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Leaders in the use of Alumina and Silica Gel

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- ▶ Alumina
- ▶ Silica
- ▶ TLC
- ▶ Flash Cartridges
- ▶ Drysphere

Frequently Asked Questions

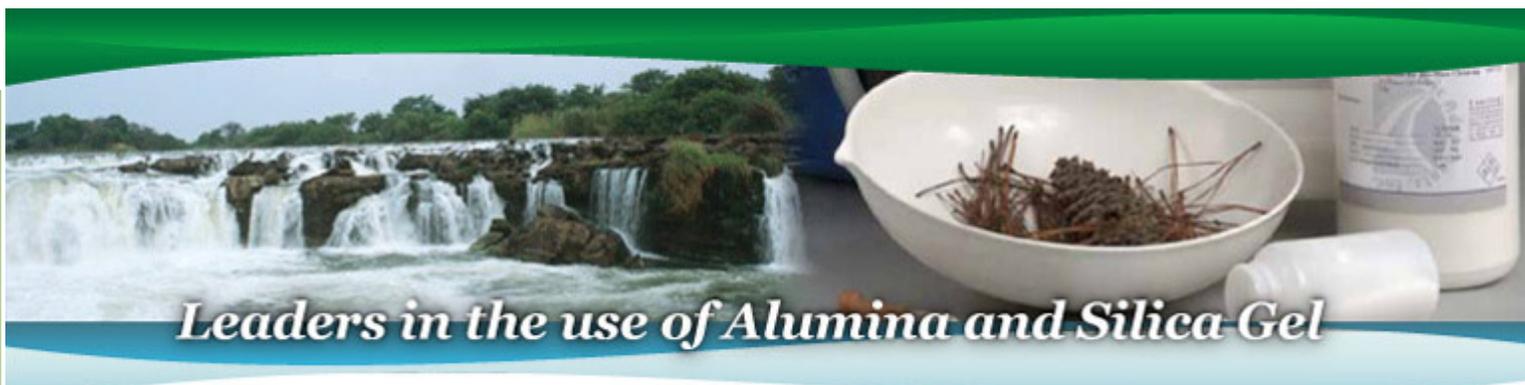
- ▶ [Alumina](#)
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Click on a link below for detailed product information.

[Download 4 page alumina brochure](#)

1.	<a href="#">Alumina</a>	2.	<a href="#">Dynamic Adsorbents' Aluminas - Analytical, Prep LC, Sample Processing</a>
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5.	<a href="#">Silica Gel / Prep LC</a>	6.	<a href="#">Gravity and Flash Column Chromatography</a>
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- ▶ [PCB Removal](#)

## Specialty Aluminas

### Alumina C (for PCB Removal)

**Alumina C** is a chemically and physically modified Alumina for the analysis and removal of PCB's. This material will find wide use and application in/for:

- Analysis
- Environmental clean-Up
- Solvent purification
- Electric utilities: transformer oil
- Soil, water studies

(Request the **Alumina Environmental Product Bulletin** for other environmental applications)

### Alumina P for Pyrogen Removal

This material was developed specifically for the removal of Pyrogens in solution. Pyrogens are typically

complex carbohydrates which preferentially sorb to **alumina P**. Ideal for antibiotic production and other types of bio-technology products.

## **Alumina R**

**Alumina R** is an **alumina** which is used for purifying, separating, and product formulations in the radioactive field; used for the production of various generators where one isotope is retained while the other is eluted. Mainly its improved exchange properties and the constant elution behavior will contribute to its reliability.

**AL 5788** has been developed for doing dioxin analysis. It is a 50-200 micron particle.

## **Alumina for Solvent Purification**

**Alumina** is an ideal media for many solvent clean-up applications.

## **Alumina for Pilot and Process**

Based on DAI's expertise, **aluminas** can be produced according to customer's specifications. They are used for batch processes as well as for production size chromatography. Please request information and technical assistance.

## **DRYSPHERE**

**Drysphere®** is new high technology, dust free, spherical activated Alumina manufactured and designed to optimize desiccant performance. Request the **Drysphere® Product Bulletin**. [More info](#)

## **AL 2000 - For Removal of Lead from Water**

**AL 2000** is a large particle (+200 micron) specially modified, chemically treated **Alumina** that has been designed for the removal of metal ions, especially dissolved lead and other cations from water. Request the **AL 2000 Product Bulletin**.

## **AL 2100 - Scavenger Alumina for Process Clean-up**

**Scavenger activated alumina** is used for process scale removal of impurities. Its high macroporosity improves diffusion rates and the high surface area provides enhanced capacity.

## **Typical Applications**

- Removal of peroxides from hydrocarbons and ethers
- Peroxide adsorption from solvents for ultraviolet spectroscopy
- Dehydration of organic solvents with super active adsorbents
- Removal of alcohol from chloroform

- Purification of organic solvents for optical purposes
- Purification of hydrocarbons and silicone oil for UV

### **AL 2300 - For Bio-Mass Clean-up**

AL 2300 is designed for removing bio-mass in nutraceutical or natural product purification.

### **AL 5000 for Removal of LEAD and other Heavy Metals from Water**

AL 5000 is a +50 micron spheroidal Alumina that can readily remove Lead and other heavy metals from Water. Metal Cation selectivity is Fe III> Cr III> Al III> Pb> Ag II> Zn II> Co II> Cd II.

### **AL 5005 for Decolorization**

AL 5005 is a 50 micron spheroidal, macroporous high surface area, high performance Alumina, for the removal of color, dyes, and clean-up of water.

### **AL 5500 for Arsenic Removal from Water**

AL 5500

### **AL 5900 Activated Wide-Pore Alumina**

**Wide-Pore aluminas** are available in various pore sizes up to a macropore of 1000Å . Ideal for Biotechnology, environmental, and petroleum uses.

