°C (lit.[37a] mp 35–39 °C); ^{1}H NMR δ 0.78 (d, 3 H, J = 7.0 Hz), 0.94 (s, 3 H), 1.06 (s, 3 H), 1.15–1.95 (m, 14 H), 1.25 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 73.3 (s), 61.5 (d), 56.3 (d), 53.4 (s), 41.9 (s), 41.8 (d), 39.9 (t), 36.9 (t), 34.3 (t), 30.6 (q), 30.5 (t), 29.1 (q), 28.2 (q), 25.4 (t), 15.5 (q); IR ($CHCl_3$) 3600, 3450, 2940, 1455, 1375, 1305, 1085, 990, 965, 890, 855 cm^{-1}.

2α,8β-Dihydroxycedrane (2a) was isolated as a white, crystalline solid by flash chromatography on silica gel (1:1 AcOEt–pentane): mp 134 °C (lit.[23b] mp 135–137 °C, lit.[24b] mp 80–82 °C);[50] ^{1}H NMR ($CDCl_3$) δ 1.04 (s, 3 H), 1.18 (s, 3 H), 1.28 (s, 3 H), 1.30 (s, 3 H), 1.15–1.96 (m, 14 H), ^{13}C NMR ($CDCl_3$) δ 79.6 (s), 74.9 (s), 59.1 (d), 57.6 (s), 53.5 (d), 45.0 (s), 41.3 (t), 36.6 (t), 35.7 (t), 30.4 (q), 30.0 (t), 28.5 (q), 27.5 (q), 24.3 (q), 21.5 (t); IR ($CHCl_3$) 3600, 3460, 2950, 1705, 1455, 1375, 1295, 1170, 1125, 1090, 1010, 975, 945, 920 cm^{-1}.

2α-Hydroxy-8β-acetoxycedrane (3a) was isolated as a white, crystalline solid by flash chromatography on silica gel (1:1 ether–pentane): $[\alpha]^{22}_D$ +20.6° (c 7.29, $CHCl_3$); mp 58 °C (lit.[23a] mp 57–58 °C, lit.[24b] mp 59–60 °C); ^{1}H NMR ($CDCl_3$) δ 1.0 (s, 3 H), 1.04 (d, 3 H, J = 7.1 Hz), 1.17 (s, 6 H), 1.10–2.16 (m, 11 H), 2.05 (s, 3 H), 5.0 (1 H, ddd, J = 11, 11, 6.6 Hz); ^{13}C NMR ($CDCl_3$) δ 170.4 (s), 85.9 (s), 79.4 (s), 57.4 (s), 54.9 (d), 53.8 (d), 44.9 (s), 41.3 (t), 35.7 (t), 33.6 (t), 29.6 (t), 28.0 (q), 26.7 (q), 25.9 (q), 24.2 (q), 22.7 (q), 21.5 (t); IR ($CHCl_3$) 3600, 3460, 2950, 1715, 1470, 1450, 1370, 1245, 1025, 1015, 975, 965 cm^{-1}.

2α-Hydroxy-8α-H-9α-acetoxycedrane (4a) was isolated as a clear, colorless oil by flash chromatography on silica gel (1:1 ether–pentane): $[\alpha]^{22}_D$ +46.5° (c 9.87, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.0 (s, 3 H), 1.05 (d, 3 H, J = 7.1 Hz), 1.1–2.2 (m, 12 H), 1.16 (s, 6 H), 2.05 (s, 3 H), 5.0 (ddd, 1 H, J = 11, 11, 6.6 Hz); ^{13}C NMR ($CDCl_3$) δ 170.8 (s), 79.6 (s), 75.4 (d), 57.9 (s), 54.8 (d), 53.1 (d), 45.1 (s), 42.4 (d), 41.1 (t), 40.6 (t), 37.8 (t), 28.1 (q), 27.6 (q), 24.1 (q), 21.7 (t), 21.1 (q), 17.3 (q); IR ($CHCl_3$) 3595, 3480, 2950, 1720, 1460, 1440, 1370, 1245, 1030, 1015, 980, 970 cm^{-1}. Anal. Calcd for $C_{17}H_{28}O_3$: C, 72.81; H, 10.06. Found: C, 72.15; H, 10.54.

