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I. Introduction

To ensure that a sample is free of residues that may accumulate from the natural environment a pretreatment of the sample must be carried out before combustion. This pretreatment can involve physical, chemical or both procedures, depend on actual conditions of the sample.

The first step for all samples submitted to the laboratory is a physical examination and cleaning. Obvious extraneous materials (surface dirt and contamination) are removed, using a variety of lab tools, such as forceps, scalpels, etc. Some samples can also be sieved if a specific size fraction is required. In some cases, the sample can be crushed or ground to increase the surface area. After the physical examination, the sample can be submitted for the chemical pretreatment.

Organic samples generally undergo a standard acid-base(alkaline)-acid treatment (Olsson, 1986). The sample is initially washed by an acid solution. This dissolves geological carbon accumulated from dust or soil (when the sample was taken from the ground). The second step is the base wash (also called an alkaline wash). In this step the alkaline soluble humics present are removed. This is observed by a change in the NaOH color, which changes from clear to brown. The third step is another acid wash. The acid strips away any atmospheric carbon that may have been accumulated during the alkaline wash. Lastly, the sample is brought back to a neutral pH using Millipore water to get rid of chloride. During the ABA process chloride is present and can cause two problems. The chloride may corrode the combustion tubes or interfere with the graphitization reaction. After the sample is neutral it is dried on a heating block and then is ready for combustion.

II. ABA Pre-treatment Procedure

II.1 First HCl wash

Set the heating block to 70°C. Turn the heating block on to the low setting using the black switch. Ensure that the low temperature setting is at 7, using the blue dial (figure 1).
1. Place sample into a previously baked disposable test tube. The tube should be labeled for identification and the UCIG index card should accompany the sample. The amount of sample required will depend on carbon content and number of sub-samples desired. However, try to avoid filling the tube more than 1/4 full with material. This ensures that all of the sample will completely come in contact with the solvent.

2. Add enough (figure 2) 1N HCl to submerge sample (add extra due to any liquids lost to evaporation).

3. Heat tube on hot plate at a maximum of 70°C for 30 minutes.

4. Centrifuge if sample is small and has not settled at the bottom of the tube (figure 3 - section III). As an alternative you can use an (ultra fine pipet) to remove solution. If you are unfamiliar with a centrifuge please ask for assistance.
5. Pipet and discard the HCl.

II.2 NaOH wash

6. Add enough 1N NaOH to submerge the sample.
7. Heat tube on hot plate at a maximum of 70°C for 1 hour.
8. Centrifuge if sample is not settled at the bottom of the tube.
9. Pipet and discard the liquid.
10. Repeat steps 6-9 until liquid appears clear. The color (right tube - figure 4) indicates that steps 6-9 should be repeated.

Fig. 4 Left tube is clear water. Right tube is discolored NaOH.

Fig. 5 pH paper used in step 18

II.3 Final HCl wash

11. Add enough 1N HCl to submerge sample.
12. Heat tube on hot plate at a maximum of 70°C for 30 minutes.
13. Centrifuge if sample is not settled at the bottom of the tube.
14. Pipet and discard the liquid.

II.4 Sample neutralization

15. Add enough Milli-Q water to submerge sample.
16. Heat tube on hot plate at a maximum of 70°C for 5 minutes.
17. Centrifuge if sample is not settled at the bottom of the tube.
18. Take the pH of the solvent using pH paper (figure 5). Discard the water and repeat steps 15-18 until the pH is 7.
19. Pipet and discard the liquid.

II.5 Drying
20. Place the tube in the heating block. Take off the cap and replace with aluminum foil hood. Allow to dry. Make a sign that includes what your sample is and that it is drying.
21. After sample is dry, place lock cap to keep the clean sample from exchanging carbon with the atmosphere.

III. Centrifuge Operation

To use the centrifuge, place disposable tubes (with caps on) into the holders. Keep in mind that the centrifuge must be balanced to operate correctly (figure 6). Tubes set opposite each other should weigh approximately the same. Close and lock lid using the black knob on the lid. Turn the “Time” knob to 1-2 minutes. Allow the centrifuge to come to a COMPLETE stop. Never open the door before the centrifuge stops moving or injury can result. The brake switch can be used to slow down the centrifuge. Once you have looked into the peek hole on the lid and determined that the centrifuge has stopped you may CAREFULLY remove your sample.

Fig. 7. Inside of the centrifuge, showing balanced positioning of the test tubes (circled in red).
IV. Appendix

Schematic of ABA procedure

Add 1N HCl

30min @ 70°C.

Add 1N NaOH

1h @ 70°C. Repeat until liquid is clear.

Add 1N HCl

30min @ 70°C.

Wash with water until it is neutral pH

5 min @ 70°C. Recheck pH. If is neutral go to the next step, if not repeat wash.

Dry

IV. References