Alkoxycarbonylimines of hexafluoroacetone in reaction of [2+4]-cycloaddition

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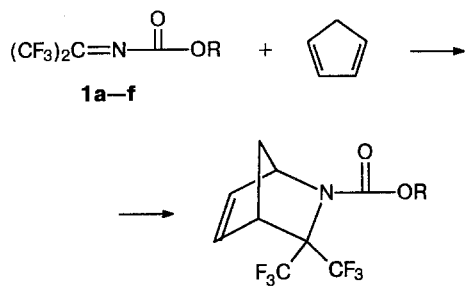
The [2+4]-cycloadditions of alkoxycarbonylimines of hexafluoroacetone to cyclopentadiene and *N*-nitriles have been studied. Alkoxycarbonylimines of hexafluoroacetone may be considered as both dienophiles and 1,3-heterodienes.

Key words: alkoxycarbonylimines of hexafluoroacetone, cyclopentadiene, reaction of [2+4]-cycloaddition; 1,3-heterodienes; dienophiles; cyanoguanidines, cyanamides.

We have elaborated earlier a convenient preparative method for the synthesis of alkoxycarbonylimines of hexafluoroacetone (AH) (**1**) and demonstrated that they react with nucleophilic agents[1–3] like acylimines of hexafluoroacetone[4,5] with the exception of the interaction between compound **1b** and trimethylphosphite.[3] Reactions of [2+4]-cycloaddition of AH with cyclopentadiene, cyanguanidines, and cyanamides have been studied in the present work.

It was established that AHs **1a—e** react with cyclopentadiene to form adducts of [2+4]-cycloaddition, acting like dienophiles (Scheme 1).

Scheme 1



1a—f

2a—f

R = Me (**a**), Et (**b**), Pr^n (**c**), Pr^i (**d**), i-C_5H_{11} (**e**), $PhCH_2$ (**f**)

The reaction products, 2-alkoxycarbonyl-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-enes (**2a—f**), are characterized by ^1H and ^{19}F NMR spectra (Table 1) that indicate that they represent mixtures of two isomers in ratios from 1 : 1 to 1 : 2.3. For the isomers the difference in chemical shifts of a proton at the C(1) atom is $\Delta\delta$ = 0.04 to 0.10 (**2a—c**) and at the C(6) atom is $\Delta\delta$ = 0.10 to 0.14 (**2a—f**). Two singlets of a MeO group are observed in the ^1H NMR spectrum of compound **2a**, two signals of methyl protons of alkoxy groups are observed in the case of compounds **2b,c**, and methylene protons of a benzyloxy group appear as a singlet for one isomer and a multiplet for the other in the case of compound **2f**. The difference in chemical shifts of the CF_3 moiety is $\Delta\delta$ 0.8 in the ^{19}F NMR spectrum of isomers **2c**. The structure of products of the dihydroxazine type[6,7] is excluded because the chemical shifts of protons and F atoms in cycloadducts **2a—f** coincide almost exactly with the chemical shifts described for 2-trifluoroacetyl-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-ene.[8] Moreover, ^1H and ^{19}F NMR spectra of a mixture of the isomers have a typical temperature dependence. In the case of compound **2c**, for example, coalescence of the signals of the isomers takes place at 95 °C. After cooling of the sample the spectra is completely restored to the initial one. Hence, mutual transformation of the isomers takes place, which is accelerated when temperature increases. Evidently, this is due to slower amide rotation as in the case of 2-acyl-3-ethoxycarbonyl-2-azabicyclo[2.2.1]hept-5-enes,[9] which are related to adducts **2a—f**.

There are bands in the IR spectra of all the compounds obtained that indicate the presence of a C=C bond and a carbonyl group (ν/cm^{-1}: 3300 (HC=); 1670—1725 (C=O)).

The compositions of the compounds synthesized are confirmed by elemental analysis data (Table 2).

Properties of AH as 1,3-heterodienes were studied by us in the reactions with cyanoguanidines and cyanamides. As mentioned earlier,[10] the presence of an activated C=N bond and 1,3-heterodiene fragment >C=N—C=O in *N*-acylimines of hexafluoroacetone allows one to suppose the possibility of competitive directions of the reactions with cyanoguanidines. It was found, that when equimolar quantities of cyanoguanidines **3** and imine **1** are mixed in ether, [2+4]-cycloaddition is realized exclusively (Scheme 2).