2α-Hydroxy-8α-H-9α-[(methoxycarbonyl)oxy]cedrane (5a) was isolated as a clear, colorless oil by flash chromatography on silica gel (1:1 AcOEt–pentane): $[\alpha]^{22}_D$ +49.9° (c 7.25, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.00 (s, 3 H), 1.10 (d, 3 H, J = 7.1 Hz), 1.17 (s, 6 H), 1.22–2.15 (m, 12 H), 3.77 (s, 3 H), 4.84 (ddd, 1 H, J = 6.5, 10.7, 10.7 Hz); ^{13}C NMR ($CDCl_3$) δ 155.8 (s), 80.1 (d), 79.7 (s), 58.1 (s), 55.0 (q), 54.5 (d), 53.2 (d), 45.3 (s), 42.5 (d), 41.2 (t), 40.7 (t), 37.9 (t), 28.3 (q), 27.7 (q), 24.3 (q), 21.9 (t), 17.4 (q); IR ($CHCl_3$) 3595, 3460, 2950, 2900, 1730, 1440, 1370, 1360, 1315, 1270, 1140, 945 cm^{-1}. Anal. Calcd for $C_{17}H_{28}O_4$: C, 68.88; H, 9.52. Found: C, 68.80; H, 9.55.

5β,9β-Oxy-8α-hydroxycedrane (6a) was isolated as a white, crystalline solid by flash chromatography on silica gel (3:7 ether–pentane): $[\alpha]^{22}_D$ −5.1° (c 5.83, $CHCl_3$); mp 101 °C; ^{1}H NMR ($CDCl_3$) δ 3.94 (1 H, d, J = 4.1 Hz), 1.43 (s, 3 H), 1.23 (s, 3 H), 1.0–2.0 (m, 11 H), 0.98 (s, 3 H), 0.92 (d, 3 H, J = 5.7 Hz); ^{13}C NMR ($CDCl_3$) δ 101.3 (s), 84.7 (d), 72.4 (s), 62.5 (d), 61.1 (s), 44.3 (s), 41.6 (d), 38.7 (t), 37.5 (t), 36.1 (t), 29.3 (q); 28.2 (q), 26.6 (t), 23.6 (q), 14.8 (q); IR ($CHCl_3$) 3590, 3430, 2935, 2855, 1475, 1445, 1365, 1225, 1160, 1115, 1085, 1050, 1005, 985, 965, 940, 915, 895 cm^{-1}. Anal. Calcd for $C_{15}H_{24}O_2$: C, 76.22; H, 10.23. Found: C, 76.25; H, 10.19.

Keto lactone 6b was isolated as a white, crystalline solid by flash chromatography on silica gel (1:1 ether–pentane): $[\alpha]^{22}_D$ −55.5° (c 8.38, $CHCl_3$); mp 87 °C; ^{1}H NMR ($CDCl_3$) δ 2.80 (m, 1 H), 2.74 (1 H, d, J = 19.3 Hz), 2.33 (1 H, d, J = 19.3 Hz), 2.17 (s, 3 H), 1.05–2.07 (m, 7 H), 1.28 (s, 3 H), 1.02 (d, 3 H, J = 6.6 Hz), 0.83 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 207.6 (s), 177.7 (s), 107.0 (s), 57.2 (d), 54.1 (s), 46.4 (s), 46.1 (d), 39.4 (t), 37.7 (t), 34.6 (t), 31.8 (t), 31.6 (q), 23.5 (q), 18.5 (q), 14.1 (q); IR ($CHCl_3$) 2960, 2860, 1760, 1705, 1415, 1360, 1215, 995 cm^{-1}. Anal. Calcd for $C_{15}H_{22}O_3$: C, 71.96; H, 8.86. Found: C, 71.93; H, 8.78.

