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Pipette Pasteur extraction: a fast, cheap, convenient, exhaustive and environmentally friendly method for the extraction of solid samples

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Abstract

A simple and fast (~ 5 min.) method has been set up to extract small soil samples (~ 300 mg) by elution of a small volume of methylene chloride (2000 µl) on a modified Pasteur pipette. Pasteur pipette extraction thus allows to minimize sample losses by evaporation, degradation, contamination, and experimental handling. Moreover, shake and ultrasonic extractions were found to give lower extract yields, amounting respectively to 1.8 and 1.9 mg/g dry weight, than pipette Pasteur extraction (3 mg/g). This method may be applied to extract the soluble organic matter of any kind of solid sample such as sedimentary rocks, coals, aerosols, food, and biological substances, prior molecular analyses such as gas chromatography coupled to mass spectrometry.

INTRODUCTION

Although extraction methods are rarely described in details in the literature, it is one the key steps of relevant biogeochemical investigations. Soxhlet extraction usually give high yields (Lindhardt *et al.*, 1994, Collister *et al.*, 1992), but since this system is operated with a boiling solvent during long time periods, organic compounds may be either removed by evaporation or degraded by oxidation, photooxidation and mineral catalysis. Shake extraction at low temperature (Radke *et al.*, 1978, Lichtfouse *et al.*, 1994) should lower the degradation of organic molecules, but losses by evaporation are still possible, especially for low molecular weight substances. Ideally, those methods should be conducted under inert atmosphere in closed systems, but from E.L. experience in several laboratories worldwide this is rarely done.

Furthermore, mixing organic compounds with minerals using either high energy apparatus, e.g. ultrasonic (Lichtfouse *et al.*, 1997), microwave (Budzinski *et al.*, 1995) , or even low temperature, e.g. shake extraction, should favor organic/organic and organo/mineral reactions. Indeed, clay-catalysed reactions can occur under very mild conditions. For example, the treatment at room temperature for 1 h. of cholest-5-ene in cyclohexane in presence of montmorillonite clay gives quantitatively rearranged cholest-13(17)-enes (Sieskind and Albrecht, 1985). Under similar conditions, cholesterol reacts with benzene to give phenylcholestenes with 50% yield in 30 min (Sieskind and Albrecht, 1993). Therefore, ideally, the following conditions should be lowered for extraction : O₂ contact, high temperature, high energy apparatus (sonication, micro-wave), extraction time, transfert and concentration steps, costly materials and solvent volume (health hazard).

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Here, we report a simple method which fulfil the following requirements : it is fast to shorten contact with minerals and atmosphere; yields are higher than shake and ultrasonic extraction; the absence transfert steps and the extraction at room temperature minimizes degradation and losses by evaporation; the use of few small materials and solvent volumes makes it cheap, environnementally friendly, and reduces possible contamination of the sample, e.g. by plasticizers and atmospheric fallout. It requires less than 1 m² of laboratory space under a ventilated hood and may be also operated in field studies.

EXPERIMENTAL

Material preparation

Silica gel 60 (Merck, particle size 0.063-0.200 mm, 70-230 mesh ASTM) and cotton wool are washed three times with methylene chloride either by soxhlet extraction or by shake extraction in a beaker. The cotton wool is allowed to dry over an aluminium foil under a ventilated hood overnight. The silica gel is allowed to dry 2 days under a ventilated hood then reactivated in a beaker in a ventilated oven 4 h at 60°C, 4 h at 90°C, 4 h at 110°C, then kept in a dessicator with blue silica gel dessicant. Methylene chloride (CH₂Cl₂) is either purchased at high purity (GC, HPLC grades) or distilled. Pasteur pipette columns are made by cutting carefully the narrow end of 150 mm Pasteur pipettes (length 95 mm, top external diameter 7 mm, see Figure 1). A small piece of cotton wool is introduced into the pipette and push firmly on the bottom. The pipette is washed with CH₂Cl₂ (3 x 500 µl) using a 1 ml syringe, then kept in a clean box until extraction. Screw top vials (Varian) are washed twice with 1 ml methylene chloride.

