**Chelation-Accelerated Sequential
Decarbonylation of Formate and
Alkoxycarbonylation of Aryl Halides by the
Combined Use of Ru and Pd Catalyst**

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Table of Contents

R = aryl, vinyl, allylic, imidoyl

X = halide, triflate, carbonate

Current Data Parameters

NAME	sj7
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	11.53
INSTRUM	Spect
PROBHD	5 mm Dual 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIDRES	0.156983 Hz
AQ	3.1850996 sec
RG	456.1
DM	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	¹ H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.1300028 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

2-pyridylmethyl benzoate (2a)

-S3-

Current Data Parameters
NAME sj7
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 500000
Time 12.04
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 231
DS 2
SWH 19841.270 Hz
FIDRES 0.302754 Hz
AQ 1.6515572 sec
RG 4096
DM 25.200 usec
DE 6.00 usec
TE 300.0 K
D12 0.0002000 sec
PL13 23.00 dB
D1 1.0000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 250.1310005 MHz
NUC2 13C
PL2 -3.00 dB
PL12 19.00 dB
P1 7.60 usec
DE 6.00 usec
SF01 62.9031573 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters

SI 32768
SF 62.8952140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 12579.04 Hz
F2P -10.000 ppm
F2 -628.95 Hz
PPMCM 10.50000 ppm/cm
HZCM 650.39972 Hz/cm

Current Data Parameters

NAME	SI
EXPNO	56
PROCNO	1

F2 - Acquisition Parameters

Date..	Time	500000
Time	6.34	
INSTRUM	spect	
PROBHD	5 mm QNP 13	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	5144.033 Hz	
FIDRES	0.156983 Hz	
AQ	3.185096 sec	
RG	256	
DM	97.200 usec	
DE	6.00 usec	
TE	300.0 K	
D1	1.00000000 sec	
P1	11.50 usec	
DE	6.00 usec	
SFO1	250.1315447 MHz	
NUC1	¹ H	
PL1	-3.00 dB	

F2 - Processing parameters

SI	16384
SF	250.130053 MHz
MDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

2-Pridylmethyl 4-methylbenzoate (2b)

-S5-

ppm

Current Data Parameters
NAME S17
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 10.03

INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
DS 2

SMH 19941.270 Hz
FIDRES 0.302754 Hz
AQ 1.651572 sec
RG 4096

DM 25.200 usec
DE 6.00 usec
TE 300.0 K

D12 0.0002000 sec
PL13 23.00 dB
D1 1.00000000 sec

CPOPRG2 waltz16
PCPD2 100.00 usec
SF02 250.131005 MHz
NUC2 1H

PL2 -3.00 dB
PL12 19.00 dB
P1 7.60 usec
DE 6.00 usec

SF01 62.9031573 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing Parameters

SI 32768
SF 62.8952140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 12579.04 Hz
F2P -10.000 ppm
F2 -628.95 Hz
PPMCM 10.50000 ppm/cm
HZCM 660.39972 Hz/cm

Current Data Parameters

NAME	SI
EXPNO	8
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
500000	7.54
INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIDRES	0.156983 Hz
AQ	3.1850966 sec
RG	574.7
DM	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	Value
SI	16384
SF	250.1300028 MHz
MDM	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

Parameter	Value
CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

Current Data Parameters
NAME sj7
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters

Date_ 500000
Time 8.00
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 434
DS 2
SMH 19841.270 Hz
FIDRES 0.302754 Hz
AQ 1.6515572 sec
RG 4096
DM 25.200 usec
DE 6.00 usec
TE 300.0 K
D12 0.00002000 sec
PL13 23.00 dB
D1 1.00000000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SFO2 250.1310005 MHz
NUC2 1H
PL2 -3.00 dB
PL12 19.00 dB
P1 7.60 usec
DE 6.00 usec
SFO1 62.9031573 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters

SI 32768
SF 62.8952140 MHz
MDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 12579.04 Hz
F2P -10.000 ppm
F2 -628.95 Hz
PPMCM 10.50000 ppm/cm
HZCM 660.39972 Hz/cm

