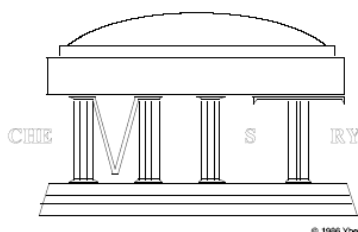


MIT

Massachusetts Institute of Technology
Department of Chemistry



Laboratory Manual

5.301
Chemistry Laboratory Techniques

Prepared by Katherine J. Franz and Kevin M. Shea
with the assistance of Professors Rick L. Danheiser and Timothy M. Swager

Revised by J. Haseltine, Kevin M. Shea, and Sarah A. Tabacco

IAP 2004

5.301

Katherine J Franz Kevin M Shea
Rich L Danheiser Timonthy M Swager
J Haseltine, Kevin M Shea Sarah A Tabacco

IAP 2004

1		
1.1.		3
1.2.		4
1.3.		5
1.4.		6
1.5.		7
1.6.		8
1.7.		9
2.		
2.1. CC "	"	14
2.2. EE		17
3.		
3.1. CC		19
3.2. EE		21
4.		
4.1. CC "	"	24
4.2. EE	?	26
5.		
5.1. CC "	"	28
5.2. EE		30
6.		
6.1. CC "	"	32
6.2. EE "	"	35
7.		
7.1. Mn(salen)		37

8.		
8.1.	FT-NMR	44
8.2	GC	46
8.3	TLC	47
8.4.		50
8.5.		53
8.6.		55
8.7.		57
8.8.		59
8.9.		63
9.		
9.1	NMR	68
9.2	IR	73
9.3	GC	74
9.4	UV-Vis	75

1.

1.1.

5.301

5.301

UROP

UROP

5.301

5

"

" "

"

CC(Competent Chemist) EE(Expert Experimentalist)

CC

CC

EE

EE

"

"

3

5.301

1.3.

1 5

5.301

Zubrick

J. Leonard, B. Lygo G. Procter

5.301

Tabacco

IAP

1 5

Zubrick—

6

1 ,2 ,4 ,6 ,9 ,10
,11 ,15 ,18 ,19 ,31
Leonard, Lygo Procter **2**
1 2 3 4 8

1.4.

" " 5.301 " " " " UROP
" " UROP

" CC "

" EE "

" CC " " EE "

5.301 " CC " " EE "
" EE "

1.5. 5.301

2004 1

				1	2	3
4	5 #1	6 #2 CC	7 #3 CC	8 #4 EE	9 CC EE	10
11	12 #5 CC	13 #6 EE	14 #7 CC	15 #8 EE 1	16 CC 2	17
18	19	20 #9 CC	21 #10 EE	22 #11	23 #11	
				Salen	Mn	

1.6.

9

		"	"	5.301		
	2~6				5	"
" "	" "	" "	" "	" "		"

2~6

	NMR		IR		GC		UV-Vis
	7		IAP				
	8		"	"			
			5.301				
	9			5.301			
NMR	GC	IR	UV-Vis				

1.7.

Mircea Gheorghiu

Scott Virgil

1.

:

1.

2.

3. /

4.

5. ---

--- 100

CO₂

1 6

_____ :

4-454

4-460

(4-450)

2.

()

:

1. —

2.

3. (5.301)

4. 5.301

()

3.

2004 1 30

\$35.00

4.

a.

——4-457

——4-457

——4-454 4-460

b.

4-454 4-460

4-454

c.

d.

_____ LS LS=Lab Supplies

17mm $5\frac{1}{2}$ cm 11cm

5.

Mircea Gheorghiu

Gheorghiu

ACGIH

40

TLV's

ACGIH

TLV

6.

1

2

3

4

5

6

7

8

9

:

a

b

10

11

2.

2.1.

:"

"

NMR

Zubrick 15 LLP 10

Zubrick 37

Zubrick 87-92

NMR Zubrick 35 LLP 15 2

5 1

6 2

100mL

(125mL)

2× 250mL

150mL

100mL

NMR

¹H NMR

¹H NMR 100mg 3- 4
3-



10 HCl 50~70mL

¹H NMR
NMR

3 69 73 " CC " 90mg 3-

¹H NMR

2.2.

