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NUCLEOPHILIC REACTIONS OF FLUOROOLEFINS. V. REGIOSELECTIVITY AND STEREOCHEMISTRY IN THE REACTIONS OF 1-PHENYLPENTAFLUOROPROPENES WITH LITHIUM DIALKYLAMIDES *

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SUMMARY

1-Phenylpentafluoropropenes 1 readily react with lithium dialkylamides to give, in most cases, mixtures of 1-aminosubstituted alkenes 2 and 2-amino-substituted alkenes 3, with the latter being the favoured products. The reactions with bulky lithium diethylamide and lithium 2-methylpiperidinoamide gave exclusively 1-amino-substituted products 2. The effect of the increased bulk of N-nucleophiles is opposite to that observed for the reactions of alkenes 1 with C-nucleophiles Increasing electronegativity of the phenyl ring substituents in alkenes 1 shifts the regioselectivity of the attack of lithium amides towards the C-2 carbon atom. The E to Z isomer ratios of enamines 2 were found to be time dependent and the slow isomerisation of the kinetic isomers E to the thermodynamic isomers Z was observed, while the ratio of isomers of enamines 3 did not change with time.

A concerted, single-step process is suggested for the reactions of alkenes 1 with lithium dialkylamides, and a tentative explanation of the different stereochemistry of enamines 2 and 3 is given.

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INTRODUCTION

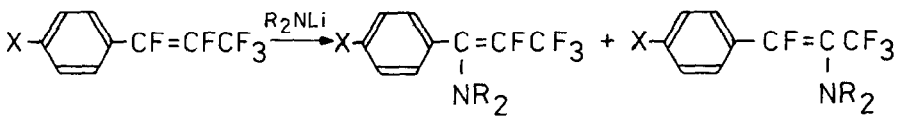
1-Phenylpentafluoropropenes 1 have proved to be good models for a study of the influence of different factors on the regioselectivity of the attack of nucleophiles on electrophilic alkenes. These factors may be both inherent in the reactant alkene, i.e. resonance, inductive, and steric effects of substituents, and external like electronic properties and the bulk of the attacking nucleophile. Results of the preceeding work have shown that in the reactions of alkenes 1 with sodium ethoxide [2] and with alkyllithium reagents [1] , the influence of the benzene ring substituents X on the regioselectivity of the attack by a nucleophile obeys the Hammett type correlation. The regioselectivity in the reactions of alkenes 1 with alkyllithium reagents has been found to be also strongly dependent on the bulk of the attacking reagent. Surprisingly enough, the increasing bulk of alkyllithium reagents, from methyl to tert-butyl, effectively directs the substitution of the alkyl group for fluorine to the more hindered carbon atom C-2 bearing the trifluoromethyl group [1] . These results are in contrast with those obtained earlier by Nguyen et al. [3] for the reaction of alkene 1a with bulky lithium diethylamide, in which a 92 % regioselectivity in favour of the substitution at benzylidene carbon atom C-1 was obtained. To clear the above discrepancy, further studies on the influence of the bulk of nucleophiles, particularly nitrogen nucleophiles, on the regioselectivity of their reactions with alkenes 1 are required.

Perfluoroalkenes are known to react readily with primary and secondary amines under mild conditions and these reactions have been treated in numerous papers. Primary amines combine with terminal fluoroalkenes like CF_2=CFCl [4] and CF_2=CF_2 [5] to give amidines, imidoyl fluorides and in some cases gem--diamidines, while with internal alkenes, like dimers of hexafluoropropene and some of its derivatives, imines are formed [6] Secondary amines form with fluoroalkenes saturated tertiary amines, enamines, or mixtures of both [4 - 11] . In some cases, instead of vinylic fluorines, a substitution of allylic fluorines by an amino group was observed [6,12] .

