Jce-88-673-11

DATASET · SEPTEMBER 2012

READS
12

1 AUTHOR:

Beniamin Pintea
Polytechnic University of Timisoara
9 PUBLICATIONS **63** CITATIONS

SEE PROFILE

JOURNAL OF CHEMICAL EDUCATION

LABORATORY EXPERIMENT

pubs.acs.org/jchemeduc

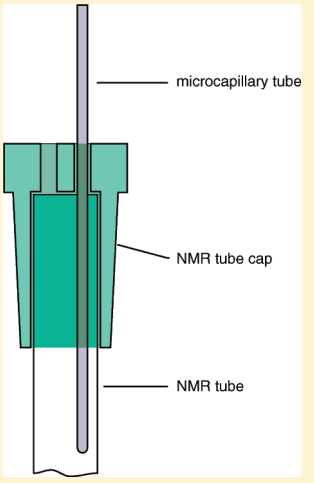
A Simple and Inexpensive Capillary Holder for Thin-Layer Chromatography

Beniamin-Nicolae V. Pintea*

CAICON Department, Faculty of Industrial Chemistry and Environmental Engineering, University Politehnica of Timişoara, Timişoara 300001, Romania

ABSTRACT: A simple and inexpensive capillary holder for applying samples onto thin-layer chromatography plates is described.

KEYWORDS: High School/Introductory Chemistry, First-Year Undergraduate/General, Second-Year Undergraduate, Upper-Division Undergraduate, Analytical Chemistry, Organic Chemistry, Hands-On Learning/Manipulatives, Laboratory Equipment/Apparatus, Thin Layer Chromatography, Qualitative Analysis



Thin-layer chromatography (TLC) is a widely used method of qualitative analysis in organic synthesis, as it uniquely combines low cost, rapidity, simplicity, versatility, small quantities of sample and low detection limits. The simplest and most economical method for the application of samples onto TLC plates is by hand, using glass capillaries. The inexpensive and widely available microcapillary glass tubes are recommended, as they are flat-ended and come in a wide range of accurate capacities. For an easy manual application, several capillary holders are commercially available (Table 1).

In certain cases, for example, when monitoring a reaction conducted in a large flask, especially when direct spotting is applicable (i.e., the sample taken from the reaction mixture is applied directly on the TLC plate), a device that can be used for both sample removal and application is useful and makes the analysis simpler and faster. A usable tool should be long enough to reach the reaction mixture and slim enough to fit easily through a 14/23-flask neck. None of the holders in Table 1 satisfies both requirements, as they are either too short (Drummond) or too thick (Camag and Analtech).

For this purpose, a simple and affordable capillary holder was devised that is easily made from parts commercially available and normally found in any organic laboratory. It consists of a microcapillary tube inserted into a polyethylene or polypropylene NMR tube cap and then attached to the NMR tube itself (Figure 1).

If the NMR tube is used as it is, that is, close-bottomed, two holes have to be bored into the cap: one to hold the capillary tube and the second to allow for pressure equalization (Figure 2). The holes should be wide enough to allow for the easy insertion of the capillary and at the same time tight enough to prevent its slippage during sample application. Sewing needles, syringe needles, or safety pins of appropriate thicknesses are suitable to create the holes. Alternatively, an NMR tube open at both ends can be used; the author's device was made from a 5 mm NMR tube after cutting off its bottom. In this case, the second hole in the cap becomes unnecessary; however, its existence does not constitute an impediment.

Broken capillary tubes can be used as long as they retain one original flat end. The capillary should extend at least 10 mm outside the NMR cap. If an even longer capillary holder is needed for sample removal, the end of the NMR tube can be easily attached to an a glass rod with a connector (e.g., a short piece of tubing).

For the sake of practicality and cost, when applying multiple samples, the capillary should not be discarded after each use, but instead a single capillary is used for all samples, rinsing it between samples to prevent any carryover contamination. Using the device in this manner, numerous qualitative TLC analyses have been performed successfully: reaction monitoring, purity analyses, identifications, checking of fractions from column chromatography, and so forth, without encountering any contamination problems.

Compared with commercial devices, this capillary holder, although less sophisticated, is also much less expensive at less than $1.20 per holder (Table 2). Moreover, considering that the capillary tubes have to be purchased anyway and that the NMR tubes can still be used for their primary purpose by simply

Published: March 03, 2011

Table 1. Examples of Commercially Available Capillary Holders

Manufacturer	Location	Product Name	Catalog No.	Price/(US $)
Drummond Scientific	Broomall, PA, USA	Bulb Dispenser	1-000-9000	1.40
Camag	Muttenz, Switzerland	Universal Capillary Holder	022.7786	215.00 (€156)
Analtech	Newark, DE, USA	Applicator Clamp	21-90	32.00

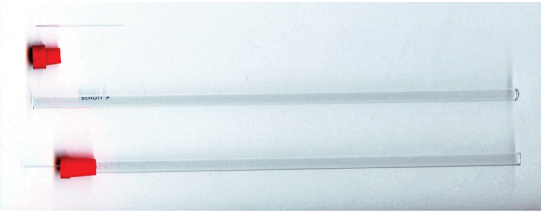


Figure 1. The components of the device (from top to bottom): capillary tube, NMR cap, and NMR tube. The assembled device is shown at the bottom.

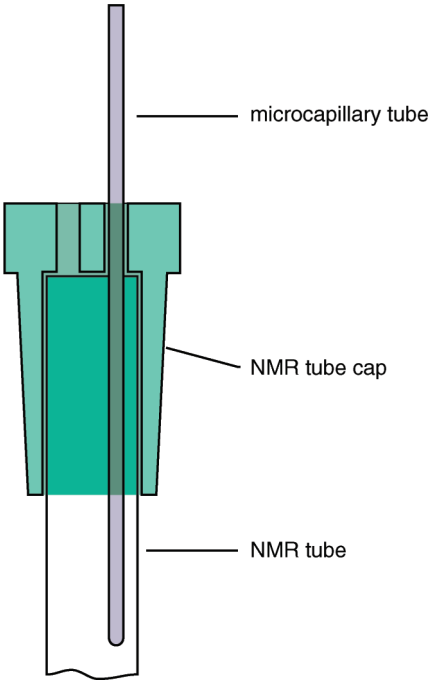


Figure 2. The assemblage of the components into the capillary holder.

Table 2. Example of Cost Estimation for the Capillary Holder Using Prices from U.S. Vendors

Component	Description	Price/(US $)
NMR tube	5 mm, 7 in., economy	<1.00 (<$100 for a pack of 100)
NMR tube cap	5 mm, polyethylene	<0.07 (<$7 for a bag of 100)
Microcapillary tube	1 μL, 32 mm	<0.13 (<$13 for a pack of 100)

detaching the capillary-holding cap (some tubes no longer suitable for spectroscopy can also be used), it follows that the actual cost of the device is the price of the NMR cap and hence literally pennies. It is also robust and easier to make, handle, carry, or fix. Its low cost and easy handling make it appropriate for student laboratories where multiple units would be necessary.

AUTHOR INFORMATION

Corresponding Author

*E-mail: bnpintea@hotmail.com.