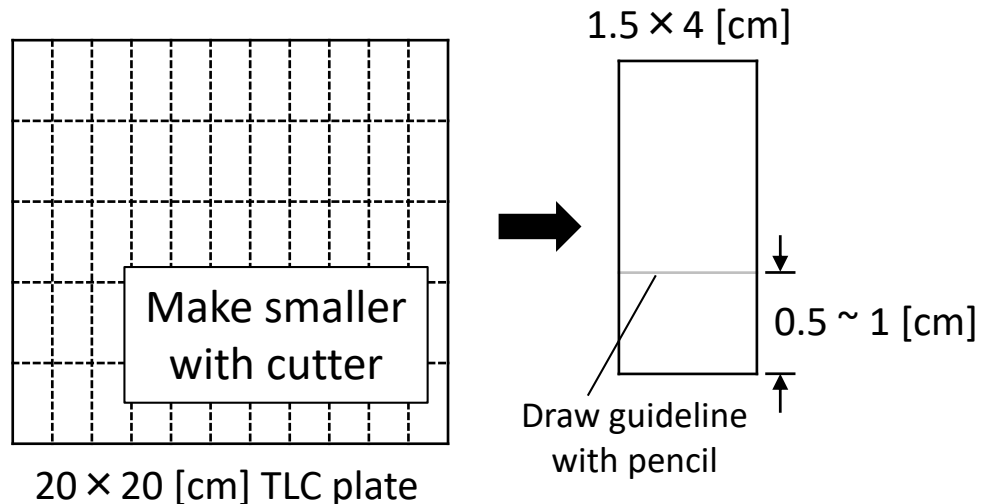


## Purpose of TLC analysis

- 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)

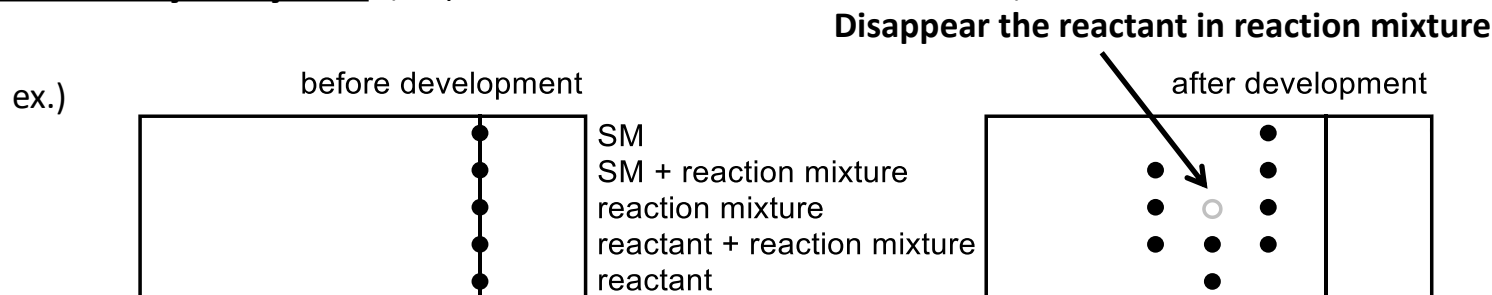
## How to prepare a "TLC plate"



## 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.)



- 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. → **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.
- CS<sub>2</sub> may improve the solubility of compound (especially suitable for large  $\pi$ -conjugated system).