In the preliminary experiments 1-phenylpentafluoropropene 1a failed to react with free n-propylamine and diethylamine, even when heated at 100 - 120° for a long time. Alkene 1a also failed to react with lithium ethylamide prepared by addition of ethylamine to commercial n-butyllithium solution. These results limit the study of reactions of alkenes 1 with N-nucleophiles to reactions with lithium dialkyl amides which were earlier found to react easily [3] . The present paper reports on results obtained in the reactions of alkene 1a with five amides, i.e. lithium dimethyl-, diethyl-, pyrrolidino-, piperidino-, and 2-methylpiperidinoamide. Additionally, in order to study the effect of the phenyl ring substituents, the reactions of p-substituted alkenes 1b-d with lithium pyrrolidinoamide and lithium piperidinoamide were carried out.

RESULTS

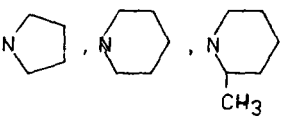
1-Phenylpentafluoropropenes 1a-d reacted readily at 0 - 20° with lithium dialkylamides to give, in general, mixtures of the E and Z isomers of 1-aminosubstituted alkenes 2 and 2-aminosubstituted alkenes 3 .



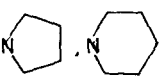
1 2 3

a: X=H , b: X=CH3O , c: X=CH3 , d: X=Cl

2a: NR2=NMe2 , NEt2 ,



3a: NR2=NMe2 ,



2b-d , 3b-d : NR2=

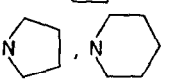
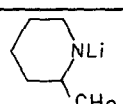


TABLE 1

Reactions of alkenes 1a-d[a] with lithium dialkylamides: ratios, physical properties, and analyses of the products

Substrates		Products ratio[b]		B.p.	ν(C=C)	Found/Calc. (%)[c]			
Alkene	Amide	2a(E/Z)	3a(E/Z)	°C(Torr)	cm^{-1}	C	H	F	N
1	2	3	4	5	6	7	8	9	10
PhCF=CFCF$_3$ 1a	$(CH_3)_2NLi$	38(0.6)	62(6.2)	39(0.2)	1650 1665	56.2 56.6	4.7 4.8	32.5 32.6	6.1 6.0
	$(C_2H_5)_2NLi$	100(3.1)	0	49-51(0.1)	1660 1700	59.7 59.7	5.9 5.8	29.2 29.1	5.3 5.4
		31(0.3)	69(5.6)	76-80(0.2)	1670 1710	60.2 60.2	5.1 5.1	29.5 29.3	5.4 5.4
		37(0.7)	63(9.4)	74-77(0.1)	1650 1665	61.6 61.5	5.6 5.5	27.8 27.8	5.0 5.1
		100(2.3)	0	101-104(0.6)	1635 1640	62.7 62.7	5.9 5.9	26.4 26.5	5.1 4.9

		2b	3b						
$p\text{-}CH_3OPhCF{=}CFCF_3$		45(0.1)	55(13.8)	104-109(0.6)	1610 1650	58.0 58.1	5.2 5.2	26.2 26.3	4.6 4.8
1b		85(2.3)	15(only E)	108-110(0.2)	1620 1710	59.1 59.4	5.6 5.7	25.3 25.1	4.3 4.6
		2c	3c						
$p\text{-}CH_3PhCF{=}CFCF_3$		32(0.3)	68(6.8)	74-76(0.1)	1620 1660 1670	61.4 61.5	5.5 5.5	27.8 27.8	5.1 5.1
1c		51(0.9)	49(11.5)	96-100(0.6)	1630 1660 1720	62.5 62.7	5.8 6.0	26.4 26.5	4.7 4.9
		2d	3d						
$p\text{-}ClPhCF{=}CFCF_3$		22(only Z)	78(3.8)	81(0.1)	1600 1655 1665	53.2 53.2	4.0 4.1	26.0 25.9	4.8 4.8
1d		29(0.5)	71(6.5)	98(0.2)	1610 1670 1715	54.3 54.6	4.4 4.6	24.8 24.7	4.6 4.6

[a] The isomer ratio E/Z = approx. 9 for alkene 1a and approx.6 for alkenes 1b-d.