Current Data Parameters

NAME sj7

EXPNO 116

PROCNO 1

F2 - Acquisition Parameters

Date_ 500000

Time 13.42

INSTRUM spect

PROBHD 5 mm Dual 13

PULPROG zg30

TD 32768

SOLVENT CDCl₃

NS 8

DS 2

SWH 5144.033 Hz

FIDRES 0.156983 Hz

AQ 3.1850936 sec

RG 512

DM 97.200 usec

DE 6.00 usec

TE 300.0 K

D1 1.0000000 sec

P1 11.50 usec

DE 8.00 usec

SFO1 250.1315447 MHz

NUC1 ¹H

PL1 -3.00 dB

F2 - Processing parameters

SF 16384

SF 250.1300000 MHz

MDM EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 22.00 cm

F1P 11.500 ppm

F1 2876.49 Hz

F2P 1.500 ppm

F2 375.19 Hz

PPMCM 0.45455 ppm/cm

HZCM 113.69546 Hz/cm

2-Pridylmethyl 4-hydroxybenzoate (2d)

-S9-

Current Data Parameters

NAME	117
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	500100
Time	12.51
INSTRUM	spc1
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIRES	0.156983 Hz
RG	3.1850996 sec
DE	574.7
TE	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.130009 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

Current Data Parameters

NAME sj7

EXPNO 6

PROCNO 1

F2 - Acquisition Parameters

Date_ 500000

Time 12.58

INSTRUM spect

PROBHD 5 mm Dual 13

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 222

DS 2

SMH 19841.270 Hz

FIDRES 0.302754 Hz

AQ 1.651572 sec

R6 4096

DM 25.200 usec

DE 6.00 usec

TE 300.0 K

D12 0.00002000 sec

PL13 23.00 dB

D1 1.00000000 sec

CPDPRG2 waltz16

PCPD2 100.00 usec

SFO2 250.131005 MHz

NUC2 1H

PL2 -3.00 dB

PL12 19.00 dB

P1 7.60 usec

DE 6.00 usec

SFO1 62.9031573 MHz

NUC1 13C

PL1 -3.00 dB

D11 0.03000000 sec

F2 - Processing parameters

SI 32768

SF 62.8952140 MHz

MDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

10 NMR plot parameters

CX 20.00 cm

F1P 220.000 ppm

F1 13836.55 Hz

F2P -10.000 ppm

F2 -628.55 Hz

PPMCM 11.50000 ppm/cm

HZCM 723.29496 Hz/cm

Current Data Parameters

NAME	SI
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
500000	11.53
INSTRUM	spect
PROBHD	5 mm Dui1 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIDRES	0.156983 Hz
AQ	3.1850996 sec
RG	574.7
DM	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	¹ H
PL1	-3.00 dB

F2 - Processing Parameters

SI	Value
SI	16384
SF	250.1300059 MHz
MDN	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

Parameter	Value
CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCH	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

ppm

Current Data Parameters
NAME S17
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 12.00

INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 472
DS 2

SMH 19841.270 MHz
FIDRES 0.302754 Hz
AQ 1.6515572 sec
RG 4096

DM 25.200 usec
DE 6.00 usec
TE 300.0 K

D12 0.0002000 sec
PL13 23.00 dB
D1 1.00000000 sec

CPDPRG2 waitz16

PCPD2 100.00 usec
SF02 250.131005 MHz

NUC2 1H
PL2 -3.00 dB
PL12 19.00 dB

P1 7.60 usec
DE 6.00 usec

SF01 62.9031573 MHz
NUC1 13C
PL1 -3.00 dB

D11 0.03000000 sec

F2 - Processing parameters

SI 32768
SF 62.8952140 MHz

MDM EM
SSB 0

LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm

F1 12579.04 Hz
F2P -10.000 ppm

F2 -628.95 Hz
PPMCH 10.50000 ppm/cm
HZCM 660.39972 Hz/cm

Current Data Parameters

NAME	sj7
EXPNO	18
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	14.35
INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIDRES	0.156983 Hz
AQ	3.185096 sec
RG	645.1
DM	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.1300037 MHz
MDN	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

ppm

165.325
155.916
149.949
137.296
136.570
133.134
132.134
130.447
128.816
123.462
122.932
122.298

78.014
77.505
76.996
68.002

Current Data Parameters
NAME sj7
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters

Date_ 500000
Time 13.00
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 277
DS 2

SNH 19841.270 Hz
FIDRES 0.302754 Hz
AQ 1.6515572 sec

RG 4096
DM 25.200 usec
DE 6.00 usec
TE 300.0 K

D12 0.00002000 sec
PL13 23.00 dB
D1 1.00000000 sec

CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 250.131005 MHz

NUC2 1H
PL2 -3.00 dB
PL12 19.00 dB

P1 7.60 usec
DE 6.00 usec
SF01 62.9031573 MHz

NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing Parameters
SI 32768
SF 62.8952140 MHz

