

Purify your crude reaction mixture.

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# Flash Chromatography and TLC Laboratory Data Guide

## Solvent Systems for Column Chromatography and TLC

Less Polar Substrates → More Polar Substrates

Indoles; Pyrroles; Azaindoles; Pyrazoles	Ketones; Indanones; Aldehydes	Alkylamino heterocycles (e.g., Alkylaminopyridines)	Phenols; Hydroxy acids; Hydroxyheterocycles	Carboxylic acids	Heteroarylamines; Diamines; Diols
Cyclohexane 60 DCM 40	Cyclohexane 95 EtOAc 5	Cyclohexane 90 TEA 7 EtOH 3	Chloroform 200 EtOH 10 AcOH 1	Toluene 90 EtOAc 8 AcOH 2	Chloroform 95 MeOH 5
Cyclohexane 40 DCM 60	Cyclohexane 90 EtOAc 10	Cyclohexane 70 TEA 20 EtOH 10	Chloroform 100 EtOH 10 AcOH 1	Toluene 83 EtOAc 14 AcOH 3	Chloroform 90 MeOH 10 NH <sub>4</sub> OH 1
Cyclohexane 20 DCM 80	Cyclohexane 75 EtOAc 25	Cyclohexane 60 TEA 20 EtOH 20		Toluene 75 EtOAc 20 AcOH 5	Cyclohexane 60 TEA 20 EtOH 20
	Cyclohexane 65 EtOAc 35	Cyclohexane 40 TEA 30 EtOH 30			Cyclohexane 40 TEA 30 EtOH 30
	Cyclohexane 50 EtOAc 50				

Less Polar System ↓ More Polar System

## Flash Chromatography

For compounds differentiated by  $\Delta R_f \geq 0.15$ ; compound of interest at  $R_f \approx 0.35$

Column Diameter (mm)	Vol. of Eluant (mL)	Typical Sample Loading (mg)		Typical Fraction Size (mL)
		$\Delta R_f \geq 0.2$	$\Delta R_f \geq 0.1$	
10	100	100	40	5
20	200	400	160	10
30	400	900	360	20
40	600	1,600	600	30
50	1,000	2,500	1,000	50

### Recommendations:

40 - 63  $\mu$ m silica gel (400 - 230 mesh silica gel 60) and a pressure-driven flow rate of 2.0 in/min (5 cm/min).

Depth of silica = 5 - 6 in. (12.5 - 15 cm).

For higher viscosity solvents (cyclohexane, dioxane, 2-propanol, ethanol, etc.) the flow rate should be reduced slightly.

Still, W. C. et al. *J. Org. Chem.*, **43**, 2923 (1978)

## Column Chromatography and Related Products



### Silica Gels

Aldrich® Chemistry offers the broadest range of silica gel products from low-cost irregular silica to high-quality bonded phases and spherical silica manufactured under highly controlled processes.

### High Purity Silica Gel

Our highest purity silica gel features:

- Narrowest particle size distribution
- Lowest moisture content
- Greatest lot-to-lot consistency and overall performance
- Minimal impurities, such as metallic oxides

Cat. No.	Particle Size	Moisture Content %	Pore Size (Å)	Particles within Distribution Range
60737	230-400 mesh 40-63 µm	3-7	60 Å	>90%
60738	220-440 mesh 35-75 µm	3-7	60 Å	>90%
60741	70-230 mesh 63-200 µm	3-7	60 Å	>90%

### Technical Grade Silica Gel

These are our most economical Aldrich silica gel products, ideal for less critical, day-to-day separations.

- Contract pricing available
- Best value product for day-to-day separations

Cat. No.	Particle Size	Pore Size (Å)
717185	230-400 mesh 40-63 µm	60 Å
717177	70-230 mesh 63-200 µm	60 Å

For more information on these products and a complete list of silica gel products, visit

[Aldrich.com/silicagel](http://Aldrich.com/silicagel)

### Column Packing Materials

To aid in the filtration process, additional column packing materials are available.



Cat. No.	Packing Material
161551	Activated Charcoal, decolorizing
22140	Celite® 545, filter aid, treated with sodium carbonate, flux calcined
22139	Celite Filter Cel, filter aid, slightly calcined
CLS3950	Pyrex® fiber glass wool
274739	Sand, -50-70 mesh particle size

### What Size Column Do I Need?

Use this handy chart to estimate the typical fraction and sample sizes when using Aldrich Chromatography Columns.

- **Filtration** – Simple separation of baseline impurity that elutes at significantly different  $R_f$  values.
- **Separation** – Removing one or more impurities that have close  $\geq 0.1 R_f$  values.

Methods		Aldrich Chromatography Columns		Collection
Filtration Sample Size (g)	Separation Sample Size (g)	Column O.D. x L (mm)	Column Capacity (mL)	Fraction Size (mL)
5	1-2	25 x 584	200	10
10	3-5	38 x 560	400	20
20	5-8	44 x 572	600	30
30	10	57 x 508	1,000	50
60	20-30	76 x 560	2,000	100
100	50	—	4,000	150-200
200	100	—	8,000	250-400
300	150-200	—	20,000	500-750

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