"

"

pKa

" CC"

" CC"

100mL

(125mL)

4× 250mL

150mL

100mL

pH

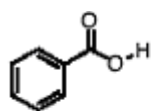
NMR

pKa

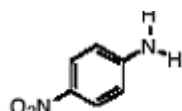
4-
1

100mg

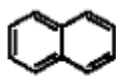
pKa



苯甲酸
MP 122–123 °C
pKa 4.2



4-硝基苯胺
MP 149–151 °C
pKa (RNH₃⁺) 1.0



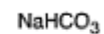
萘
MP 80–82 °C
(no acidic protons)



水
pK_a 15.7



盐酸
pK_a -7



碳酸氢钠
pK_a (H₂CO₃) 6.4

—

—

—

— Na₂CO₃

—6M HCl

—1M NaOH

" EE "

90mg

3

2

¹ Gilbert J. C; Martin S F. *Experimental Organic Chemistry: A Miniscale & Microscale Approach*; 3th ed.; Brooks/Cole: Pacific Grove, CA 93950; p. 141.

3.

3.1.

:"

"

Zubrick 13 LLP 11.2

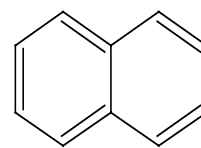
9.

(5 13× 100 mm)
(2× 50-mL, 1× 125-mL)

/
(250mL)

2.00g

1



I.

Zubrick 104-105

II.

50mL

20mL

I

" CC "

1.30g

3

77

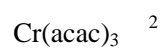
83

¹ Fieser, L. F.; Williamson, K. L. *Organic Experiments*; 7th ed.; D. C. Heath and Company: Lexington, MA, 1992; p. 40.

3.2.

" "

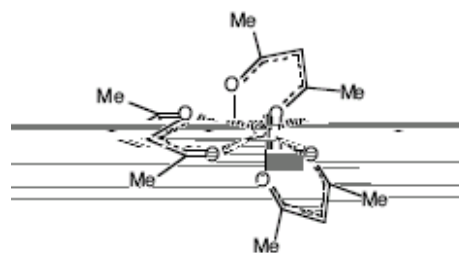
X-



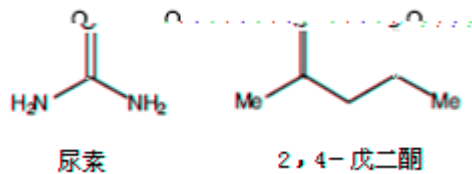
50mL
2mL

250mL
3 4
2

D



² Szafran, Z.; Pike, R. M.; Sing, M. M. *Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience*, Wiley: New York, 1991: "synthesis of Metal Acetylacetonates" p. 224-229.



CrCl ₃ · 6H ₂ O					1.00mmol	1
						17
2,4-						8
Cr(acac) ₃						

2mL 50mL CrCl₃ · 6H₂O

2,4-

1

D

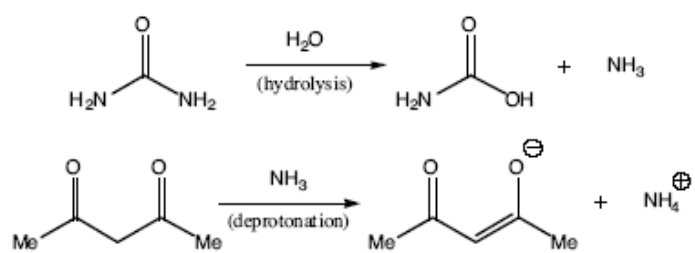
X-

NH₃ pH NH₃

acac 2,4-

2,4-

Cr(acac)₃



X- " EE " 50 Cr(acac)₃

4.

4.1.

"

?"

