KJM3200 / 4200 - Organic Chemistry II

Form of teaching

Laboratory course (120 hours / 7 weeks) Lectures (20 hours). Colloquiums (10 hours.)

Form of exam

Written exam (3 hours) Wednesday June 13.

Recommended prerequisites

KJ120 or KJ101 or KJM1010+1020 or KJM1011+KJM1021

http://www.uio.no/studier/emner/matnat/kjemi/KJM3200/v07/

Lecture plan (also on the web)

General info

Writing a lab journal

Alkene / alkyne related chemistry (McM chapt. 6, 7, 8, 11)

Organometallic coupling reactions (McM chapt 10 + lab manual)

Protecting groups (McM chapt. 17.9)

Dienes, Aromatic compounds (McM chapt. 14, 17)

Carbonyl compounds (McM chapt. 19, 21, 22, 23)

Amines (McM chapt. 24)

Amino acids etc. (McM chapt. 26)

Karbohydrates (McM chapt. 25)

Amino acids etc. (McM chapt. 26)

Lipids (McM chapt. 27)

Heterocycles etc (McM chapt 28)

Pericyclic Reactions (McM chapt 30)

KJM 3200 / 4200 - Journal writing
(Lab. manual p 9→)

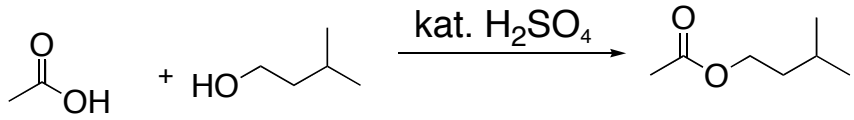
- **Date**
- **Synthesis No.**
- **Literature references**
- **Reaction scheme**
- **Reaction mechanism**
- **Table: Reagents used and amounts**
- **Detailed procedure / description of what you have done**
- **Yield (g / mg and %)**
- **Mp or bp. (+ litt. values if available)**
- **Interpretation of chromatograms**
- **Interpretation of spectra**

- **Appendixes: Spectra, chromatograms. TLC-plates glued into the journal**

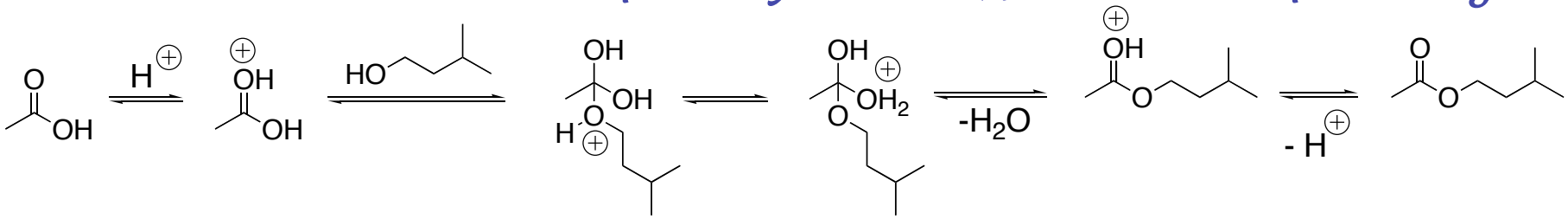
LLG 01 15.01.07

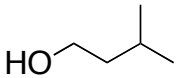
Litt: Harwood, L. M. og Moody, C. J

"Experimental Organic Chemistry, 2nd Ed. s 430



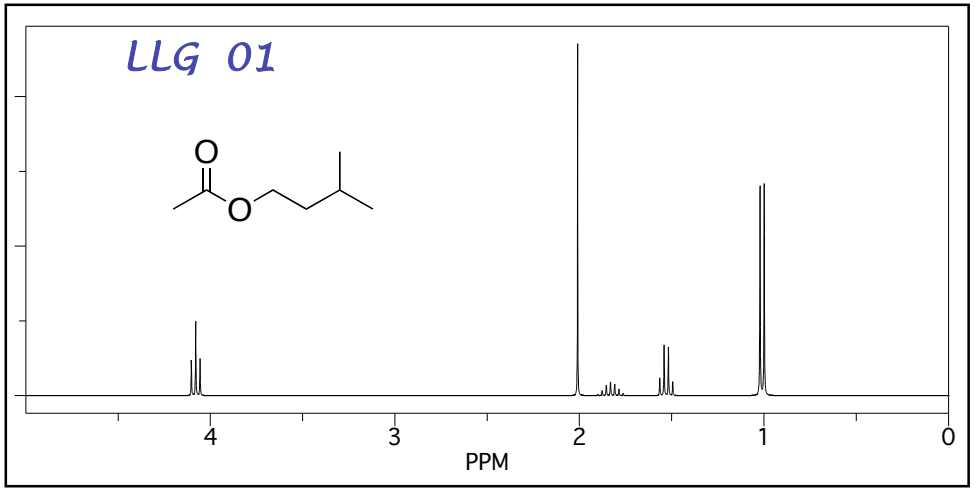
Mekanisme: Nukleofil acylsubst. /Fisher estrifisering