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Registry No. 1, 13567-54-9; **1a**, 19903-73-2; **2**, 77-53-2; **2a**, 67152-03-8; **3**, 77-54-3; **3a**, 65669-75-2; **4**, 19903-81-2; **4a**, 127558-82-1; **5**, 127558-83-2; **5a**, 127558-84-3; **6**, 61234-88-6; **6a**, 142745-21-9; **6b**, 142745-22-0; **7**, 13567-39-0; **8**, 13567-40-3; **9**, 142864-21-9; **10**, 469-61-4; R_4O_4, 20427-56-9.

(50) The melting point reported in ref 24b for 2α,2β-dihydroxycedrane is apparently erroneous.

Tautomerization of 1-(*N*-Methoxyamino)cyclohepta-1,3,5-triene into 3,5-Cycloheptadien-1-one *O*-Methyloxime: A Regiospecific Enamine–Imine Interconversion

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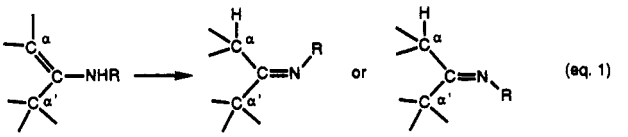
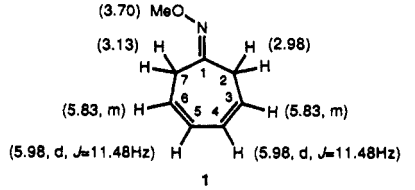
Although there have been many investigations of stereospecificity and regiospecificity in the reactions of enamines,[1] there appears to have been no investigation of the steric course of an enamine–imine interconversion (e.g., eq 1), presumably because imines are normally hydrolyzed much faster than they are formed from the enamines.[2] As shown in eq 1, the R group attached to the nitrogen of the imine product can lie either on the same or the opposite side of the double bond of the original enamine (syn or anti to the group which was protonated).



(eq. 1)

The signals in the ^{1}H NMR spectrum of 3,5-cycloheptadien-1-one *O*-methyloxime in DMSO-d_6 were assigned as shown in **1**. The protons at positions 2 and 7 had different chemical shifts as a result of hindered rotation around the C=N bond, but those at positions 3 and 6 and at positions 4 and 5 were indistinguishable from one another in the 270-MHz spectrum. A nuclear Overhauser effect (NOE) experiment indicated that the low-field methylene group signal at δ 3.13 was that of the protons syn to the methoxyl group whereas the high-field signal at δ 2.98 was due to the protons anti to the methoxyl group. Irradiation of the low-field signal (δ 3.13) gave a 10.4% enhancement of the signal of the methoxyl group (δ 3.70), but irradiation of the high-field signal (δ 2.98) gave no enhancement.



1

The N-deuterated enamine form (**3**) of this *O*-methyloxime was generated by hydrolysis of its *N*-trimethylsilyl derivative **2** in DMSO-d_6–D_2O (95:5 v/v) at 25 °C which

