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ANIONIC ACTIVATION BY FLUORIDE ION IN SOLID-LIQUID SYSTEMS. SYNTHESIS OF 3(2H)-FURANONES AND 2 (5H)-FURANONES.

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Abstract - The evolution of 2-acyloxy 2-methyl 3-oxobutanamides **1** and 2-acyloxy 3-oxo 2,3-diphenylpropanamides **13** under anionic activation by cesium fluoride was studied. The fluoride ion is an efficient base for the heterocyclization of **1** into 3 (2H)-furanones and 2 (5H)-furanones, but the hydrolysis of the ester group lowered the selectivity of the reaction. However, the cleavage of **13** into the esters **14** and the cyclization of 3-benzoyloxy 3-methyl 2-butanone into bullatenone are very selective.

Introduction

Alkali metal fluorides are, in many cases, very effective bases. They allow reactions like alkylation, esterification, condensation and elimination.[1,2] They show frequently a high selectivity and has been used for the synthesis of β-lactams without protection of the electrophilic group present in the starting compounds.[3,4] Cesium fluoride is an alkali metal fluoride with the largest cation diameter and thus, the anion is nearly a naked fluoride. Alumina supported potassium fluoride is also an efficient base.[2,5-7]

With a view to evaluate the selectivity of the reactions, we have examined the anionic activation of compounds bearing several functional groups : 2-acyloxy 2-methyl 3-oxobutanamides **1** and 2-acyloxy 3-oxo 2,3-diphenylpropanamides **13**, by cesium fluoride. In addition, we have compared the ability of alumina-supported potassium fluoride, dihydrated potassium fluoride and potassium hydroxyde to induce the anionic activation of **1** in solid-liquid systems.

Results and discussion

The esters **1**, treated in THF by powdered CsF in the presence of benzyltriethylammonium chloride (**2**), gave the hydroxyketones **3** and the 3 (2H)-furanone **4**, the 2 (5H)-furanones **5** or the 4-methylene 2-oxazolidinone **12** (table I, schemes I and III). The anionic activation with different reagents was investigated and the results are summarized in table II. CsF in "dry media" (without solvent) and CsF suspended in THF, with **2**, gave the same results. No reaction was observed when dried CsF was used. KF has a very low reactivity ; KF, $2H_2O$ [8] with **2** is as reactive as alumina-supported KF (table II).

The hydroxyketones **3**, formed in all cases, arised from the hydrolysis of the ester group of **1**. This hydrolysis was more important with KF, $2H_2O$ and alumina - supported KF than with commercial CsF, and was not observed with dried CsF (table II). The anionic activation of a substrate by CsF requires a little amount of water. In these conditions, the fluoride ion induces an anionic activation of the water, which can cleave the ester group if the rates of the other ways of evolution are slow. The fluoride ion of alumina-supported KF gives some AlO^- species or HO^- which cleave the ester group (scheme II). The rate of the hydrolysis was decreased when the electrophilic acetyl group was removed. Thus,

Table I

Evolution of the esters **1** in refluxing THF, in the presence of CsF and **2**.

	Esters 1		time (h)	Product yields (%) [a]				
	R^1	R^2		**3**	**5**	**4**	**12**	others
a	Me	t-Bu	72	30	70			
b	Ph	t-Bu	48	64		36		
c	PhCH=CH	t-Bu	72	45				$tBuNH_2$ [c]
d	Me	Ar [b]	4	50	40			$ArNH_2$, ArNHCONHAr
e	Ph	Ar [b]	4	40			35	$ArNH_2$, ArNHCONHAr
f	PhCH=CH	Ar [b]	24	80				$ArNH_2$

a - Isolated yield.
b - Ar = 2,6-$Me_2C_6H_3$.
c - Degradation products.

Table II

Evolution of **1b** in the presence of various reagents.