Reagents	Mw	δ	mmol	amout
	88.15	0.809 g/mL	49	5.3 ml
kons. eddiksyre	60.05		201	12.05 g
kons H_2SO_4				2 ml

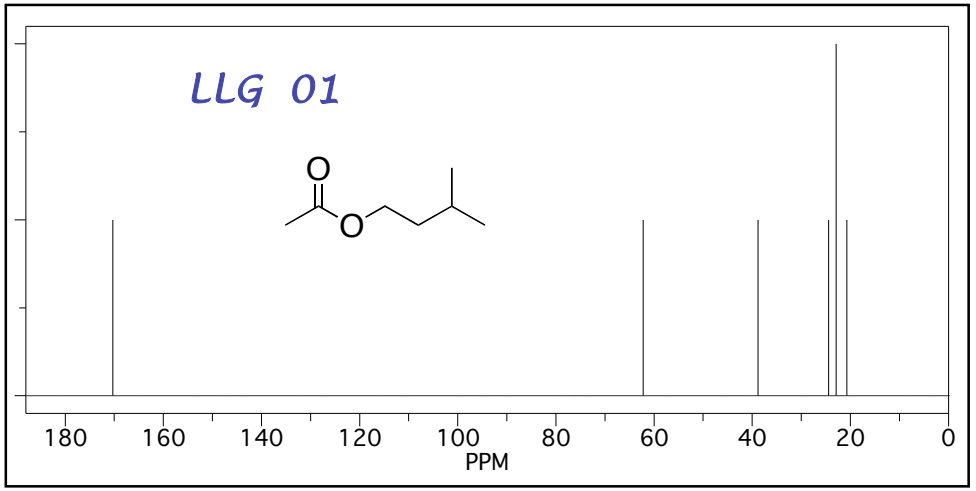
^{1}H NMR (200 MHz, $CDCl_3$) δ

4.08	2H	t	*J* 7.0 Hz	CH2O
2.05	3H	s		CH3CO
1.61-1.69	1H	m		CH
1.52	2H	q	*J* 7.0 Hz	CH2
0.82	6H	d	J 6.0Hz	CH3



