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Communication

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Simple and Automatic Closed Grinding and Extraction System

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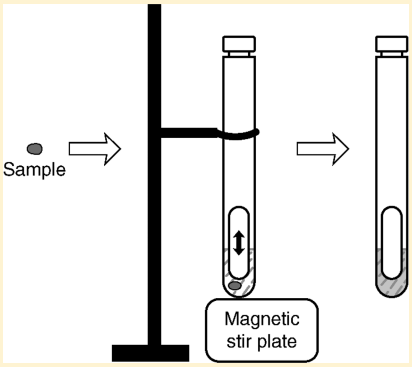
Supporting Information

ABSTRACT: This article describes a simple, automatic, and closed grinding system for small samples using common laboratory equipment that is particularly useful for air-sensitive samples or volatile compounds analyses.



KEYWORDS: *Second-Year Undergraduate, Upper-Division Undergraduate, Analytical Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Agricultural Chemistry, Bioanalytical Chemistry, Laboratory Equipment/Apparatus*

A great number of chemical analyses start by grinding and extracting the sample. This apparently elementary step can become tricky when dealing with small quantities of air-sensitive or volatile organic compounds (VOC) emitting materials. Most common grinding methods involve manual mortar and pestle or bladed devices. None of those are easy to get airtight. Moreover, the first one cannot be automated, and the second one is often difficult to clean, which could lead to cross contamination and analyte losses. Furthermore, dedicated grinding systems can sometimes be costly. This article describes a simple protocol to solve this problem using common laboratory equipment. This protocol has been successfully applied to the lossless extraction of insects' secondary metabolites[1] and to plant VOC sampling and could be used for a large number of applications involving the crushing or grinding of VOC-emitting or air-sensitive samples.

MATERIALS

This method requires a narrow tube with a tightly closing top (e.g., a Sovirel-type tube), a stand with a holder, a magnetic stir bar fitting vertically the inside of the tube, and a magnetic stir plate (Figure 1). Any kind of narrow strong-walled glassware could be used, but best results were obtained with round-bottomed glass tubes that fit the stir bars in a vertical position and where sample particles remain in the center of the tube where the stir bar action is the strongest. Tubes should be long enough to allow the stir bar move freely up and down without hitting the top. Suitable glassware size is defined by the stir bar−stir plate interaction. The optimal stir bar should fit the inside of the tube, although not too tightly to avoid being stuck by the sample particles. Large glassware would require a large stir bar that could be too heavy for the stir plate and would move vertically too slowly or even not move depending on the stir plate strength. For hard samples, a tip of Pasteur pipet (or any fine piece of glass that could be easily broken and reduced to a fine powder) can be added to improve the grinding.

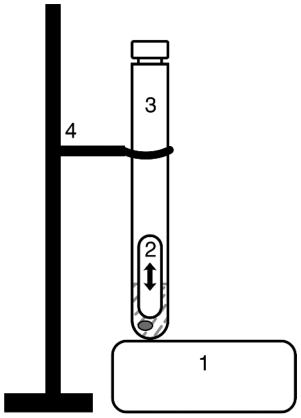


Figure 1. Extraction system: (1) magnetic stir plate, (2) magnetic stir bar, (3) Sovirel-type tube with sample and extraction solvent, if needed, and (4) holder. The solid sample and extraction solvent are shown in the tube.

METHOD

The sample is placed in the tube. If needed, a small quantity of extraction solvent can be added. The total volume should not

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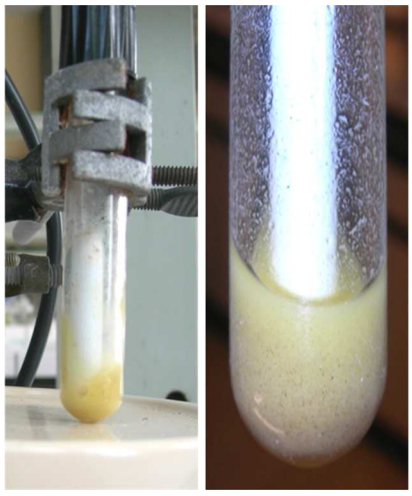

Figure 2. Ladybird beetle after a two minutes grinding in methanol.

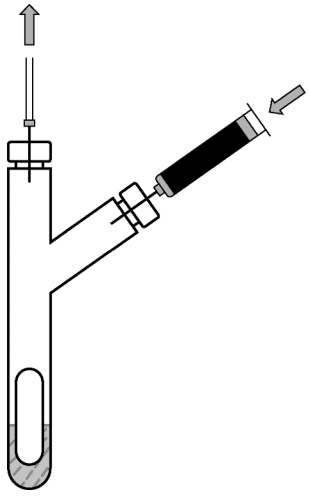

Figure 3. Adapted system for active VOCs sampling. Incoming air is cleaned, for example, on activated charcoal maintained in a glass syringe by two glass wool plugs. In this case, caps with septa are used. Arrows indicate the airflow in this system.

exceed the third of the tube, as too much volume would slow down the motion of the stir bar. The magnetic stir bar is added and the tip of a Pasteur pipet for the fine grinding of hard samples can be added if needed. The tube is positioned off center on the magnetic stir plate and the stir plate is turned on. Space or particles (sand, glass fragments, ...) should be avoided between plate and tube to prevent breakage. When the tube is at the correct location on the stir plate with the correct agitation frequency (both determined by trial and error), the magnetic stir bar will alternately be propelled upward and attracted downward because of the alternating magnetic field, acting like an automatic pestle. Quickly powdered, the Pasteur pipet tip will enhance the system efficiency by its abrasive properties. This system is left to extract until the desired grinding is obtained. Using a 45 mm stir bar with a IKA labortechnik RCT basic magnetic plate (Filter Service, Eupen, Belgium), fine grinding of small insects is completed in less than 2 min. Figure 2 shows a ladybird beetle after a two minutes grinding in methanol. The crushing of plants to analyze VOC is achieved in a matter of seconds.

Being completely closed, this system avoids any analyte losses. For air-sensitive compounds, the tube should be filled with nitrogen prior to extraction. This system is also suitable for VOC analyses by using septum caps. For passive solid-phase microextraction (SPME) sampling, the fiber is simply exposed by piercing the septum. For active sampling, incoming air has to be cleaned, which can be done using the setup presented in Figure 3.

■ ASSOCIATED CONTENT

Supporting Information

Two videos of the described system: (1) the grinding of a ladybird in solvent under nitrogen atmosphere to study its secondary metabolites and (2) the sampling of crushed *Arabidopsis thaliana* volatile compounds. This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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■ REFERENCES

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