9.3-1. Separation of a Mixture of Fluorene and Fluorenone.[1]

CAUTION!! Since the solvents used in this experiment are all highly flammable, no lighted burners or any other flames are permitted in the laboratory while this experiment is in progress.

Obtain from the stockroom a chromatographic column, a 250 mL erlenmeyer flask (2 are required in the experiment), and the fluorene-fluorenone mixture. Provide the stockroom with a *clean* and *dry* 8 inch test tube for the mixture.

Pack the column with alumina by the dry packing method (**9.2-1B**) as follows: Pour 10 mL of petroleum ether into the column (*stopcock closed!*). Push a plug of cotton through the solvent to the bottom of the column with a dowel rod or glass rod. Pour enough sand into the column to form a 1 cm layer over the cotton. Use a little petroleum ether to wash the sand from the sides of the column. Level the surface of the sand by tapping the column gently. Fill the column with petroleum ether and *slowly* pour 55-60 g of *dry* alumina into the top of the column. Tap the column as the adsorbent settles to prevent formation of air pockets during the addition and open the stopcock to allow some solvent to flow out. Add more solvent, if necessary, to prevent the solvent level from falling below the adsorbent level.

When all of the alumina has been added and has settled completely, adjust the solvent level so that there is about 10 mL of solvent above the alumina surface. Pour enough sand into the column to form a 1 cm layer on top of the alumina. Finally, open the stopcock again and let the solvent level fall to just above the upper layer of sand and then stop the flow. The column is now ready for use.

Carefully pour the solution (which contains 0.75 g of fluorene and 0.75 g of fluorenone in 30 mL of cyclopentane and 5 mL of diisopropyl ether) into the column without disturbing the sand layer at the top of the column. Again, let the liquid level drop to the top of the packing and start eluting with the following solutions in the order indicated:[2]

1. 100 mL of petroleum ether (utilize the petroleum ether drawn from the column during its preparation);
2. 40 mL of a solution of 30 mL of petroleum ether and 10 mL of diisopropyl ether;
3. 30 mL of a solution of 20 mL of petroleum ether and 10 mL of diisopropyl ether;
4. 20 mL of a solution of 10 mL of petroleum ether and 10 mL of diisopropyl ether;
5. pure diisopropyl ether to completion.

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[1] The chromatographic portions of this experiment must be completed in one laboratory period. Evaporation of solvent and weighing of products can be delayed.

[2] Air pressure can be used to accelerate the elution process. Attach a length of pressure tubing to the compressed air and to a suction adapter fitted onto the top of the column. Turn the air on very low and plug the opening in the suction adapter. (Holding your finger over the opeing in the suction adapter when the air is turned on is an effective way to accelerate elution.)

When the elution has been started, test for the presence of fluorene after every 10 mL of eluent has been collected. Collect one drop onto a watch glass from the tip of the column . The solvent will evaporate quickly. If fluorene, a colorless solid, has started coming off the column, a residue will be seen where the drop evaporated.

At the first sign of the presence of fluorene in the eluent, change receivers collecting the eluent in a clean 250 mL erlenmeyer flask. Continue eluting, testing periodically for the presence of fluorene, until no more can be detected in the solution coming off the column.[3] Change receivers again, setting aside the solution for eventual isolation of the fluorene.

In order to elute the fluorenone as rapidly as possible, begin the use of pure diisopropyl ether at this point, whether or not all of the petroleum ether-diisopropyl ether solutions have been utilized. Since fluorenone is a bright yellow color, it can be seen coming off the column. When the fluorenone first begins to appear in the eluent, change receivers once again, collecting the fluorenone fraction in a clean 250 mL erlenmeyer flask.

Carefully evaporate the solvent from the solutions of fluorene and fluorenone on a steam bath in the hood. Remember to use boiling stones. Isolate the solids, weigh them to determine the percent recovery (did you separate out the boiling stones first?), and determine the melting points.

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[3] If any of the yellow color (due to fluorenone) begins to appear in the eluent, change receivers immediately.