Reagent	CsF [a]	CsF [b]	KF/THF	$KF,2H_2O$/THF	KF/Al_2O_3	KOH/THF	$Ca(OH)_2$THF
Temp.(°C)	85-90	85-90	66	66	66	20	66
Time (h)	4	4	72	72	48	0,25	72
3 (yield %)	0	65	0	90	90	c	90
4 (yield %)	0	35	0	0	0		0

a - Without benzyltriethylammonium chloride and solvent, CsF dried at 150°C/0.05 torr for 18 h.
b - Without benzyltriethylammonium chloride and solvent. Commercial CsF.
c - Degradation of **1b**.

the ester **10** was stable in the presence of CsF at 70°C for 48 h.

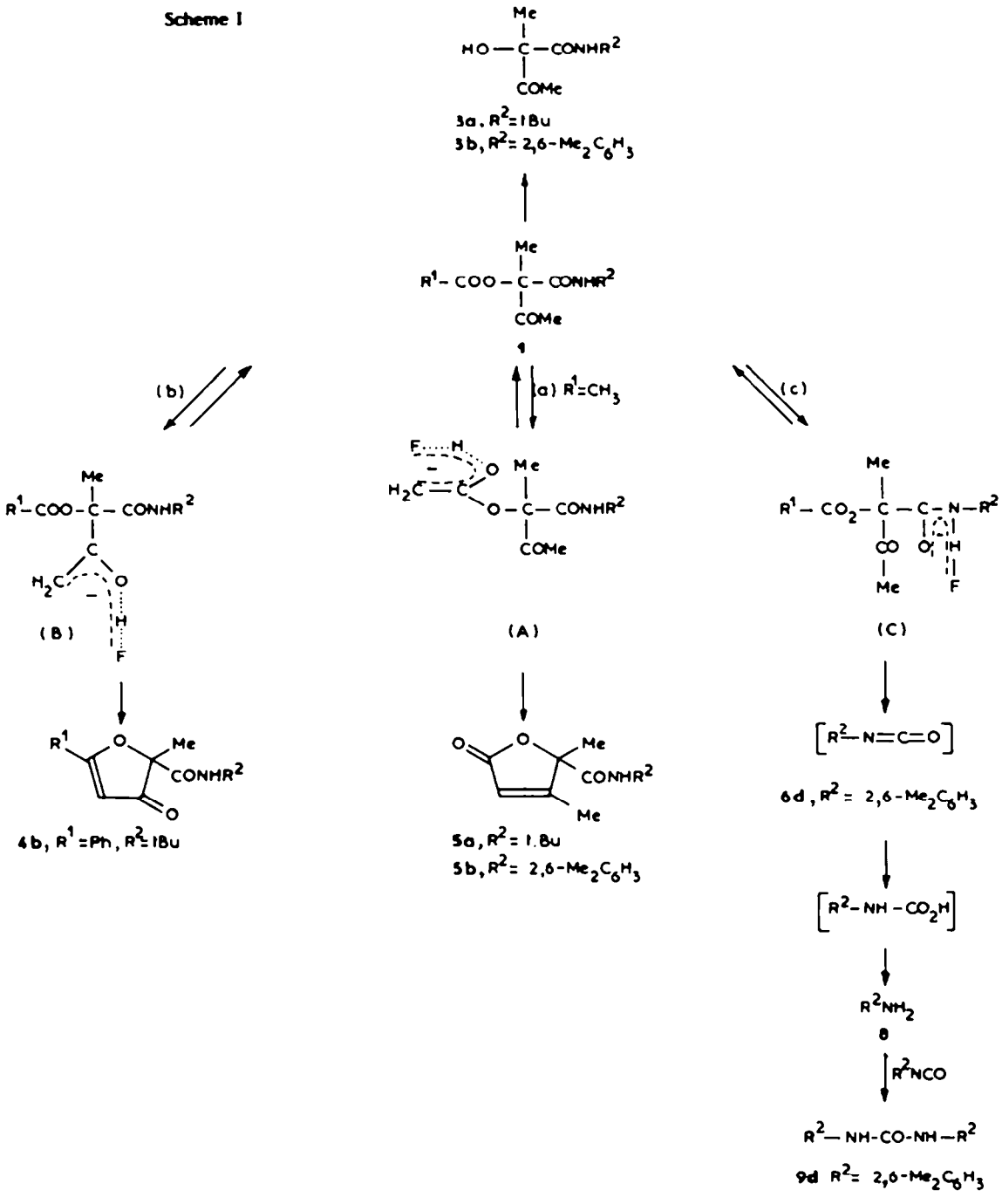
When R^1 = Me, the carbanion (B) was more easily formed than (A) (scheme I). However, (A) and (B) are in equilibrium and (A) is cyclized faster than (B).[9] This explains the formation of **5a** and **5d** from **1a** and **1d**. When R^1 = Ph, the cyclization of the anion (B) gives the 3 (2H)-furanone **4b**.

