

Column Chromatography

Summary

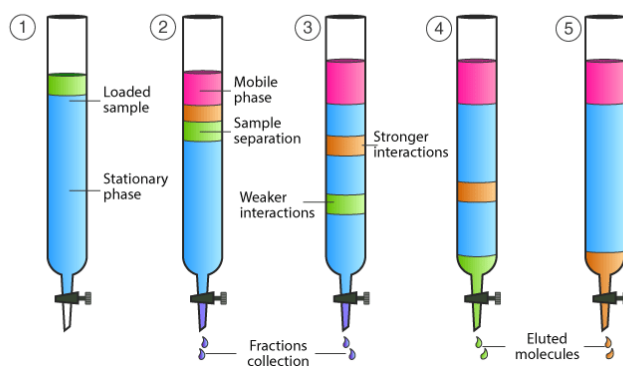
- Column chromatography can use a multitude of different solvents, which carry a lot of the risk involved when working with these.
- Automated columns can be substituted for manual run columns to minimize exposure and solvent use.
- Labelling contents of fume hoods and racks is key to ensuring everybody is aware of the hazards involved with a particular setup.

What is column chromatography?

Column chromatography is a common technique in many synthetic laboratories used to purify a compound from different reactants and byproducts of reactions. Mixtures are eluted through the column with appropriate solvent (the mobile phase) and interact with the solid medium (the stationary phase) to move at different rates depending on particular chemical and physical properties of each constituent. Because the materials move at differing rates through the column, this provides an effective means of separating the constituents by collection of the eluent at different points in time (i.e., fractions). Common forms of media include silica gel and alumina for charge neutral organic compounds. A multitude of different solvents and solvent proportions can be used to achieve separation based largely on polarity. Other types of media and mobile phases can be used to separate proteins, inorganic compounds, charged compounds and other materials. There is no “one size fits all” approach, so please consult your senior lab members and PI before running a column.

COLUMN CHROMATOGRAPHY

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What are the hazards?

The primary hazards associated with column chromatography would be the hazards innate to the products and solvents used in this process. These hazards can include flammability, irritation, and various other health hazards depending on the solvent(s). The media can also present a hazard, such as silica being a skin/eye irritant, as well as a respiratory irritant due to the dust. It is important to note that silica gel does not contain crystalline silica, and therefore does not pose the risks associated with exposure to respirable crystalline silica. There may also be cut hazards present when handling the glass column, particularly if you are doing flash chromatography where pressure from a gas (typically compressed air) is used to facilitate rapid movement of material through the column to speed up the process. When flash chromatography is performed manually, the adapter used to provide the gas to the column is typically a ground glass fitted joint that is held by the column operator at the top of the column. Because the adapter is under pressure from the gas, any weakness in the glass can result in failure leading to lacerations for the column operator, and this is particularly possible when adapters that do not bleed off air are used. Flash columns can also become over pressurized, resulting in potential for solvent spray and exposure.

Use of automated column chromatography to minimize risk

As stated above, traditional column chromatography consists of packing and manually running a column with various solvents, and manual flash columns involve holding an adapter supplying air to the top of the column. However, there are automated systems that can do this job even more efficiently and safely for the user. There are numerous companies that offer automated flash chromatography systems with prepacked columns to separate synthetic products. These products can be used safely with minimum exhaust ventilation, as there is little exposure to the researcher. In fact, these methods can also save chemicals and are often greener processes, using less overall material than a traditional manual setup. Not only are these columns safer, but they can provide an increase in productivity, since they help eliminate the need for standing near the column to collect small fractions. Please reach out to EH&S if you would like help in evaluating different products.



device, in the gas supply line to avoid over pressurization.