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drysphere photos Alumina for Pilot and Process

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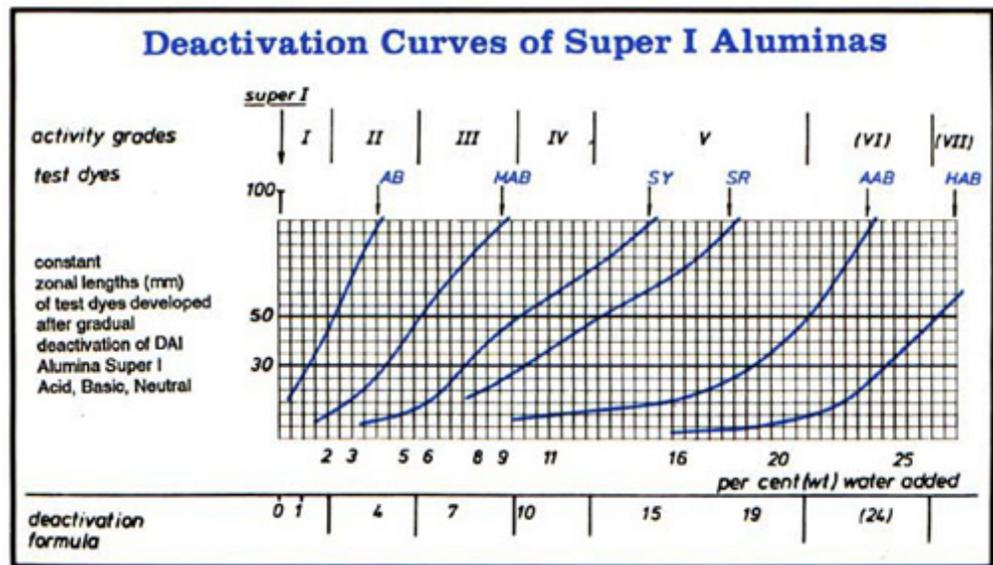
## Dynamic Adsorbents' Aluminas Analytical, Prep LC, Sample Processing Super I Activity

**Super activity I aluminas** are unique as DAI's products; they demonstrate approximately twice the capacity as compared to **Standard activity I**; Surface modifications available are "A" (Acid), "B" (Basic), and "N" (Neutral). **Super activity I aluminas** constitute the starting material for the **Dynamic Adsorbents** line of aluminas. Therefore, it is easy to change between various modes of chromatography. A special feature of Super Activity I is absolutely constant deactivation behavior valid for the deactivation process as well as when in contact with the chromatographic solvent.

### Standard Activity I

**Alumina standard activity I** is available with various surface modifications to facilitate the separation of a wide range of compounds. In addition to pH the activity of the surface of alumina can mediate the separation. It is simple to adjust the activity by adjusting the water content of the material. (Alternatively other polar media can replace water)

- Use **high activity alumina (std act I, super act I)** for the separation of polar samples in nonpolar solvent systems and for the purification of solvents. (see next page)...
- Use **lower activity alumina** for less polar samples. (See Deactivation Protocols Pg. 6)



Symbols of test dyes on the deactivation curves:

AB	Azobenzene	MAB	Methoxy azobenzene	SY	Sudan yellow
SR	Sudan red	AAB	p-amino azobenzene	HAB	p-hydroxy azobenzene



### DCC Alumina

DCC - Dry column chromatography is a versatile **Prep LC** method that bridges the gap between **analytical TLC** and preparative **column chromatography**. (Request DCC Application Guide)

### ‘Flash’ Alumina

Flash Chromatography is a rapid **prep LC** technique that facilitates the separation of 0.1 - 10 g of

material via simple economical laboratory protocols. (Request "Flash" Application Guide)

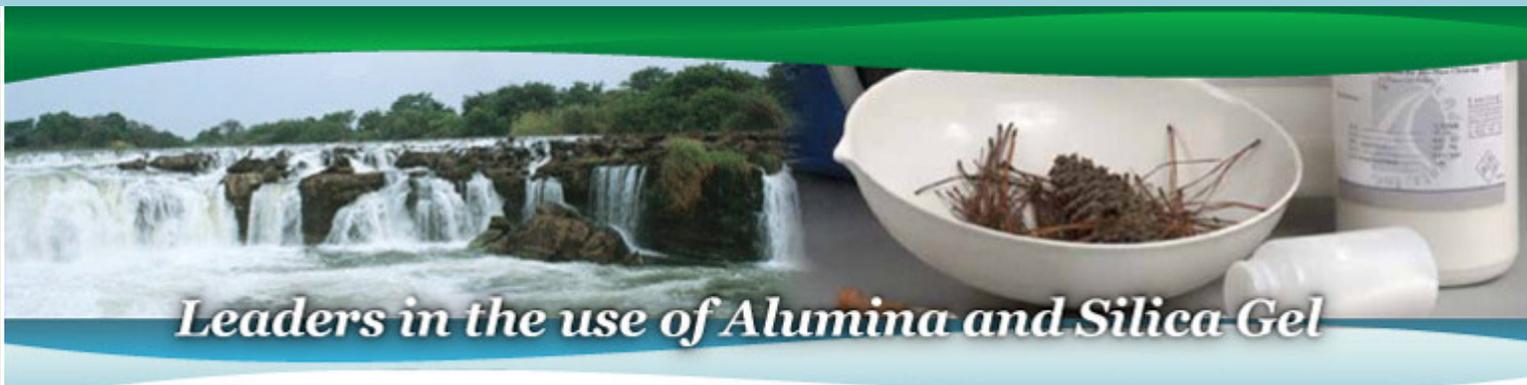
### **Activity II, III**

**Alumina II - III sorbents** are economical adsorbents of medium activity. Use this material for general purpose scouting and in cases where the use of carbon black is precluded due to its organic nature. Also, use **alumina II - III** as a replacement for organic/polymeric ion exchangers, especially when it is necessary to overcome temperature and radiation cleavage problems.

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**Introduction**

Dynamic Adsorbents, Inc. aluminas are unique products; e.g., Super I, Std Act I, etc: **High activity alumina** can be used for polar samples in nonpolar solvents, and for the purification of solvents. Lower activities of **alumina** can readily be obtained by the addition of polar media, especially water. Thus, each problem can be resolved via the adjustment of the sorption system, as required for each problem.