The reaction of the esters **1 d-f** with CsF gave the amidure (C) stabilized by the 2,6-dimethylphenyl group (scheme I). The isocyanate **6** is formed by β-elimination of a stabilized carbanion from (C). Hydrolysis of **6** followed by decarboxylation produces the amine **8** and the urea **9**. The reaction of **3e** with the isocyanate **6** gave the carbamate **11** which was converted by CsF into the 4-methylene 2-oxazolidinone **12** [10] (scheme III).

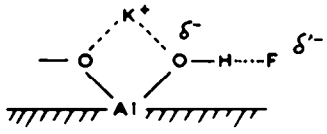
The keto group of the ester **1c** has a low electrophilicity, thus the evolution of **1c** followed the way (c) (scheme I) ; t-$BuNH_2$ was isolated.

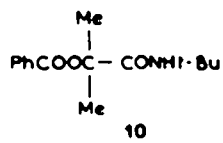
Esters **13**, treated with CsF, gave selectively the esters **14** (90-95 %) and the isocyanates **6** which are converted into ureas **9** (scheme IV). The low electrophilicity of the keto group did not allow the formation of the corresponding 2 (5H)-furanones from **13a** and **13c**.

Scheme I

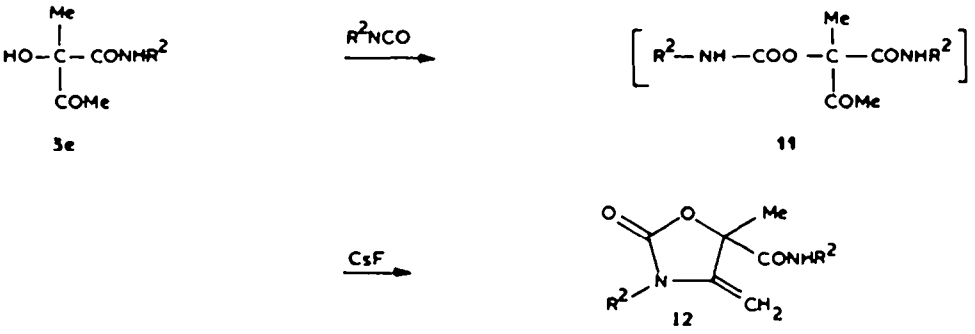


Scheme II

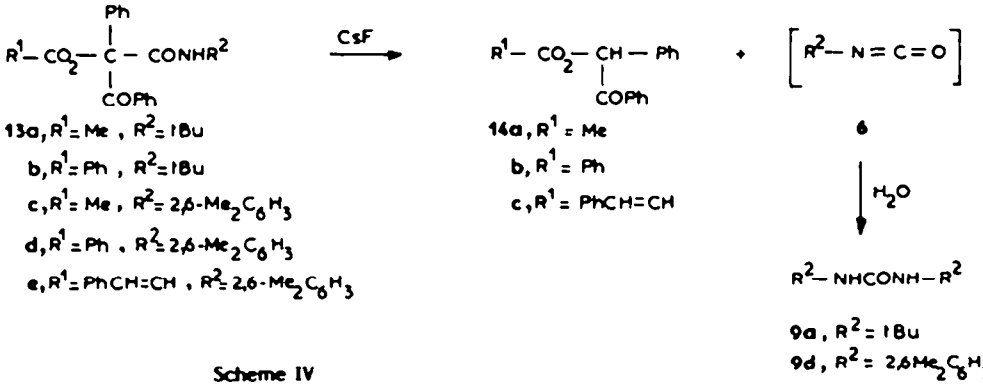




10



Scheme III
($R^2 = 2,6\text{-}Me_2C_6H_3$)



Scheme IV

The reaction of powdered KOH with 13 gave a complex mixture of degradation products : the nucleophilic attack of HO^- on the ester group and the keto group afforded 15, 16 and 17 (table III).

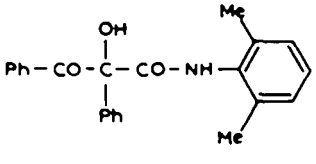
The high selectivity of the reactions induced by the anionic activation with the fluoride ion, previously reported in the case of the preparation of β-lactames from α-acyloxycarboxamides [3] is observed with the conversion of α-acyloxyketones 13 into 14. The acyloxyketones 1 have several possibilities of evolution with similar rates ; their