GC

Zubrick 36 LLP 11 3
 Zubrick 20

GC Zubrick 32

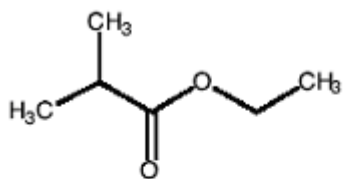
1× 25mL, 1× 50mL

11.20g

40

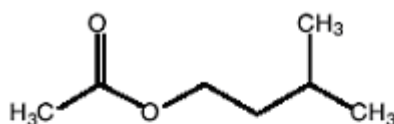
GC

GC



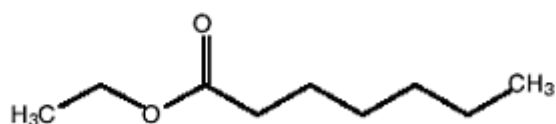
异丁酸乙酯 (桃子)

(BP 142-143 °C)



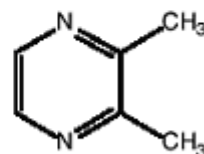
乙酸异戊酯 (香蕉)

(BP 112-113 °C)



庚酸乙酯

(BP 188-189 °C)



2,3-二甲基吡啶

(BP 156 °C)

GC

92 " CC "

7.00g

4.2.

"

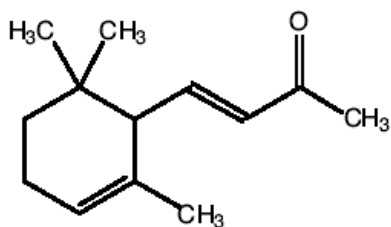
?"

25mL

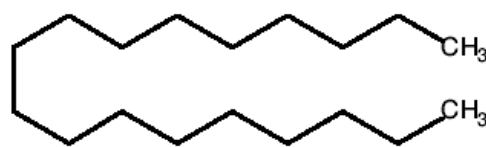
Vigreux

-

7.50g
" CC "



α -Ionone
(BP ~ 265 °C)

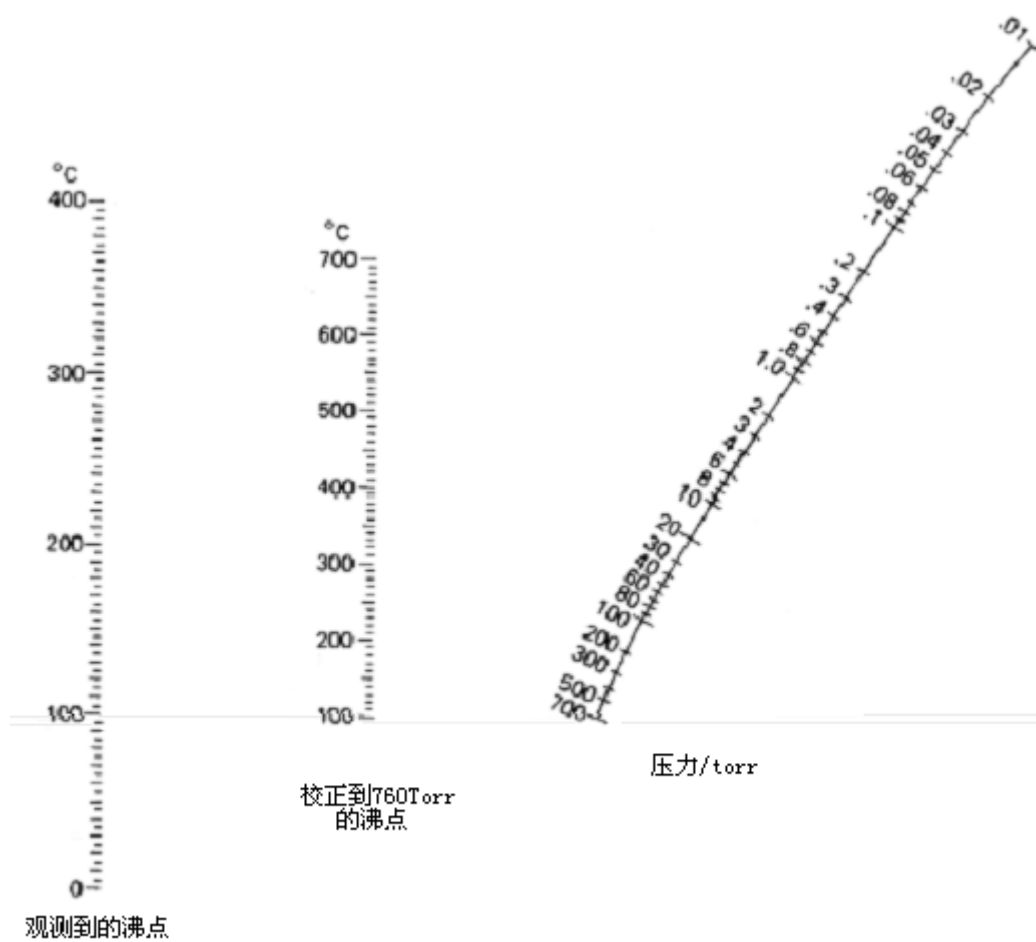


Octadecane
(BP 317 °C)

0.5torr
93

" EE "

4.00g -



5.

