10 Hz) and 132.8 (m, J 10 Hz) (2 F, CF_2), 215.8 (dqm, J 42, 10 Hz), and 219.9 (d sextet, J 44, 10 Hz) (1 F, CF); m/z 207 ($M^+ - 1$, 3%) and 57 (100).

Additions with Oxane.—Oxane (9.8 g, 114 mmol) and hexafluoropropene (1) (10.7 g, 71 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)oxane⁹ (12.3 g, 61%) (mixture of diastereoisomers), b.p. 86 °C (70 mmHg) and 2,5-bis-(1,1,2,3,3,3-hexafluoropropyl)oxane⁹ (3.8 g, 35%) (mixture of diastereoisomers).

Similarly, oxane (7.8 g, 91 mmol) and octafluorocyclopentene (2) (5.2 g, 25 mmol) gave 2-(1,1,2,3,3,4,4,5,5-octafluorocyclopentyl)oxane (5.6 g, 76%) (mixture of diastereoisomers), b.p. 75 °C (15 mmHg) (Found: C, 41.1; H, 3.9; F, 50.0. $\text{C}_{10}\text{H}_{10}\text{F}_8\text{O}$ requires C, 40.3; H, 3.4; F, 51.0%); δ_{H} 1.47 (6 H, m, $3 \times \text{CH}_2$), 3.30, 3.77 (3 H, m, CH_2O , CHO), and 4.77 (1 H, dm, J 42 Hz, CHF); δ_{F} 115 to 138 (6 F, overlapping br, $3 \times \text{CF}_3$), 189.3, 194.5, 197.5, 198.2 (1 F, m, CF), 210.7, 211.7, 224.3, and 229.7 (1 F, dm, J 43 Hz, CFH); m/z 297 ($M^+ - 1$, 0.7%), and 85 (59).

Oxane (7.9 g, 92 mmol) and octafluorobut-2-ene (3) (5.8 g, 29 mmol) gave recovered (3) (2.6 g) and 2-(1,1,2,3,3,3-pentafluoro-1-trifluoromethylpropyl)oxane (1.8 g, 21%) (mixture of diastereoisomers), b.p. 65 °C (20 mmHg) (Found: C, 38.1; H, 3.3; F, 53.4. $\text{C}_9\text{H}_{10}\text{F}_8\text{O}$ requires C, 37.8; H, 3.5; F, 53.2%); δ_{H} 1.40 (6 H, m, $3 \times \text{CH}_2$), 3.32 and 3.72 (3 H, m, CH_2O , CHO), and 5.0 (1 H, br, CHF); δ_{F} 73.5, 74.7, and 75.5 (6 F, m, $2 \times \text{CF}_3$), 185.7 and 191.3 (1 F, m, CF), 213.3 and 217.8 (1 F, dm, J 38 Hz, CFH); m/z 285 ($M^+ - 1$, 0.7%), and 85 (100).

Additions with Oxepane.—Oxepane (8.6 g, 86 mmol) and hexafluoropropene (1) (4.3 g, 29 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)oxepane (5.0 g, 70%) (mixture of diastereoisomers), b.p. 79–81 °C (24 mmHg) (Found: C, 43.4; H, 5.2; F, 45.1. $\text{C}_9\text{H}_{12}\text{F}_6\text{O}$ requires C, 43.2; H, 4.8; F, 45.6%); δ_{H} 1.47 (8 H, m, $4 \times \text{CH}_2$), 3.47 (3 H, m, CH_2O , CHO), and 4.87 (1 H, dm, J 44 Hz, CHF); δ_{F} 76.5, 76.8 (3 F, m, CF_3), 117–134 (2 F, overlapping br, CF_2), 214.8, 219.5 (1 F, dm, J 44 Hz, CFH); m/z 189 (0.6%) and 99 (100).

Similarly, oxepane (7.8 g, 77 mmol) and octafluorocyclopentene (2) (5.7 g, 27 mmol) gave 2-(1,1,2,3,3,4,4,5,5-octafluorocyclopentyl)oxepane (5.9 g, 71%) (mixture of diastereoisomers), b.p. 106 °C (23 mmHg) (Found: C, 42.6; H, 4.1; F, 48.0. $\text{C}_{11}\text{H}_{12}\text{F}_8\text{O}$ requires C, 42.3; H, 3.9; F, 48.7%); δ_{H} 1.57 (8 H, m, $4 \times \text{CH}_2$), 3.57 (3 H, m, CH_2O , CHO), and 4.87 (1 H, dm, J 44 Hz, CHF); δ_{F} 115 to 138 (6 F, overlapping br, $3 \times \text{CF}_2$), 189.8, 191.7 (*trans*), 195, 195.5 (*cis*) (1 F, m, CF), 210.3, 211.7 (*trans*), 225.2, and 229.5 (*cis*) (1 F, dm, J 44 Hz, CFH); m/z 183 (0.4%) and 99 (100).

Oxepane (8.0 g, 80 mmol) and octafluorobut-2-ene (3) (5.7 g, 28 mmol) gave 2-(1,1,2,3,3,3-pentafluoro-1-propyl)oxepane (5.1 g,

60%) (mixture of diastereoisomers), b.p. 84–85 °C (24 mmHg) (Found: C, 40.3; H, 3.7; F, 50.7. $\text{C}_{10}\text{H}_{12}\text{F}_8\text{O}$ requires C, 40.0; H, 4.0; F, 50.7%); δ_{H} 1.57 (8 H, m, $4 \times \text{CH}_2$), 3.60 (3 H, m, CH_2O , CHO), and 5.17 (1 H, dm, J 44 Hz, CHF); δ_{F} 74.0, 76.0 (6 F, m, 2CF_3), 183.0, 188.5, 190.0 (1 F, m, CF), 212.3, 213.0, and 215.2 (1 F, dm, J 44 Hz, CFH); m/z 185 (0.4%), 99 (90), and 42 (100).