anionic activation is not selective. However, a selective evolution is expected with the acyloxyketone 18,[9] what can be used for a simple preparation of the bullatenone 19,[13] a naturally occuring furanone isolated from essential oil of *Myrtus bullata*, which has been frequently prepared.[9,12,14,15]

Table III

Reaction of 13 with powdered KOH.

Reactant	Product yield (%) [a]		
13a			17a (40 %)
13b			17a (20 %)
13c	15 (30 %)	16c (30 %)	17c (30 %)
13d	15 (80 %)		17c (10 %)
13e	15 (20 %)	16e (60 %)	17c (5 %)

a - Isolated product.



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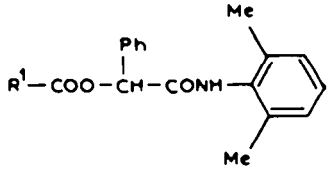
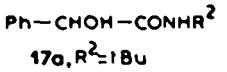


16c, R^1 = Me
16e, R^1 = PhCH=CH



17a, R^2 = tBu
17c, R^2 = 2,6-$Me_2C_6H_3$

Experimental section

Melting points are uncorrected. IR spectra were recorded as suspensions in Nujol, unless otherwise indicated, with a Perkin-Elmer 225 Spectrometer. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker WP 80 CW instrument at 80 MHz and a Bruker WP 80 DS spectrometer at 20.115 MHz, respectively. Chemical shifts are reported in δ relative to Me_4Si as an internal standard. Mass spectra were obtained with a Varian MAT 311 mass spectrometer at 70 eV. Microanalyses were performed by the analytical laboratory, Centre National de la Recherche Scientifique. The acyloxy amides 1, 10 and 13 were prepared from the isocyanides R^2NC, the acids R^1COOH and the corresponding α-diketones or the acetone, by using the reaction of Passerini[11]. Alumina-supported KF was prepared as previously described.[6]

1a, yield 90 %, m.p. 61-62° ; IR, ν : 1650, 1715, 1725, 3375 cm^{-1}.
^{1}H NMR, δ : 1.36 (s, 9H) ; 1.74 (s, 3H) ; 2.20 (s, 3H) ; 2.25 (s, 3H) ; 6.95 (br, 1H).

