Samples for analysis (Microscopy, XRD)

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SOP for Air Sensitive or Pyrophoric Materials

The Standard Operating Procedures outlined here refer specifically to the handling of air sensitive and pyrophoric materials. For general operating procedures of the microscopes and diffractometer, refer to the respective laboratory manuals and instructions received during initial training.

No air-sensitive or pyrophoric Material can be taken for analysis in shared lab facilities (XRD, SEM, Raman, IR, AFM, etc.)

1. Before a sample can be taken for analysis, it must be tested whether it is air-sensitive, and if it is, it must be passivated.
2. To determine whether a material can be handled in air, dry a small (<10 mg) quantity of the material in the airlock of the glovebox and expose it to air.
    i. If the test sample self-heats or ignites, **it must be passivated** before it can be analyzed in any shared characterization facility. → *Step 3*
    ii. If no self-heating occurs, small quantities of the material can be handled in air. **Larger quantities may still be pyrophoric or flammable.** Always handle with care. → *Step 4*
3. To passivate a pyrophoric sample, store the smallest amount needed for further analysis in an open container in the glove box. Let it dry for at least 12 hours, then test again if it remains pyrophoric. Repeat this if needed.
4. When taking a sample from storage for analysis, first assess the quantity needed for analysis, **typically, less than 10 mg**.
5. Bring sample holder from the outside instrument to Lab 221, where the material is stored and prepare samples for analysis in Lab 221.
6. Leave **prepared sample exposed to air for at least 10 min in Lab 221**.
7. For samples greater than 5 mg, enter the record in the registry for "*Material samples actively used in experiments*".
8. Move prepared/mounted samples between the labs using a secondary container.
9. For X-ray diffraction, take **no more than 3 sample holders with samples** outside of Lab 221 at any time.
10. After analysis, move sample holders/mounts back to Lab 221, and clean or dispose as appropriate.
11. Properly dispose the analyzed sample after analysis.
12. After sample is disposed, e-mail the PI to remove the entry from the registry of "*Material samples actively used in experiments*". Include sample ID and time stamp.