Preparation of Pressed Powder Samples for XRF Trace Element Analysis

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Supplies:

Rock powder sample Polyvinyl alcohol (PVA) 2.5% solution Boric acid powder (H₃BO₃) extra pure crystals Isopropyl alcohol

Steps:

1) Remove all metal parts from the dessicator under the fume hood and clean with alcohol (Fig 1). Assemble parts.

2) Using the digital balance weigh 3 grams of rock powder into a paper cup. Add 5 drops of Polyvinyl Alcohol (PVA) to the powder and mix in thoroughly using a metal spatula. The PVA will not mix easily and it is necessary to press the powder against the sides of the cup in order to homogenize the mixture.

3) Fold the rim of the cup into a pour spout and pour the powder into the metal assembly, making sure that both the inner and outer sleeves are in place. Insert the plastic plunger into the sleeve and twist so as to tamp the powder into a pellet. Twist the inner aluminum sleeve and *pull* it up slightly, then remove both the sleeve and the plunger together. You may find that the pellet sticks to the plunger rather than staying on the bottom (a common and annoying problem with some samples). If this happens, tamp down the powder with the bent spatula and then add a little dry powder to the surface before you use the plunger.

4) After removing the plastic plunger and the inner sleeve add Boric Acid powder (H_3BO_3) so that it fills in the space around the sample pellet and completely covers its top.

5) Now insert the large metal plunger into the outer sleeve, twist slightly and place the entire assembly into the Spex hydraulic press. Increase pressure to 10 tons, wait 1-2 minutes, increase the pressure back to 10 tons if it has decreased, and then slowly release the pressure.

6) Tip the assembly upside down and carefully remove the heavy outer sleeve. Then remove the flat metal plate, exposing the surface of the pressed pellet. Cover with the recessed side of the white plastic disc and insert back into the Spex press, and press down using the top crank just enough to eject the sample pellet.

7) Wipe off excess Boric acid powder. Round the edges of the pellet by scraping with the metal spatula.

8) Label with a sharpie both the back (Boric Acid side) of the sample pellet and its container (Figure 2). Place pellets in dessicator for 24 hours.

9) Clean all parts and spatulas with alcohol and repeat the process for each sample.



Figure 1. Spex press and supplies used for producing pressed powder pellets.



Figure 2. A pressed powder pellet with labeled container.