1b, yield 93 %, m.p. 58-60° ; IR, ν: 1680, 1700, 1730, 3360 cm^{-1}.
^{1}H NMR, δ : 1.41 (s, 9H) ; 1.86 (s, 3H) ; 2.30 (s, 3H) ; 7.10 (br, 1H) ; 7.50 (m, 3H) ; 8.05 (m, 2H).

1c, yield 85 %, m.p. 158-159° ; IR, ν : 1625, 1655, 1710, 1735, 3250 cm^{-1}.
^{1}H NMR, δ : 1.41 (s, 9H) ; 1.83 (s, 3H) ; 2.30 (s, 3H) ; 6.45 (d, J = 16 Hz, 1H) ; 7.15 (br, 1H) ; 7.40 (m, 5H) ; 7.76 (d, J = 16 Hz, 1H).

1d, yield 88 %, m.p. 100-101° ; IR, ν : 1655, 1725, 1745, 3280 cm^{-1}.
^{1}H NMR, δ : 1.82 (s, 3H) ; 2.19 (s, 9H) ; 2.29 (s, 3H) ; 7.05 (s, 3H) ; 8.95 (br, 1H).

1e, yield 75 %, m.p. 103-104° ; IR, ν: 1690, 1715, 1725, 3280 cm^{-1}.
^{1}H NMR, δ : 1.93 (s, 3H) ; 2.22 (s, 6H) ; 2.32 (s, 3H) ; 7.03 (s, 3H) ; 7.45 (m, 3H) ; 8.05 (m, 2H) ; 8.90 (br, 1H).

1f, yield 79 %, m.p. 100-101° ; IR, ν : 1630, 1685, 1700, 1715, 3330 cm^{-1}.
^{1}H NMR, δ : 1.93 (s, 3H) ; 2.26 (s, 6H) ; 2.36 (s, 3H) ; 6.56 (d, J = 16 Hz, 1H) ; 7.05 (s, 3H) ; 7.40 (m, 5H) ; 7.80 (d, J = 16 Hz, 1H) ; 9.00 (br, 1H).

10, yield 80 %, m.p. 72-73°. ^{1}H NMR, δ : 1.35 (s, 9H) ; 1.73 (s, 6H) ; 5.95 (br, 1H) ; 7.50 (m, 3H) ; 8.00 (m, 2H).

13a, yield 85 %, m.p. 111-112°, IR, ν : 1660, 1690, 1745, 3310 cm^{-1}.
^{1}H NMR, δ : 1.35 (s, 9H) ; 2.07 (s, 3H) ; 7.5-7.8 (m, 10H) ; 8.35 (br, 1H).

13b, yield 94 %, m.p. 185-186° ; IR, ν : 1665, 1695, 1715, 3320 cm^{-1}.
^{1}H NMR, δ : 1.36 (s, 9H) ; 7.45 (m, 9H) ; 7.75 (m, 4H) ;8.05 (m, 2H) ; 8.45 (br, 1H).

13c, yield 82 %, m.p. 153-154° ; IR, ν : 1665, 1695, 1745, 3300 cm^{-1}.
^{1}H NMR, δ : 2.05, 2.08 (2s, 9H) ; 7.00 (s, 3H) ; 7.45 (m, 6H) ; 7.75 (m, 4H).

13d, yield 90 %, m.p. 220° ; IR, ν : 1670, 1690, 1725, 3320 cm^{-1}.
^{1}H NMR, δ : 2.08 (s, 6H) ; 6.98 (s, 3H) ; 7.25-8.10 (m, 15H) ; 10.0 (br, 1H).

13e, yield 89 %, m.p. 100-110° ; IR, ν : 1630, 1670, 3310 cm^{-1}.
^{1}H NMR, δ : 2.08 (s, 6H) ; 6.50 (d, J = 16 Hz, 1H) ; 7.00 (s, 3H) ; 7.30-7.95 (m, 16H) ; 9.95 (br, 1H).

