Simple Preparation of α -Diazo Esters

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Experimental Section

General. ^1H NMR (at 300 MHz) and ^{13}C NMR (at 300 MHz) spectra were obtained as solutions in deuteriochloroform (CDCl_3). The infrared (IR) spectra were determined as neat oils. R_f values indicated refer to thin layer chromatography (TLC) on 5.0 x 10 cm, 250 μm analytical plates coated with silica gel 60, F₂₅₄, developed in the solvent system indicated. Column chromatography was carried out on MPLC using silica gel 60 particle size 0.015 – 0.040 mm. The solvent mixtures reported are volume/volume mixtures. H is hexane. EtOAc is ethyl acetate. All glassware was oven dried and reactions were carried out under a flow of nitrogen. All reactions were stirred magnetically, under dry N_2) unless otherwise noted.

Amplified on: Unity Plus-300-M

Amplified by: G. Green / H. Elton

Acquired on Unity Plus-300-MB

Acquired by: J. Groves / M. Kilian

000002-04A

000000

DATE: 06-28-04

INSTRUMENT: CPMX-3

C13 PPM: 79.43 MHz.

FILE: 0000020004.231304

Acquired on: Dufay Plus-300-M

Acquired by: S. Gross / M. Kline

CD3 acetone

CD3 acetone

CD3 acetone

all protonated acetone

Acquired on Unity Plus-300-M

Acquired by: J. Guo / M. Elise

Acquired on: NMR-300-M

Acquired by: J. Gross / M. Kilm

4b

NAME: 4b

DATE: 06-21-04

INSTRUMENT: CPMX-3

CDCl₃ solvent: 75.43 MHz

PROB: 1H/13C QNP 300

Acquired on: July 25-2004 Acquired by: S. Green / R. Kline

CD3 acetone

CD3 acetone

CD3 acetone

All protonated carbons

Reprinted by: J. Simon / M. Kilian

Acquired on: Nalby Plus-100-M

Acquired by: V. Brown / K. Allen

CDCl₃ solution

CDCl₃ solution

CDCl₃ solution

all protonated carbons

Acquisition on: 04/15/2004 15:00:00

Acquisition by: J. G. G. / M. K. K.

CDCl₃ spectrum

CDCl₃ spectrum

CDCl₃ spectrum

all protonated carbons

NAME: 4c

FORMULA: C₁₀H₁₀O₂

DATE: 04-15-04

INSTRUMENT: Bruker

CDCl₃ solvent: 75.43 MHz

FILE: 0404170004.2137304

Acquired on: Unity 300-400 Acquired by: J. S. Green / M. Kline

800000-832A

XXXXXXXX

DATE: 06-28-04

SOLVENT: CDCl₃

CL1 Prog: 75.43 MHz.