MDM EM
SSB 0
LB 1.00 Hz

GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm

F1 12579.04 Hz
F2P -10.000 ppm
F2 -628.95 Hz

PPMCM 10.50000 ppm/cm
HZCM 650.39972 Hz/cm

Current Data Parameters

NAME	sj7
EXPNO	24
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	10.45
INSTRUM	spect
PROBHD	5 mm QNP 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIDRES	0.155983 Hz
AQ	3.185096 sec
RG	645.1
DM	97.200 usec
DE	5.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.1300950 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

Current Data Parameters
NAME sj7
EXPNO 25
PROCNO 1

F2 - Acquisition Parameters

Date_ 500000
Time 10.52
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 963
DS 2
FIDRES 19841.270 Hz
AQ 0.302754 Hz
RG 1.6515572 sec
DE 4096
DM 25.200 usec
TE 6.00 usec
D12 0.0002000 sec
PL13 23.00 dB
D1 1.0000000 sec
CPDPRG2 waitz16
PCPD2 100.00 usec
SF02 250.131005 MHz
NUC2 1H
PL2 -3.00 dB
PL12 19.00 dB
P1 7.60 usec
DE 6.00 usec
SF01 62.9031573 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.0300000 sec

F2 - Processing parameters

SI 32768
SF 62.8952140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 175.000 ppm
F1 11006.66 Hz
F2P 60.000 ppm
F2 3773.71 Hz
PPMCM 5.75000 ppm/cm
HZCM 361.64749 Hz/cm

Current Data Parameters

NAME	516
EXPNO	114
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	13.48
INSTRUM	spect
PROBHD	5 mm Dn1 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SMH	5144.033 Hz
FIDRES	0.156983 Hz
AQ	3.185096 sec
RG	161.3
DM	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.130015 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	9.131 ppm
F1	2283.84 Hz
F2P	-0.317 ppm
F2	-79.33 Hz
PPMCK	0.42944 ppm/cm
HZCM	107.41675 Hz/cm

Current Data Parameters

NAME sj7

EXPNO 96

PROCNO 1

F2 - Acquisition Parameters

Date_ 500000

Time 0.24

INSTRUM spect

PROBHD 5 mm Dual 13

PULPROG zgpg30

TD 65536

SOLVENT CDCl₃

NS 15360

DS 2

SMH 19841.270 Hz

FIDRES 0.302754 Hz

AQ 1.6515572 sec

RG 6502

DM 25.200 usec

DE 6.00 usec

TE 300.0 K

D12 0.0002000 sec

PL13 23.00 dB

D1 1.00000000 sec

CPDPRG2 temp

P31 100.00 usec

SFO2 250.1310005 MHz

NUC2 ¹³C

PL2 -3.00 dB

PL12 19.00 dB

P1 7.60 usec

DE 6.00 usec

SFO1 62.9031573 MHz

NUC1 ¹³C

PL1 -3.00 dB

D11 0.03000000 sec

F2 - Processing parameters

SI 32768

SF 62.8952140 MHz

MDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

10 NMR plot parameters

CX 20.00 cm

F1P 192.016 ppm

F1 12076.88 Hz

F2P -10.992 ppm

F2 -691.35 Hz

PRNCM 10.15039 ppm/cm

HZCM 638.41119 Hz/cm

Current Data Parameters
 NAME sj7
 EXPNO 27
 PROCNO 1

F2 - Acquisition Parameters

Date_ 500000
 Time 11.49
 INSTRUM spect
 PROBD 5 mm Dual 13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1243
 DS 2
 SWH 19841.270 Hz
 FIDRES 0.302754 Hz
 AQ 1.6515572 sec
 RG 4096
 DM 25.200 usec
 DE 6.00 usec
 TE 300.0 K
 D12 0.0002000 sec
 PL13 23.00 dB
 D1 1.0000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 250.131005 MHz
 NUC2 ¹H
 PL2 -3.00 dB
 PL12 19.00 dB
 P1 7.60 usec
 DE 6.00 usec
 SF01 62.9031573 MHz
 NUC1 ¹³C
 PL1 -3.00 dB
 D11 0.03000000 sec

