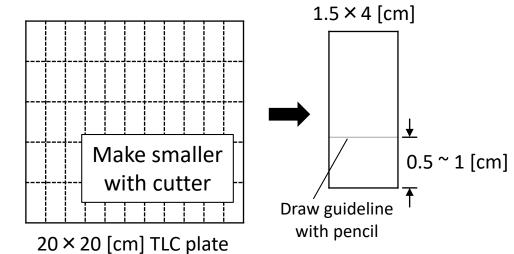
Purpose of TLC analysis

- To check purity of sample
- To detect FG of sample
- To analyze polarity for purification
- To trace reaction progress
- To check stability for storage (2D)

How to prepare a "TLC plate"



Points

- Do not touch the surface of TLC by bare hands. (treat with glove or tweezers)
- Do not trace notch with cutter if you fail to cut.
- •When you cut TLC, do not press cutter strongly on TLC.
- Store TLC at sealed bottle to avoid moisture.

How to analyze by TLC (3-Spot TLC is sufficient for most of cases.)

ex.) before development SM SM + reaction mixture reaction mixture reaction mixture reactant + reaction mixture reactant Figure 1 The proper the reactant in reaction mixture after development The properties of the properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in reaction mixture after development The properties of the reactant in re

- You can obtain some information from TLC.
 - ex1. If TLC is like above right figure, more loading of the reactant possibly facilitate the reaction.
 - ex2. SM and reactant are remained, but the reaction stop. \rightarrow cause: deactivation of catalyst etc...
- Develop TLCs as often as possible, at least when the reaction time is 5 min and 1 h.
- If you cannot speculate polarity of sample, try hexane/EtOAc = 2:1 as eluent.
- If you want to know the analytical information of TLC spot,
 - 1. scratch the TLC spot
 - 2. extract with MeOH or AcOEt
 - 3. filter off silica gel
 - 4. analyze by MS, LC-MS or GC-MS
- Filter paper in developing chamber makes the development faster.
- CS₂ may improve the solubility of compound (especially suitable for large π -conjugated system).

How to detect TLC spot

1st: UV (254 nm) Background = green UV active = black (no FL)

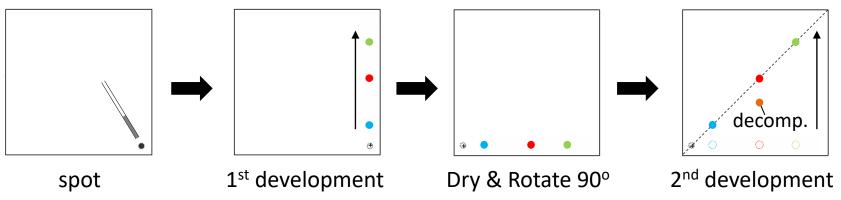
 2^{nd} : Iodine $(I_2 - SiO_2)$ Background = white I_2 active = brown

3rd : stains (Depends on each method, also see "TLC発色試薬の調製法" by K. Oisaki)

How to choose stains

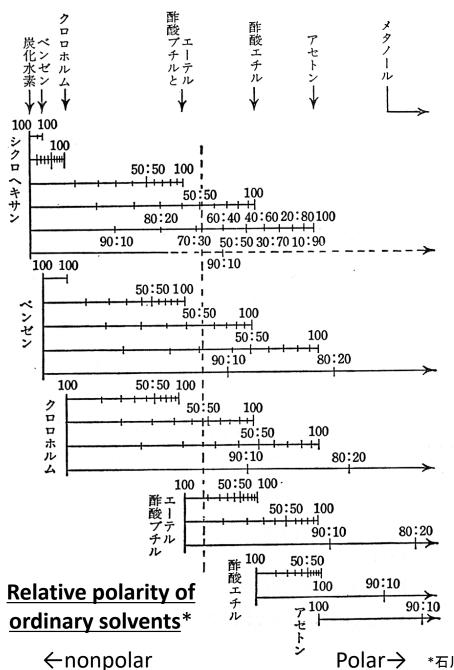
Stain name	Target compound	Treatment	
iodine	For most of functional groups	Bury into iodine/SiO ₂	
p-anisaldehyde	For most of functional groups (Especially, Ar-OH, sugar)		
Phosphomolybdic acid	For most of functional groups		
KMnO ₄	For [O].(e.g. –CH ₂ =CH ₂ -, R-OH, -NH ₂ , -SH, etc.)		
ninhydrin	For amine, amino acid	Dip in stain → heat	
2,4-dinitrophenylhydrazine	For only aldehyde, ketone		
Ceric ammonium molybdate	For most of functional groups	ps	
vanilin	For alcohol, phenol		

2D TLC analysis



Other TIPS

- 1) "Hexane Et_3N or pyridine (1% v/v) soaked then dried" normal TLC is also effective to analyze basic sample.
- 2) Toluene is sometimes effective to affect Rf value of aromatic compound (due to π - π interaction).
- 3) HFIP and TFE is sometimes effective to separate peptide and carboxylic acid. (due to break hydrogen bond)
- 4) Considering "additive" in solvents (e.g. ~1% EtOH in CHCl₃)
- 5) Only UV detection and "イイモリ"(improved Phosphomolybdic acid) are available to analyze NH2-TLC.



How to select TLC type

Normal TLC: Nonpolar sample

(standard)

 C_{18} -TLC: Polar sample

Neutralized TLC: H⁺-sensitive sample

 Al_2O_3 , NH_2 -TLC : Basic sample

NH₂-TLC: Reaction monitor is often easier because excess reagents or SM (containing -CHO, COOH, -COCI, and acidic FG) can be trapped at the baseline.

ex.) -OH →-OTs: trap TsCl amide formation (with HOBt): trap HOBt

How to select solvent

1st choice: Hexane/AcOEt system

(for high polar compound: + MeOH ~10%)

(solvent for C₁₈-TLC : H₂O/MeOH or MeCN)

If possible, you should not use CH_2Cl_2 and $CHCl_3$ because of problematic environmental burden.

How to select additive (for normal TLC)

Acidic sample : + AcOH (1% v/v)

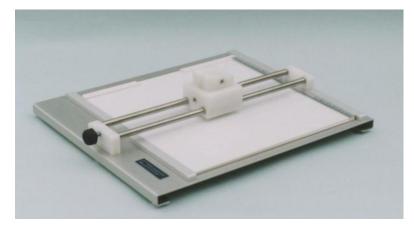
Basic sample : $+ Et_3N$ or Pyridine (1% v/v)

*石川正幸、原昭二、古谷力、中沢泰男編 "薄層クロマトグラフィー" 南山堂 (1972) R. Neher "Steroid Chromatography" p.249, Elsevier, Amsterdam (1964)

Cost of TLCs

Merck (20 cm x 20 cm)	catalog #	Price (on catalog)
Silica gel TLC (0.5 mm) Silica gel TLC (1 mm) C_{18} -TLC basic Al_2O_3 -TLC NH_2 -TLC	1.05715.0001 1.13895.0001 1.15389.0001 1.05550.0001 1.05533.0001	¥14,400/box (25 sheets) ¥34,800/box (15 sheets) ¥57,760/box (25 sheets) ¥35,500/box (100 sheets) ¥48,200/box (20 sheets)

Cost of Accessories



TLC plate cutter (KN3315765): ¥46,550 Diamond blade (KN3315766): ¥6,460



TLC developing tank 30cm x 30cm : ¥43,000 (Sansyo) 10cm x 10cm : ¥12,100 (Yazawa)