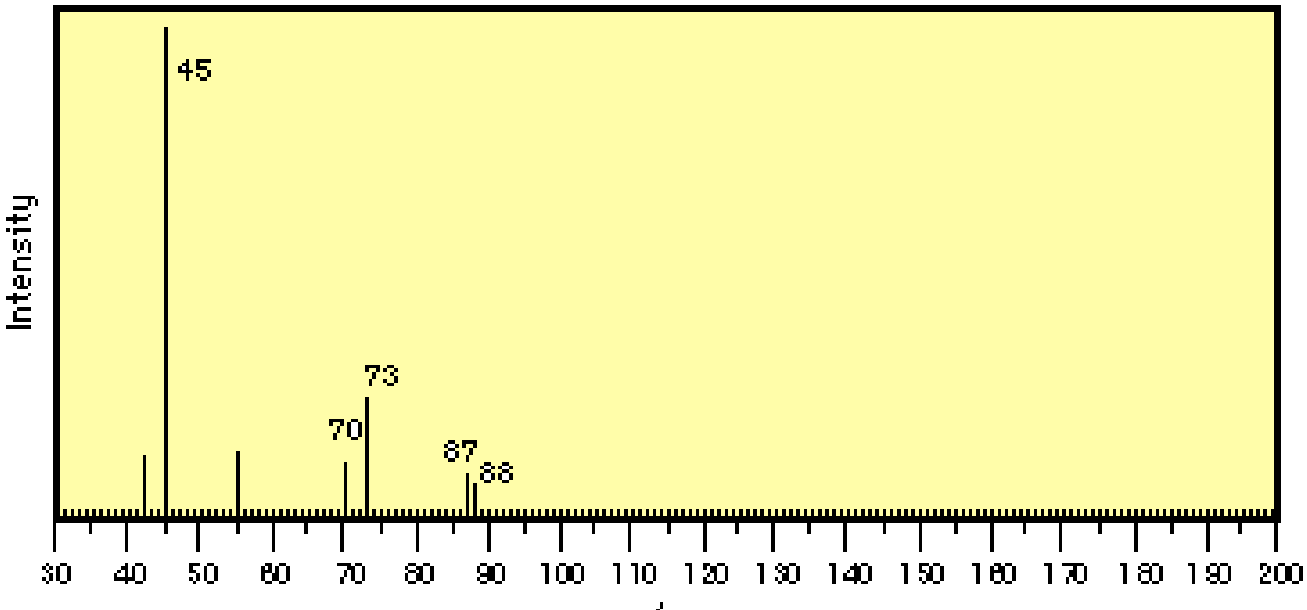
^{13}C NMR (50 MHz, $CDCl_3$) δ

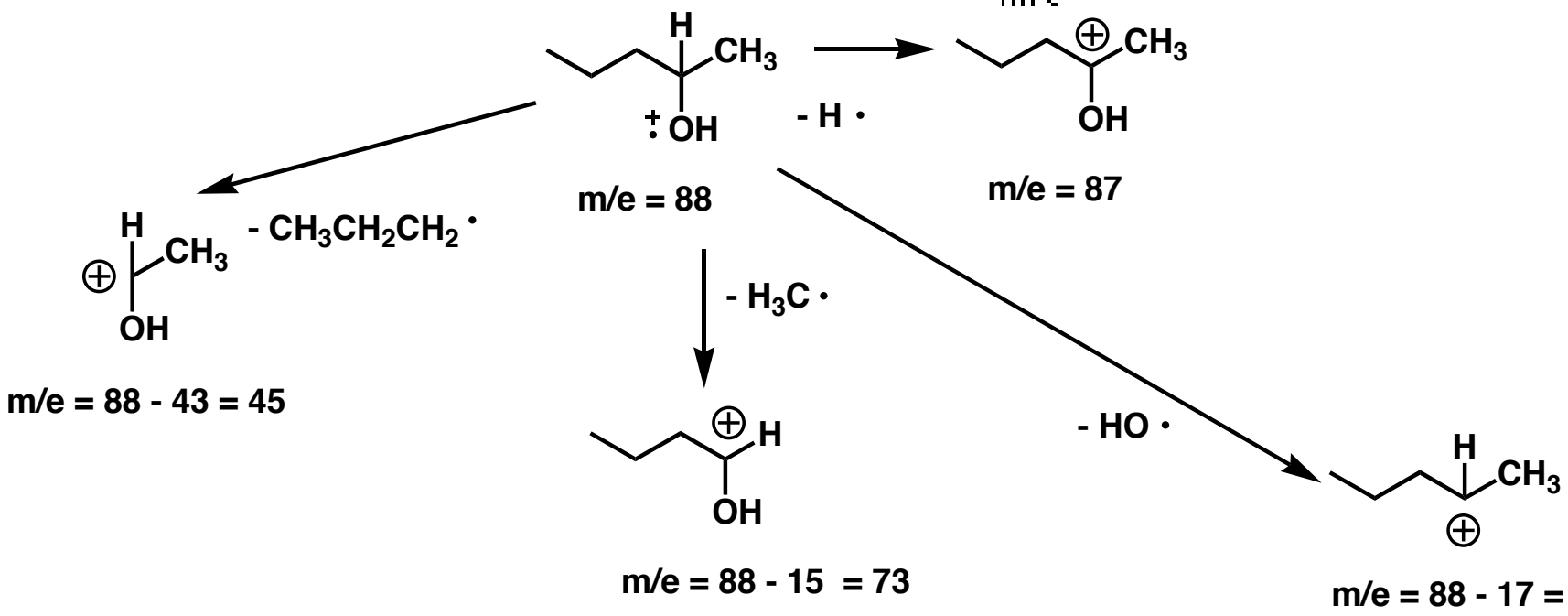
174.2	C=O
62.9	CH2O
37.1	CH3CO
24.8	CH2
22.2	CH3
21.3	CH