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Table 1. ^1H and ^{19}F NMR spectra of compounds **2a—f**

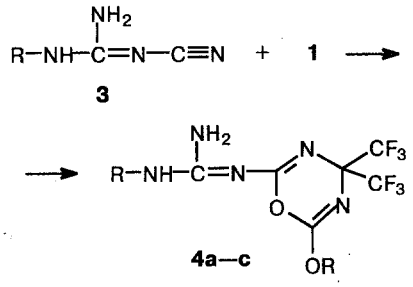
Compound	δ^1H (*J*/Hz)	$\delta^{19}F$ (*J*/Hz)
2a	1.47 (d, 1 H, C(7)H_2, J = 9.2); 2.14 (d, 1 H, C(7)H_2, J = 9.2); 3.60 (br.s, 1 H, C(4)H); 3.62 (3.68)* (s, 3 H, OMe); 4.92 (4.82) (br.s, 1 H, C(1)H); 6.33 (br.s, 1 H, C(5)H); 6.73 (6.61) (d, 1 H, C(6)H)	8.4 (m, 3 F, CF_3); 16.9 (17.9)* (m, 3 F, CF_3) [1.00 : 1.00]**
2b	1.16 (1.20) (t, 3 H, OCH_2Me); 1.48 (d, 1 H, C(7)H_2, J = 9.0); 2.13 (d, 1 H, C(7)H_2, J = 9.0); 3.60 (br.s, 1 H, C(4)H); 4.07 (q, 2 H, OCH_2); 4.88 (4.78) (s, 1 H, C(1)H); 6.31 (br.s, 1 H, C(5)H); 6.71 (6.62) (br.s, 1 H, C(6)H)	8.05 (m, 3 F, CF_3); 16.94 (17.67) (q, 3 F, CF_3, $J_{F,F}$ = 9.0) [1.46 : 1.00]
2c	0.92 (0.96) (t, 3 H, CH_2Me); 1.54 (d, 1 H, C(7)H_2, J = 9.4); 1.67 (m, 2 H, CH_2Me); 2.23 (d, 1 H, C(7)H_2, J = 9.4); 3.66 (br.s, 1 H, C(4)H); 4.05 (m, 2 H, OCH_2); 5.03 (4.90) (s, 1 H, C(1)H); 6.39 (br.s, 1 H, C(5)H); 6.78 (6.66) (d, 1 H, C(6)H, $J_{H(5),H(6)}$ = 3.8)	7.32 (q, 3 F, CF_3); 16.95 (17.75) (q, 3 F, CF_3, $J_{F,F}$ = 10.0) [1.35 : 1.00]
2c* **	0.94 (t, 3 H, CH_2Me); 1.42 (d, 1 H, C(7)H_2, J = 9.4); 1.66 (m, 2 H, CH_2Me); 2.20 (d, 1 H, C(7)H_2, J = 9.4); 3.65 (br.s, 1 H, C(4)H); 4.04 (m, 2 H, OCH_2); 4.94 (br.s, 1 H, C(1)H); 6.36 (br.s, 1 H, C(5)H); 6.70 (br.s, 1 H, C(6)H)	8.57 (br.s, 3 F, CF_3); 17.31 (br.s, 3 F, CF_3)
2d	1.14 (1.11) (d, 6 H, $OCHMe_2$); 1.43 (d, 1 H, C(7)H_2, J = 9.2); 2.13 (d, 1 H, C(7)H_2, J = 9.2); 3.55 (br.s, 1 H, C(4)H); 4.86 (m, 1 H, OCH); 4.92 (s, 1 H, C(1)H); 6.32 (br.s, 1 H, C(5)H); 6.75 (6.62) (br.s, 1 H, C(6)H)	8.43 (8.11) (q, 3 F, CF_3); 17.47 (17.92) (q, 3 F, CF_3, $J_{F,F}$ = 9.2) [2.29 : 1.00]
2e	0.93 (d, 6 H, $CHMe_2$); ~1.51 (m, 2 H, CH_2CHMe_2); 1.56 (d, 1 H, C(7)H_2, J = 10.0); 1.70 (m, 1 H, $CHMe_2$); 2.19 (d, 1 H, C(7)H_2, J = 10.0); 3.65 (3.63) (m, 1 H, C(4)H); 4.16 (m, 2 H, OCH_2); 4.94 (4.89) (br.s, 1 H, C(1)H); 6.35 (br.s, 1 H, C(5)H); 6.75 (6.65) (br.s, 1 H, C(6)H)	8.41 (q, 3F, CF_3) 17.25 (18.20) (q, 3 F, CF_3, $J_{F,F}$ = 9.6) [1.45 : 1.00]
2f	1.48 (d, 1 H, C(7)H_2, J = 10.0); 2.12 (2.15) (d, 1 H, C(7)H_2, J = 10.0); 3.60 (s, 1 H, C(1)H); 4.96 (4.90) (br.s, C(4)H); 5.13 (s + m, 2 H, OCH_2Ph); 6.31 (br.s, 1 H, C(5)H); 6.72 (6.58) (br.s, 1 H, C(6)H); 7.29 (br.s, 5 H, Ph)	8.13 (m, 3 F, CF_3); 17.13 (17.94) (q, 3 F, CF_3, $J_{F,F}$ = 9.0) [1.00 : 1.70]

