

UV-LIGHT INDUCED CYCLOADDITION OF 3,4-DICHLORO-3,4,4-TRIFLUORO-2-BUTANONE TO 2-METHYL-2-BUTENE*

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Photochemically initiated reaction of 3,4-dichloro-3,4,4-trifluoro-2-butanone (*I*) with 2-methyl-2-butene in the liquid phase gave a mixture of isomeric oxetanes *Xa*, *Xb* (26% rel), reduction products of the starting ketone, *XI* and *XII*, and the recombination products *XIVa*, *XIVb*, *XV*. Irradiation of halogenoketone *I* in the gaseous phase by an ultraviolet discharge tube gave a mixture of compounds *III*—*VIII*; their structures indicate that the Norrish cleavage of "type *I*", cleavage of C—Cl bonds and reduction of C—Cl bonds were operative in their formation.

The photochemical (2 + 2)-cycloadditions of olefinic compounds to carbonyl compounds, giving rise to products with the oxetane skeleton, have been studied to a wide extent¹⁻³. The reactions were studied with various pairs of non-fluorinated or fluorinated reactants and afforded different yields of oxetane derivatives³. The cycloadditions were conducted both in the liquid and in the gaseous phases, medium-pressure and high-pressure discharge tubes being used as sources of radiation^{2,3}. There are rare reports⁴ in the literature on cycloaddition of an aliphatic fluorinated carbonyl compounds to a non-fluorinated olefinic component. Such a pair of reactants is treated in the present paper.

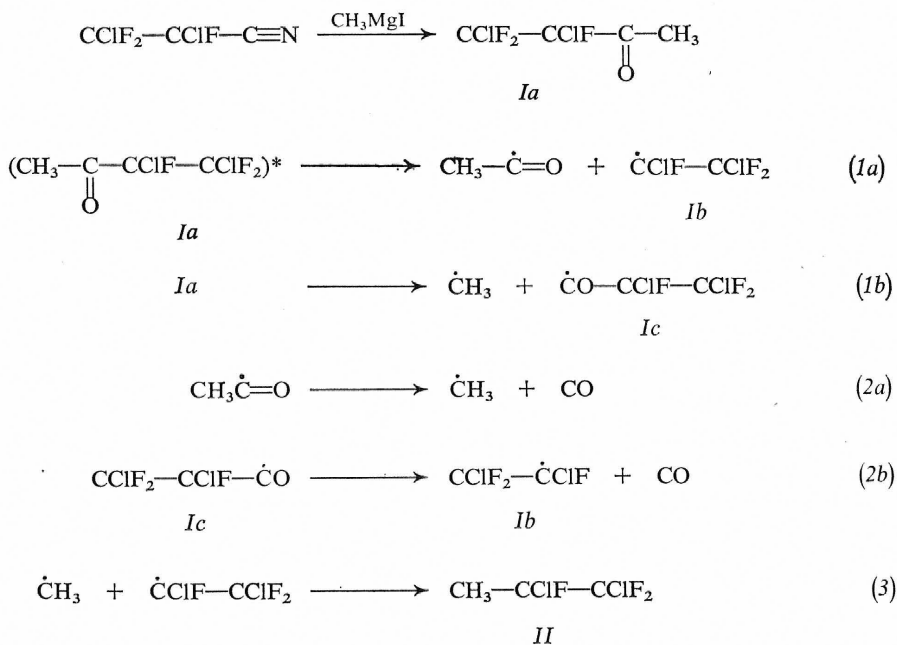
The chosen olefinic component was 2-methyl-2-butene, known by high reactivity in a number of cycloadditions^{2,3}. The chosen carbonyl component was 3,4-dichloro-3,4,4-trifluoro-2-butanone (*I*), whose boiling point permitted to conduct the reaction in liquid phase at room temperature. The triplet energy T_1 of trimethylethylene⁵ is 357.2 kJ mol⁻¹. The energy T_1 of the halogenoketone *I* is not known, but can be supposed to be between those of acetone^{6,7} (335 kJ mol⁻¹) and hexafluoroacetone⁸ (291.2 kJ mol⁻¹) and to be lower than T_1 of the olefin. This fulfils the principal photochemical condition⁹ for cycloaddition of an oxo compound to an olefin.