Reaction of CsF with the 2-acyloxy 2-methyl 3-oxobutanamides **1**. The reagent (CsF, KF, KF/Al_2O_3 or $Ca(OH)_2$, 15 mmol) was added to a solution of **1** (5 mmol) and **2** (0.5 mmol) in dry THF (20 ml). The resulting mixture was refluxed for the time indicated in the tables I or II. Filtration of the suspension and evaporation of the solvent let to a residue which was washed with water.

When the reaction was performed without solvent, the reagents were mixed, then heated for the time and at the temperature indicated in the table II. The products were extracted with dichloromethane. The evaporation of the solvent gave the crude products which were separated by Kugelrohr distillation under 0.02 mm.

N-*tert*.Butyl 2-acetyl 2-hydroxy propanamide **3a** : m.p. 65°C (petroleum ether). IR, ν : 1650, 1725, 3240, 3380 cm^{-1}. ^{1}H NMR, δ : 1.37 (s, 9H) ; 1.62 (s, 3H) ; 2.46 (s, 3H) ; 4.65 (br, 1H) ; 6.75 (br, 1H). M.S. M$^+$ m/e 187.1207. $C_9H_{13}NO_3$ requires 187.1208.

N-(2,6-dimethylphenyl) 2-acetyl 2-hydroxypropanamide **3d** : m.p. 104°C (ether). IR, ν : 1655, 1710, 3280, 3415 cm^{-1}. ^{1}H NMR, δ : 1.68 (s, 3H) ; 2.11 (s, 6H) ; 2.45 (s, 3H) ; 5.00 (s, 1H) ; 7.03 (s, 3H) ; 8.30 (br, 1H). M.S. m/e (relative intensity) 235 (M$^+$, 24), 193 (17), 192 (37), 148 (32), 122 (48), 88 (100).

2(5H)-furanone **5a** : m.p. 94°C (ether/petroleum ether). IR, ν : 1635, 1670, 1760, 3345 cm^{-1}. ^{1}H NMR, δ : 1.35 (s, 9H) ; 1.67 (s, 3H) ; 2.24 (d, J = 2 Hz, 3H) ; 5.76 (q, J = 2 Hz, 1H) ; 6.35 (br, 1H). ^{13}C NMR, δ : 14.1 ; 22.6 ; 28.5 ; 51.6 ; 89.2 ; 115.2 ; 167.2 ; 171.4. M.S. : (M$^+$-CH_3)/e calcd 196.0974, found 196.0969.

2(5H)-furanone **5d** : m.p. 131°C (ether). IR, ν : 1630, 1680, 3325 cm^{-1}. ^{1}H NMR, δ : 1.83 (s, 3H) ; 2.17 (s, 6H) ; 2.29 (d, J = 2 Hz, 3H) ; 5.84 (q, J = 2 Hz, 1H) ; 7.08 (s, 3H) ; 7.80 (br, 1H). ^{13}C NMR, δ : 14.0 ; 18.0 ; 22.4 ; 89.6 ; 115.5 ; 127.8 ; 128.4 ; 132.6 ; 135.4 ; 166.6 ; 171.2. M.S. M$^+$ m/e 259.1215, $C_{15}H_{17}NO_3$ requires 259.1208.

3(2H)-furanone **6b** : m.p. 125°C (ether). IR (CCl_4), ν : 1675, 1695, 1720, 3325 cm^{-1}. ^{1}H NMR, δ : 1.40 (s, 9H) ; 1.79 (s, 3H) ; 6.05 (s, 1H) ; 6.60 (br, 1H) ; 7.60 (m, 3H) ; 7.95 (m, 2H). ^{13}C NMR, δ : 23.2 ; 28.6 ; 51.6 ; 90.0 ; 99.2 ; 127.7 ; 128.5 ; 129.1 ; 133.4 ; 164.3 ; 185.2 ; 201.8. M.S. : (M$^+$ - CH_3)/e calcd 258.1130, found 258.1132.

