Unexpected and expeditious entry into trifluoromethyl aziridines from substituted β -dicarbonyl compounds

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General Experimental Methods. GC analyses were performed with a gas chromatograph equipped with a capillary column (methyl silicone, 12.5 m x 0.2 mm). GC-MS analyses were performed using a chromatograph equipped with a capillary column (phenyl methyl silicone, 30 m x 0.25 mm). ESI MS analyses were performed using a quadrupole-time of flight (TOF) mass spectrometer equipped with an ESI source and a syringe pump; the experiments were conducted in the positive ion mode. IR spectra were recorded using CHCl₃ as the solvent. ¹H NMR and ¹³C NMR spectra were recorded by a spectrometer at 300 and 75 MHz respectively, using CDCl₃ as the solvent and CHCl₃ as the internal standard. ¹⁹F NMR spectra were recorded by a spectrometer at 282.2 MHz, using CDCl₃ as the solvent and C₆F₆ as the internal standard; chemical shifts are given in ppm relative to CFC₃. HPLC analyses were performed with an instrument equipped with a differential refractometer, using an analytical column (3.9 x 300 mm, flow rate 1.3 mL/min). Eluents were HPLC grade. The synthesis of **2**,¹ **10**,² and NsONH-Z³ were performed following reported procedures. To synthesize **1** we had to use more drastic conditions (CF₃CO₂H, toluene, reflux, 7 days) than those reported.¹

(1) Blond, G.; Billard, T.; Langlois, B. R. *J. Org. Chem.* **2001**, *66*, 4826-4830.

(2) Fioravanti, S.; Colantoni, D.; Pellacani, L.; Tardella, P. A. *J. Org. Chem.* **2005**, *70*, 3296-3298.

(3) Fioravanti, S.; Morreale, A.; Pellacani, L.; Tardella, P. A. *Synlett* **2004**, 1083-1085.

STANDARD 1H OBSERVE

exp5 std1h

SAMPLE		DEC. & VT	
date	Jul 21 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.942	dm	nnn
tn	H1	dum	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	lb	0.50
fb	2600	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	7.0		
d1	0	werr	
tof	400.0	wexp	
nt	320	wbs	
ct	240	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-317.4		
wp	4506.5		
vs	178		
sc	0		
wc	265		
hzmm	2.02		
is	302.75		
xf1	317.4		
xfp	0		
th	38		
ins	100.000		
nm	ph

3

STANDARD 1H OBSERVE

exp2 std1h

SAMPLE DEC. & VT
 date Nov 26 2002 dfrq 299.942
 solvent CDCl3 dn H1
 file exp dpr 30
 ACQUISITION dof 0
 sfrq 299.942 dn nnn
 tn H1 dnm c
 at 1.998 dmf PROCESSING 200
 np 18008
 sw 4506.5 wtfile
 fb 2600 proc ft
 bs 16 fn not used
 tpwr 55
 pw 7.0 weir
 d1 0 wexp
 tof 400.0 wbs
 nt 320 wnt
 ct 320
 alock n
 gain not used
 FLAGS
 il n
 in n
 dp Y
 DISPLAY
 sp -317.6
 wp 4506.5
 vs 185
 sc 0
 wc 265
 hzmm 0.26
 ls 290.86
 xfl 317.6
 xfp 0
 th 20
 ins 100.000
 nm ph

13C OBSERVE

exp6 std13c

SAMPLE		DEC. & VT	
date	Mar 25 2005	dfrq	299.942
solvent	CDCl3	dn	R1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.428	dm	YYY
tn	C13	dmm	w
at	1.815	dmf	9900
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	5.5		
d1	1.000	werr	
tof	400.0	wexp	
nt	5000	wbs	
ct	5000	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1432.4		
wp	18761.4		
vs	173		
sc	8		
wc	268		
hzmm	23.10		
is	500.00		
rfl	7240.1		
rfd	5807.3		
th	23		
ins	100.000		
nm	ph