**Special Features**

**Super I aluminas** show an approximate double capacity as compared to **Activity I. Super I** does not have to be deactivated in steps. By following the appropriate deactivation curves, deactivation can be achieved in minute increments. Deactivation behavior by the following procedures makes it relatively easy to obtain the desired activity.

**Deactivation Behavior**

By following the procedures below, it is relatively easy to obtain the desired activity.

Deactivation Behavior - Alumina						
Activity Grade						
Alumina Type	Super I	I	II	III	IV	V
Super I - A,B,N	0	1	4	7	10	19% Water Added
Std Act I - A,B,N	na	0	3	6	10	15% Water Added
A = Acid, B = Basic, N = Neutral						

**Deactivation Procedure(s)**

The % water addition shown above are based upon

weight / weight relationships; these relationships are critical and any deviation will/could result in obtaining improper activities.

To reproducibly obtain the desired activity, weigh an appropriate amount of **alumina** into a stoppered glass bottle. Add the appropriate weight of water to the **alumina** and close the bottle. For example, 97 g of **alumina** + 3 g H<sub>2</sub>O = 3% water addition.

Shake well until all lumps disappear. Wait until the mixture has cooled to room temperature. Keep the container closed so that equilibrium conditions remain constant.

alumina deactivation protocols plates



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### Dynamic Adsorbents, Inc. silica gels

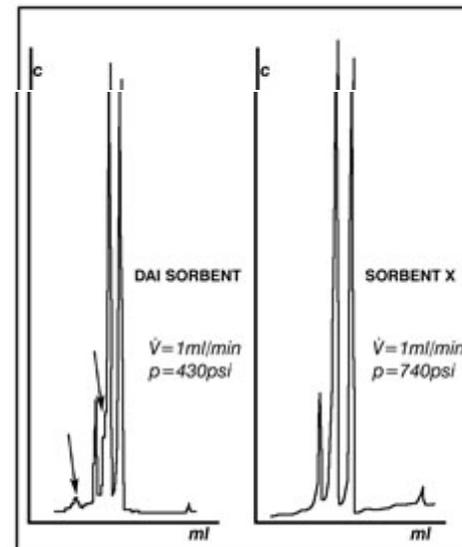
are carefully manufactured and quality assured to provide the ideal laboratory and pilot Process chromatographic material. We control the manufacturing process from raw material to finished product. We carefully control the physical characteristics of pore size, surface area, particle size and surface chemistry ensuring reproducible optimized chromatographic behavior for:

- k' - uniform capacity
- reproducible selectivity
- R<sub>S</sub>- improved resolution
- N - excellent performance

Reproducible performance is delivered regardless of the technique used, especially when transferring from one technique to another.

Technique	Application
“Flash” Chromatography	Prep LC. Request “Flash”,
Column Chromatography	DCC, Application Guide(s)
DCC - Dry Column Chromatography	Pilot - Prep - Process
Large Column Chromatography	Analytical QC Methods Development
TLC, HPTLC, HPLC	

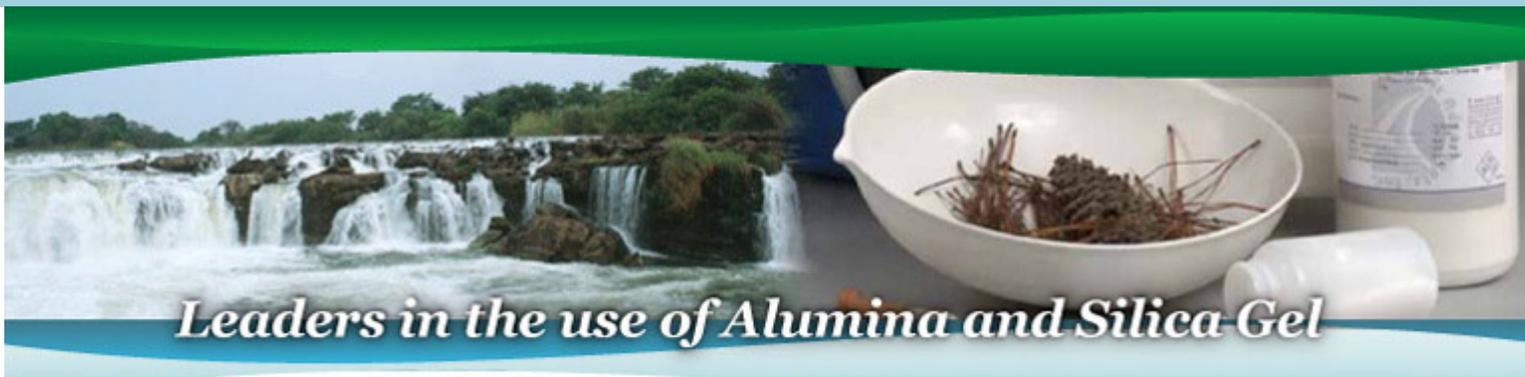
Flash Chromatography



“Flash chromatography” is a rapid form of **preparative column chromatography - prep LC** based upon “an air pressure driven hybrid of **medium and short column chromatography** optimized for rapid separation.” This approach was pioneered by W.C. Still at Columbia University, and described in J. Org Chem 43, 2923 (1978). Separation was based upon the relatively inexpensive apparatus used.

**Flash chromatography** is typically used to prepare 0.1-10.0 g of material in less than 15 minutes and is especially useful when the differences on TLC are greater than 0.15 Rf units. Clearly, **Flash chromatography** is a simple and economical approach to Prep LC.