Ex. 2-pentanol,
Mw = 88

MS





Ex. 2-pentanol,
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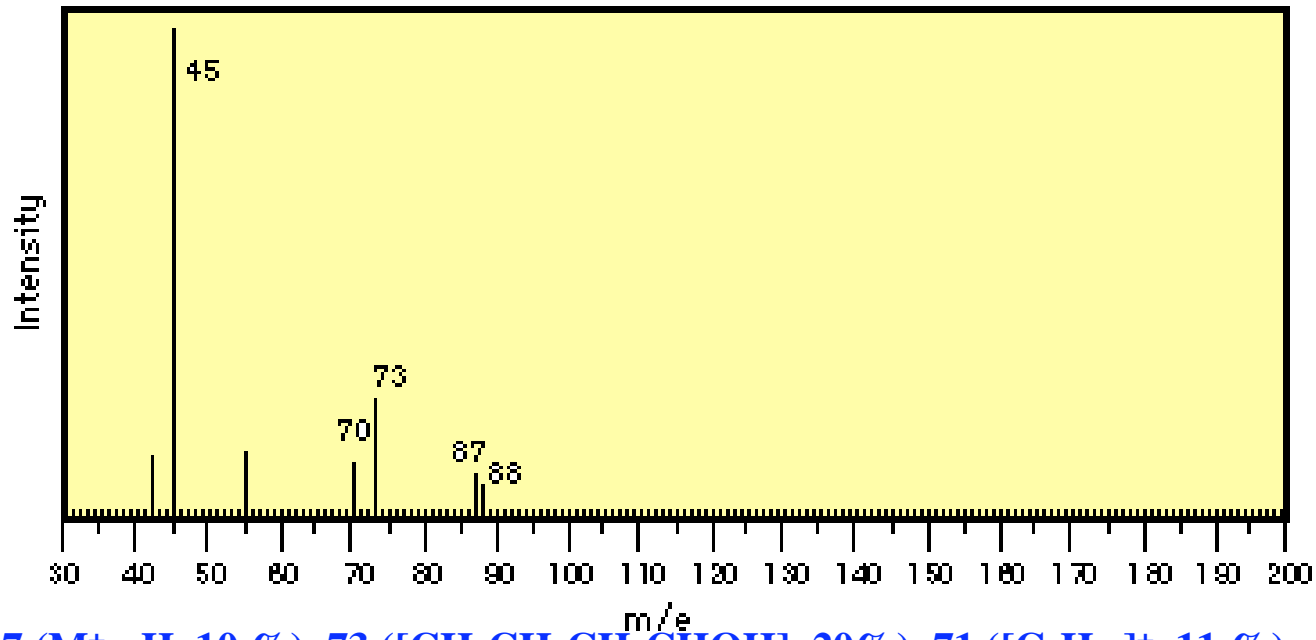
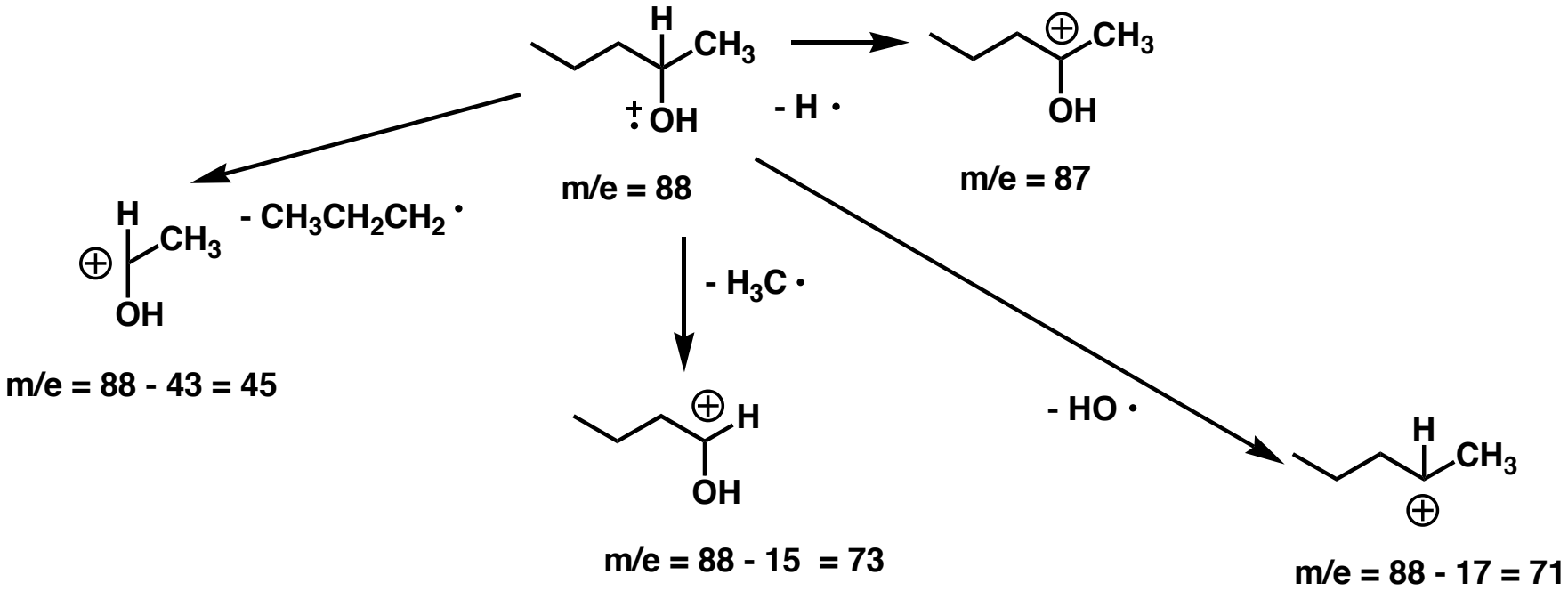