2-oxazolidinone **12** : m.p. 201-202°C (ether). IR, ν : 1630, 1690, 1765, 3340 cm^{-1}. ^{1}H NMR, δ : 2.02 (s, 3H) ; 2.23 (s, 12H) ; 3.93 (d, J = 3 Hz, 1H) ; 4.70 (d, J = 3 Hz, 1H) ; 7.1 (s, 3H) ; 7.25 (m, 3H) ; 8.0 (br, 1H). ^{13}C NMR, δ : 17.2 ; 17.3 ; 18.1 ; 26.3 ; 84.0 ; 85.4 ; 127.8 ; 128.4 ; 129.0 ; 129.8 ; 130.3 ; 132.6 ; 135.5 ; 136.9 ; 144.1 ; 153.1 ; 167.2. M.S. M$^+$ m/e 364.1803, $C_{22}H_{24}N_2O_3$ requires 364.1787.

Reaction of esters **13** with CsF. To a THF solution (20 ml) of **13** (5 mmol) and **2** (0.1 g, 0.44 mmol) was added CsF (2.30 g, 15 mmol). The heteregeneous mixture was refluxed for 30 min., then poured into 10 ml of water. The organic layer was separated and dried (Na_2SO_4). Evaporation of the solvent gave the crude products which were separated by distillation under reduced pressure.

Amines **8** were identified by comparison of their NMR spectra with those obtained for authentic samples.

N,N'-di *tert* butyl urea **9a** : $E_{0.02}$ = 95-100°C ; ^{1}H NMR, δ : 1.50 (s, 9H) ; 10.08 (s, 1H) ; MS m/e (relative intensity) 172 (M$^+$, 4), 157 (11), 57 (100).

N,N'(-di (2,6 -dimethylphenyl) urea **9d** : $E_{0.02}$ = 150°C. ^{1}H NMR ($CDCl_3$ + CF_3CO_2H) δ : 2.23 (s, 3H) ; 2.46 (s, 3H) ; 5.95 (br, 1H) ; 7.05 (s, 3H) ; 7.29 (s, 3H).

Anal. calcd for $C_{17}H_{20}N_2O$: C, 76.12 ; H, 7.46 ; N, 10.45. Found C, 75.61 ; H, 7.27 ; N, 10.16.

O-acylbenzoïnes **14**

14a, m.p. 75-76°C ; IR, ν : 1690, 1725 cm^{-1}. ^{1}H NMR, δ : 2.24 (s, 3H) ; 6.86 (s, 1H) ; 7.40 (m, 8H) ; 7.95 (m, 2H). ^{13}C NMR, δ : 20.8 ; 77.8 ; 128.9 ; 129.2 ; 129.5 ; 133.6 ; 133.9 ; 134.9 ; 170.6 ; 194.0.

M.S. : M^+ m/e 254.0938, $C_{16}H_{14}O_3$ requires 254.0943.

14b, m.p. 120-121°C ; IR, ν : 1715, 1790 cm^{-1}. ^1H NMR, δ : 7.10 (s, 1H) ; 7.45 (m, 11H) ; 8.05 (m, 4H). ^{13}C NMR, δ : 78.1 ; 128.5 ; 128.8 ; 129.0 ; 129.2 ; 129.4 ; 129.7 ; 130.1 ; 133.5 ; 134.1 ; 135.0 ; 166.1 ; 193.9.
Anal. calcd for $C_{21}H_{16}O_3$: C, 79.72 ; H 5.21. Found : C, 79.73 ; H, 5.10.