* The δ values for the other isomer are given in parentheses (if their chemical shifts differ). ** The ratio of the isomers is given in brackets. *** The spectrum of the compound was obtained at 95 °C.

Table 2. Yields, physical characteristics, and elemental analysis data of compounds **2a—f**

Compound	Yield (%)	B.p./°C (*p*/Torr) [M.p./°C]	n_D^{20}	Found / Calculated (%) C	H	N	Empirical formula
2a	70.0	[58—60]	—	41.61 / 41.53	3.08 / 3.14	4.78 / 4.84	$C_{10}H_9F_6NO_2$
2b	68.0	97 (11)	1.4150	43.62 / 43.57	3.58 / 3.66	4.60 / 4.62	$C_{11}H_{11}F_6NO_2$
2c	63.8	126 (12)	1.4167	45.40 / 45.43	4.15 / 4.13	4.40 / 4.42	$C_{12}H_{13}F_6NO_2$
2d	53.1	119 (11)	1.4090	45.41 / 45.43	4.10 / 4.13	4.39 / 4.42	$C_{12}H_{13}F_6NO_2$
2e	41.1	86 (10)	1.4166	48.81 / 48.70	5.03 / 4.96	4.10 / 4.06	$C_{14}H_{17}F_6NO_2$
2f	64.3	137 (0.1)	1.4758	52.72 / 52.61	3.49 / 3.59	3.79 / 3.84	$C_{16}H_{13}F_6NO_2$

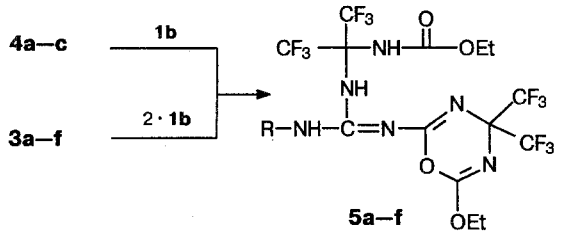
Scheme 2



The NMR spectra of the 1,3,5-oxadiazines **4** obtained were described earlier.[10]

Addition of a guanidine amino group of adducts **4** to an activated C=N bond of an AH occurs during the reaction of adducts **4a—c** with the AH as well as on treatment of cyanoguanidines **3a—f** with two moles of AH **1b** (Scheme 3).