MS (EI): 88 (M^+, 8 %), 87 (M^+ - H, 10 %), 73 ([$CH_3CH_2CH_2CHOH$], 20%) 71 ([C_5H_{11}]$^+$, 11 %), 55 (10), 45 ([CH_3CHOH]$^+$, 100%) .



MS of chlorides and bromides

Cl: ^{35}Cl and ^{37}Cl, ca 3:1

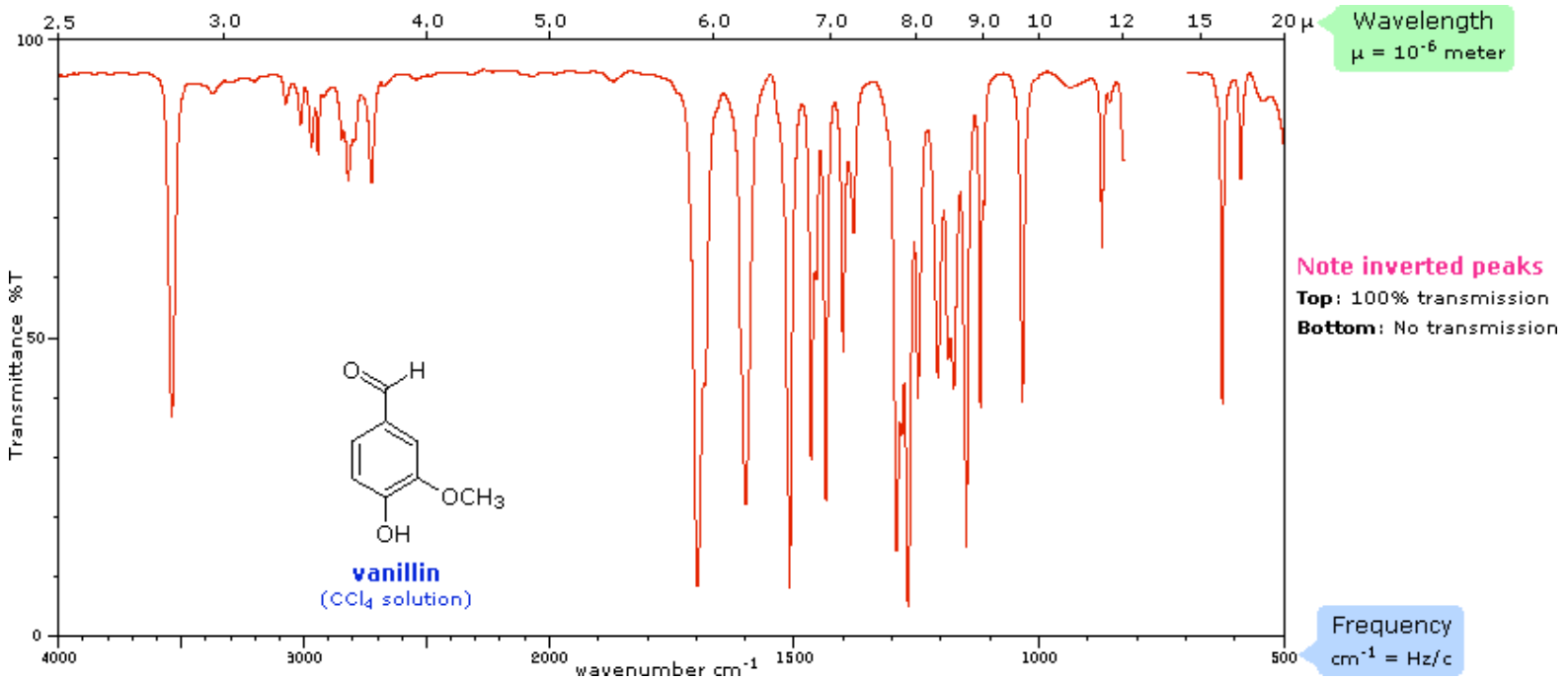
Br: ^{79}Br and ^{81}Br, ca 1:1

Ex. MS of $CH_3CH_2CH_2CH_2Br$, Mw 137.02

MS (EI): 138/136 (M^+, 12/13 %), 109/107 ($[CH_2CH_2Br]^+$, 13/14 %),

95/93 ($[CH_2CH_2Br]^+$, 30/28 %), 57 ($[CH_3CH_2CH_2CH_2]+$, 100 %).

IR



Peaks in fingerprint region (below 1400 cm^{-1}) are normally less important

IR (CCl_4) ν_{max} 3500 (m, OH), etc

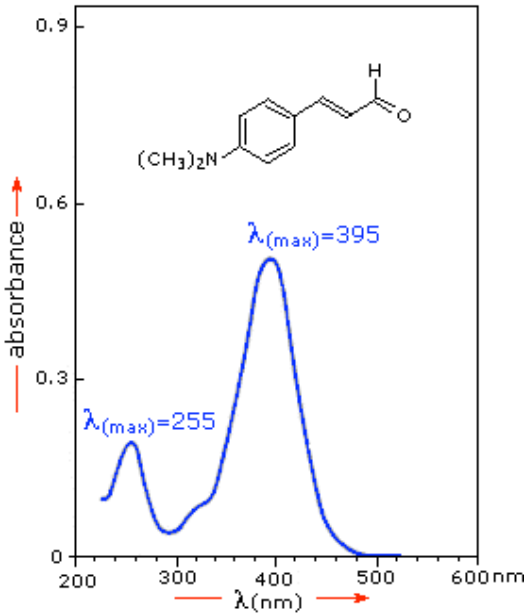
(Film, KBR)