F2 - Processing parameters

SI 32768
 SF 62.8952140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 200.000 ppm
 F1 12579.04 Hz
 F2P -10.000 ppm
 F2 -628.95 Hz
 PRNOM 10.50000 ppm/cm
 HZCM 650.39972 Hz/cm

Current Data Parameters

NAME	SI7
EXPNO	30
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
500000	14.45

INSTRUM spect

PROBHD 5 mm Du1 13

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 8

DS 2

SWH 5144.033 Hz

FIDRES 0.156983 Hz

AQ 3.185096 sec

RG 812.7

DM 97.200 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

P1 11.50 usec

DE 6.00 usec

SFO1 250.1315447 MHz

NUC1 1H

PL1 -3.00 dB

F2 - Processing parameters

SI	16384
SF	250.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

2-Pridylmethyl-(E)-4-phenyl-3-butenate (2k)

-S23-

2-Pridylmethyl 1-pyrroline-2-carboxylate (21)

-S24-

Current Data Parameters

NAME	s17
EXPNO	39
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	9.36
INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5144.033 Hz
FIDRES	0.156963 Hz
AQ	3.1850996 sec
RG	1625.5
DM	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

Current Data Parameters

NAME 517

EXPNO 64

PROCNO 1

F2 - Acquisition Parameters

Date_ 500000

Time 3.36

INSTRUM spect

PROBHD 5 mm Dual 13

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 15360

DS 2

SWH 19841.270 Hz

FIDRES 0.302754 Hz

AQ 1.6515572 sec

RG 8192

DM 25.200 usec

DE 6.00 usec

TE 300.0 K

D12 0.00002000 sec

PL13 23.00 dB

D1 1.00000000 sec

CPDPRG2 waltz16

PCPD2 100.00 usec

SFO2 250.131005 MHz

NUC2 1H

PL2 -3.00 dB

PL12 19.00 dB

P1 7.60 usec

DE 6.00 usec

SFO1 62.9031573 MHz

NUC1 13C

PL1 -3.00 dB

D11 0.03000000 sec

F2 - Processing parameters

SI 32768

SF 62.8952140 MHz

MDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

F1P 190.000 ppm

F1 11950.09 Hz

F2P 10.000 ppm

F2 628.95 Hz

PPMCM 9.00000 ppm/cm

HZCM 566.05695 Hz/cm

2-Pridylmethyl-3,4,5,6-tetrahydropyridinecarboxylate (2m)

-S26-

Current Data Parameters

NAME	sj7
EXPNO	22
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	4.47
INSTRUM	spect
PROBHD	5 mm QNP 13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SWH	5144.033 Hz
FIDRES	0.165863 Hz
AQ	3.185096 sec
RG	812.7
DN	97.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	11.50 usec
DE	6.00 usec
SFO1	250.1315447 MHz
NUC1	1H
PL1	-3.00 dB

F2 - Processing parameters

SI	16384
SF	250.1300043 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-0.300 ppm
F2	-75.04 Hz
PPMCM	0.46818 ppm/cm
HZCM	117.10632 Hz/cm

2-Pridylmethyl-3,4,5,6-tetrahydropyridinecarboxylate (2m) -S27-

Current Data Parameters

NAME	s17
EXPNO	23
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	4.54
INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1681
DS	2
SWH	19841.270 Hz
FIDRES	0.302754 Hz
AQ	1.6515572 sec
RG	8192
DM	25.200 usec
DE	6.00 usec
TE	300.0 K
D12	0.00002000 sec
PL13	23.00 dB
D1	1.00000000 sec
CPOPRG2	waitz16
PCPD2	100.00 usec
SFO2	250.131005 MHz
NUC2	13C
PL2	-3.00 dB
PL12	19.00 dB
P1	7.60 usec
DE	6.00 usec
SFO1	62.9031573 MHz
NUC1	13C
PL1	-3.00 dB
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952140 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	12579.04 Hz
F2P	-10.000 ppm
F2	-628.95 Hz
PPMCM	10.50000 ppm/cm
HZCM	650.39972 Hz/cm

Current Data Parameters
NAME s17
EXPNO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 12.28
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 5144.033 Hz
FIDRES 0.156983 Hz
AQ 3.1650996 sec
RG 812.7
DM 97.200 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 11.50 usec
DE 6.00 usec
SF01 250.1315447 MHz
NUC1 1H
PL1 -3.00 dB