[b] Determined from the crude reaction mixtures by integration of the CF_3 group signals in the ^{19}F NMR spectra. [c] Determined from the distilled mixtures of products.

The reactions proceeded even more readily than similar reactions with alkyllithium reagents [1] and they were complete in less than one hour. The reactions with lithium diethylamide and lithium 2-methylpiperidinoamide gave exclusively 1-amino-substituted products 2, while with other lithium amides investigated both types of products 2 and 3 were obtained in ratios depending on the amide used. Results, together with some physical data and analyses of products are summarised in Table 1.

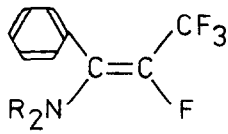
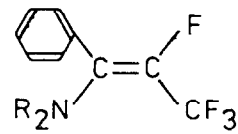
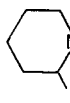
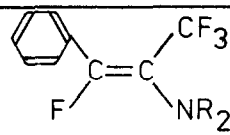
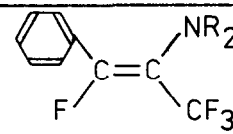
Compounds 2 and 3, with the exception of 2a where R = NEt_2 and $CH_3.\overline{CH(CH_2)_4N}$ which were isolated in pure state, were identified as their mixtures by the ^{19}F and ^{1}H NMR spectrometry (Table 2) and elemental analysis (Table 1). Similarly to alkyl-substituted 1-phenylpentafluoropropenes reported in the previous paper [1], the E and Z isomers of compounds 3 are easily distinguished from each other by the characteristic magnitude of the vinylic fluorine-to-CF_3 group coupling constants. Assigment of the geometric isomers of compounds 2, because of little difference of chemical shifts and coupling constants in the ^{19}F NMR spectra of these compounds, is somewhat ambiguous.

The ratios of geometric isomers quoted in Table 1 were determined from the crude reaction mixtures after 22 - 24 hours after the completion of the reaction. It has been found that the ratio of isomers of compounds 2 changes slowly on standing at ambient temperature and finally only the thermodynamically stable compounds 2(Z) remain. In contrast, both isomers 3(E) and 3(Z) are stable and their ratios were time-independent. This phenomenon is exemplified in Table 3 which shows the time dependance of the composition of mixtures obtained from the reactions of alkene 1a with lithium dimethylamide and lithium diethylamide. The isomerisation of compounds 2(E) to 2(Z) occured also during distillation.

Enamines 2 and 3 are stable in neutral and basic media but they are sensitive to acids. When treated with concentrated hydrochloric acid at 20°, compounds 2 hydrolyse to 2,3,3,3-tetrafluoropropiophenone 4, while compound 3 gives 1,3,3,3-tetrafluoro-1-phenyl-2-propanone 5.

TABLE 2

^{19}F NMR data of compounds 2a and 3a [a]

Compound R_2N-	$\phi(CF_3)$ ppm	$\phi(F)$ ppm	$J(F-CF_3)$ Hz	$\phi(CF_3)$ ppm	$\phi(F)$ ppm	$J(F-CF_3)$ Hz
	 2a(Z)			 2a(E)		
$(CH_3)_2N$-	61.3	159.6 m	13.5	64.1	spt 161.0	12.8
$(C_2H_5)_2N$-	61.5	156.9	13.0	65.6	156.8	11.7
	60.7	168.1 qq	13.6	61.6	165.0	11.2
	61.8	156.6 qm	13.3	64.4	159.0	12.5
	61.6	155.3	12.2	63.9	152.2	10.9
	 3a(Z)			 3a(E)		
$(CH_3)_2N$-	61.7	94.4	10.3	59.8	115.2	22.4
	60.2	95.5	9.2	59.9	115.5	24.0
	62.9	96.9	10.4	59.4	117.4	23.0

[a] ^{19}F NMR data of compounds 2b-d and 3b-d were within the ranges reported for compounds 2a and 3a. Chemical shifts are frc internal CCl_3F, positive upfield. If not indicated, the CF_3 group signals appeared as doublets, and signals of vinylic fluorines as quartets.