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## Gravity and Flash Column Chromatography

**Flash chromatography** is a type of preparative liquid chromatography used for the separation of organic compounds. This is **adsorption chromatography** for the routine purification of organic compounds. By using the **flash technique chromatographers** can scale up normal phase chemistries from **thin layer chromatography (TLC)** helping to satisfy the demands of the pharmaceutical and biotech industries in the transition to large scale purification of organic compounds and peptides. The technique utilizes an air pressure driven hybrid of medium pressure and **short column chromatography** optimized for particularly rapid separations.<sup>1</sup>

Flash is very similar to traditional **column chromatography** except that solvent is driven through the column by applying positive pressure. Resolution is measured in terms of the ratio of retention time ( $r$ ) to peak width ( $w$ ,  $w/2$ ). The technique simply uses a set of **chromatography columns** and flow controller valves. Modern **flash chromatography** systems are very convenient, being sold as prepackaged plastic cartridges with solvent being pumped through the cartridge.



**Column chromatography** (which is the basis for **flash chromatography**) follows the same principles as thin layer chromatography (TLC). The main difference is that TLC separates miniscule amounts of material whereas **column chromatography** can be used to separate large amounts of material. If the solvent flows down the column by gravity or percolation the technique is called **gravity column chromatography**. If the solvent is forced down the column by positive air pressure it is called **flash chromatography**. The term **flash chromatography** was first used by Dr. W. Clark at Columbia University because the technique allows organic compounds to be purified "in a flash".

**Column chromatography** involves stationary and mobile phases. In **column chromatography** the stationary phase (a solid absorbent) is placed in a vertical column and the mobile phase (liquid) is added to the top and flows down through the column by either gravity or external pressure. In **column chromatography** the stationary phase is most commonly either **silica** (SiO<sub>2</sub>) or **alumina** (Al<sub>2</sub>O<sub>3</sub>). The columns packed with **silica** usually have a defined particle size of 40-60 microns. The mobile phase is normally a mixture of hexane and ethyl acetate. Mobile phases with low viscosity require smaller particle sizes. The stationary phase is normally more polar than the mobile phase.

By increasing the polarity of the solvent system all components of the mixture move faster. By lowering the polarity all components move more slowly.

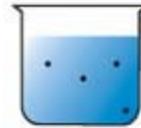
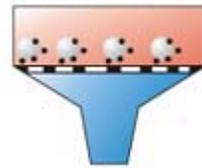
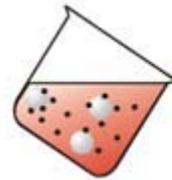
The eluting power of organic solvents

The highest polarity being the most powerful eluters (at the top of the list)

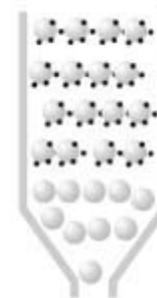
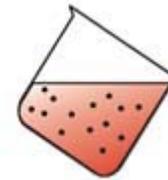
- Acetic acid
- Alcohol
- Acetone
- Ethyl acetate
- Diethyl ether
- Halogenated hydrocarbons (methylene chloride)
- Toluene
- Alkanes (hexanes, petroleum ether)

# Purification by Adsorption

Batch Process



Column Process



Purity Of  
Samples  
Running

Through

Batch Process: Equilibrium  
Concentrations of Contaminant  
Still Present

Column Process:  
Chromatographically  
Pure Substance



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### The Dynamic TLC Program

Dynamic Adsorbents Inc.'s technology and experience has resulted in one of the broadest **TLC-HPTLC-"S"** HPTLC programs in the world. Our **TLC-HPTLC program** is one of the most complete. Included in the program are **silica gels, aluminas, cellulose and PEI cellulose**. In addition, we supply these materials in a broad variety of layers and plate types.

Layer Code/Type	Layer Description	Feature/Benefits
Analytical TLC		
HLO	Hard-Layer: Organic Binder HLO the most abrasion resistant high resolution TLC product available in our program. Write directly on the plate. Outstanding detectability, sensitivity; Minimal breakage.	<ul style="list-style-type: none"> <li>• High resolution</li> <li>• Standard of the industry</li> <li>• Durable reflective surface</li> </ul>

<b>Alumina A, B, N</b>	Select the pH most appropriate to your separation, A=Acid, B=Basic, N=Neutral. <b>Alumina</b> is stable a pH 4 - 14 and can be used to separate most compounds, especially basic.	<ul style="list-style-type: none"> <li>• Ideal for the separation of basic compounds</li> <li>• Standardized Particle for TLC, prep TLC</li> <li>• Stable reproducible layer</li> </ul>
<b>PEI - Cellulose</b>	Ideal Anion ion-exchanger for many life science applications e.g. nucleic acid compositions. Keep refrigerated at 4° Celcius to avoid discolorization.	<ul style="list-style-type: none"> <li>• Long chain anion exchanger</li> <li>• Bio-life science applications</li> <li>• Stable reproducible layer</li> </ul>
<b>Cellulose</b>	Available as microcrystalline, Avicel, and Native (MN layers for the separation of polar compounds via liquid - liquid partition chromatography.	<ul style="list-style-type: none"> <li>• Liquid - liquid partition separation mechanism</li> <li>• Ideal for polar analytes</li> <li>• Available as crystalline or native fibers</li> </ul>
HPTLC and "S" - HPTLC Advanced Layers/td>		

<b>HPTLC</b>	A 5 micron particle, 200 micron thick layer, suitable for very difficult separations. Spots of 1-2 mm will optimize separations. Three to five times the resolving power of TLC. Fast development time.	<ul style="list-style-type: none"> <li>• Obtain 3-5,000 theoretical plates /5 cm/ div&gt;</li> <li>• Ideal for the most difficult separations</li> <li>• Resolution similar to HPTLC</li> </ul>
<b>"S" HPTLC</b>	The ultimate in separating power; 3-10 times the resolving power of TLC. Technology and separation dependant on a 3 micron particle; 100 micron layer. Separate nanogram - picogram quantities. Spots of 1-2 mm will optimize separations.	<ul style="list-style-type: none"> <li>• Smallest TLC particle (micron), highest resolution</li> <li>• Fast analyses</li> <li>• Thin, highly reflective surface</li> </ul>
<b>Prep TLC</b>		
<b>Prep TLC</b>	Select 100, 200, 250, 500, 1,000, and 2000 micron layers according to the amount of material to be separated.	<ul style="list-style-type: none"> <li>• Readily Isolate mg - gms</li> <li>• Standardized particle for prep TLC, Prep LC/div&gt;</li> <li>• Wide variety of prep TLC Layers</li> </ul>