19F OBSERVE
STANDARD PARAMETERS

exp5 s2pul

SAMPLE		DEC. & VT	
date	Nar 25 2005	dfreq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfreq	282.197	dm	nan
tn	F19	dmm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	lb	0.30
fb	not used	wtfile	
bs	8	proc	ft
tpwr	58	fn	not used
pw	10.0		
d1	4.000	werr	
tof	400.0	wexp	
nt	320	wbs	
ct	320	wnt	
alock	n		
gain	2		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	5698.6		
wp	49996.9		
vs	626		
sc	0		
wc	265		
hmm	0.63		
ls	40276.65		
rfl	-5695.5		
rtp	0		
th	28		
ins	100.000		
nm	no	ph

67.281
67.302
67.583
67.605

-30 -50 -70 -90 -110 -130 -150 -170 ppm

7a

13C OBSERVE

exp2 std13c

SAMPLE		DEC. & VT	
date	May 25 2005	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpr	43
ACQUISITION		dof	0
sfrq	75.428	dm	Y7Y
tn	C13	dmm	W
at	1.815	dmf	9900
np	68106	PROCESSING	
ew	18761.7	lb	1.00
fb	10400	wtfile	
be	16	proc	ft
tpvr	35	fn	not used
pw	5.5		
d1	0.500	weir	
tof	400.0	weap	
nt	3000	wbe	
ct	3000	wnt	
alock	n		
gain	28		
FLAGS			
al	n		
in	n		
dp	y		
DISPLAY			
sp	-1434.4		
wp	18761.4		
vs	200		
sc	0		
wc	265		
harm	1.73		
ie	500.00		
rf1	7242.1		
rfp	5807.3		
th	39		
lne	100.000		
nm	ph

19F OBSERVE
STANDARD PARAMETERS

exp7 s2pul

SAMPLE		DEC. & VT	
date	Apr 11 2005	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	282.197	dm	nmn
tn	F19	dmm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	lb	0.50
fb	not used	wtfile	
bs	4	proc	ft
tpwr	58	fn	not used
pw	5.0		
d1	4.000	werr	
tof	400.0	wexp	
nt	320	wbs	
ct	320	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	5781.0		
wp	49996.9		
vs	2050		
sc	0		
wc	265		
hazmm	188.67		
is	807.33		
rfl	8020.0		
rfp	13797.9		
th	165		
ins	100.000		
nm	ph

7a

-70.082
-70.417

ppm

13C OBSERVE

exp2 std13c

SAMPLE DEC. & VT
date Nov 15 2004 dfrq 299.942
solvent CDCl3 dn H1
file exp 43
ACQUISITION dof 0
sfrq 75.428 dm yyy
tn Cl3 dnm w
at 1.815 dmf 9900
np 68106 PROCESSING
sw 18761.7 lb 1.00
fb 10400 wfile
bs 16 proc
tpwr 55 fn not used
pw 5.5
dl 0.500 werr
tof 400.0 wexp
nt 10000 wbs
ct 2000 wnt
alock n
gain 28
FLAGS
il n
in n
dp y
DISPLAY
sp -1435.3
vp 18761.4
vs 176
sc 0
wc 265
hmm 5.24
is 500.00
rf1 7242.9
rfp 5807.3
th 18
ins 100.000
nm ph

19F OBSERVE
STANDARD PARAMETERS

exp4 s2pul

SAMPLE		DEC. & VT	
date	May 20 2005	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpr	30
ACQUISITION		dof	0
sfrq	282.197	dm	nnn
tn	F19	dmm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	lb	0.30
fb	not used	wtfile	
bs	16	proc	ft
tpwr	58	fn	not used
pw	5.0		
d1	4.000	werr	
tof	400.0	wexp	
nt	320	whs	
ct	288	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	5611.6		
wp	49996.9		
vs	200		
sc	0		
wc	265		
hzmm	2.55		
is	1001.26		
rfl	8132.9		
rfp	13741.5		
th	20		
ins	100.000		
nm	ph

-64.501
-64.533
-66.394
-66.426

ppm