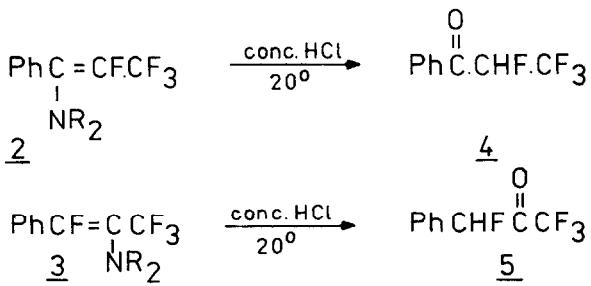
TABLE 3

Time dependance of the composition of mixtures obtained from the reaction of alkene 1a with $(CH_3)_2NLi$ and $(C_2H_5)_2NLi$.

Time (hrs)*	Products composition (GLC %)			
	$PhC(N(CH_3)_2)=CFCF_3$ (2a)		$PhCF=C(N(CH_3)_2)CF_3$ (3a)	
	Z	E	Z	E
24	23.4	14.8	8.6	53.2
168	36.0	0.0	8.9	55.1

	$PhC(N(C_2H_5)_2)=CFCF_3$	
	Z	E
22	24	76
70	31	69
166	43	57
238	53	47
670	100	0

* After the completion of the reaction



The new compound, ketone 5, was obtained on a preparative scale by hydrolysis of a mixture obtained from the reaction of alkene 1a with lithium pyrrolidinoamide which gave the highest yield of enamine 3 (Table 1).

DISCUSSION

The influence of the bulk of lithium dialkylamides on the regioselectivity of their reactions with 1-phenylpentafluoropropenes

The regioselectivity in the reactions of alkenes 1 which lithium dialkylamides (Table 1) is in agreement with that what could be expected by considering the structure of the substrate. In the case of smaller N-nucleophiles (dimethylamino-, pyrrolidino-, and piperidino-anion), similarly to the reaction with the ethoxide ion [2] and with small C-nucleophiles like methyllithium [1] , the regioselectivity is governed mainly by electronic properties of the alkene, i.e. polarisation of the double bond, and thus, relative susceptibility of carbon atoms C-1 and C-2 to the nucleophilic attack, with C-2 being the favoured position. In the case of large N-nucleophiles (diethylamino-, and 2-methylpiperidino-anion) the electronic effects are overwhelmed by steric interaction between the nucleophile and the substrate and the attack occures exclusively on the less hindered carbon atom C-1 bearing the phenyl substituent. * Steric effects directing N-nucleophiles, but not C-nucleophiles, to position C-1 are also noticed when the reactions of alkene 1a with anions $(CH_3)_2\bar{C}H$ and $(CH_3)_2\bar{N}$ of similar size are compared; the C-1/C-2 ratios of 0.315 and 0.612 were obtained respectively (reference [1] and Table 1).

In the preceeding paper [1] , the unusual effect of the bulk of C-nucleophiles which,otherwise than expected favours the reaction at the more hindered position C-2, has been interpreted in terms of steric strains influencing the geometry and, thus, free-energy of activation for the formation of the transition states leading to 1-substituted and 2-substituted products. Structures of those transition states should resemble the intermediate carbanions,rather than the substrates.