<b>"S" HPTLC</b>	The ultimate in separating power; 3-10 times the resolving power of TLC. Technology and separation dependant on a 3 micron particle; 100 micron layer. Separate nanogram - picogram quantities. Spots of 1-2 mm will optimize separations.	<ul style="list-style-type: none"> <li>• Smallest TLC particle (micron), highest resolution</li> <li>• Fast analyses</li> <li>• Thin, highly reflective surface</li> </ul>
<b>Selected Backings</b>		
<b>Glass Backing</b>	Use glass for optimum separation and with aggressive mobile phases. Inert backing will not react with selected detection sprays. Easy to handle. Best resolution.	<ul style="list-style-type: none"> <li>• Resistant to virtually all sprays, eluants</li> <li>• Rigid support for optimum Resolution</li> <li>• Available in micro-macro sizes</li> </ul>
<b>Plastic and Aluminum Backing</b>	Unbreakable and easy to handle. Cut into any size. Easy to isolate one spot for subsequent elution/ detection. Can be easily included (attached) to lab reports.	<ul style="list-style-type: none"> <li>• Cut into virtually any size</li> <li>• Readily isolate any spot for</li> <li>• Subsequent detection</li> <li>• Ideal for documentation</li> </ul>

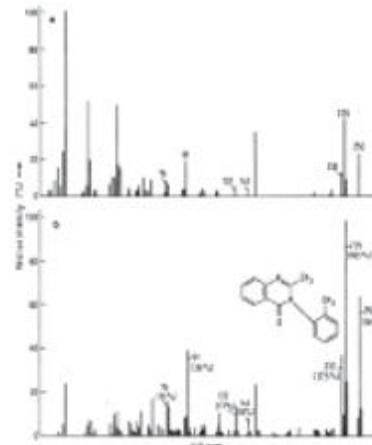
## Applications

Separation of Phenothiazine Derivatives on Basic Aluminum Oxide TLC Plates

Phenothiazine salts migrate little, if at all, on acid aluminum oxide plates. On layers of neutral and more particularly basic Aluminum Oxide TLC layers, good migration is achieved by virtue of exchange processes (similar to those with alkaloid salts on aluminum oxide layers). Benzene is a suitable developing solvent with the addition of 5% acetone. Dragendorff reagent is used as a developer. If the acetone content is increased, the Rf-value becomes greater.

	Pure substance	Drops	Ampoules
Phenothiazine	RF-value	RF-value	RF-value
Megaphen	0.51	0.54	0.53
Verophen	0.31	0.36	0.40
Atosil	0.58	0.56	0.61
Lorusil	0.22	—	0.24
Randolectil	0.23	—	0.23
Neurocil	0.71	—	0.71
Latibon	0.84	—	0.85
Andantol	0.42	—	0.48

#### Identification of Methaqualone in Tissue and Blood via TLC and Mass Spectrometry



It is difficult to distinguish between methaqualone and substances with similar Rf-values via thin-layer chromatography. If this problem arises, methaqualone may be identified by the mass spectrum of the substances adhering to the adsorbent.

Chromatographic examination of autopsy-blood extract contaminated with decomposition products of hemoglobin, was carried out on Silica Gel F TLC, using chloroform/acetone 9+1 (v/v) and Dragendorff reagent, and showed a substance spot at Rf=0.80-0.83.

The reference substances showed the following Rf values:

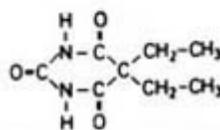
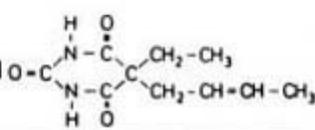
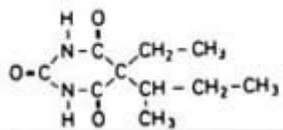
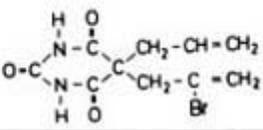
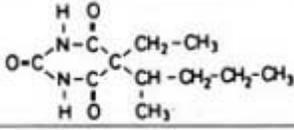
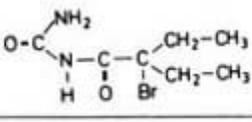
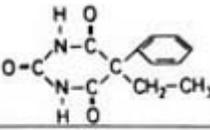
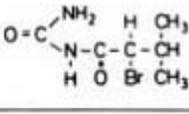
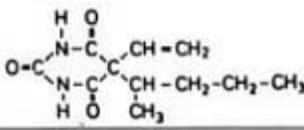
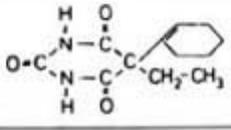
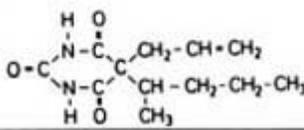
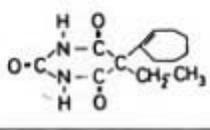
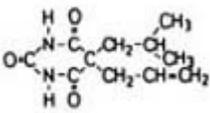
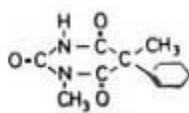
Methaqualone=0.84  
 Gluethimide=0.78

For improving the differentiation, the spot detected on the plate under UV-light was scraped off, the sample was extracted with diethyl ether, decanted, enriched in a small amount of Silica Gel and placed directly into the ion-source of the mass spectrometer.

The attached figure shows the mass spectra of the sample and of the pure substance methaqualone. Quantities of about 15-20 µg. of methaqualone can be reliably detected by means of this procedure.