Interpretation if possible

Intensity: w (weak), m (medium), s (strong),

Interval if broad peak

UV



Solvent, conc., λ_{max}, ε

$\varepsilon = A / cl$

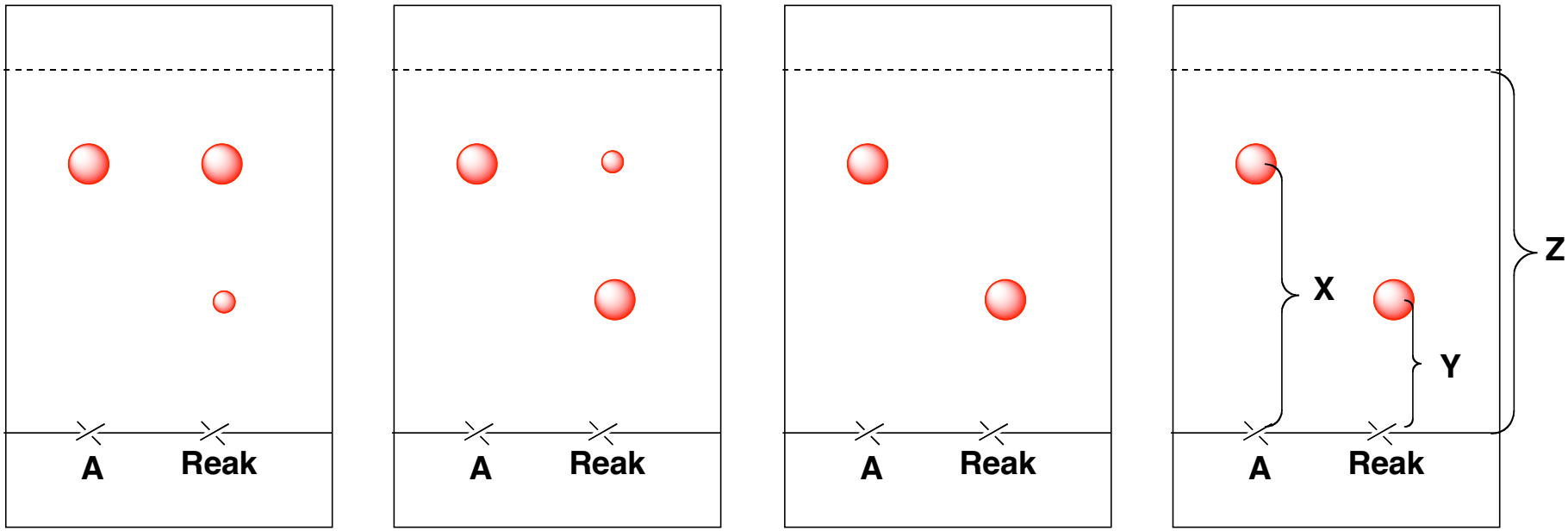
c: conc (M)
l: length of light path thru cuvette (cm)

TLC
(for reaction A→B)

- TLC plate (ex SiO_2)
- Eluent (ex. EtOAc - heksan 1:4)
- R_f values
- Detection (UV, I_2, e.l.)

$$R_f(A) = \frac{X}{Z}$$

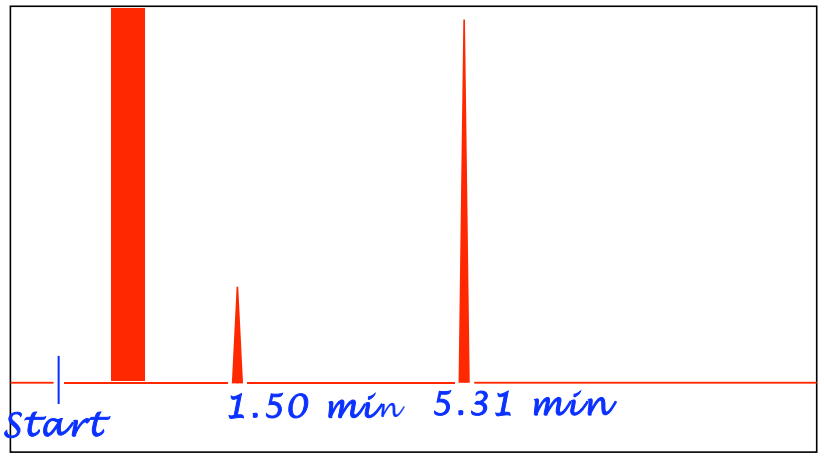
$$R_f(B) = \frac{Y}{Z}$$



Reak. etter 10 min Reak. etter 3 h Råprod. Omkryst. prod.

GC
(for reaction A→B)

- **Coulumn**
- **Injector temp**
- **Coulums temp (+. gradient program)**
- **Detector temp**
- **Detector type**
- **Solvent**
- **Retention times**
- **%**



Råprod.
Rt (A)= 1.50 min (20%)
Rt (B)= 5.31 min (80%)

Conclusions

Write down how you can be sure that you have the right product
(based on spectra and chromatograms)