Administrative Controls

- If a column is being left unattended for any period of time, be sure to label the fume hood with the contents inside. This would include any solvents and products being separated. It is a good idea to add contact information if anything is left in the hood for a longer period.
- Be sure to use bulk labelling on fractions that are sitting in the hood while awaiting testing. This could include labelling the rack, tubes, hood, or a combination of the three. Please be sure to be consistent in the lab.
- Before use, be sure to inspect all glassware and equipment being used. This would ensure the integrity of the materials, reducing the risk of broken glassware and exposure.
- Be sure to use proper techniques with the fume hood and equipment. Ensure new members of the lab are competent with these procedures using a training system. A written SOP on how to pack and run a column should be in place.
- When applying tubing to glass adapters, use an appropriate lubricant to avoid breaking the adapter.
- Do not over pressurize columns. Maintain appropriate flow rates.

Personal Protective Equipment

- Always wear proper PPE when working with a column:
 - Wear chemical safety glasses with ANSI z87+ rating at the minimum, you may consider chemical splash goggles and a face shield. Check the EH&S safety glasses program (: <https://ehs.umass.edu/eye-protection-program>)
 - A properly fitting (preferably flame resistant depending on what solvent and quantity is being worked with) lab coat is required. Sleeves must be the correct length and not rolled up. Check the EH&S lab coat program (<https://ehs.umass.edu/lab-coat-management-program>)
 - Closed toe shoes and long pants are required, tie back long hair.
 - Wear appropriate gloves for the material being worked with. Check the EH&S glove selection page (<https://ehs.umass.edu/glove-selection>) for more information.

Waste Handling

Proper waste management is required for all the chemicals worked with. Consult the SOP and SDS of the material you are working with to ensure proper waste disposal occurs. If there are ever any questions, please reach out to EH&S for help on proper disposal. Dried mobile phase should generally be handled and solid hazardous waste, and used solvent should be handled as ignitable liquid hazardous waste.

Exposure and Spill Procedure

In the event of a spill involving a column that does not involve the contamination of a person, the material may be cleaned up if it is safe to do so following the general procedure for small spills detailed in the University's Chemical Hygiene Plan.

- Ensure that it is safe to clean up the spill.
 - Spills of more than 1 L should be immediately referred to EH&S (413-545-2682) and the area should be evacuated. Do not permit entry to the area until EH&S arrives.
- Use absorbent material to contain and remove the liquid. After the liquid is removed rinse the area with water and contain and remove with absorbent material.
- Place all items used for cleanup in a labeled hazardous waste container and request a pickup through CEMS.
- If at any point you are uncomfortable cleaning up the spill or require assistance, stop and call EH&S (413-545-2682).

Exposures to hazardous materials should follow the general procedures for exposures outlined in the University's Chemical Hygiene Plan.

For a major exposure or fire requiring the use of a drench shower or eyewash:

- Have someone call 911 (report the building name, room number, and street address) or 413-545-3111 (or simply 5-3111 from a campus line) to report the incident and request medical help. If possible, communicate to the first responders that there was an exposure to whatever chemical was being used and provide the SDS
- Help the affected individual to position their head over the eyewash and activate it, or position them under the drench shower and activate it as appropriate.
 - Always ensure your own safety before helping others. Only help if it is safe for you to do so.
 - Wear gloves, safety glasses, and a lab coat.
- If using an eyewash: Instruct the affected individual to open their eyes and roll them around while the water is flowing. Help them to hold their eyes open if necessary and safe to do so.
- If using a drench shower: Remove all clothing from the affected area while under the shower.
- Flush the affected area for 15 minutes with water.

For minor exposures such as a spill to readily accessible extremities (e.g., hand):

- Flush the affected area in a sink equipped with potable water for at least 15 minutes.
- Go to University Health Services (UHS) for medical evaluation, and tell them you have had a lab exposure to whichever chemical being used.

- Notify EH&S (413-545-2682) as soon as possible and complete the lab incident form (<https://ehs.umass.edu/lab-incidents-and-lab-incident-report-form>).

Report any spill, skin or eye contact, or inhalation of vapors to EH&S as soon as possible by calling 413-545-2682 and submit a lab incident form. Call 911 for fire reporting.

References

UC San Diego instructional video on flash columns: <https://www.youtube.com/watch?v=fF1gXUvyGb4>

U Wisconsin SOP for Column Chromatography: <https://safety.engr.wisc.edu/wp-content/uploads/sites/706/2018/07/Flash-Column-Chromatography-SOP-15.pdf>