The Many Uses of a Seven-inch Side Arm Test Tube

Eugene J. McDevitt
Siena College
515 Loudon Road
Loudonville, NY 12211

The decrease in scale of laboratory experiments has decreased the expenses for the purchase of chemicals but often increases the cost of equipment. Typical microglassware kits sell for about $200, which makes equipping all organic student lockers with kits a huge financial outlay for many departments. This cost can be avoided by using a 7-in. side arm test tube (SISATT) (cost~$5) described below.

The dimensions of the test tubes are 7 in. × ½ in. (180 mm × 13 mm), longer and thinner than a normal test tube. They have a side tubulation bent down at a 75° angle, the usual angle one associates with distillation flask side arms and the typical distillation head side arm. These tubes were fabricated to my order by the C. S. Glass Company, now known as The Glassblowers, Inc., P. O. Box 8089, Turnerville, NJ 08012.

The particular dimensions were chosen for several reasons.

1. The usual rubber thermometer adapter used with most 19/22 glassware kits will fit over the top of the tube.
2. The tube is thin enough to fit into a 6-in. test tube.
3. The tube will fit into 19/22 glassware.
4. A ½-in. magnetic stirrer will function satisfactorily within the tube.
5. The tube is long enough to hold a sample of up to 5 mL and yet function as a distilling flask, with room for some column packing.

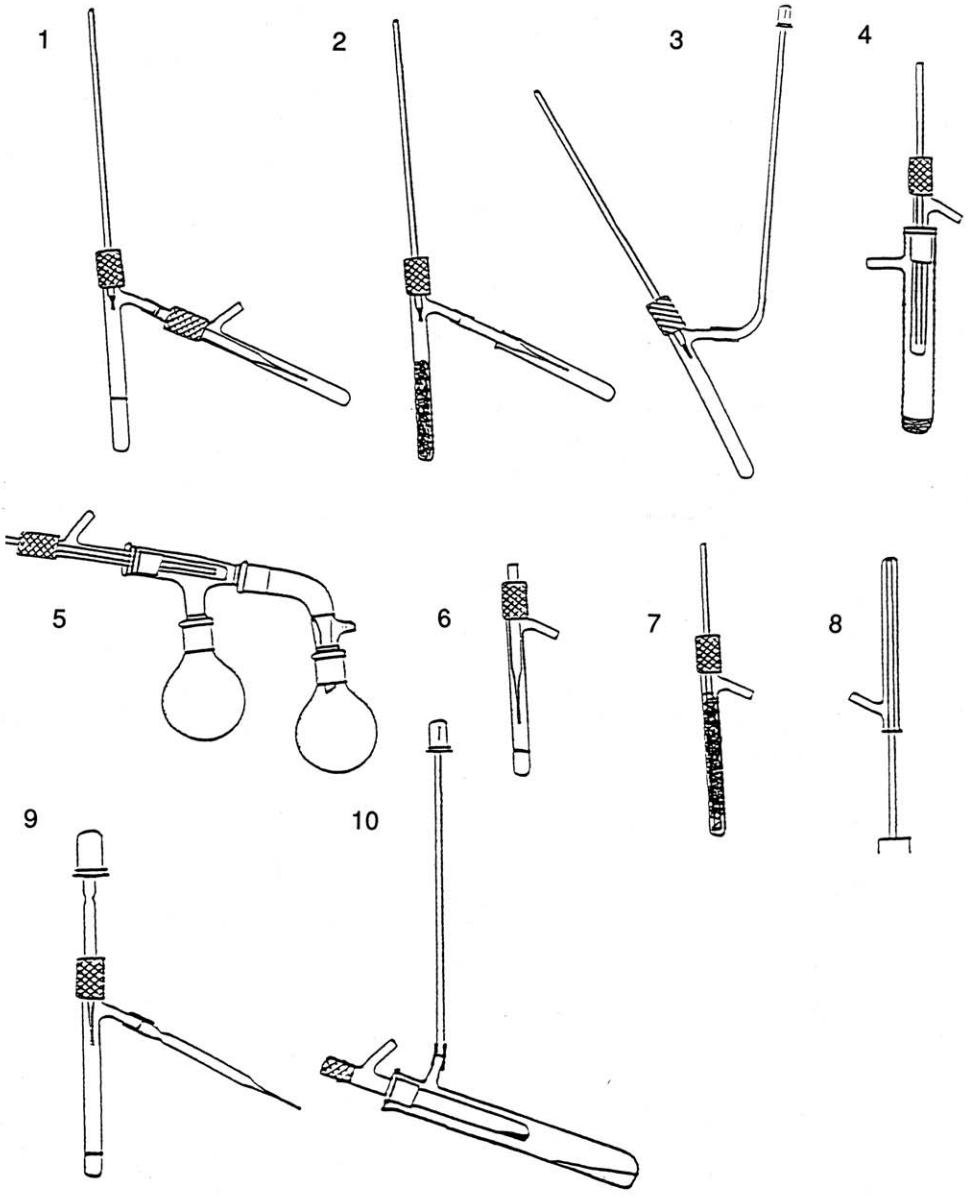
This device can be used for a variety of operations as described below and illustrated in the accompanying diagrams.

The SISATT can function as a normal or vacuum distillation flask. The collection tube can be set into a beaker or flask filled with ice to cool the distillate. See Figure 1.