The starting ketone *I* was prepared by reaction of methylmagnesium iodide with 2,3-dichlorotrifluoropropionitrile^{10,11} in a yield of 20.2%. The literature¹² reports

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good yields of ketones obtained by reaction of Grignard agents with nitriles of perfluorinated acids. In our case the yield was probably reduced by side reactions of methylmagnesium iodide¹³ with C—Cl bonds of chlorofluoropropionitrile as was inferred from considerable liberation of elemental iodine in the reaction and from a considerable amount of chloride ions in the reaction mixture.

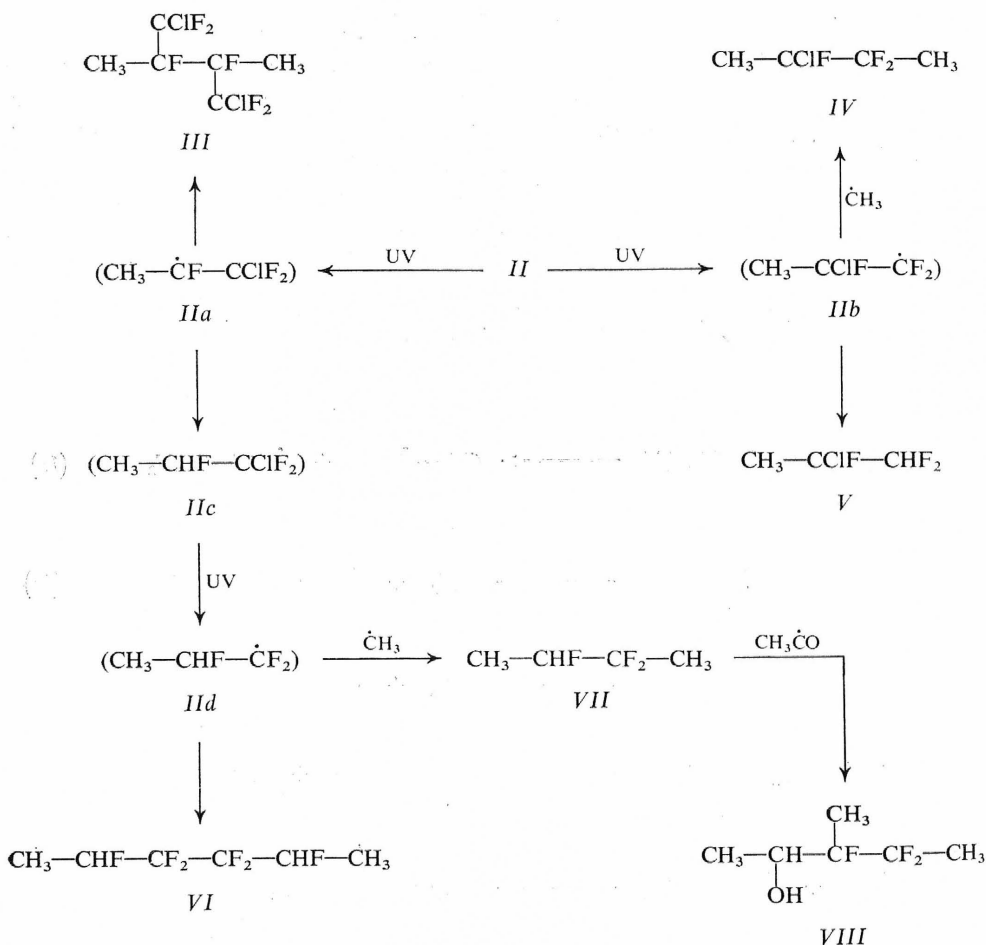
Prior to performing the cycloaddition reactions we studied qualitatively the behaviour of the halogenoketone *I* in irradiation by ultraviolet light in a solution and in the gaseous phase. A solution of the ketone *I* in benzene showed no change after 15 hours' irradiation, but irradiation in the gaseous phase by a high-pressure discharge tube at 110°C produced strong photolysis. The principal routes of the photolysis were deduced from the final products *III*–*VIII* (Scheme 1). The main primary