F2 - Processing Parameters
SI 16384
SF 250.1300053 MHz
MDM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
FAP 10.000 ppm
F1 2501.30 Hz
F2P -0.300 ppm
F2 -75.04 Hz
PPMCM 0.46818 ppm/cm
HZCM 117.10632 Hz/cm

ppm

173.717

155.589

149.634

137.723

123.740

123.131

122.781

117.671

77.973

77.465

76.956

67.086

44.195

34.027

29.766

25.581

24.226

Current Data Parameters
NAME s17
EXPNO 76
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000

Time 3.45

INSTRUM spect

PROBHD 5 mm Dual 13

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 15360

DS 2

SMH 19841.270 Hz

FIDRES 0.302754 Hz

AQ 1.651572 sec

RG 5792.6

DN 25.200 usec

DE 6.00 usec

TE 300.0 K

D12 0.0002000 sec

PL13 23.00 dB

D1 1.00000000 sec

CPDPRG2 mltz16

PCPD2 100.00 usec

SFO2 250.1310005 MHz

NUC2 1H

PL2 -3.00 dB

PL12 19.00 dB

P1 7.60 usec

DE 6.00 usec

SFO1 62.9031573 MHz

NUC1 13C

PL1 -3.00 dB

D11 0.03000000 sec

F2 - Processing parameters

SI 32768

SF 62.8952140 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

F1P 187.889 ppm

F1 11817.31 Hz

F2P 11.963 ppm

F2 752.41 Hz

PPMCM 8.79630 ppm/cm

HZCM 553.24493 Hz/cm

2-Pridylmethyl 1-aza-1-cyclononene-2-carboxylate (2o)

-S30-

2-Pridylmethyl 1-aza-1-cyclononene-2-carboxylate (2o)

-S31-

ORTEP of the ruthenium cluster 3

Table 1. Crystal data and structure refinement for 3.

Empirical formula	$C_{20} H_{12} N_2 O_{10} Ru_3$	
Formula weight	270.37	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Monoclinic, $P2(1)/c$	
Unit cell dimensions	$a = 13.097(3)$ Å	$\alpha = 90$ deg.
	$b = 9.137(2)$ Å	$\beta = 101.843(4)$ deg.
	$c = 20.287(5)$ Å	$\gamma = 90$ deg.
Volume	$2376.0(10)$ Å ³	
Z, Calculated density	11, 2.079 Mg/m ³	
Absorption coefficient	1.940 mm ⁻¹	
F(000)	1432	
Crystal size	0.28 x 0.26 x 0.16 mm	
Theta range for data collection	2.05 to 25.52 deg.	
Limiting indices	$-13 \leq h \leq 15$, $-11 \leq k \leq 11$, $-24 \leq l \leq 22$	
Reflections collected / unique	17585 / 4416 [$R(\text{int}) = 0.0519$]	
Completeness to $\theta = 25.52$	99.8 %	
Max. and min. transmission	0.7466 and 0.6127	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4416 / 0 / 316	
Goodness-of-fit on F^2	1.180	
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0435$, $wR2 = 0.0718$	
R indices (all data)	$R1 = 0.0576$, $wR2 = 0.0754$	
Largest diff. peak and hole	0.539 and -0.469 e. Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 3. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
Ru(1)	3371(1)	3204(1)	1727(1)	29(1)
Ru(2)	2639(1)	1067(1)	768(1)	29(1)
Ru(3)	2764(1)	4271(1)	427(1)	28(1)
O(1)	3499(2)	2291(3)	203(2)	30(1)