The addition of packing to the tube will allow its use as a fractionating still. The packing can extend down to the bottom of the tube without altering the efficiency of the process. See Figure 2.

A long section of glass tubing can be attached to the side tubulation to act as an effective air cooled condenser. Capping off the tube with a rubber bulb with a vertical cut, a Bunsen valve (*1*), prevents most vapor loss. When a 5 g sample of acetone was refluxed for 4 h using a 4-ft section of 8-mm glass tubing, the loss of weight was only 2½%. See Figure 3.

Adding a section of glass tubing to the SISATT equipped with a rubber adapter converts it into a cold finger con-



Microscale apparatus assembled from a side-arm test tube.

denser. Placing the cold finger condenser into a 6-in. side arm test tube forms a sublimation apparatus. Sublimations can be carried out at reduced pressure by sealing the SISATT into position with a rubber washer made by cutting off the top of a 1-mL rubber dropper bulb. See Figure 4.

Seating the cold finger condenser into a three-way adapter gives a short path distillation apparatus capable of handling larger volumes of distillate. See Figure 5.

Solvents can be evaporated quickly by attaching an aspirator to the side arm of a SISATT that has a rubber adapter with a Pasteur pipet through it. See Figure 6.

An inexpensive drying tube can be constructed by placing a small amount of cotton in the bottom of a SISATT, adding a section of glass tubing, drying agent and more cotton. See Figure 7.

Gas vapors that escape from the top of a reflux condenser can be removed by inverting a SISATT over the top of the glass tubing used as a condenser and attaching the side arm of the SISATT to an aspirator. The system is not under any reduction of pressure because there is no seal between the glass tubing and the SISATT. See Figure 8.

A medicine dropper on top of the SISATT may be used to add liquid to a reaction. This apparatus has been used as a mini steam distillation apparatus and to generate acetylene gas. See Figure 9.

While not as elegant as an Abderhalden Drying Apparatus, this setup will serve the same purpose for a small sample and is much cheaper. See Figure 10.

The SISATT also can act as a vacuum flask for suction filtrations using a small Hirsch funnel.

Acknowledgment

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Literature Cited

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Microscale Column Chromatographic Isolation of a Red Pigment from Paprika

Kevin J. West[1] and Paul Rauch[2]
University of Wisconsin–Whitewater
Whitewater, WI 53190

The isolation of a red pigment from paprika using a macroscale procedure by Miller and Neuzil (*1*) was satisfactory with only a few problems. However, the difficulties encountered with the microscale experiment prompted an effort to devise a better microscale version. The problems associated with the procedure included the quantity of solvent needed for the chromatographic separation (50–100 mL ligroin, 300 mL methylene chloride and 50–100 mL ethanol) and the time necessary to make the chromatographic column and perform the separation (often more than one laboratory period, resulting in poor separations). The method described here overcomes these difficulties while reducing the volume of solvents needed to about 100 mL total for the experiment.

Procedure

Isolation of the Pigment Mixture

The pigment mixture is obtained by refluxing 0.25 g of paprika in 10 mL of methylene chloride for 20 min using a 10-mL round-bottomed flask and condenser. The solid is removed by vacuum filtration using a Buchner funnel, and the solid is discarded. The filtrate is transferred to a 50-mL Erlenmeyer flask and concentrated to 1–2 mL using a steam cone.

Column Preparation

Approximately 50 mL of a methylene chloride:ligroin mixture (1:3 by volume) is prepared. An 18-gauge hypodermic needle (usually included in the microscale kit) is inserted through a rubber stopper that will fit a 125- or 250-mL vacuum filtration flask. (A drop of mineral oil on the stopper at the point of insertion makes this easier.) The stopper is inserted in the filter flask, and a solid phase extraction/filtration column with a filter frit is placed in the luer fitting of the hypodermic needle. (A 6-mL Bakerbond spe Disposable Filtration Column with a 20-μm frit works well.) Silica gel (1.0–1.5 g) is slurry packet using 10–20 mL of the methylene chloride:ligroin mixture. A gentle vacuum may be applied to the filtration flask to pack the column but a 1-cm solvent head should remain on the column. A layer of sand may be added to the top of the column to protect the sorbent layer.

Separation of Pigments

The pigment mixture is mixed with 1–2 mL of the mixed solvent and placed on the column using a Pasteur pipet. A gentle vacuum is applied and the mixed solvent is added until the yellow bands are removed. The vacuum is stopped and the contents of the filter flask are transferred to a 125-mL Erlenmeyer flask. The filter flask is rinsed with 1–2 mL of methylene chloride that is added to the flask containing the yellow band solution. The column is reassembled and developed with neat methylene chloride (20–30 mL), using a gentle vacuum if necessary, until the solvent is no longer colored.

The two fractions obtained from the column are concentrated to 1–2 mL each. The concentrated pigment fractions may be compared with the original pigment mixture using thin-layer chromatography (silica gel plate and methylene chloride solvent). The red pigment (mostly fatty acid esters of capsanthin) in the second fraction is analyzed by infrared spectrometry by evaporation of the solvent from the solution on AgCl windows. An ultraviolet/visible spectrum (260–600 nm) also is obtained by dissolving one drop of the

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[1]Author to whom correspondence should be addressed.

[2]Undergraduate research student. Current address: Department of Chemistry, Ohio University, Athens, OH 45701.