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# A Simple, Semiquantitative Device for Liquid–Liquid Separations

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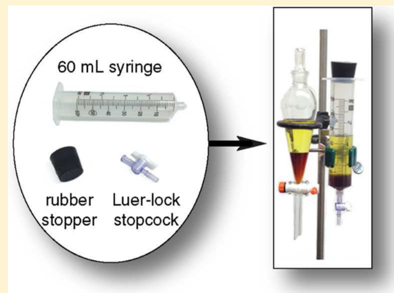
**ABSTRACT:** A simple liquid–liquid separation device is prepared using a plastic syringe and compatible plastic stopcock. The design allows students to carry out quantitative extractions using a variety of organic solvents. Extraction yields are found to be equally precise and more accurate compared to glass separatory devices. The plastic separatory device is much more economical than the glass counterpart and is far more resistant to breakage. The design allows for simple adaptation to a variety of different syringes, further lowering material costs in the teaching lab.

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The separatory funnel has long been an important piece of glassware in the organic chemistry laboratory.[1] Its design allows the user to easily visualize and separate liquid phases, most typically organic and aqueous, by draining individual layers from the funnel. Organic extraction is a common laboratory practice that uses moderately expensive and fragile glass separatory funnels. In the teaching lab, however, it is most economical to use equipment that is both reusable and durable. Although the common glass separatory funnel is reusable, its durability cannot be guaranteed and can be easily broken by inexperienced hands.

A reusable and durable separatory device was designed by attaching a plastic Luer-lock stopcock to a polypropylene syringe of desired volume. The other end of the stopcock has a slip-tip. The syringe plunger is removed and replaced with a rubber stopper of appropriate size to allow for mixing components in the syringe (Figure 1). Delivery and venting is accomplished in the same manner as a glass separatory funnel by inverting the syringe and opening the stopcock to release the pressure.

The syringe is used as the reservoir for the separation. The graduations on the syringe provide a reasonably quantitative estimate of the volume of solution in the separatory device and allow the user to visualize and estimate the volume of solution drained from the separatory device. This eliminates the additional step of transferring the solution to a graduated cylinder for measurement. Plastic syringes are readily available in volumes from 1 to 60 mL, aiding in micro- and miniscale extractions. The typical glass separatory funnel is designed with a conical shape with a small cross-sectional area near the stopcock to ease in phase identification during separation. The base of a syringe, however, is cylindrical and does not allow for this level of precision and limits a student's ability to extract the lower phase from the separatory device. Still, the benefits of graduations on microscale extractions may outweigh this deficiency.

**Figure 1.** Separation of the glycerol phase (bottom) from the biodiesel phase (top) following a synthesis of biodiesel from canola oil.[3] The as-synthesized volume ratio of glycerol to biodiesel is modified to show contrast.

With respect to durability, a glass separatory funnel allows for the use of a wide variety of organic solvents, but plastic syringes are inferior with respect to chemical compatibility. Common organic solvents such as acetone, methanol, ethanol, isopropyl alcohol, ethyl acetate, and diethyl ether have no effect on polypropylene syringes, whereas toluene, ether, pentane, and heptane cannot be used without softening and deterioration of the plastic.[2] Glass separatory funnels must be used with noncompatible solvents. Although these limitations may prevent the syringe separatory device from being used in some organic lab experiments, the general chemistry teaching lab and high school laboratories generally do not deploy such aggressive solvents.

As an example, we have used the plastic syringe separatory device to extract the glycerol phase from a common synthesis

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**Table 1. Cost Breakdown of a Glass Separatory Funnel Compared to a Plastic Syringe Device of Equal Volume**

| | | Part number | Vendor | Cost[a]/$ | |
|---|---|---|---|---|---|
| **Glass separatory funnel, 60 mL** | | 60003−992 | VWR | 50.68 | |
| | | | | **Total cost** | **50.68** |
| **Plastic syringe separatory funnel** | 60 mL Luer-lock syringe | 82002−318 | VWR | 1.18 | |
| | #6 stopper | 59580−229 | VWR | 2.10 | |
| | Male Luer-lock stopcock | EW-30600−01 | Cole−Parmer | 1.55 | |
| | | | | **Total cost** | **4.83** |

[a]Prices reflect standard retail costs as of 2/1/12.

of biodiesel from canola oil.[3] The separation is clearly visible (Figure 1) and an analysis of extracted yield indicates both methods are equally precise ($\sigma = 0.11$), but the accuracy of the plastic graduated syringe is superior (4.4% vs 13.4% error).

The reusable plastic separatory syringe device is more economical than a glass separatory funnel. Students can use any size syringe and matching rubber stopper because the stopcock is interchangeable. This greatly reduces the cost associated with purchasing a large number of glass separatory funnels in many different sizes. A home-built plastic syringe separatory device can be assembled for approximately 10% of the cost of a similarly sized glass separatory funnel (Table 1). There are a few plastic Nalgene or Teflon separatory funnels available for purchase, though the cost of these devices is even higher than glass funnels and relatively inaccessible to the general chemistry lab.[4]

## SUMMARY

The teaching lab requires durable and sustainable equipment that can be easily cleaned and reused to reduce cost and waste, and that is ideal for large enrollment courses. The interchangeable syringes accommodate varying volume sizes and several compatible solvent systems without purchasing entirely new setups. The semiquantitative delivery of liquids from the graduated syringe provides the user with added control and precision for full extraction and analysis.

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### Notes

The authors declare no competing financial interest.

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