5.1.

" "

TLC

TLC	/	Zubrick 27			
		Zubrick 28	LLP	9.3.1	
		Zubrick 29	LLP	11.6	

T

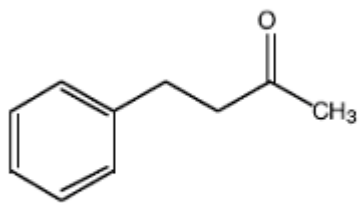
100mL
18× 150 mm

3. TLC

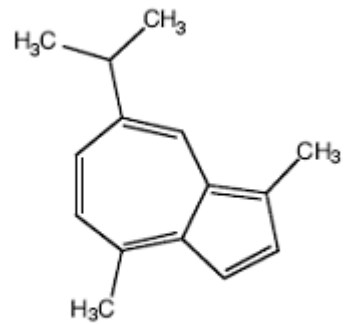
10.

2mL (benzylacetone) / (guaiazulene) 4- -2-
 TLC 10% / TLC
 TLC
 R_f 10% / 50g 5
 10mL 1mL

TLC 4- -2- (benzylacetone) GC
 TLC



4-苯基-2-丁酮



愈创蓝油烃

0.95g 4- -2-

" CC " 95

5.2.

" "

" CC"

3-	20mL	0.2g	(guaiazulene) 0.2g	(fluorenone) 0.2g
	/		TLC	TLC

TLC

TLC

R_f

" EE " "

R_f

TLC

6.

6.1.

"

"

- UV-Vis

100P 1000P

8
Eppendorf
UV-Vis

5mL

Protein Assay C Pierce Coomassie® Plus

Eppendorf

pH 7 3 50µL
25mM MOPS

C

2 Coomassie®

Coomassie® Plus

Coomassie® Plus

3

0.05mL

Eppendorf

3

0.05mL

25mM MOPS

pH 7,

1.5mL Coomassie® Plus

595nm

595nm

595nm

595nm

μ g/mL

" CC "

R 0.930

0.048

6.2.

“ ”

UV-Vis 1mL
20P 100P 1000P

Eppendorf

Eppendorf

“ CC”
C

Ferrozine

UV-Vis 562nm (Fe) -
 A_{562}

Fe AA AA
25mM MOPS PH 7
HNO₃ (5 M)
75 mM C
10 mM

1.

μL of Fe AA standard (99 μg/mL)	μL of Buffer to add
0	300
6	294
12	288
18	282
24	276

30μ L (5M)

Eppendorf Pyrex

30

1 2

300μ L

1020μ L

60μ L 75mM C

60μ L 10mM

60μ L

10 15

1.5mL 562nm

A_{562}

A_{562} [Fe]

[Fe]

" EE " "

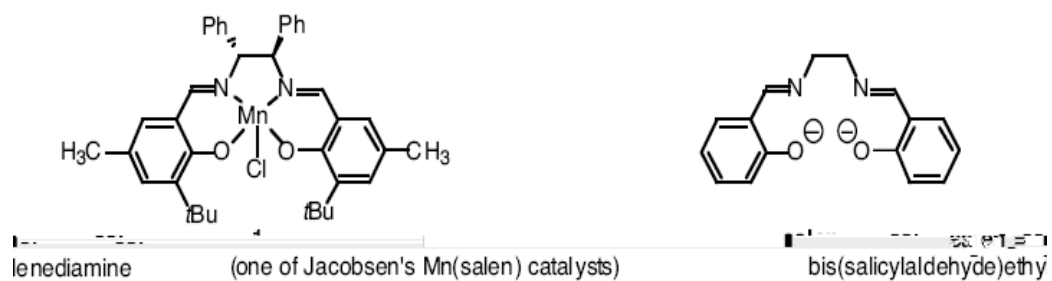
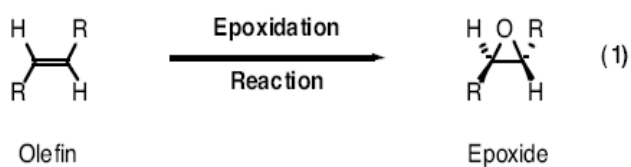
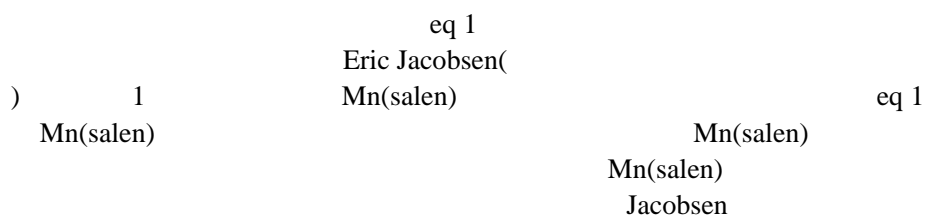
0.995