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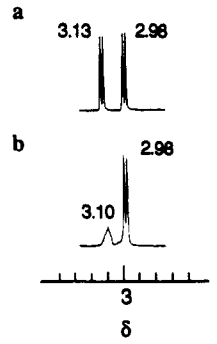
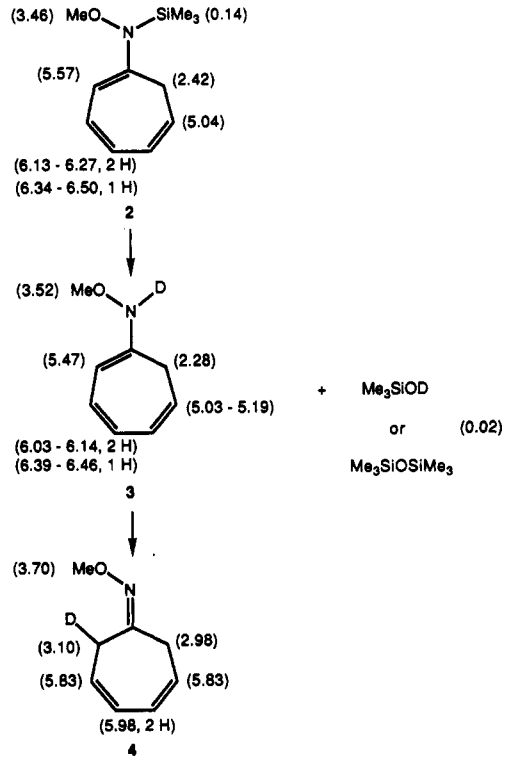


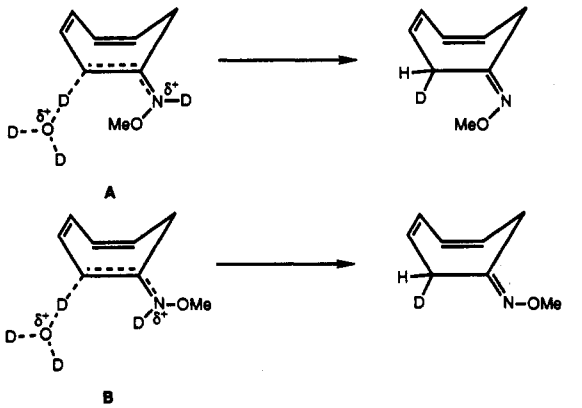
Figure 1. 270-MHz ^{1}H NMR spectra of 3,5-cycloheptadien-1-one *O*-methyloxime at the region of 3 ppm. (a) Pure authentic sample in DMSO-d_6. (b) Sample from enamine–imine conversion of 1-(*N*-^{2}H-*N*-methoxyamino)cyclohepta-1,3,5-triene in DMSO-d_6–D_2O (95:5 v/v) containing 2×10^{-4} M DCl.

Scheme I



contained DCl (2×10^{-4} M). The changes in chemical shift that occurred in the ^{1}H NMR spectra are shown in Scheme I. Hydrolysis of the trimethylsilyl group was indicated by a decrease in the signal at δ 0.14 and the formation of trimethylsilanol-*d* or hexamethyldisiloxane (δ 0.02).[3,4] This was accompanied, not by the formation of the oxime, but of another species with a ^{1}H NMR spectrum similar to that of the precursor in which most of the signals of the ring protons were shifted slightly upfield. This is similar to what was found previously on generation of cyclohexenyl amines,[2] and the new species was considered to be the secondary enamine **3**. This appears to be the first time that the enamine of an *O*-methyloxime has been detected, although recently the enamine forms of some heterocyclic oximes have been generated.[5] After 30 min, the signals of the precursor had disappeared and the solution contained 70% enamine **3** and 30% oxime **4**. After 10 h, all the enamine had been converted to oxime, but although the signal of the methylene protons anti to the methoxyl group of the oxime (δ 2.98) was a sharp doublet with an intensity equivalent to two protons, that of the methylene protons syn to the methoxyl group was a broad multiplet (δ 3.10) with an intensity equivalent to one proton (see Figure 1). It was concluded that the oxime was **4** with the deuterated methylene group syn to the methoxyl group formed to the extent of at least 99%.

There are two extreme structures possible for the transition state for protonation (or deuteration) of the double bond of the enamine: one with the methoxyl group syn to the double bond (**A**) and one with it anti to the methylene group (**B**). The experimental results indicate that



A is more stable by at least 2.7 kcal mol^{-1}. Possibly, this arises from an unfavorable steric interaction between the methoxyl group and ring methylene group which destabilizes transition state **B**. The alternative transition state **A** may be stabilized by a stereoelectronic effect similar to the one which causes the s-cis conformation of vinyl alcohol to be more stable than the s-trans.[6]

High syn preference has been well established and explained for the formation of and stereoselective alkylation of carbanions of N-derivatives of carbonyl compounds such as oximes and oxime ethers.[7] Our study suggests that the electronic structure of the enamine in DMSO may very well resemble that of its conjugate base, which of course is the carbanion formed by C_α-deprotonation of the oxime ether.