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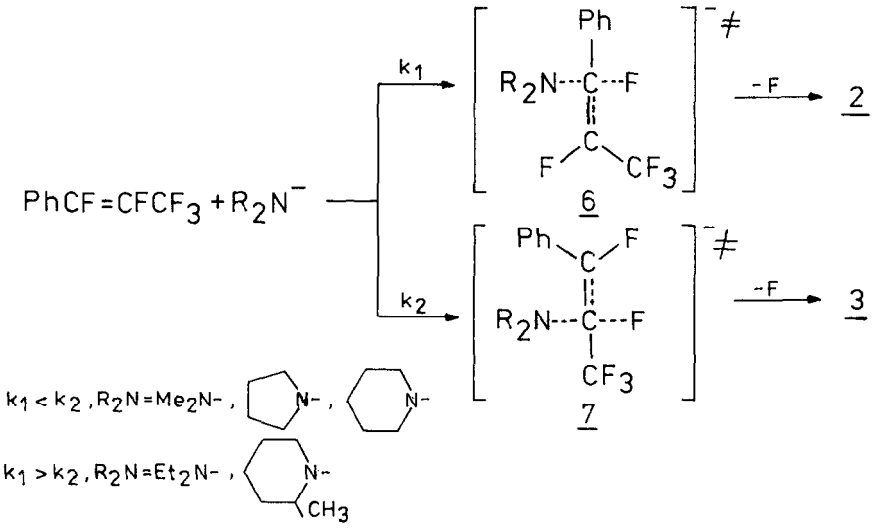
* Steric hindrances created by the CF_3 group have been discussed in detail in the preceeding paper [1] .

Steric strains should also have a substantial influence on the geometry and the activation energy of the formation of the transition states in the reactions with N-nucleophiles. Since the steric effect of N-nucleophiles is in agreement with the structure of the attacked alkene, it should be expected that the geometry of the corresponding transition states resemble the substrate. Thus, according to the Hammond postulate [13] this is a situation involving early transition states which are closer in free-energy to the substrate than to any intermediate or product. Thus, it may be assumed that, in contrast to the reactions with alkyllithiums [1] and also with sodium ethoxide [2], the reaction of 1-phenylpentafluoropropenes 1 with lithium dialkylamides is a concerted process involving transition states 6 and 7.



Concerted processes were recently postulated by Rappoport [14] to be involved in those cases where intermediate carbanions cannot be sufficiently stabilised. In the reactions of 1-phenylpentafluoropropenes 1 with lithium dialkylamides the unstability of the carbanionic intermediates is rationalised by strong donation of electrons from the lone-pair on the nitrogen atom to the carbon atom bearing fluorine which is due to leave. This effect forces the fluoride ion to leave before the negative charge on the carbanionic centre is fully developed.

In conclusion, it is suggested that the reverse effect of the bulk of alkyllithium reagents and lithium dialkylamides on the regioselectivity in their reactions with 1-phenylpentafluoropropenes may be interpreted by a shift in mechanism from a carbanionic two-step process for the former to a concerted process for the latter reagents. Further studies on this problem, including carbanion trapping reactions, stereochemistry, and the element effect are being conducted and will be reported in a forthcoming paper.

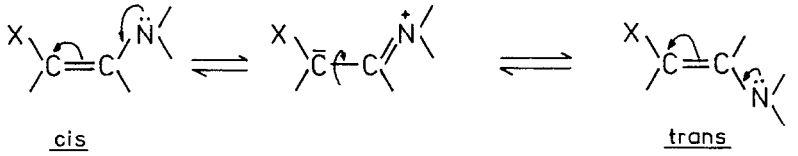
The effect of the phenyl ring substituents

The increasing electronegativity of the phenyl ring substituents X, similarly to the reactions with sodium ethoxide [2] and with alkyllithium reagents [1], effectively shifts the regioselectivity in the reactions of 1-phenylpentafluoropropenes 1 with lithium dialkylamides towards a substitution at the C-2 carbon atom. The ratios of 2-substituted products 3 to 1-substituted products 2 (C-2/C-1) were approximately 2.3 times higher for the reactions of p-chlorosubstituted alkene 1d than for p-methoxysubstituted alkene 1b, both with lithium pyrrolidineamide and with lithium piperidineamide (Table 1). However, in contrast to the previously reported reactions [1,2] no straight-line correlation between the C-2/C-1 ratios and the σ_p values of the phenyl ring substituents was found.