0.035

7.

7.1. Mn(salen)

:
5.301



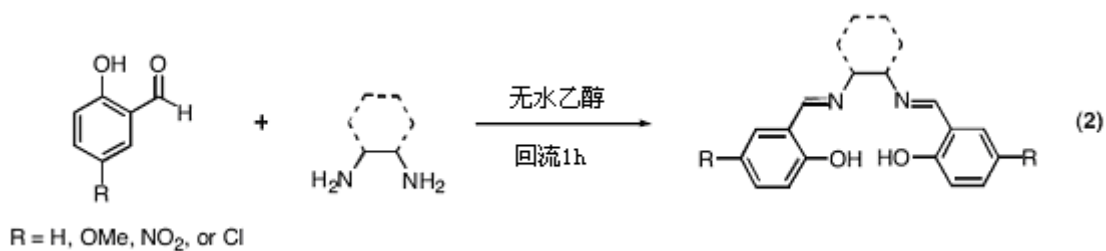
salen (eq 2) ¹H NMR
Mn(salen) (eq 3)

Jacobsen ¹H NMR 1,2-

¹H NMR

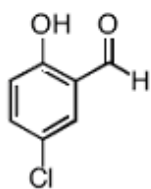
1 **Salen** (eq 2)

Mn(salen)

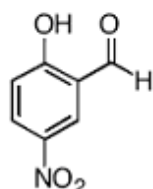


原料:

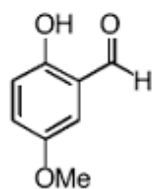
水杨醛衍生物:



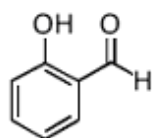
FW = 156.57



FW = 167.12

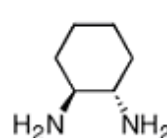


FW = 152.15
d = 1.219

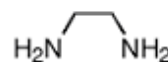


FW = 122.12
d = 1.146

二胺:

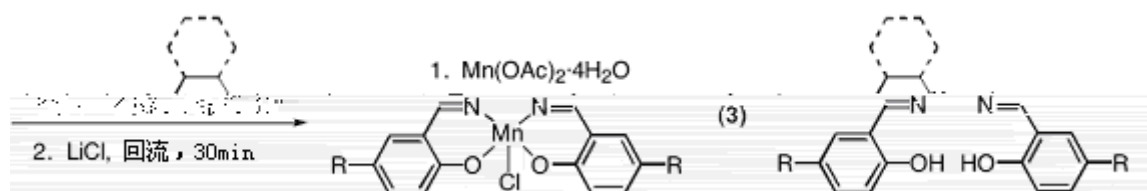


FW = 114.19
d = 0.951



FW = 60.10
d = 0.899

2. Mn(salen) (eq 3)



3. 1,2-

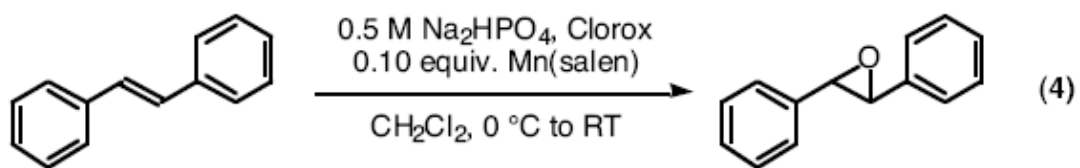
pH

pH

eq 4

pH

TLC



- 1)
- 2) NMR 10mL
- 3) NMR Kimwipe
Pasteur Pasteur Kimwipe
- 4)
- 5) NMR 10
- 6)
- 7) NMR NMR