Experimental Section

General. A FT-IR spectrum was recorded with a Nicolet 20sxc FT spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded in deuterated solvents on a JEOL FX-90Q (90-MHz) or a JEOL GSX-270 (270-MHz) FT-NMR spectrometer. Chemical shifts were measured downfield from internal tetramethylsilane and were quoted in δ. An ordinary mass spectrum was run on a Hitachi RMS-4 spectrometer. Accurate mass was determined on a VG 7070F Micro-Mass spectrometer. Elemental analysis was performed at the Butterworth Laboratories Ltd., UK.

Materials. Triethylamine was dried over sodium hydroxide for at least 3 days before use. Dichloromethane was distilled from calcium hydride. *n*-Hexane was dried over sodium wire. Other commercial chemicals were used without further purification.

3,5-Cycloheptadien-1-one *O*-Methyloxime (1). To a stirring solution of 3,5-cycloheptadienone[8] (1 g, 9.3 mmol) and sodium acetate (0.9 g, 11 mmol) in methanol–H_2O (1:1 v/v, 20 mL) was added a solution of *N*-methoxyamino hydrochloride (0.9 g, 11 mmol) in the same solvent (20 mL). The mixture was stirred for 30 min. Water (40 mL) was added, and the resultant mixture

(1) Hickmott, P. W. *Tetrahedron* **1982**, *38*, 1975; **1982**, *38*, 3363; **1984**, *40*, 2989.
(2) Capon, B.; Wu, Z.-P. *J. Org. Chem.* **1990**, *55*, 2317.
(3) See Fleming, I. In *Comprehensive Organic Chemistry*; Barton, D. H. R., Ollis, W. D., Eds.; Pergaman Press: Oxford, 1979; Vol. 3, p 576.
(4) Capon, B.; Kwok, F.-C. *J. Am. Chem. Soc.* **1989**, *111*, 5346.
(5) Huang, Y. Ph.D. Thesis, University of Hong Kong, 1991.
(6) Capon, B.; Siddhanta, A. K. *J. Org. Chem.* **1984**, *49*, 255.
(7) Glaser, R.; Streitwieser, A., Jr. *J. Am. Chem. Soc.* **1989**, *111*, 7340 and 8799 and references cited therein.
(8) Garbisch, E. W., Jr. *J. Org. Chem.* **1965**, *30*, 2109.

was extracted with diethyl ether (3 × 30 mL). The combined organic layer was dried ($MgSO_4$) and concentrated. The residue was purified by column chromatography on silica gel (petroleum spirit:diethyl ether = 3:1) to give 1.1 g of pure product (86% yield). ^1H NMR (90 MHz, $CDCl_3$): δ 3.03 (d, 2 H, J = 5.25 Hz, CH_2), 3.2 (d, 2 H, J = 4.16 Hz, CH_2), 3.8 (s, 3 H, OMe), 5.78–5.99 (m, 4 H, olefinic protons). ^{13}C NMR (22.5 MHz, $CDCl_3$): δ 29.44 (t), 35.27 (t), 61.19 (q, OMe), 127.39 (d), 127.52 (d), 127.77 (d), 127.96 (d), 164.41 (s, C-7). IR (neat, cm^{-1}): 3024, 2937, 1627, 1424, 1052. HRMS for $C_8H_{11}NO$: calcd 137.0885, found 137.0821. Anal. Calcd for $C_8H_{11}NO$: C, 70.04; H, 8.08; N, 10.21. Found: C, 70.60; H, 8.13; N, 9.88.