SCHEME 1

process was evidently the Norrish cleavage¹⁴ of “type I” (*1a*), (*1b*). The main secondary process, (*2a*) and (*2b*), was cleavage of the acetyl radical and the acyl radical *Ic* to carbon monoxide and a methyl or halogenoethyl radical (*Ib*). The terminal reaction (*3*) probably gave rise to the main intermediate, *II*, from which all the identified products, *III*–*VIII*, can be derived (Scheme 2). The individual processes involve cleavage of the bond C—Cl, leading to intermediates *IIa* and *IIb*, cleavage of C—H bonds, producing the radical *IIc*, reduction of the C—Cl bond, giving rise to compounds *IIc* and *V*, recombination of the alkyl radicals to products *III* and *IV*, and

finally the "reductive acetylation", affording the alcohol *VIII*. The proportion of the products *III* – *VIII* in the reaction mixture is given in Table I.



SCHEME 2

The suitability of the photochemical reactor and the irradiation source for the cycloaddition of the ketone *I* was tested on cycloaddition of benzaldehyde to 2-methyl-2-butene¹⁵⁻¹⁷. The reaction gave a mixture of isomeric cycloadducts *IXa* and *IXb* in a yield of 42.8%. The proportion of the two isomers was 1.9 : 1, in good agreement with the ratio 1.6 : 1 found previously^{16,17}. Attempts at a photochemically initiated reaction of the ketone *I* with 2-methyl-2-butene gave a mixture of compounds (*Xa*, *Xb*, *XI*, *XII*, *XIVa*, *XIVb*, *XV*), the proportion of which is given in Table I. The mixture contained the cycloadducts *Xa* and *Xb*, recombination products

XIVa and *XIVb*, as well as the fragmentation products *XI*, *XII* and *XV*. The probable ways of formation of some of the compounds are indicated in Scheme 3. Attempts to separate the cycloaddition isomers *Xa* and *Xb* were not successful.

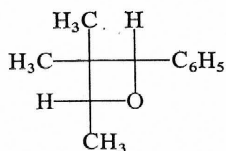
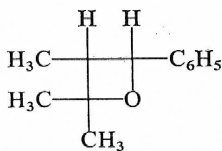
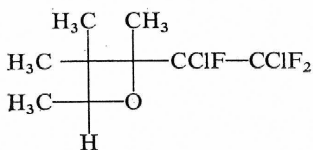
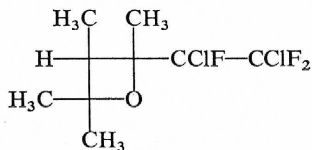
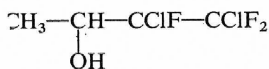
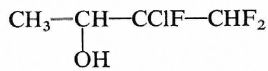
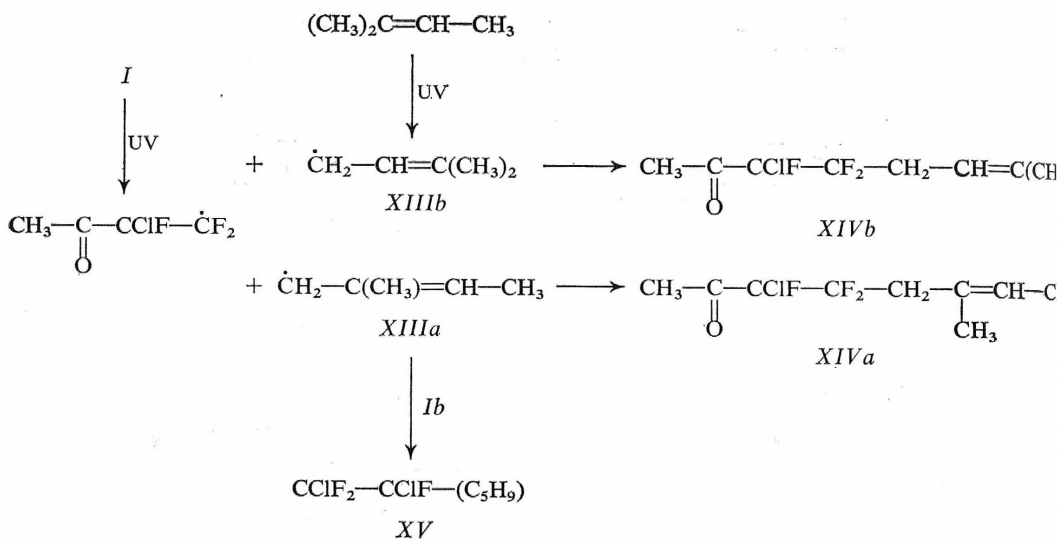
*IXa**IXb**Xa**Xb**XI**XII*