N(2)	2006(3)	4385(4)	-624(2)	32(1)
O(9)	1211(3)	-344(4)	1554(2)	50(1)
N(1)	2226(3)	37(4)	-216(2)	33(1)
O(7)	4632(3)	6176(5)	487(2)	63(1)
C(35)	1145(4)	3555(5)	-788(2)	32(1)
C(36)	787(4)	2822(5)	-211(2)	35(1)
C(31)	2350(4)	5108(5)	-1114(2)	39(1)
C(25)	2734(4)	581(5)	-676(3)	35(1)
C(24)	2504(4)	119(6)	-1336(3)	47(1)
C(7)	1770(4)	183(5)	1257(2)	35(1)
C(5)	3925(4)	5449(5)	469(3)	37(1)
O(2)	1545(2)	2763(3)	382(2)	30(1)
O(6)	1060(3)	3382(4)	1792(2)	49(1)
O(10)	4372(4)	-894(5)	1431(2)	69(1)
O(5)	5472(3)	2852(5)	1276(2)	54(1)
O(4)	3785(3)	6240(5)	2364(2)	64(1)
O(3)	3988(3)	1227(5)	2963(2)	65(1)
C(4)	1908(4)	3297(5)	1754(2)	36(1)
C(34)	595(4)	3459(6)	-1442(2)	41(1)
C(8)	3701(4)	-159(6)	1174(3)	41(1)
C(6)	2141(4)	5808(5)	760(2)	34(1)
C(26)	3579(4)	1692(5)	-416(2)	37(1)
C(32)	1832(4)	5052(6)	-1773(3)	46(1)
C(3)	4691(4)	2987(6)	1436(2)	38(1)
C(21)	1477(4)	-974(5)	-416(3)	42(1)
C(1)	3758(4)	1949(6)	2503(3)	40(1)
C(33)	946(4)	4220(6)	-1946(3)	48(1)
C(2)	3677(4)	5096(6)	2131(3)	42(1)
O(8)	1760(3)	6772(4)	984(2)	54(1)
C(22)	1218(4)	-1472(6)	-1068(3)	48(1)
C(23)	1742(5)	-905(6)	-1538(3)	59(2)

Table 3. Bond lengths [Å] and angles [deg] for 3.

Ru(1)-C(2)	1.921(5)	Ru(1)-C(4)	1.930(6)
Ru(1)-C(1)	1.930(6)	Ru(1)-C(3)	1.949(6)
Ru(1)-Ru(3)	2.7693(8)	Ru(1)-Ru(2)	2.7841(7)
Ru(2)-C(8)	1.843(6)	Ru(2)-C(7)	1.843(5)
Ru(2)-O(1)	2.088(3)	Ru(2)-O(2)	2.147(3)
Ru(2)-N(1)	2.171(4)	Ru(2)-Ru(3)	3.0197(9)
Ru(3)-C(6)	1.821(5)	Ru(3)-C(5)	1.851(5)
Ru(3)-O(2)	2.096(3)	Ru(3)-O(1)	2.142(3)
Ru(3)-N(2)	2.163(4)	O(1)-C(26)	1.395(5)
N(2)-C(35)	1.342(6)	N(2)-C(31)	1.346(6)
O(9)-C(7)	1.145(6)	N(1)-C(25)	1.347(6)
N(1)-C(21)	1.348(6)	O(7)-C(5)	1.134(6)
C(35)-C(34)	1.377(7)	C(35)-C(36)	1.505(6)
C(36)-O(2)	1.394(5)	C(31)-C(32)	1.370(7)
C(25)-C(24)	1.379(7)	C(25)-C(26)	1.514(6)
C(24)-C(23)	1.368(8)	O(6)-C(4)	1.131(6)
O(10)-C(8)	1.144(6)	O(5)-C(3)	1.140(6)
O(4)-C(2)	1.144(6)	C(3)-C(1)	1.132(6)
C(34)-C(33)	1.391(7)	C(6)-O(8)	1.151(6)
C(32)-C(33)	1.371(7)	C(21)-C(22)	1.373(7)
C(22)-C(23)	1.385(8)		
C(2)-Ru(1)-C(4)	94.0(2)	C(2)-Ru(1)-C(1)	100.9(2)
C(4)-Ru(1)-C(1)	95.7(2)	C(2)-Ru(1)-C(3)	95.8(2)
C(4)-Ru(1)-C(3)	164.0(2)	C(1)-Ru(1)-C(3)	94.9(2)
C(2)-Ru(1)-Ru(3)	94.80(16)	C(4)-Ru(1)-Ru(3)	85.36(15)
C(1)-Ru(1)-Ru(3)	164.14(16)	C(3)-Ru(1)-Ru(3)	81.20(14)
C(2)-Ru(1)-Ru(2)	160.33(16)	C(4)-Ru(1)-Ru(2)	81.36(14)
C(1)-Ru(1)-Ru(2)	98.57(16)	C(3)-Ru(1)-Ru(2)	85.21(15)
Ru(3)-Ru(1)-Ru(2)	65.88(2)	C(8)-Ru(2)-C(7)	89.2(2)
C(8)-Ru(2)-O(1)	97.36(19)	C(7)-Ru(2)-O(1)	172.88(17)
C(8)-Ru(2)-O(2)	171.01(18)	C(7)-Ru(2)-O(2)	94.20(17)
O(1)-Ru(2)-O(2)	78.92(12)	C(8)-Ru(2)-N(1)	99.81(19)
C(7)-Ru(2)-N(1)	103.93(18)	O(1)-Ru(2)-N(1)	77.81(13)
O(2)-Ru(2)-N(1)	87.47(13)	C(8)-Ru(2)-Ru(1)	89.64(16)