1-(*N*-Methoxy-*N*-(trimethylsilyl)amino)cyclohepta-1,3,5-triene (2). This compound is highly sensitive to moisture and was prepared and worked up by the method described below.

Trimethylsilyl trifluoromethanesulfonate (1.22 g, 5.5 mmol) was added cautiously with a syringe to a stirring solution of the *O*-methyloxime 1 (0.69 g, 5 mmol) and triethylamine (0.61 g, 6 mmol) in dried *n*-hexane (15 mL) under dry nitrogen atmosphere. Stirring was continued until two immiscible layers were formed. The lower layer was darkish brown in color. About 500 μL of the upper clear layer was withdrawn from the reaction flask and transferred immediately to a NMR tube dried in a desiccator over P_2O_5 for at least 1 day. The NMR tube was transferred quickly to a vacuum trap, and the organic solvent was removed at reduced pressure with great care to prevent the solution from shooting out of the NMR tube. After all the organic solvent was removed, dry air was admitted to the vacuum trap through an anhydrous $CaCl_2$ guard tube. Deuterated solvent (DMSO-d_6) was added, and the spectra were recorded immediately. MS (m/e): 225 (M^+).

Registry No. 1, 142868-78-8; 2, 142868-79-9; 3, 142868-80-2; 4, 142868-81-3; $MeONH_2$·HCl, 593-56-6; 3,5-cycloheptadien-1-one, 1121-65-9.

Anti-Michael Addition and Fluoride Ion Elimination on β,β-Bis(trifluoromethyl)acrylic Esters. Preparation of α-Perfluoroisopropenyl α-Substituted Acetic Acid Esters[1]

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In the course of our studies on fluorinated analogues of prenyl derivatives[2] the allylic alcohol **2** was needed. This compound was already described in a patent[3] and by Taguchi.[4] Both reported methods referred to a metal hydride reduction of the readily prepared ethyl β,β-bis(trifluoromethyl)acrylic ester (**1**).[5] In our hands, all attempts to repeat these described procedures failed. With the recommended diisobutylaluminum hydride[6] (DIBALH) in toluene at −70 °C we obtained the perfluoroisopropenyl compound **4a** resulting from an anti-Michael addition[7] of hydride ion followed by a fluoride ion elimination. Two other unidentified fluorinated products were also formed. The inverted polarization of the double bond in β,β-bis(trifluoromethyl)acrylic esters has already been observed by Knunyants in the addition of amines which apparently was not followed by fluoride ion elimination.[8] This unexpected addition–elimination reaction was extended to various nucleophiles, and we report here a convenient route to the new α-perfluoroisopropenyl α-substituted acetic acid esters **4**.[9]

Compound **4a** was obtained as a single product when using the less reactive lithium triethoxyaluminum hydride instead of DIBALH for the reduction. The reaction was conducted in ether at −78 °C, and **4a** was isolated in 77% yield. The sodium salt of diethyl malonate in tetrahydrofuran at 10 °C gave **4b** in 70% yield. Under the same conditions, the sulfide **4c** was obtained with 67% yield from the sodium phenyl sulfide. Organolithium reagents reacted in this fashion as well. For example, methyllithium in tetrahydrofuran at −78 °C gave **4d** in 54% yield. In all cases, the hexafluoro compounds **5a–d** were formed in less than 5% as estimated from the ^{19}F NMR spectra of the crude reaction product, but could not be isolated.[10] With piperidine, only the saturated compound **5e** was isolated as previously reported by Knunyants.[8] However careful monitoring during the course of the reaction by ^{19}F NMR showed the formation of **4e** as major product at the beginning of the condensation. We noticed that increasing the reaction time led only to **5e** which could be considered as the thermodynamic product. All attempts to isolate **4e** failed.