8) NMR

- NMR**
- 1 1-3
 - 2 10mg
 - 3 1mL
 - 4 NMR
 - 5 6-8

- NMR**
- 1 NMR
 - 2 NMR 1
 - 3 NMR

8.2

GC

GC

Zubrick 252

- | | | | |
|----|---------|-----|------|
| 1) | Pasteur | | 10mg |
| 2) | 1mL | | |
| 3) | | | |
| 4) | 1mL | | |
| 5) | | | |
| | | | |
| 1) | 10mg | 1mL | |
| 2) | | 3-5 | |

8.3. TLC

TLC

5.301

" "

R_f 0 1

LLP145-152

TLC

1)

2)

/ 0~30%
 / 0~40%
 / 5~30%
 / 5~30%

3) 1~2mL

4)

Pasteur

UROP

5)

90%

6)

R_f

7)

8)

5.301
UROP

9)

5.301

10)

R_f

R_f

11)

TLC

R_f

8.4.

Zubrick 127-138

1

2

10g

500mL 1L

125mL 250mL

1

3

50 500mg

25 100mL

4

2~3

10 HCl

1/10~1/2

NaHCO₃ 10 NaOH

5

TLC

6

NaCl

"

"

7

MgSO₄ MgSO₄
Na₂SO₄

Na₂SO₄

8

Zubrick 136-138

9)

10)

11)

12

30

/

13

30

15

NMR

8.5.

LLP 9.2

Schlenk

1

!

14/20

Keck

2

3

!

4

8.6.

1

2

Zubrick 114-117

1)

2)

3)

4) 2mL

5)

15mL

6)

7)

2

2

2

2

2

1

8)

9)

10)

11)

8.7.

1

2

1

2

Celite

Pasture

3

4

3

CH_2Cl_2

/

CHCl_3 /

THF/

/

8.8.

5.301

Vigreux

5.301

1

— LLP 196

2

20 30°C

200°C

3

4

1/2 2/3

5

5.301

UROP

6

7

8

70

70°C

9

10

11

12

10

13

12

14

5°C

15

16

17

1

Perkin

5.301

3

4

2

2~4

3

Zubrick 53-55

4

6~7

5

6

8.9.

(Flash Column Chromatography)

5.301

1g

LLP 205-214

1

2

R_f 0.2 0.3

R_f 0.2 0.3

3

R_f 0

1.5mL

4
1

120 1 LLP

30~50

5)

5.301

5.301

6)

4

200mL

50mL

7)

8)

9

10)

1cm

11)

100g 200mL

0.5 g/mL
1/3

12)

1.5

13)

14)

15)

16

2cm

17

18

19

2cm

20

1cm

21

" "

22) TCL

23

R_f

100mL

5

24

25

26

NMR

9.1. NMR

Varian Mercury 300 Plus FT (NMR)
()

(NMR) 5.301 ()
2~7 1 7 ¹H NMR

¹H NMR

IAP
10

¹H NMR

7 ¹H NMR

1) 5)
2) 6)
3) 7)
4) FID

1) NMR

NMR

NMR

[eject]

NMR [insert]

[on]

3)

" shim"

()

20 /

XY

Z

(4cm)

/

NMR

4)

FID()

~~XXXXXXXXXXXX~~ A A A U U e e - e ' i # A 0

6)

NMR

7)

9.2. IR

5.301 FT-IR

X Y Z

X Y Z

IR " Plot"

9.4. - UV-Vis

HP 8542

UV-Vis

:

- 1) HP 89532A **Kinetics**
- 2) **Functions** **Time/Cell** 2 6
596 562
- 3) **Scan Screen**
Pre-Run **Meas. Blank** 5
- 4) **Run** **Begin** **Start**
- 5) **Traces** **Return, Tabulate** **Time**
- 6) **Screen** *Escape* **Scan**
- 7) 4~6