TABLE I

Composition of the Reaction Mixture in Photolysis and Cycloaddition of Ketone I

Compounds	Photolysis		Cycloaddition	
	Compounds	% rel. ^a	compound	% rel. ^a
<i>III</i>		90	<i>Xa, Xb</i>	26
<i>IV</i>		3	<i>XI</i>	11
<i>V</i>		2.5	<i>XII</i>	22
<i>VI</i>		2	<i>XIVa</i>	20.5
<i>VII</i>		1	<i>XIVb</i>	9
<i>VIII</i>		1.5	<i>XV</i>	11.5

^a Determined by gas chromatography without calibration; average of two measurements.



SCHEME 3

The fundamental analytical tool for identifying the products in the reaction mixture was mass spectrometry combined with a chromatographic separation. By analysis of the mass spectra we derived the structures of products *III*–*VIII* (Table II). The structures of the isomeric oxetanes *IXa* and *IXb* were deduced from elemental analysis of a mixture of the isomers and from identity of the mass spectrum of the separated isomers *IXa* and *IXb* (Table II). In the $^1\text{H-NMR}$ spectrum of a mixture of isomers *IXa* and *IXb* the content of either isomer was determined from the integral intensities of signals of the hydrogen atoms bound directly to the oxetane ring. The structure of the cycloadducts *Xa* and *Xb* were determined analogously. The $^{19}\text{F-NMR}$ spectrum of the same mixture of isomers revealed that the signals of fluorine in the groups CClF and CClF_2 were split in the same way and that they had the same chemical shifts as the ketone *I* (Table III). The $^1\text{H-NMR}$ spectrum exhibited dihedral interactions due to methyl groups ($^4J_{\text{HH}} = 2 \text{ Hz}$). However, the respective isomers *Xa* and *Xb* were not identified. The propounded structures of the products *XI*, *XII*, *XIVa*, *XIVb* and *XV* are based on mass spectra of the separated substances (Table II). Correctness of these structures is confirmed by infrared spectra of the mixtures enriched in the individual predominant compounds.

TABLE II
 Mass Spectra of Compounds III—VIII, X—XII, XIV, XV
 (Ionic species m/e , relative intensity %, probable composition).