Scheme 3



R = Bu^t (**a**), $(MeO)_2P(O)—CMe_2$ (**b**), $(EtO)_2P(O)—CMe_2$ (**c**), Ph (**d**), 2-MeC_6H_4 (**e**), 4-MeC_6H_4 (**f**)

Table 3. ^1H and ^{19}F NMR spectra of compounds **5a—f**

Compound	δ^1H (J/Hz)	$\delta^{19}F$
5a	1.27 (t, 3 H, OCH_2Me); 1.48 (t + s, 12 H, OCH_2Me + Bu^t); 4.20 (q, 2 H, OCH_2); 4.42 (q, 2 H, OCH_2); 5.88 (br.s, 1 H, NH); 8.42 (br.s, 1 H, NH); 11.44 (m, 1 H, NH)	−3.50 (s, 6 F, 2 CF_3); 1.80 (s, 6 F, 2 CF_3)
5b*	1.20 (t, 3 H, OCH_2Me); 1.35 (t, 3 H, OCH_2Me); 1.56 (d, 6 H, 2 Me, $J_{P,H}$ = 14.8); 3.84 (d, 6 H, 2 OMe, $J_{P,H}$ = 10.0); 4.11 (q, 2 H, OCH_2); 4.38 (q, 2 H, OCH_2); 7.12 (s, 1 H, NH); 9.15 (s, 1 H, NH); 10.38 (d, 1 H, NH—C—P, $J_{P,H}$ = 8.8)	−3.11 (s, 6 F, 2 CF_3); 3.57 (s, 6 F, 2 CF_3)
5c**	1.20 (t, 3 H, OCH_2Me); 1.37 (m, 9 H, 3 OCH_2Me); 1.60 (d, 6 H, 2 Me, $J_{P,H}$ = 14.8); 4.09 (q, 2 H, OCH_2); 4.20 (q, 2 H, OCH_2); 4.31 (m, 4 H, 2 P—OCH_2); 6.78 (s, 1 H, NH); 9.44 (s, 1 H, NH); 9.97 (s, 1 H, NH—C—P, J = 8.0)	−3.15 (s, 6 F, 2 CF_3); 3.52 (s, 6 F, 2 CF_3)
5d	1.23 (t, 3 H, OCH_2Me); 1.36 (t, 3 H, OCH_2Me); 4.13 (q, 2 H, OCH_2); 4.37 (q, 2 H, OCH_2); 5.66 (br.s, 1 H, NH); ~7.38 (m, 6 H, Ph + NH); 11.02 (br.s, 1 H, NH)	−2.97 (s, 6 F, 2 CF_3); 3.11 (s, 6 F, 2 CF_3)
5e	1.27 (t, 3 H, OCH_2Me); 1.38 (t, 3 H, OCH_2Me); 2.26 (s, 3 H, MeC_6H_4); 4.14 (q, 2 H, OCH_2); 4.37 (q, 2 H, OCH_2); 5.60 (s, 1 H, NH); 7.33 (m, 4 H, C_6H_4); 7.55 (s, 1 H, NH); 10.80 (br.s, 1 H, NH)	−2.89 (s, 6 F, 2 CF_3); 3.03 (s, 6 F, 2 CF_3)
5f	1.27 (t, 3 H, OCH_2Me); 1.40 (t, 3 H, OCH_2Me); 2.38 (s, 3 H, MeC_6H_4); 4.15 (q, 2 H, OCH_2); 4.37 (q, 2 H, OCH_2); 5.63 (br.s, 1 H, NH); 7.24 (m, 4 H, C_6H_4); 7.60 (br.s, 1 H, NH); 11.47 (br.s, 1 H, NH)	−2.97 (s, 6 F, 2 CF_3); 3.08 (s, 6 F, 2 CF_3)

* δP (**5b**) 30.10. ** δP (**5c**) 28.37.

Table 4. Yields, physical characteristics, and elemental analysis data of compounds **5a—f**

Compound	Yield (%)	M.p../°C	Found / Calculated (%)				Empirical formula
			C	H	N	P	
5a	88.8	117—120	35.39 35.19	3.83 3.61	13.76 13.68		$C_{18}H_{22}F_{12}N_6O_4$
5b	59.4	100—103	32.07 32.21	3.59 3.56	11.76 11.86	4.30 4.37	$C_{19}H_{25}F_{12}N_6O_7P$
5c	61.8	119—121	34.17 34.25	4.02 3.97	11.51 11.41	4.11 4.20	$C_{21}H_{29}F_{12}N_6O_7P$
5d	84.2	125—127	37.72 37.87	2.74 2.86	13.12 13.25		$C_{20}H_{18}F_{12}N_6O_4$
5e	68.5	91—93	38.98 38.90	3.12 3.11	12.78 12.96		$C_{21}H_{20}F_{12}N_6O_4$
5f	67.1	131—133	38.77 38.90	3.22 3.11	12.84 12.96		$C_{21}H_{20}F_{12}N_6O_4$

Parameters of NMR spectra of products **5a—f** are given in Table 3; the compositions of the compounds obtained are confirmed by elemental analysis data (Table 4).