### Detection of Barbituric Acid Derivatives by TLC and Mass Spectrometry in Autopsy Material

The following substances could be identified:

Barbital		Crotylbarbital	
Secbutabarbital		Vesperone®	
Pentobarbital		Carbromal	
Phenobarbital		Bromisoval	
Vinylbitalum		Cyclobarbital	
Secobarbital		Heptabarbum	
Butalbital		Hexobarbital	

The identification of about 20-25µg of 12 barbiturates as well as Cabromal and Bromisoval, which are often present in pharmaceutical specialties together with 4 barbituric acids, is possible by mean of a combination of thin-layer chromatography and mass spectrometry.

Autopsy material is extracted with a solution of tartaric 5. acid in ethanol after homogenization. the ethanol is evaporated and the residue dissolved by warm water.

After filtration, the tartaric filtrate is extracted with ether and the ether dried over sodium sulfate and evaporated. Urine, after addition of hydrochloric acid (pH 3-4), is exhaustively extracted by ether. The ether is dried over sodium sulfate, treated with a small amount of active carbon and Aluminum Oxide neutral, Act. 1, for a short time, and finely evaporated.

The residue is chromatographed on Silica Gel GF TLC with the solvent chloroform/acetone 9:1. For the detection of substance spots the thin-layer chromatograms are sprayed with mercurous-(I)-nitrate, Zwikkers reagent, and mercurous-(II) sulfate/diphenylcarbazone.

wo samples each of the test material are spotted adjacent to each other. Both samples are primarily evaluated under UV-light. One sample is used for a color test and the corresponding zones of the second sample for the mass spectrometry. For this purpose the single spots are scrapped off, extracted by ether, and the ether is decanted and evaporated. The substances so enriched are brought directly into the ion source of the mass spectrometer. They allow mass spectra, which can be reliably evaluated.

### Identification of Selected Pesticides via Thin-Layer Chromatography

For the detection of pesticide residues in food many methods are published, which in most cases require a considerable amount of apparatus, reagents and time. The separation technique should allow quick detection of the quantity of pesticide residue without much expediture, and only with small amounts of solvents. This preliminary data will then dictate whether a precise determination of the identified pesticide should follow or whether the approximate value obtained by spot comparison is sufficient.

#### Summary of 15 substances to be detected include:

1. Chlorinated hydrocarbons:  
DDT, deildrin, aldrin, lidane, endsulfan (I and II) as well as pentachloronitrobenzene(PCNB) and tetrachloronitrobenzene (TCNB)
2. Phosphoric acid esters:  
Parathion, dimethoate, bromophos
3. Fungicides:  
Pentachloronitrobenzene (PCNB)  
tetrachloronitrobenzene (TCNB), dichlofluanid  
as well as its metabolite DMSA
4. Bacteriostatics:  
IPC (N-phenyl isopropyl carbamate; propham
5. Herbicides:  
N-(3-chloro-4methypheny)  
-2-methypentanamide (solan)

**Technique:** The plant material is macerated with hexaneisopropyl alcohol (70:30); active substances are

transferred into the hexane phase. After drying and removal of pigments a combination column (Alumina basic, activity V and Na<sub>2</sub>SO<sub>4</sub> on top) the yellow extract yield is directly spotted on a thin layer plate. Length of run always 17 cm. If too much wax is present, it should first be treated with acetonitrile. The sensitivity is usually at 2-6 µg of each active substance, but with DDT even 0.5 µg can be detected.

1. Chlorinated hydrocarbons are separated on **silica gel G TLC** in hexane/chloroform (9:1). Detection by spraying with AgNO<sub>3</sub>\*

Aldrin	R <sub>1</sub> 0.83
PCNB	R <sub>1</sub> 0.71
DDT	R <sub>1</sub> 0.64
Lindane	R <sub>1</sub> 0.22
Endosulfan	R <sub>1</sub> 0.15
Dieldrin	R <sub>1</sub> 0.08

2. Phosphoric acid esters are separated on **silica gel G TLC** or on **TLC-plates**, pre-coated with **silica gel F 254** in hexane/acetone (4:1).

Parathon	R <sub>1</sub> 0.45
Bromophos	R <sub>1</sub> 0.70
Dimethoate	R <sub>1</sub> 0.66

3-5. Fungicides, bacteriostatics and herbicides are separated in the same manner P-esters on **TLC plates**, pre-coated with **silica gel F 254**, then diazotised, coupled and the color products evaluated in UV and visible light.

PCNB	R <sub>1</sub> 0.97
TCNB	R <sub>1</sub> 0.97 reddish
Solan	R <sub>1</sub> 0.49 blue
IPC	R <sub>1</sub> 0.52 yellowish
Dichlofluanid	R <sub>1</sub> 0.39
DMSA	R <sub>1</sub> 0.19 violet red

## Thin-Layer Chromatography of Selected Indanol Derivatives of Pharmaceutical Interest

7-Chloro-4-hydroxy indan, 4-hydroxy-1, 5, 7-trimethyl indan and other indanol derivatives demonstrate excellent bactericidal, fungicidal and amebicidal properties. **Thin-layer chromatography** was found to be ideal for qualitative and quantitative control of these substances in pharmaceutical specialties.