Compound (molecular mass)	Main ionic species
III $C_6H_6Cl_2F_6$ (262)	262/0.06 (M) ⁺ , 177/3 ($C_5H_3F_6$) ⁺ , 162/2 (C_4F_6) ⁺ , 154/1 ($C_6H_6F_4$) ⁺ , 142/3 ($C_5H_6F_4$) ⁺ , 123/22 ($C_5H_6F_3$) ⁺ , 116/10 (C_2ClF_3) ⁺ , 96/2 ($C_3H_3F_3$) ⁺ , 85/100 ($CClF_2$) ⁺ , 77/14 ($C_3H_3F_2$) ⁺ , 66/4 ($CClF$) ⁺ , 54/15 (C_4H_6) ⁺ , 50/20 (CF_2) ⁺ , 46/20 (C_2H_3F) ⁺ , 45/7 (C_2H_2F) ⁺ , 44/20 (C_2HF) ⁺ , 43 (C_2F) ⁺
IV $C_4H_6ClF_3$ (146)	146/0.08 (M) ⁺ , 116/2 (C_2ClF_3) ⁺ , 96/13 ($C_3H_3F_3$) ⁺ , 81/95 (C_2F_3) ⁺ , 77/20 ($C_3H_3F_2$) ⁺ , 66/3 ($CClF$) ⁺ , 46/49 (C_2H_3F) ⁺ , 45/100 (C_2H_2F) ⁺ , 44/25 (C_2HF) ⁺ , 43/22 (C_2F) ⁺ , 40/10 (C_3H_4) ⁺ , 33/12 (CHF) ⁺ , 31/95 (CF) ⁺
V $C_3H_4ClF_2$ (132)	132/0.1 (M) ⁺ , 117/5 (M—CH ₃) ⁺ , 97/15 (M—Cl) ⁺ , 82/8 (C_2HF_3) ⁺ , 81/100 (C_2F_3) ⁺ , 77/9 ($C_3H_3F_2$) ⁺ , 66/10 ($CClF$) ⁺ , 51/5 (CHF_2) ⁺ , 46/17 (C_2H_3F) ⁺ , 44/27 (C_2HF) ⁺ , 44/10 (C_3H_4) ⁺ , 33/12 (CHF) ⁺ , 31/8 (CF) ⁺
VI $C_6H_8F_6$ (194)	194/0.2 (M) ⁺ , 159/100 ($C_5H_4F_5$) ⁺ , 128/8 ($C_4H_4F_4$) ⁺ , 126/10 ($C_4H_2F_4$) ⁺ , 81/14 (C_2F_3) ⁺ , 77/10 ($C_3H_3F_2$) ⁺ , 59/5 (C_3H_4F) ⁺ , 47/7 (C_2H_4F) ⁺ , 45/5 (C_2H_2F) ⁺ , 44/6 (C_2HF) ⁺ , 43/4 (C_2F) ⁺ , 40/8 (C_3H_4) ⁺ , 32/10 (CHF) ⁺ , 31/8 (CF) ⁺
VII $C_4H_7F_3$ (112)	112/0.15 (M) ⁺ , 97/1 (M—CH ₃) ⁺ , 96/11 (M—CH ₄) ⁺ , 82/100 (C_2HF_3) ⁺ , 81/5 (C_2F_3) ⁺ , 77/5 ($C_3H_3F_2$) ⁺ , 65/3 ($C_2H_3F_2$) ⁺ , 47/18 (C_2H_4F) ⁺ , 44/14 (C_2HF) ⁺ , 40/4 (C_3H_4) ⁺ , 32/5 (CHF) ⁺
VIII $C_6H_{11}F_3O$ (156)	157/2 (M + 1) ⁺ , 156/16 (M) ⁺ , 111/2 ($C_3H_2F_3O$) ⁺ , 89/7 ($C_4H_3F_2$) ⁺ , 85/3 (C_3H_4FO) ⁺ , 73/4 (C_4H_5F) ⁺ , 61/19 (C_2H_3FO) ⁺ , 46/22 (C_2H_3F) ⁺ , 44/5 (C_2HF) ⁺ , 43/100 (C_2H_3O) ⁺ , 32/3 (CHF) ⁺ , 31/3 (CF) ⁺
Xa, Xb $C_9H_{13}Cl_2F_3O$ (264)	266/1 (M + 2) ⁺ , 264/2 (M) ⁺ , 248/3 (M—CH ₄) ⁺ , 116/5 (C_2ClF_3) ⁺ , 71/89 (C_4H_7O) ⁺ , 69/26 (C_5H_9) ⁺ , 55/12 (C_4H_7) ⁺ , 45/14 (C_2H_5O) ⁺ , 43/100 (C_2H_3O) ⁺ , 41/25 (C_3H_5) ⁺
XI $C_4H_5Cl_2F_3O$ (196)	198/2 (M + 2) ⁺ , 196/12 (M) ⁺ , 194/20 (M—2) ⁺ , 90/9 ($C_4H_4F_2$) ⁺ , 62/16 (C_2F_2) ⁺ , 58/100 (C_3H_3F) ⁺ , 43/50 (C_2H_3O) ⁺ , 41/22 (C_2HO) ⁺
XII $C_4H_6ClF_3O$ (162)	108/6 ($C_4H_6F_2O$) ⁺ , 106/12 (C_4H_4ClF) ⁺ , 58/80 (C_3H_3F) ⁺ , 45/100 (C_2H_5O) ⁺ , 43/58 (C_2H_3O) ⁺ , 41/26 (C_2HO) ⁺
XIVa $C_9H_{12}ClF_3O$ (228)	228/0.3 (M) ⁺ , 150/3 ($C_7H_9F_3$) ⁺ , 135/12 ($C_6H_6F_3$) ⁺ , 69/14 (C_5H_9) ⁺ , 43/100 (C_2H_3O) ⁺ , 41/19 (C_3H_5) ⁺ , 39/7 (C_3H_3) ⁺