The reactions of [2+4]-cycloaddition of AH take place only with dienophiles possessing the sufficient donor properties. The behavior of AH towards different compounds with nitrile group illustrates this clearly (Scheme 4). If diethylcyanamide reacts with AH exothermically, and *N*-cyanomorpholine reacts at ~20 °C for 5—7 h, so chloroacetonitrile and MeCN can not be involved into the reaction with AH even on prolonged heating (3 days) at 130—140 °C.

Yields, characteristics, and ^1H and ^{19}F NMR spectral data of compounds **6a—h** are given in Table 5.

Thus, AHs demonstrate biphilic properties and act like 1,3-heterodienes or like dienophiles in [2+4]-cycloaddition reactions.

Scheme 4

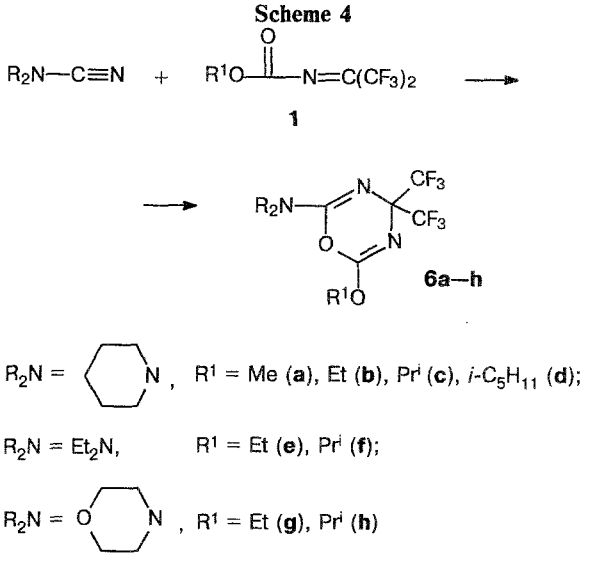


Table 5. Yields, physical characteristics, and ^1H and ^{19}F NMR spectra of compounds **6a—h**

Compound	Yield (%)	B.p./°C (*p*/Torr) [M.p./°C]	n_D^{20}	δ^1H	$\delta^{19}F$
6a	83.6	92 (0.1)	1.4240	1.52 (m, 6 H, $C_5H_{10}N$); 3.40 (m, 4 H, $C_5H_{10}N$); 3.90 (s, 3 H, OMe)	−3.27 (s)
6b	85.7	110 (0.1)	1.4250	1.36 (t, 3 H, OCH_2Me); 1.60 (m, 6 H, $C_5H_{10}N$); 3.47 (m, 4 H, $C_5H_{10}N$); 4.36 (q, 2 H, OCH_2)	−3.15 (s)
6c	78.4	[58—60]	—	1.25 (d, 6 H, $CHMe_2$); 1.48 (br.s, 6 H, $C_5H_{10}N$); 3.35 (br.s, 4 H, $C_5H_{10}N$); 5.05 (m, 1 H, $CHMe_2$)	−3.08 (s)
6d	70.4	Oil	—	0.93 (d, 6 H, $CHMe_2$); 1.52 (m, 6 H + 2 H, $C_5H_{10}N + CH_2CH$); 1.66 (m, 1 H, CH); 3.38 (m, 4 H, $C_5H_{10}N$); 4.30 (t, 2 H, OCH_2)	−2.02 (s)
6e	73.3	96 (1)	1.4022	1.14 (t, 6 H, $N(CH_2Me)_2$); 1.34 (t, 3 H, OCH_2Me); 3.35 (q, 4 H, $N(CH_2Me)_2$); 4.35 (q, 2 H, OCH_2)	−3.38 (s)
6f	84.2	92(1)	1.4019	1.16 (t, 6 H, $N(CH_2Me)_2$); 1.34 (d, 6 H, $CHMe_2$); 3.36 (q, 4 H, $N(CH_2Me)_2$); 5.13 (m, 1 H, $OCHMe_2$)	−3.21 (s)
6g	78.0	[32—33]	—	1.37 (t, 3 H, OCH_2Me); 3.51 (t, 4 H, C_4H_8NO); 3.71 (t, 4 H, C_4H_8NO); 4.36 (q, 2 H, OCH_2)	−3.00 (s)
6h	74.5	[55—57]	—	1.37 (d, 6 H, $OCHMe_2$); 3.52 (t, 4 H, C_4H_8NO); 3.72 (t, 4 H, C_4H_8NO); 5.15 (m, 1 H, $OCHMe_2$)	−2.97 (s)

