A Novel Redox Reaction between 8-Aza-5,7-dimethyl-2-trifluoromethylchromone and Alkyl Mercaptoacetates. Facile Synthesis of CF₃-containing 2-Pyridone Derivatives

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General experimental methods

^1H , ^{19}F and ^{13}C NMR spectra were recorded at 400, 376 and 100 MHz, respectively; chemical shifts are given in δ ppm downfield from tetramethylsilane and hexafluorobenzene as internal standards. IR spectra were measured in Nujol or KBr. Melting points are uncorrected. The starting 8-azachromones **5a,b** were prepared by condensation of 3-acetyl-4,6-dimethyl-2-pyridone with $\text{R}^{\text{F}}\text{CO}_2\text{Et}$ according to the described procedure.¹⁰ Pyrazol **9** and aminoenone **10** were described previously.¹³

S3

ppm

6.7302

4.4508
4.3558
4.3579
4.3290
4.3207
4.3117
4.3023
4.2932
4.2772
4.2683
4.2594
4.2504
4.2416
4.2326
4.2247
4.1834

3.2734
3.2040

2.8417
2.6430
2.6223
2.6136
2.5937
2.4187

1.6558
1.3395
1.3321
1.3042

Current Data Parameters
NAME S03547
EXPNO 1
PROCNO 1
USER utatomr

F2 - Acquisition Parameters
Date_ 20020417
Time 14.58
INSTRUM dxt400
PROBHD 5 mm BBO BB/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SW 11.9692 ppm
FIDRES 0.146157 Hz
AQ 3.4210251 sec
RG 362
TW 104.403 usec
DE 5.00 usec
TE 300.2 K
IC 1.2030000 sec

----- CHANNEL f1 -----
NUC1 1H
P 10.10 usec
PL1 -2.00 dB
SF01 400.1120017 MHz

F2 Processing Parameters
SI 32768
SF 430.1320274 MHz
SA 7.41 Hz
RG 20
H2PPT 0.146157 Hz
KDA 20
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.50 cm
FIP 7.030 ppm
F2P 1.360 ppm
F3P 0.27273 ppm/cm
H2CM 109.12637 Hz/cm

Spectrum No 37 7735

S4

Nitro

13.17

F2 - Acquisition Parameters
 Date_ 20021030
 Time 11:11
 INSTRUM spect
 PROBE 5 mm SBC 3B/7
 PULPROG zgpg30
 TD 32768
 SFO100 100.625
 RG 16
 DS 2
 SO 16.3703 270m
 OLDF 7.000 200
 FIDRES 5.195625 Hz
 AQ 2.5559420 sec
 RG 326
 IN 18.000 usec
 DE 6.00 usec
 TE 298.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUCL 13C
 P1 10.00 usec
 PL -2.00 dB
 SFO1 400.528000 MHz

F2 - Processing parameters
 SI 32768
 SF 500.130088 MHz
 SP 5.78 Hz
 SFO2 0.195625 Hz
 WDW EM
 SSF 0
 LB 0.00 Hz
 GB 0
 PC 4.00
 1D NMR plot parameters
 CX 23.00 C
 ZF 14.000 200
 ZG 0.500 200
 PPMCM 0.61364 ppm/cm
 BACK 205.53452 Hz/cm

6.62

X

Integrat

Page: 5*10933

473-474

mez;B*13473

neg:3*13539

Current Data Parameters	
INME	\$08.58
EXPO	1
PRCNO	1
USER	prajmug

F2 - Acquisition Parameter=

[illegible]

===== TJ GREENE =====

KCl	13
PI	9.35 μ sec
PLI	2.30 dB
3502	40% 1.92809 MHz

K2 - Processing parameter:

ST	32769	
SF	400.1502087 MHz	
SE	8.71 Hz	
52qpc	0.19565 Hz	
WFW	EM	
SSE	0	
L3	0.00 Hz	
G5	0	
TC	4.02	

10 THE 101 PARAMETERS

OX	22.00 cm
F-P	13.500 ppm
F2P	1.070 ppm
P25TM	3.56818 ppm/cm
H2O	227.34559 Hz/cm

era: B*13333