TABLE II
 (Continued)

Compound (molecular mass)	Main ionic species
<i>XIVb</i> $C_9H_{12}ClF_3O$ (228)	228/0.1 (M) ⁺ , 226/0.2 (M-2) ⁺ , 152/19 (C ₆ H ₇ F ₃ O) ⁺ , 125/14 (C ₃ ClF ₃ O) ⁺ , 97/27 (C ₂ ClF ₂) ⁺ , 70/19 (C ₄ H ₃ F) ⁺ , 69/40 (C ₅ H ₉) ⁺ , 68/15 (C ₅ H ₈) ⁺ , 55/26 (C ₄ H ₇) ⁺ , 43/100 (C ₂ H ₃ O) ⁺ , 41/62 (C ₃ H ₅) ⁺
<i>XV</i> $C_7H_9Cl_2F_3$ (220)	222/0.1 (M+2) ⁺ , 220/0.1 (M) ⁺ , 218/0.5 (M-2) ⁺ , 85/16 (CClF ₂) ⁺ , 70/42 (C ₃ H ₄ F) ⁺ , 69/90 (C ₅ H ₉) ⁺ , 57/89 (C ₃ H ₂ F) ⁺ , 55/32 (C ₄ H ₇) ⁺ , 43/95 (C ₃ H ₇) ⁺ , 41/100 (C ₃ H ₅) ⁺

 TABLE III
 NMR Spectra of Compounds *I*, *IX* and *X*

Chemical shift ppm (number of protons); s singlet, d doublet, q quarter, m multiplet; interaction constants, J Hz. ¹⁹F-NMR spectrum related to CCl₃F.