14c, m.p. 102-103°C ; IR, ν : 1635, 1690, 1715 cm^{-1}. ^1H NMR, δ : 6.58 (d, J = 16 Hz, 1H) ; 7.45 (m, 13H) ; 7.77 (d, J = 16 Hz, 1H). 7.95 (m, 2H). ^{13}C NMR, δ : 77.7 ; 117.3 ; 128.3 ; 129.0 ; 129.2 ; 129.4 ; 130.6 ; 133.5 ; 134.0 ; 134.4 ; 134.9 ; 146.2 ; 166.4 ; 194.0.
M.S. : M^+ m/e 342.1240, $C_{23}H_{18}O_3$ requires 342.1256.

Reaction of esters 13 with KOH

Potassium hydroxyde (0.28 g, 5 mmol) was added to a solution of **13** (2.5 mmol) in dichloromethane (25 ml). The reaction mixture was stirred for 30 min., then washed with water (20 ml). Removal of the solvent gave the crude products which were separated by distillation under reduced pressure.

N-(2,6-dimethylphenyl) 2,3-diphenyl 2-hydroxy propanamide 15

m.p. 192-193°C (ether). IR, ν : 1665, 1725 ; 3230, 3250 cm^{-1}. ^1H NMR, δ : 2.11 (s, 6H) ; 6.42 (s, 1H) ; 7.00 (s, 3H) ; 7.50 (m, 8H) ; 8.15 (m, 2H).
M.S. : M^+ m/e 359.1515, $C_{23}H_{21}NO_3$ requires 359.1521.

16c, $E_{0.02}$ - 165-170°C ; m.p. 138-139°C (ether). IR, ν : 1660, 1735, 3250 cm^{-1}. ^1H NMR, δ : 2.07 (s, 6H) ; 2.21 (s, 3H) ; 6.15 (s, 1H) ; 7.00 (s, 3H) ; 7.40 (m, 6H).
M.S. : $M^{+\cdot}$ m/e 297.1351, $C_{18}H_{19}NO_3$ requires 297.1348.

16e, m.p. 164-166°C (ether). IR, ν : 1640, 1665, 1720, 3235 cm^{-1}. ^1H NMR, δ : 2.16 (s, 6H) ; 6.37 (s, 1H) ; 6.60 (d, J = 15 Hz, 1H) ; 7.04 (s, 3H) ; 7.45 (m, 11H) ; 7.83 (d, J = 15 Hz, 1H).
M.S. : m/e (relative intensity) 385 (M^+, 5) ; 238 (14) ; 237 (12) ; 265 (5) ; 131 (100).

17a, $E_{0.02}$ - 105 -110°C ; IR, ν : 1645, 3245, 3345 cm^{-1}. ^1H NMR, δ : 1.35 (s, 9H) ; 4.15 (d, J = 3 Hz, 1H) ; 4.84 (d, J = 3 Hz, 1H) ; 6.15 (br, 1H) ; 7.33 (s, 5H).

17c, $E_{0.03}$ = 160-165°C ; IR, ν 1660, 3240, 3350 cm^{-1}. ^1H NMR, δ : 2.07 (s, 6H) ; 4.20 (br, 1H) ; 5.10 (s, 1H) ; 7.00 (s, 3H) ; 7.40 (m, 6H).

Bullatenone 19

A mixture of ester **18** [9] (1.03 g, 5 mmol) and CsF (3 g, 20 mmol), without solvent, was heated at 150-155°C for 64 h. The reaction mixture was cooled at room temperature and extracted with dichloromethane. The extract, concentrated in vacuo, gave the bullatenone **19**, m.p. 65°C (ether/petroleum ether) in 65 % yield. **19** had spectroscopic properties (^1H NMR, IR) compatible with those reported.[9,15]
^{13}C NMR, δ : 23.2 ; 89.0 ; 97.7 ; 127.3 ; 128.9 ; 129.4 ; 132.7 ; 183.5 ; 207.0. M.S. $M^{+\cdot}$ m/e 188.0836, $C_{12}H_{22}O_2$ requires 188.0837. Anal. calcd for $C_{12}H_{22}O_2C$, 76.57 ; H, 6.43. Found C, 76.25 ; H, 6.43.

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