Stereochemistry of enamines 2 and 3

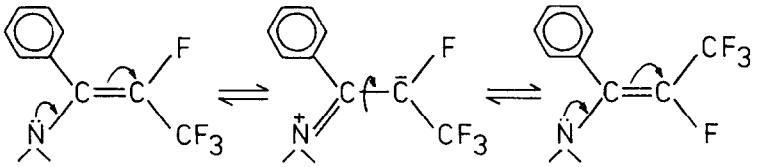
Analysis of the composition of mixtures obtained from the reactions of alkenes 1a-d with lithium dialkylamides (Table 1) and particularly the observation of the time dependence of the composition of some of these mixtures (Table 3) suggests that both types of enamines 2 and 3 are originally formed as kinetic products, mostly in the E form in which the amino group is *trans* to the vinylic fluorine. The isomerisation to the thermodynamically stable form is a common feature for enamines [15], but there is a question why only enamines 2, but not 3, undergo

the E to Z isomerisation. Enamines have a three-atom π system and the isomerisation proceeds via the immonium type resonance form (zwitterion) in which the double bond is shifted towards the nitrogen atom, thus enabling the carbon-carbon bond rotation [16-17].



cis trans

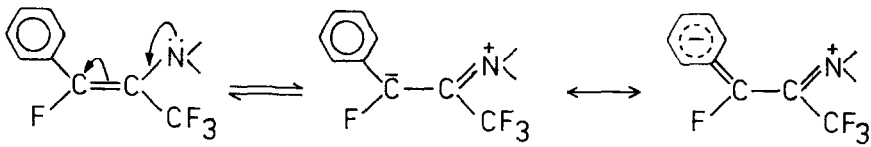
A common explanation concerning the isomerisation of enamines is the relative thermodynamic stability of the cis and trans isomers. McMullen and Stirling [16] argued that, when X is an electron-attracting group the trans-isomer is favoured by the opposition of the dipoles of substituents X and the C-N bond, and by the absence of non-bonded interaction between group X and the alkylamino-group. The steric interaction between the substituents can also help to shift the equilibrium [18]. This is just the case of enamines 2 and gives satisfactory explanation to the facile isomerisation of the kinetic isomer E to the thermodynamic isomer Z in which the dialkylamino-group is trans to the CF_3 group. In the intermediate zwitterion 8 the negative charge on the sp^3-hybridised carbon atom is stabilised by the inductive effect of the trifluoromethyl group.



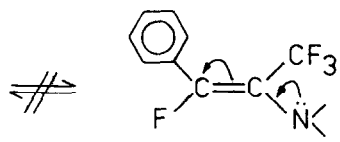
2(E) 8 2(Z)

By analogy to 2-(4-morpholino)-1,2-diphenylethene [19], for which the stability of the isomer having the morpholino-group trans to one of the phenyl substituents was attributed to the extention of the π-electron system to the phenyl group, the Z isomer of enamines 3 should also be expected to be thermodynamically more stable than the E isomer. However, the experimental

results showed high stability of the E form of enamines 3 . This is in agreement with another hypothesis which says that for such resonance systems the energetically favourable form is that in which the positive and negative dipoles are in the cis rather than trans form [20] . Irrespective of which of the above hypotheses is right, it seems reasonable that the minimum energy resonance form of intermediate immonium zwitterion 9 must be planar, thus inhibiting the carbon-carbon bond rotation.



3(E) 9



3(Z)

EXPERIMENTAL

Boiling points are uncorrected. NMR spectra were recorded with a JEOL JNM-4H-100 spectrometer and IR spectra with a Beckman Acculab-TM-1 spectrometer. The reactions were monitored with a Chromatron GHF.18.3.4 GLC instrument using a 3.5 m x 4 mm column packed with Chromosorb G coated with 3 % Silicon Oil SE-52.