Note. Elemental analysis data correspond to the molecular formulas for all of the compounds obtained.

Experimental

^1H, ^{19}F, and ^{31}P NMR spectra were obtained on a Bruker CXP-200 instrument (200, 188, and 81 MHz, respectively) with respect to TMS (internal standard), CF_3COOH, and 85 % H_3PO_4 (external standards) in $CDCl_3$ (compounds **2a—f**, **5b—f**, **6a—c**, **6e—h**) and in acetone-d_6 (compounds **5a**, **6d**). IR spectra were registered on a Specord IR-75 spectrometer (film or suspension in Vaseline oil, KBr plates). Melting points were determined in a glass capillary.

The synthesis of AH **1** is described earlier.[10]

2-Methoxycarbonyl-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-ene (2a). A solution of methoxycarbonylimine of hexafluoroacetone (3.1 g, 14 mmol) in 10 mL of anhydrous ether was added to a solution of 1.0 g (15 mmol) of cyclopentadiene in 7 mL of anhydrous ether. The ampoule was heated at 80 °C for 5 h, then the solvent was evaporated, and the residue was recrystallized from hexane. Compound **2a** (2.81 g) was obtained.

Compound **2c** was synthesized analogously to the above procedure.

2-Ethoxycarbonyl-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-ene (2b). A solution of 3.04 g (13 mmol) of ethoxycarbonylimine of hexafluoroacetone in 25 mL of anhydrous ether was added dropwise to a solution of 1.1 g (17 mmol) of cyclopentadiene in 10 mL of anhydrous ether at 20 °C, and the mixture was stirred for 4 h. The solvent was removed after ~24 h, and the residue was distilled *in vacuo*. Compound **2b** (2.64 g) was obtained in the form of a viscous colorless oil.

Compounds **2d—f** were synthesized analogously to the above procedure.

2-[1-(2-Ethoxycarbonylaminoperfluoroprop-2-yl)-3-*tert*-butylguanidino]-4,4-bis(trifluoromethyl)-6-ethoxy-1,3,5-oxadiazine (5a). A solution of 3.39 g (14 mmol) of ethoxycarbonylimine of hexafluoroacetone in 12 mL of anhydrous ether was added to a suspension of 1.0 g (7 mmol) of guanidine **3** in 40 mL of anhydrous ether, and the mixture was stirred until complete dissolution of a solid precipitate took place. The solvent was evaporated after ~24 h, and the residue was recrystallized from hexane. Compound **5a** (3.89 g) was obtained.

Compounds **5b—f** were synthesized analogously to the above procedure.

2-(Piperidin-1-yl)-4,4-bis(trifluoromethyl)-6-methoxy-1,3,5-oxadiazine (6a). A solution of 2.23 (10 mmol) of methoxycarbonylimine of hexafluoroacetone in 12 mL of anhydrous ether was added dropwise to a solution of 1.0 g (9 mmol) of *N*-cyanopiperidine in 5 mL of anhydrous ether, and the mixture was stirred at 20 °C for 3 h. The solvent was evaporated after ~24 h, and the residue was recrystallized from hexane. Compound **6a** (2.53 g) was obtained.

Compounds **6b—h** were synthesized analogously to the above procedure.

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