Pasteur pipette extraction

The extraction apparatus is drawn on Figure 1. Typically, 5 kg of soil from an ancient gaswork are well-mixed. 50 g are dried under a ventilated hood at room temperature, sieved to 2 mm then finely grounded. 300 mg are weighted in a 10 ml beaker then mixed with 300 mg of silicagel to easy solvent elution in later extraction. A small amount of silica gel is loaded onto the modified pipette Pasteur column above the cotton wool (silica height ~ 3 mm) in order to prevent fine mineral particles from falling into the 2 ml vial during extraction. The soil/silica gel mixture is loaded using a glass funnel (external diameters : 8 mm, 57 mm) plugged in a teflon tube (length 2 cm, diameter 8 mm). The column is gently tapped with a fingernail (3x) then loaded with 1000 µl CH₂Cl₂ using a 1 ml glass syringe (Hamilton # 1001). Elution is allowed until the solvent surface reaches the sol/silicagel top. A low pressure is applied using a pear-shaped rubber (taken from a soap gas flowmeter) filled with cotton wool to prevent rubber particles from falling into the Pasteur pipette column, and fitted with a 5 cm teflon tube (length 5 cm, diameter 8 mm). Care should be taken to apply a low pressure even when removing the pear after elution, otherwise vacuum may pull the soil/silica gel upward. A dark-brown colored extract typical of heavy fossil fuel products is observed. The elution is repeated twice with 500 µl CH₂Cl₂ : the second load is allowed to elute until the last drop of CH₂Cl₂ (colorless). The elution time is about 5 min. The elution volume (1439 µl) is calculated using extract and solvent weight (1.9 g) and CH₂Cl₂ density (1.32). At this point, the sample may be directly analysed by gas chromatography coupled to mass spectrometry in order to prevent losses of low-molecular weight substances by evaporation. Internal standards may also be introduced for further analytical steps. The mixture in the 2 ml vial is allowed to concentrate to dryness under a ventilated hood, then rapidly weighted. Alternatively, a low pressure of nitrogen may be applied provided that care is taken to stop the N₂ flow when dryness is reached, otherwise low molecular weight substances will be rapidly lost by evaporation.

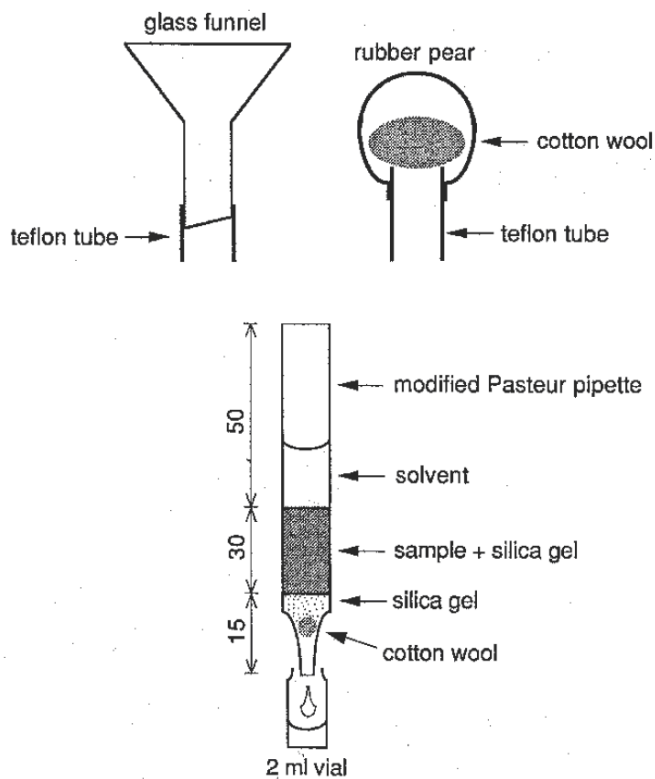


Figure 1. Apparatus for Pasteur pipette extraction of solid samples. Lengths in mm

Shake extraction

The same solvent volume/soil weight ratio was used in all extractions : pipette Pasteur, shake, ultrasonic. 1000 mg of the same soil were extracted three times 10 min. at room temperature with 2220 μl CH_2Cl_2 in a 10 ml beaker using a magnetic stirrer. The supernatants were filtered using a glass funnel and a filter paper, collected in a 50 ml round-bottom flask, then concentrated under reduced pressure at 20°C to near dryness. The extract was then transferred with 4 x 400 μl CH_2Cl_2 into a 2 ml vial using two 1 ml syringes (one for the sample, one for the solvent), then allowed to concentrated as described above.

Ultrasonic extraction

1000 mg of the same soil were extracted 10 min. three times with 2220 μl CH_2Cl_2 in a 10 ml beaker using an ultrasonic bath. The solution is then treated as in the shake extraction (see above).

RESULTS AND DISCUSSION

Nine samples (3 replicates) of the same soil were extracted giving 3.0, 3.0 and 3.0 mg/g averaging at 3.0 mg/g for the Pasteur pipette method, 1.8, 1.7 and 1.8 mg/g averaging at 1.8 mg/g for the shake method, and 1.9, 1.9 and 1.9 mg/g averaging at 1.9 mg/g for the ultrasonic method. Obviously, the Pasteur pipette method shows the highest yields. Several explanations may explain this difference. First, shake and ultrasonic extraction are prone to losses by evaporation because of the vigorous mixing and of the higher contact surface with atmosphere during extraction and filtration. Second, the additional step of transfert should also increase experimental handling losses. Third, reactions such as degradation and polymerisation are more likely to occur due to atmosphere contact (O₂) during stirring with minerals. Whatever the case, the pipette Pasteur is clearly more exhaustive, faster, more convenient, cheaper, and may be applied to any kind of solid samples such as soil, sediment, plant, food and biological samples. Moreover, this method using a small amount of sample (300 mg) is particularly well suited for the preparation of samples for current analytical tools such as gas chromatography coupled to mass spectrometry because detection levels are now very low, e.g. in the range of 1 pg to 1 ng.

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