**Method: Silica Gel GF TLC**

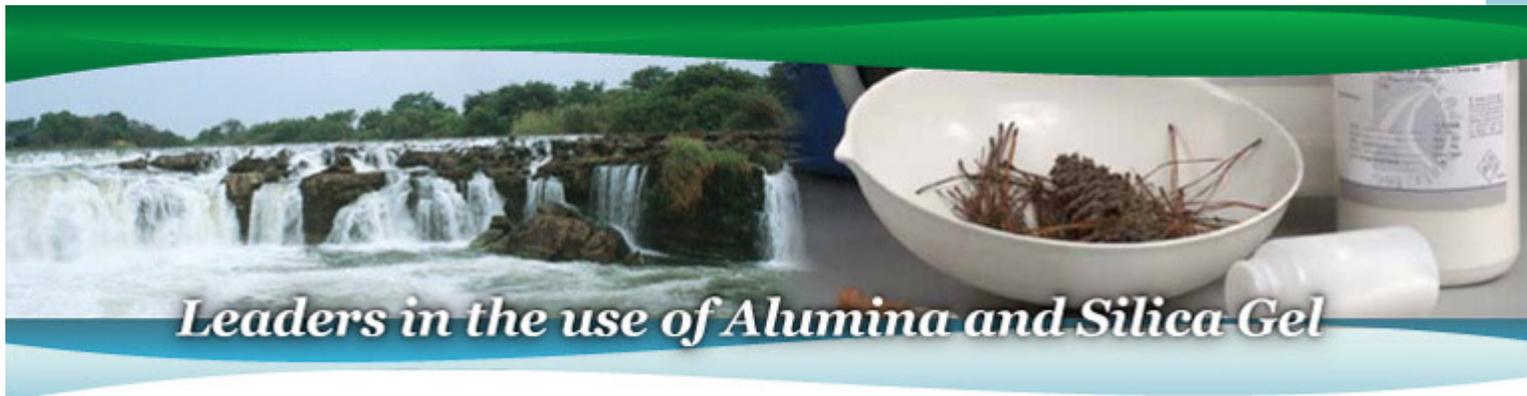
**Solvent Systems:**

- **I Water-saturated chloroform**
- **II Benzen/chloroform/abs, alcohol 4**
- **III Chloroform/abs. alcohol 4:1:1**
- **IV Benzene**
- **V Carbon tetrachloride**

**Direction:** After development the thin-layer plates should be dried. Under UV 254 nm the substances appeared as dark spots against the greenish fluorescent background. If the fluorescent indicator is not available, the plates should be sprayed with an aqueous potassium permanganate solution (1%): yellow spots indicate the position of the various compounds on violet brown background.

Substances	R1 - Values with various Solvent systems on Silica Gel F-254				
	I	II	III	IV	V
4-Hydroxy Indan	0.31	0.84	0.78	0.25	Start
5-Hydroxy Indan	0.22	0.82	0.72	0.18	Start
7-chloro-4-hydroxy Indan	0.28	0.78	0.72	0.23	Start
5,7-Dichloro-4-hydroxy Indan	0.69	0.89	0.91	0.63	0.31
7-chloro-4-hydroxy Indan-on (1)	0.60	0.92	>0.94	0.34	0.08
5-Acetyl Indan	0.60	0.92	0.94	0.34	0.05
5-Amino Indan	0.79	Front	0.94	0.83	0.38
4-Hydroxy - 1,5,7-trimethyl Indan	0.59	0.89	0.84	0.44	0.07

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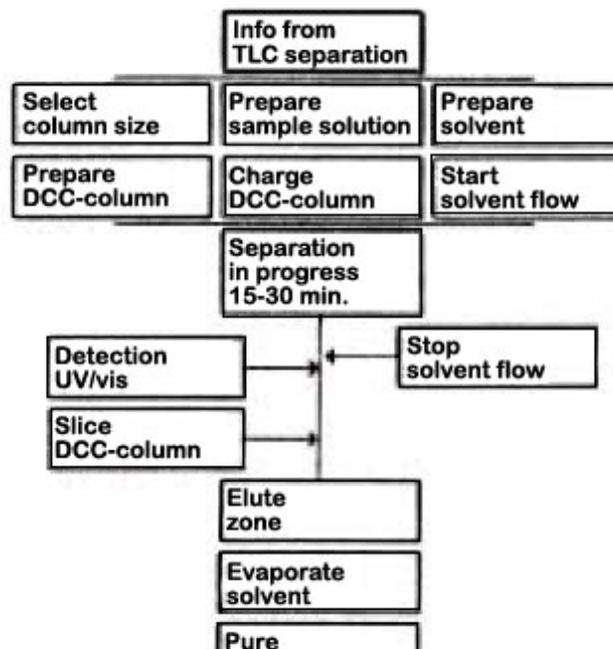
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## Dry Column Chromatography

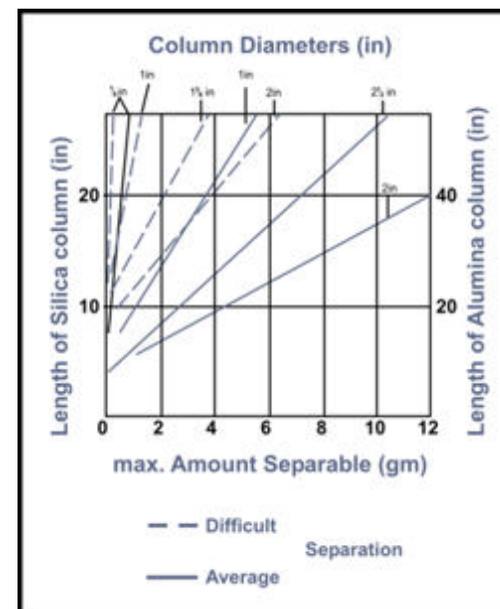
Dry column chromatography (DCC) is a versatile Prep LC method. Basically, any sample that can be separated on silica gel or neutral alumina TLC plate can also be separated by the corresponding DCC-setup. The dry-column procedure has been successfully applied for the preparation of dye-stuffs, alkaloids, and other heterocyclic substances which are known to be separated on other types of columns, but, with considerable difficulties. Lipids have also been successfully separated.



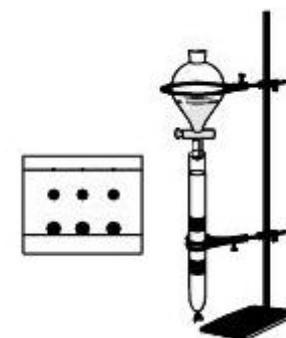
**Dry column chromatography** bridges the gap between analytical TLC and preparative classical column chromatography. The cost is much less than the cost incurred in instrumental pressure associated with preparative liquid chromatography.



The load sample versus adsorbent is maintained at approximately less than 1:500 in TLC while the ratio is 1:300 or even higher for **dry column chromatography**.



## The Dry Column Technique



Bridges the gap between preparation column chromatography and analytical thin-layer chromatography.

## Dry Column Chromatography

This is a unique and simple method for purifying material. It is inexpensive and fast. It is single column elution technique. Below is a schematic form of the method.