Compound	Kind of spectrum	Character of signals
<i>I</i>	¹⁹ F	dd—127.9 CClF, ³ J _{F_aF_c} 8.5, ³ J _{F_aF_b} 10.5, ⁴ J _{HF} 3.5; dd—64.8 CClF ₂ , ² J _{F_bF_c} 39.5
<i>IXa</i> , <i>IXb</i>	¹ H	m 1.15 (9) CH ₃ ; d 2.97 (1) 3-H (<i>IXb</i>); q 4.69 (1) 2-H (<i>IXa</i>); 7.30 (5) C ₆ H ₅
<i>Xa</i> , <i>Xb</i>	¹ H	d 0.9 (3) CH ₃ ; d 0.97 (3) CH ₃ , ⁴ J _{HH} 2.0; dd 1.19 (3) CH ₃ , ³ J _{HH} 6.0; q 1.54 (3) CH ₃ , ⁴ J _{HH} 2.0; q 4.87 (1) 4-H (<i>Xa</i>)
	¹⁹ F	dd—122.7 CClF; dd—63.7 CClF ₂ , ³ J _{F_aF_c} 8.0, ³ J _{F_aF_b} 10, ² J _{F_bF_c} c. 15

 TABLE IV
 Conditions and Results of the Reaction of Methylmagnesium Iodide with 2,3-Dichlorotrifluoropropionitrile

Nitrile g/mol	CH ₃ Mgl/Nitrile mol/mol	Temperature °C	Product <i>I</i>	
			g	%
60/0.420	1.15	-10	11.2	20.6
103.5/0.73	2	-40	4.05	7.4
13.9/0.147	1	-40	2.59	11.8

EXPERIMENTAL

The temperature data were not corrected. Gas chromatography was carried out in an apparatus Chrom 3 (Laboratorní přístroje, Prague; flame ionization, ID of the column 0.6 cm, nitrogen as carrier, poly(1,4-butanediolsuccinate), stationary support Chromaton N-AW-DMCS, Lachema. Brno). Preparative gas chromatography was conducted in a modified apparatus Chrom 2 (flame ionization, preparative columns I.D. 1 cm, length 500 cm). The infrared spectra were measured in a Perkin-Elmer apparatus 325 (Bodenseewerk). The NMR spectra were measured with an apparatus Varian XL-100-15 (Palo Alto, deuteriochloroform). The mass spectra were measured in an apparatus Gas Chromatograph — Mass Spectrometer LKB 9000 (Stockholm; one focus, energy of electrons 70 eV, chromatographic inlet through poly(ethyleneglycoladipate), helium as carrier). The photochemical reactions of the liquid components were conducted at 12–15°C in a stream of nitrogen in a photochemical reactor¹⁸ with an inner source of radiation (high-pressure mercury discharge tube RVK, 120 W, Tesla).

Chemicals Employed

2,3-Dichlorotrifluoropropionitrile was prepared as previously described^{10,11}. 2-Methyl-2-butene was prepared according to a reported procedure^{19–22} by dehydration^{21,22} of 2-methyl-2-butanol, obtained by reaction of ethylmagnesium bromide with acetone^{19,20}. The formed mixture of 2-methyl-2-butene and 2-methyl-1-butene (86% and 14% respectively) was resolved in a rotary rectification column (Perkin-Elmer, 251 Auto Annular Still).

3,4-Dichloro-3,4,4-trifluoro-2-butanone (*I*)

To a stirred solution of 2,3-dichlorotrifluoropropionitrile (50 g, 0.28 mol) in ether (100 ml) was added in the course of 2 h a solution of the Grignard agent in ether (350 ml) under a stream of nitrogen. The mixture was then stirred for 1 h under cooling, another hour at 20°C and decomposed by the addition of 20% sulphuric acid (300 ml). The ethereal layer was separated, the aqueous layer was washed with two 100 ml portions of ether. The ethereal extracts were washed with a solution of sodium sulphite and dried over anhydrous magnesium sulphate. The ethereal solution was distilled at 1.6 kPa (12 Torr); the distillate was collected in two receivers, cooled with Dry Ice. In the second distillation the product *I* was taken in the range 55–60°C/21 kPa (160 Torr). For yields see Table IV, purity 96–98% (gas chromatography: 15% poly(1,4-butanediolsuccinate) and 15% Carbowax 6000, column length 240 cm). The infrared spectrum (carbon disulphide, tetrachloromethane, cm⁻¹): 565 m, 803 s, 899 s, 975 m, 1025 m; 1063 s, 1120 s, 1145 s, 1168 s, 1204 s $\nu(\text{C}-\text{F})$; 1232 m, 1362 m, 1420 m, 1756 s $\nu(\text{C}=\text{O})$. For the ¹⁹F-NMR spectrum see Table III. For C₄H₃Cl₂F₃O (195.0) calculated: 24.62% C, 1.55% H, 36.40% Cl, 29.22% F; found: 25.57% C, 1.82% H, 36.30% Cl, 29.97% F.