1-Phenylpentafluoropropenes 1a-d were prepared according to the previously described procedure [21] . The amines used for the preparation of lithium dialkylamides were purified by storage over potassium hydroxide for a few days followed by distillation. n-Butyllithium was a commercial 1 N solution in hexane.

Reactions of 1-phenylpentafluoropropenes 1a-d with lithium dialkylamides

Solutions of lithium dialkylamides were prepared at 0° by adding a solution of 0.058 mole of the amine in 40 ml of dry diethyl ether to 30 ml of commercial n-butyllithium reagent (1.0 N solution in hexane).

A solution of 0.03 mole of alkene 1 in 15 ml of dry ether was cooled down to 0° and then, while stirring, the solution of lithium dialkylamide was added dropwise during a period of ca. 15 minutes. The reaction mixture was allowed to warm up to ambient temperature. The reaction, as shown by GLC, was completed in 45 - 60 minutes. Then, the reaction mixture was poured on cold water, the organic layer was separated, washed with water and dried over magnesium sulphate. On the next day, after evaporation of solvents, the crude mixture of products was subjected to ^{19}F NMR investigations. Samples for elemental analysis were obtained by distillation under reduced pressure. Yields of the distilled products were within the range of 35 - 50 % .

Hydrolysis of enamines 2 and 3

A sample of distilled or crude mixture of products obtained from the reaction of alkenes 1 with lithium dialkylamides was mixed with five volumes of concentrated hydrochloric acid and stirred together at 20° for 18 hours, then neutralised with sodium carbonate and extracted with ether. The extract was dried over magnesium sulphate and after evaporation of ether, the residue was subjected to GLC and spectral investigations which showed the presence of ketones 4 and 5 . Neither compound 2 nor 3 was present. Analytical samples of 4 and of a mixture of 4 and 5 were obtained by distillation under reduced pressure. Elemental analysis and spectral data for ketone 4 were in agreement with those previously reported [2] . Analytical and spectral data for compound 5 are given below.

Preparation of 1,3,3,3-tetrafluoro-1-phenyl-2-propanone 5 (nc)

A solution of 12.6 g (0.06 mole) of 1-phenylpentafluoropropene 1a in 30 ml of ether was reacted as described above with a solution of lithium pyrrolidinoamide prepared from 10 g (0.14 mole) of pyrrolidine, 80 ml of ether, and 60 ml of 1.0 N n-butyllithium reagent. The reaction was carried out three times and combined mixtures of products (40.6 g) were distilled to give 31.2 g of a fraction boiling at 68 - 70°(0.4 Torr). This fraction, which was shown by intergrated ^{19}F NMR spectrum to consist of 1-phenyl-1-pyrrolidinotetrafluoropropene (33 %) and 1-phenyl-2-pyrrolidinotetrafluoropropene (67 %) (compounds 2a and 3a where $NR_2 = N(CH_2)_4$), was hydrolised at 20° with 60 ml of concentrated hydrochloric acid for 6 hours and worked up as above to give a mixture of ketones 4 and 5 (23.0 g). Fractional distillation gave pure ketone 5 (7.5 g) boiling at 58 - 60° (10 Torr). Calculated for $C_9H_6F_4O$: C,52.4 ; H,2.9 ; F,36.9 %. Found: C,52.2 ; H,2.8 ; F,37.0 %. ^1H and ^{19}F NMR: $\delta(C\underline{H}F) = 5.55$ ppm(d), $\phi(CF_3) = 82.8$ppm(d), $\phi(C\underline{H}F) = 192.7$ppm(dq), $^2J(H-F) = 45.5$ Hz, $^4J(F-F) = 11.7$ Hz. IR(film): $\nu(C=O) = 1695$ and 1790 cm^{-1}.

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