Additions with 1,4-Dioxane.—Dioxane (9.4 g, 106 mmol) and hexafluoropropene (1) (5.0 g, 33 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)-1,4-dioxane⁸ (6.3 g, 79%) (mixture of diastereoisomers), b.p. 80 °C (46 mmHg).

Similarly, dioxane (11.9 g, 135 mmol) and octafluorocyclopentene (2) (8.9 g, 42 mmol) gave 2-(1,1,2,3,3,4,4,5,5-octafluorocyclopentyl)-1,4-dioxane (6.2 g, 49%) (mixture of diastereoisomers), b.p. 95–98 °C (15 mmHg), δ_{H} 3.53 (7 H, br, $3 \times \text{CH}_2$, CHO) and 5.14 (1 H, dm, J 42 Hz, CHF); δ_{F} 115 to 139 (6 F, overlapping br, $3 \times \text{CF}_2$), 186.7, 193.7 (*trans*), 198.3, 199.5 (*cis*) (1 F, m, CF), 210.0, 211.2 (*trans*), 225.0, and 229.3 (*cis*) (1 F, dm, J 44 Hz, CFH); m/z 300 (M^+ , 4%) and 87 (42).

Dioxane (11.8 g, 135 mmol) and octafluorobut-2-ene (3) (8.2 g, 41 mmol) gave recovered (3) (3.4 g), 2-(1,1,2,3,3,3-pentafluoro-1-trifluoromethylpropyl)-1,4-dioxane (2.2 g, 19%) (mixture of diastereoisomers), b.p. 70 °C (15 mmHg), δ_{H} 3.45 (6 H, s, $3 \times \text{CH}_2$), 3.70 (1 H, m, CHO) and 5.43 (1 H, dm, J 44 Hz, CHF); δ_{F} 73.3, 74.3 (6 F, m, $2 \times \text{CF}_3$), 187.0, 191.3, 193.7 (1 F, m, CF), 212.3 and 216.7 (1 F, m, CFH); m/z 288 (M^+ , 2%), 87 (32); and a mixture of bis-adducts (1.7 g, 15%).

Additions to 2-Methyloxolane.—2-Methyloxolane (2.7 g, 32 mmol) and hexafluoropropene (1) (3.1 g, 20 mmol) gave 5-(1,1,2,3,3,3-hexafluoropropyl)-2-methyloxolane (31%) (mixture of diastereoisomers), b.p. 86 °C (100 mmHg) (Found: C, 41.3; H, 4.1; F, 48.3. $\text{C}_8\text{H}_{10}\text{F}_6\text{O}$ requires C, 40.7; H, 4.2; F, 48.3%); δ_{H} 1.25 (3 H, d, Me), 2.18 (4 H, m, $2 \times \text{CH}_2$), 4.18 (1 H, m, OCHMe), 4.58 (1 H, m, CHO), and 5.18 (1 H, dm, J 43 Hz, CHF); δ_{F} 74.3 (3 F, m, CF_3), 124 to 133 (2 F, overlapping br, CF_2) and 213.2 (1 F, dm, J 43 Hz, CFH); m/z 221 ($M^+ - \text{Me}$, 17%), and 85 (100); and 2-(1,1,2,3,3,3-hexafluoropropyl)-2-methyloxolane (64%) (mixture of diastereoisomers), b.p. 88 °C (100 mmHg); δ_{H} 1.07 (3 H, s, Me), 1.72 (4 H, m, $2 \times \text{CH}_2$), 3.68 (2 H, t, J 6 Hz, CH_2O), and 4.92 (1 H, dm, J 44 Hz, CHF); δ_{F} 75.5 (3 F, m, CF_3), 125.0, 118.1 (2 F, AB, J 282 Hz, CF_2), and 210.5 (1 F, d sextet, J 43, 9 Hz, CFH); m/z 221 ($M^+ - \text{Me}$, 5%), 85 (43), and 43 (100).

Additions with Oxirane.—Oxirane (6.3 g, 144 mmol) and hexafluoropropene (1) (5.5 g, 37 mmol) gave a product (3.6 g) consisting of 1,4-dioxane (16%), 3,3,4,4,5,5-hexafluoropentane-2-one (0.8%), and 2-(1,1,2,3,3,3-hexafluoropropyl)dioxane (5%), identified by comparison with authentic samples.⁸

Radical Competition Reactions.—A mixture containing oxolane (3.2 g, 44 mmol), 1,4-dioxane (3.9 g, 44 mmol), and hexafluoropropene (2.4 g, 16 mmol), in a sealed Carius tube, was irradiated with gamma rays in a similar manner to the preparative experiments outlined above. The product was analysed by g.l.c. (Krytox fluid-perfluoropolyether, DuPont on Chromosorb P, 100 °C) which showed that 2-(1,1,2,3,3,3-hexafluoropropyl)oxolane was the only radical product. Competitions between other pairs of ethers were conducted in a similar manner.

Oxolane (0.45 g, 6.2 mmol), oxane (0.53 g, 6.2 mmol), and hexafluoropropene (0.28 g, 1.9 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)oxalane as the only product (>95%).

Oxane (3.72 g, 43 mmol), oxepane (4.33 g, 43 mmol), and hexafluoropropene (3.3 g, 22 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)-oxane (33%) and -oxepane (67%).

Oxolane (2.52 g, 35 mmol), oxepane (3.68 g, 37 mmol), and

hexafluoropropene (3.2 g, 21 mmol) gave 2-(1,1,2,3,3,3-hexafluoropropyl)-oxolane (70%) and -oxepane (30%).

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