Photolysis of 3,4-Dichloro-3,4,4-trifluoro-2-butanone (*I*)

A. In the gaseous phase. The reaction was allowed to proceed for 7 h at 110°C (reactor volume 4000 ml). During irradiation of vapour of the ketone *I* (2.0 g, 10.3 mmol) samples of the reaction mixture were taken for analysis at 1 h intervals (see *B*, 110°C). After the photolysis the gaseous mixture was condensed in a flask with Dry Ice and chromatographed. The structures of compounds *III*–*VIII* were derived from mass spectra (Table II).

B. In the liquid phase. A mixture of the halogenoketone *I* (1.31 g, 6.6 mmol) and benzene (28 g,

0.36 mol) was irradiated for 15 h. Gas chromatography (Carbowax 6000, column length 240 cm, 70°C) detected no change in composition of the reaction mixture during and after this time.

2-Phenyl-3,3,4-trimethyloxetane (*IXa*) and 4-Phenyl-2,2,3-trimethyloxetane (*IXb*)

A mixture of 2-methyl-2-butene (8.81 g, 0.126 mol) and benzaldehyde (22 g, 0.207 mol) was irradiated for 63 h. The mixture was then distilled over a column, the following fractions being taken: 120—125°C/3.2 kPa (24 Torr), 14.3 g, 76% of the oxetanes; 125—140°C/3.2 kPa, 0.52 g, 80% of the oxetanes, 68—73°C/10.7 Pa (0.08 Torr), 3.2 g, 15% of the oxetanes. The calculated overall yield of the oxetanes *IXa* and *IXb* was 11.8 g (42.8%). Redistillation of the first two fractions gave a mixture of the oxetanes of a 94% purity, b.p. 107—110°C/1.33 kPa (10 Torr) (gas chromatography as in the preceding reaction, 175°C). The two isomers were separated by preparative gas chromatography. The infrared spectrum is given in refs^{15,17}, the NMR spectrum in Table III. For C₁₂H₁₆O (176.6) calculated: 81.80% C, 9.15% H; found: 80.23% C, 8.86% H.

Reaction of Halogeno Ketone *I* with 2-Methyl-2-butene

A mixture of the ketone *I* (11 g, 56 mmol) and 2-methyl-2-butene (18 g, 0.26 mol) was irradiated for 24 h. The unreacted components were distilled off at 21 kPa (160 Torr) and the residue (5.01 g) was fractionated by preparative gas chromatography in an analytical column (10% "OV" phase, column length 240 cm). One of the fractions was a mixture of the isomeric cycloadducts *Xa* and *Xb* (purity 94%). The mass spectra of compounds *X—XII*, *XIV* and *XV* are given in Table II, the NMR spectra of the mixture *Xa* + *Xb* in Table III. For the mixture *Xa* + *Xb*, C₉H₁₃Cl₂F₃O (265.1), calculated: 40.77% C, 4.96% H, 26.75% Cl, 21.50% F; found: 41.03% C, 5.12% H, 25.98% Cl, 20.69% F.

The spectral measurements were performed at the departments of NMR spectra (head Dr P. Trška), mass spectra (head Dr V. Kubelka) and infrared spectroscopy (head Dr A. Muck); the elemental analysis were carried out at the department of organic analysis (head Dr L. Helešic), Institute of Chemical Technology, Prague. Their assistance is gratefully acknowledged.

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