These observations allow us to postulate the formation of the carbanionic intermediate **3** which, in the absence of a proton source, will eliminate fluoride ion to form **4**. In the case of the amine, a piperidinium hydrofluoride salt was formed and the fluoride ion became nucleophilic enough to add to the fluorinated double bond[11] of the kinetic product **4e** to give **5e**. The analogous tetrabutylammonium fluoride addition to **4** leading to the bistrifluoromethyl compounds **5**, which was followed by ^{19}F NMR, was in agreement with this mechanism.

This anti-Michael addition requires two trihalogenomethyl electron-withdrawing groups as Walborsky[12] showed that γ,γ,γ-trifluorocrotonate reacted in the same manner as its nonfluorinated analog. In the competition between a single trifluoromethyl group and carboxyl group, the latter one determined the regioselectivity of the addition.

A similar anti-Michael addition of nucleophiles followed by halide ion elimination has been observed with hindered γ-bromo α,β-unsaturated esters.[13] An allylic displacement reaction was also observed when treating highly halogenated olefins with lithium aluminum hydride since terminal perfluoromethylene olefins were formed.[14] Recently, α-perfluoroisopropenyl ketones have been obtained from

(1) Presented in part at the XIIIth International Symposium on Fluorine Chemistry, Bochum (FRG), September 1991.

(2) 1-Bromo-3-(trifluoromethyl)but-2-ene: synthesis and electrophilic reactivity. Martin, V.; Molines, H.; Wakselman, C. *J. Fluorine. Chem.*, in press.

(3) Noyori, R.; Morisawa, Y.; Maeda, T.; Yasuda, A.; Uchida, K. JP 02 56,462 [90 56,462]; *Chem. Abstr.* **1990**, *113*, 59620g.

(4) Taguchi, T.; Hosada, A.; Torisawa, Y.; Shimazaki, A.; Kobayashi, Y.; Tsushima, K. *Chem. Pharm. Bull.* **1985**, *33*, 4085.

(5) Abele, H.; Haas, A.; Lieb, M. *Chem. Ber.* **1986**, *119*, 3502.

(6) We thank Professor Takeo Taguchi for the personnal communication giving us a detailed experimental procedure.

(7) For anti-Michael additions see; Klumpp, G. W.; Mierop, A. J. C.; Vrielink, J. J.; Brugman, A.; Schakel, M. *J. Am. Chem. Soc.* **1985**, *107*, 6740 and references cited therein.

(8) Knunyants, I. L.; Cheburkov, Yu. A. *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1960**, 2162.

(9) Only the acid of **4a** has yet been obtained as a mixture with three other fluorinated compounds, by treatment of perfluoro-2,2-dimethylbutadiene with water. Kaz'mina, N. B.; Krasnikova, G. S.; Lur'e, E. P.; Mysov, E. I.; Knunyants, I. L. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1975**, 2525.

(10) Compound **5a** was isolated by preparative GC (90 °C): ^1H NMR (200 MHz) 1.35 (t, 3 H, $^3J_{HH}$ = 7.1 Hz), 2.8 (d, 2 H, $^3J_{HH}$ = 6.04 Hz), 3.78 (septuplet × d, 1 H, $^3J_{HH}$ = 6.04 Hz, $^3J_{HF}$ = 8.5 Hz), 4.25 (q, 2 H, $^3J_{HH}$ = 7.1 Hz); ^{19}F NMR −68.63 (d, $^3J_{HF}$ = 8.5 Hz).

(11) Molines, H.; Wakselman, C. *J. Fluorine Chem.* **1984**, *25*, 447.

(12) Walborsky, H. M.; Schwarz, M. *J. Am. Chem. Soc.* **1953**, *75*, 3241.

(13) Roux-Schmitt, M. C.; Sevin, A.; Seyden-Penne, J. *Bull. Soc. Chim. Fr.* **1990**, *127*, 857.

(14) Kaufman, M. H.; Braun, J. D.; Shdo, J. G. *J. Org. Chem.* **1967**, *32*, 2749.

0022-3263/92/1957-5530$03.00/0