C(7)-Ru(2)-Ru(1)	95.29(15)	O(1)-Ru(2)-Ru(1)	81.99(8)
O(2)-Ru(2)-Ru(1)	81.77(8)	N(1)-Ru(2)-Ru(1)	158.60(10)
C(8)-Ru(2)-Ru(3)	128.31(16)	C(7)-Ru(2)-Ru(3)	128.02(14)
O(1)-Ru(2)-Ru(3)	45.16(8)	O(2)-Ru(2)-Ru(3)	43.94(8)
N(1)-Ru(2)-Ru(3)	103.01(10)	Ru(1)-Ru(2)-Ru(3)	56.825(18)
C(6)-Ru(3)-C(5)	78.8(2)	C(6)-Ru(3)-O(2)	97.37(18)
C(5)-Ru(3)-O(2)	174.46(18)	C(6)-Ru(3)-O(1)	170.03(17)
C(5)-Ru(3)-O(1)	95.73(18)	O(2)-Ru(3)-O(1)	78.86(12)
C(6)-Ru(3)-N(2)	100.27(18)	C(5)-Ru(3)-N(2)	102.90(18)
O(2)-Ru(3)-N(2)	78.28(13)	O(1)-Ru(3)-N(2)	88.07(13)
C(6)-Ru(3)-Ru(1)	88.98(15)	C(5)-Ru(3)-Ru(1)	95.08(16)
O(2)-Ru(3)-Ru(1)	83.00(8)	O(1)-Ru(3)-Ru(1)	81.43(8)
N(2)-Ru(3)-Ru(1)	160.01(10)	C(6)-Ru(3)-Ru(2)	127.84(15)
C(5)-Ru(3)-Ru(2)	129.47(16)	O(2)-Ru(3)-Ru(2)	45.30(9)
O(1)-Ru(3)-Ru(2)	43.72(8)	N(2)-Ru(3)-Ru(2)	103.69(10)
Ru(1)-Ru(3)-Ru(2)	57.296(13)	C(26)-O(1)-Ru(2)	115.7(3)
C(26)-O(1)-Ru(3)	130.0(3)	Ru(2)-O(1)-Ru(3)	91.11(12)
C(35)-N(2)-C(31)	119.1(4)	C(35)-N(2)-Ru(3)	114.1(3)
C(31)-N(2)-Ru(3)	126.6(3)	C(25)-N(1)-C(21)	118.4(4)
C(25)-N(1)-Ru(2)	114.3(3)	C(21)-N(1)-Ru(2)	27.1(3)
N(2)-C(35)-C(34)	121.4(4)	N(2)-C(35)-C(36)	116.1(4)
C(34)-C(35)-C(36)	122.4(4)	O(2)-C(36)-C(35)	114.0(4)
N(2)-C(31)-C(32)	121.9(5)	N(1)-C(25)-C(24)	121.3(5)
N(1)-C(25)-C(26)	115.9(4)	C(24)-C(25)-C(26)	122.8(5)
C(23)-C(24)-C(25)	120.0(5)	O(9)-C(7)-Ru(2)	178.3(5)
O(7)-C(5)-Ru(3)	179.2(5)	C(36)-O(2)-Ru(3)	114.5(3)
C(36)-O(2)-Ru(2)	130.3(3)	Ru(3)-O(2)-Ru(2)	90.76(12)
O(6)-C(4)-Ru(1)	177.4(5)	C(35)-C(34)-C(33)	119.3(5)
O(10)-C(8)-Ru(2)	178.6(5)	O(8)-C(6)-Ru(3)	178.5(5)
O(1)-C(26)-C(25)	113.0(4)	C(31)-C(32)-C(33)	119.6(5)
O(5)-C(3)-Ru(1)	178.9(4)	N(1)-C(21)-C(22)	122.6(5)
O(3)-C(1)-Ru(1)	179.2(5)	C(32)-C(33)-C(34)	118.7(5)
O(4)-C(2)-Ru(1)	175.2(5)	C(21)-C(22)-C(23)	118.7(5)
C(24)-C(23)-C(22)	119.0(5)		