### Dry Column Chromatography: The Procedure

#### Preparation

##### Simplified Procedures

1. Use the same solvent system that was developed on a TLC plate
2. Cut the nylon tube to the desired length.

Special note: to isolate 1 gram of material use approx. 300 grams of sorbent in a 1 meter x 40 mm tube.

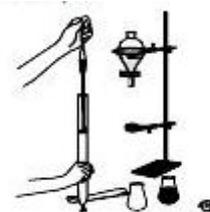


#### Filling the Column

3. Close the tube by rolling up the end and securing it by a seal or clip/staple.
4. Insert a small pad or wad of glass wool at the bottom of the column; pierce holes at the bottom with a needle.
5. Dry fill the column to  $\frac{3}{4}$  of the length.

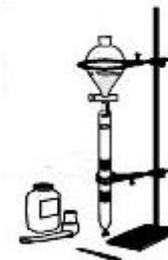


6. The sample to be separated should be combined with at least ten times its weight of the same sorbent in a conical test tube.
7. Add an additional cm of sorbent on top of the sample followed by a small pad of glass wool or a carefully placed cm layer of sorbent.

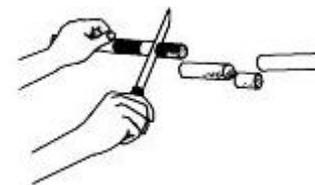


#### Adding Appropriate Solvent

8. Fasten the tube to a clamp on a stand.
9. Open the stopcock of the solvent reservoir and add solvent until it reaches the bottom of the column. Then Stop. Elapsed time approximately 30 minutes.
10. Find the location of the separated bands by visible, UV, UV quenching. Alternatively, cut a 1/16" vertical slice off the tube. Spray the exposed area with a visualization reagent and align with the untreated column to identify (mark) the separated bands.
11. Mark the location of the bands on the nylon tube.
12. Remove the column from the clamp.
13. Slice the column into the desired sections.
14. Elute the pure compounds from the sliced sections with polar solvents



#### Recovery of the Sample



### Nylon Foil Tubing for Dry Column Chromatography

**Dry column chromatography** is very simple and economical because the adsorbent filled into nylon tubing (other types of columns, such as, glass, etc., may also be used). This tube is sold folded and in rolls. It is easy to remove possible creases by blowing a hot air stream through the tubing. Shaking the tubing in acetone prior to the hot air treatment facilitates this "ironing" of the nylon tube.

### Dry Column Chromatography DCC Compared to TLC

Chromatographic Parameters	TLC	DCC
Solvent Reservoir	tank	overhead
Solvent Force	capillary	gravity
"Charge" Addition of Sample	pipette	pipette
Support	glass, plastic	nylon tubes
Adsorbent	silica, alumina, polyamide	silica, alumina
Adsorbent Activity	low	low
Equilibrium with solvent vapor	partial	none
Dimensions of width: thickness: length	(sometimes controlled)	1 :1: 20
Adsorbent bed	width: thickness: length	visible, UV
Detection	200 :1: 200	
Techniques for Recovery	visible, UV spray techniques scrape off elute	cut into sections elute

References:

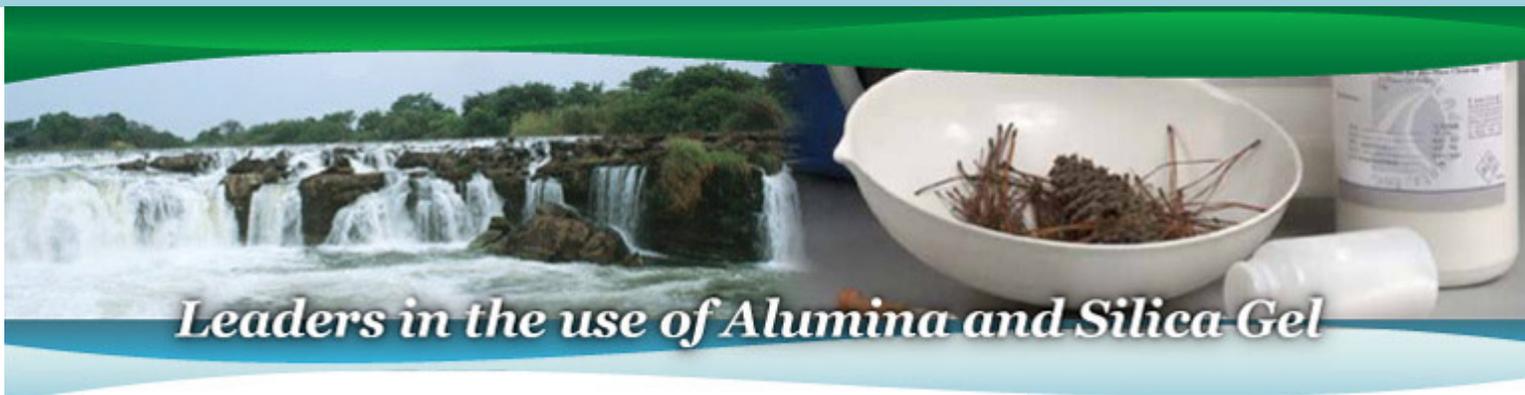
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### Polyamide

**Polyamide** is based on Nylon 6. Due to its activation process it exhibits a constant selectivity toward flavones, chalcones, anthraquinones, aromatic nitro compounds, DNP amino acids, phenols, carbonic acids, acid amides, sulphonic acids and amides of sulphonic acids as well as towards amines and quinones. Forces which contribute to the separation involve hydrogen bonding between the nitro groups, the phenolic protons, the carboxyl groups etc. of the sample and the free amino groups of the adsorbent.

### Florisil PR

**Florisil PR** is a new selective adsorbent, specially processed to give consistent results when used for column cleanup and separation of chlorinated pesticide residue prior to identification and measurement of the pesticide by gas, thin layer or paper chromatography. This material is packed in Alumina Bottles to ensure purity during storage, shipment, use.



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