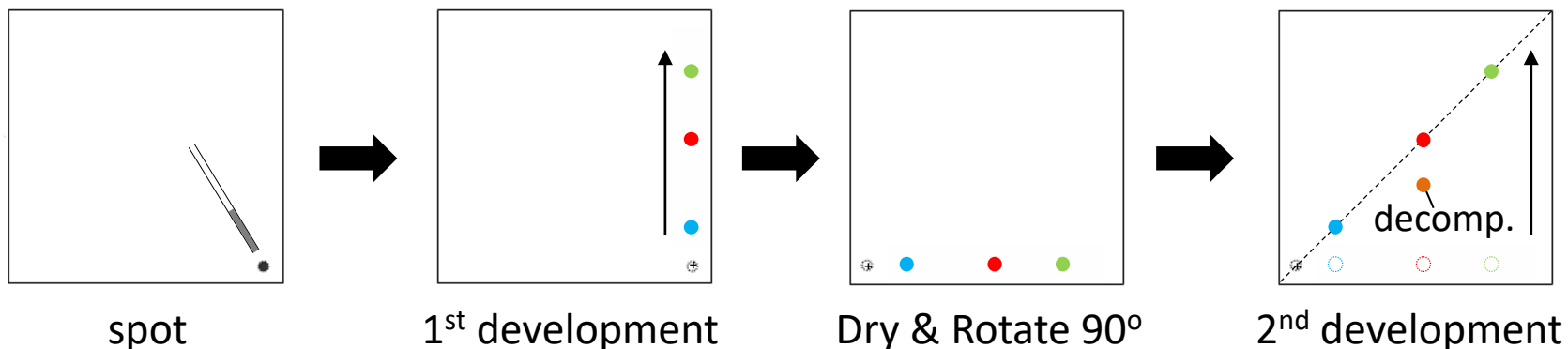
## How to detect TLC spot

- |   |   |                               |
|---|---|-------------------------------|
| 1 <sup>st</sup> : UV (254 nm)                                 | Background = green  | UV active = black (no FL)     |
| 2 <sup>nd</sup> : Iodine (I <sub>2</sub> - SiO <sub>2</sub> ) | Background = white  | I <sub>2</sub> active = brown |
| 3 <sup>rd</sup> : 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 <sub>2</sub>
p-anisaldehyde	For most of functional groups (Especially, Ar-OH, sugar)	Dip in stain → heat
Phosphomolybdic acid	For most of functional groups	
KMnO <sub>4</sub>	For [O].(e.g. -CH <sub>2</sub> =CH <sub>2</sub> -, R-OH, -NH <sub>2</sub> , -SH, etc.)	
ninhydrin	For amine, amino acid	
2,4-dinitrophenylhydrazine	For only aldehyde, ketone	
Ceric ammonium molybdate	For most of functional groups	
vanilin	For alcohol, phenol	

## 2D TLC analysis



## Other TIPS

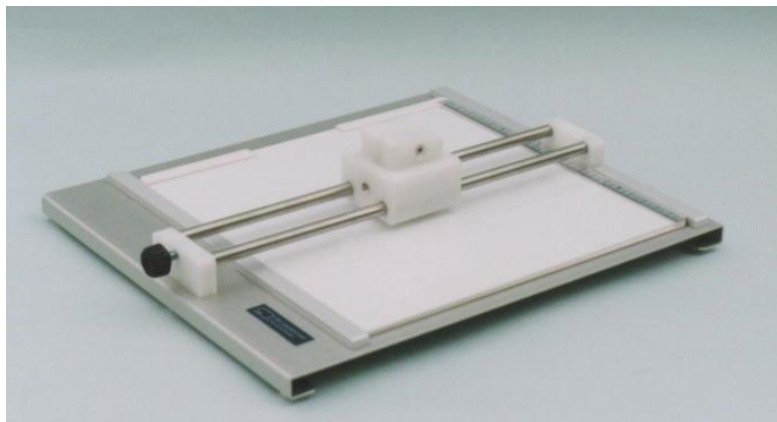
- 1) "Hexane - Et<sub>3</sub>N or pyridine (1% v/v) soaked then dried" normal TLC is also effective to analyze basic sample.
- 2) Toluene is sometimes effective to affect R<sub>f</sub> value of aromatic compound (due to π-π interaction).
- 3) HFIP and TFE is sometimes effective to separate peptide and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



## Cost of TLCs

Merck (20 cm x 20 cm)	catalog #	Price (on catalog)
Silica gel TLC (0.5 mm)	1.05715.0001	¥14,400/box (25 sheets)
Silica gel TLC (1 mm)	1.13895.0001	¥34,800/box (15 sheets)
C <sub>18</sub> -TLC	1.15389.0001	¥57,760/box (25 sheets)
basic Al <sub>2</sub> O <sub>3</sub> -TLC	1.05550.0001	¥35,500/box (100 sheets)
NH <sub>2</sub> -TLC	1.05533.0001	¥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)