FILE: 0000000004.1000000

Acquired on: Unity Plus-300-M

Acquired by: S. Brown / M. Kline

4d

CD3 carbons

CD2 carbons

CH carbons

all protonated carbons

220 200 180 160 140 120 100 80 60 40 20 ppm

NAME: 4d

EXPNO: 1

DATE_06-28-04

PROBHD: 5mm

P1: 7.00 sec

PCPD: 1.00 sec

Regulated by: J. Gross / M. Elms

Acquired on: Sat May 17 1996 10:00 AM

Acquired by: J. S. Jones / N. K. Jones

INSTRUMENT: Bruker

PROBHD: 5mm

NAME: 4e-13-04

ACQUISITION: 00013

13C Freq: 125.76 MHz

1H Freq: 500.13 MHz

Acquired on: Unity Plus-300-MX Acquired by: S. Grove / M. Elias

CD3 carbons

CD2 carbons

CD carbons

all protonated carbons

Acquired on: Valley Plus-300-MB

Acquired by: J. S. Groves / H. Kilian

Acquired on: May 21 2004

Acquired by: J. Groves / M. Hill

EXPNO: 80A

PROCNO: 1

DATE_1: 07-01-04

PROBHD: 5MM QNP 1H/13

C13 PPM: 75.43 MHz

PCPD: 4007913804.1856140

Acquired on: Daily 31st-100-4m

Acquired by: J. Groves / M. Elise

CD3 acetone

CD2 acetone

CD acetone

all protonated acetone

PROBHD-80A

PROBHD-80A

DATE: 07-01-04

ACQUANT: CQCL3

Q13 PPM: 75.43 MHz

FILE: 000701004.100010

Acquired on: NMR 200-03

Acquired by: J. Green / M. Elise

000001-03

00000000

DATE: 07-26-04

ACQUAINT: CROCK

EX PRGM: 200.03 MHz

FILE: 0007000004.1010400

Acquired on: Delsy Plus-500-M

Acquired by: J. Groves / M. Elias

NAME: 02A

DATE: 06-29-04

INSTRUMENT: CQX-500

PROB: 5mm QNP 1H/13

CT: 13 PPM: 75.43 MHz

FILE: h006282804.104370

Acquired on: Unity Plus-300-M

Acquired by: J. Stoeck / M. Elias

CD3 carbons

CD3 carbons

CD3 carbons

all protonated carbons

Acquired on: NMR-300-M

Acquired by: J. S. Brown / W. Kline

Acetylated and methylated

acetylated and methylated

INSTRUMENT: JEOL

DATE: 06-28-04

SOLVENT: CDCl3

C13 freq: 75.45 MHz

FILE: 0000000001.105150

Acquired on: Unity Flow-300-M

Acquired by: J. Simon / M. Klean

2h

060323-36A

00000000

DATE: 11-11-03

060323-36A

EX: 17000: 100.02 MHz.

FILE: 060323-36A

Acquired on Varity Plus-300-M

Acquired by: J. Gross / M. Elias

2h

000023-36A

INSTR: 11-11-03

ACQNAME: C00023

C13 PPM: 75.45 MM.

PTIME: 001113003.180314

Registered on: Valley Plan-300-MA

Acquired by: J. Gross / H. Illies

END OF THE LINE

CMF carbons

CO₂ emissions

all protonated carbons

00000000-0000

Abstract

INDEX: 11-11-03

PLACING IN JAIL

Call Freq: 75.65 MHz.

FILE: 2011112003, 1960460

Acquired on: Daltz 250-300-M

Acquired by: G. Weiss / M. Kline

000012-200

00000000

00000000 10-20-03

00000000 00000000 00000000

00000000 00000000 00000000

00000000 00000000 00000000

CC(C)(C)OC(=O)C1=CC=CC=C1

21

21

21

21

21

21

21

21

21

21

21

21

21

21

21

21

21

21

Acquired on: Bruker 500-100-M

Acquired by: S. Simon / M. Elise

CDCl₃ carbons

CDCl₃ carbons

CDCl₃ carbons

all protonated carbons

400132-10A 00000000

DATE: 10-21-03

ACQ/PRG: CDE/LS

CDCl₃ PPM: 75.43 MHz

FILE: 0010312003.1040040

INDEX: 07-26-04

POWELL: CORDA

CLD Page: 79.00

FILE: 2007212006, 20082434

6

Acquired on: Bruker Plus-300-M

Acquired by: J. Gross / M. Kline

040413-230

NAME:

DATE: 10-01-03

SOLVENT: CDCl3

IL Prog: 300.03 MHz.

FILE: mol0030003.1951313

Acquired on Unity Plus-300-MK

Acquired by: J. Simon / M. Elias

6

March-2000

NAME: 18-01-03

SEQUENCE: C13C13

C13 PPM: 75.48 MHz.

FILE: m01002003.000210

Acquired on: Bruker P100-300-M

Acquired by: J. Garcia / M. Kline

6

CDCl₃ spectrum

CDCl₃ spectrum

CDCl₃ spectrum

All protonated carbons

Acquired on: Unity Plus-300-MX

Acquired by: J. Groves / M. Elise

8

400000-420

00000000

DATE: 08-21-04

SOLVENT: CDCl3

CD3 Proq: 75.65 mm.

FILE: M0512004.2035120

Acquired on: Unity Plus-300-M

Acquired by: J. Green / B. Ellis

CD3 carbon

CD3 carbon

CD3 carbon

all protonated carbon

Acq: 03-03-04

DATE: 08-21-04

NAME: CHCL3

CD3 Procs: 75.45 MHz.

FILE: 000000000.194330

Acquired on: Unity Plus-300-M

Acquired by: Z. Green / R. Hines

NAME: 17C

NAME

DATE: 00-17-03

PROC/TYPE: CDECA

IN: PPM: 200.01 MHz.

FILE: 00010001.101724h

sh06b3-17e

NAME: 08-22-03

ANALYST: CXCAS

CL3 FROM: 75.43 MHz.

FILE: sh06b323003.188703e

Acquired on: Unity Plus-300-M

Acquired by: J. Brown / M. Kline

CD3 carbons

CD2 carbons

CH carbons

All protonated carbons