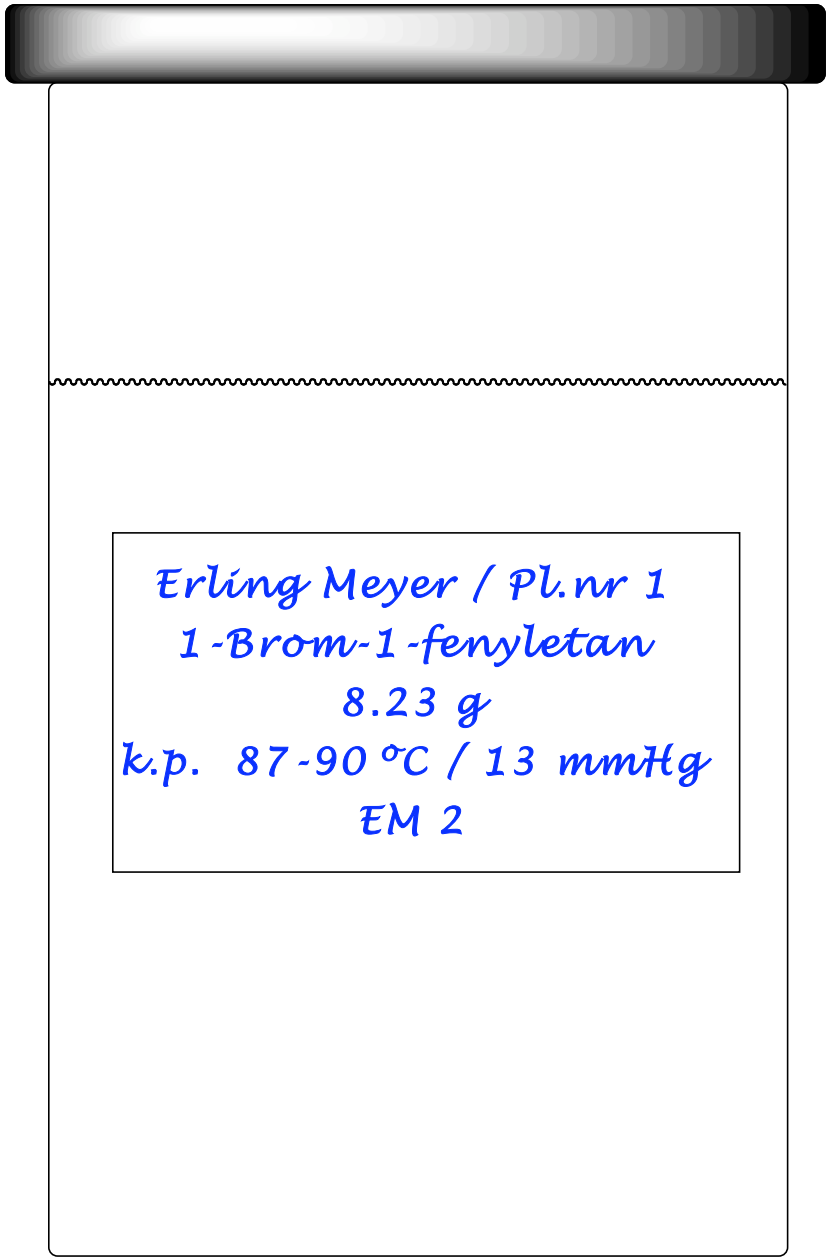
Write down ca how pure your compound is
(based on spectra and chromatograms)

(Try to) write down identity of impurities (if there are any)

Discuss reasons for poor yields, impure product
or other problems if relevant

Handing in your product (Lab manual p 5)

1. **Name Seat No.**
2. **Drawn structure or name of compound.**
3. **Amount of compound handed in**
4. **mp or bp**
5. **Synthesis No. (cf journal)**



Planning your day

Make a plan for the day before entering the lab

Get started in the morning

Have breaks preferably when something is going on
like reaktion stirring, solution drying with $MgSO_4$ etc

Try to work with more than one react at the time (see next page)

Avoid huge back logs with the journal

Mark “everything” with synth no.

- Starting react.
- Work-up, extractions etc. (crude product)
- Purification of product (recryst., distil., chromatography)
- Characterisation of product (NMR etc)
- Finalizing the journal

Monday

Start react. A
NMR and mp etc of prod from last week
Journal writing etc
Work up react. A

Tuesday

Start reaction B
Chromatographic purification of react. A
Evaporation of fractions from chrom.
Work up reaction B
NMR etc react A
Purification react B if time
(otherwise leave crude prod to mext week)
Finalizing journal react. A