IRMS solid sample preparation

This fact sheet explains how to prepare solid samples for isotope-ratio mass spectrometry (IRMS). It explains how to prepare the balance and weighing tools, weigh samples, fold capsules and load them correctly into trays.

Preparing the balance and weighing tools

Balance display screen

Correctly centred balance (bubble in middle of circle)

Using kimwipes and ethanol, clean the balance by disassembling it and gently wiping all surfaces. Aim to remove all the dust/dirt from the balance. Reassemble the balance and ensure it is centred correctly. Perform an internal calibration on the balance by pressing the CAL button. Do not touch anything (including the bench) while balance is calibrating.

Use ethanol and kimwipes to clean the mirror, tweezers and spatula. It is also good practice to ensure the bench in front of the balance is cleaned.

The mirror and weighing tools must be cleaned between each unique sample.

Do not remove weighing tools from balance area.

Weighing samples

Tin capsules

Weighing tools (L to R): Curved tweezers, flat tweezers and spatula

Ensure you have a clean, dry sample tray. Note down tray number for your records. Zero the balance.

Using tweezers, place an 8 x 5mm tin capsule on the balance. Make sure base of capsule is as flat as possible to minimise the effects on weight stability. When weight reading is stable, zero the balance.

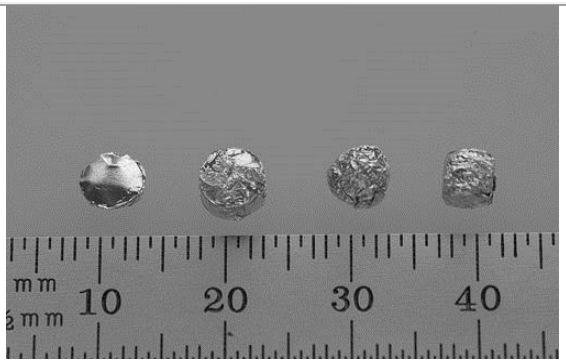
Remove the capsule using tweezers and begin adding sample. The ideal sample amount will depend on your project, what your sample is and what you expect it will contain. Discuss this with your lab contact prior to beginning work.

Once ideal mass has been achieved and weight reading is stable, press print to send the weight to the storage device attached to the balance.

Folding the capsule: encapsulation tips

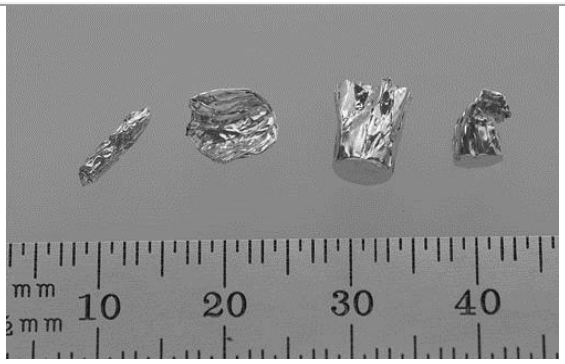
Crimp the top of the capsule and remove from balance with tweezers. Fold the top over itself. Continue folding the tin until it resembles a cube shape. Make sure there are no pieces jutting out as this may cause capsules to drop from the auto-sampler into the combustion chamber resulting in missed samples. Note that capsules folded too flat are more subject to piercing during sample transferring/loading which may results in sample contamination.

Do...	Don't...
• Crimp samples into a compact spherical, cylindrical, or cubic shape, with maximum dimensions of 6 mm for 5x9 mm tins (or 8mm for 9x10 mm tins) • Make sure tin capsule openings are folded over more than once if you can't compress the samples • Place an index card over the tray before securing the cover if you have small samples • Use clean equipment to handle the samples and tins	• Shape your samples into very flat disks (<1mm) or thin tube/cigar shapes • Ship capsules that only have their openings pinched closed or folded once • Ship samples that are leaking • Over-fill capsules (excess filter paper can be trimmed off to reduce volume) • Contaminate samples by handling with bare hands or by using sandpaper to grind plant or wood samples



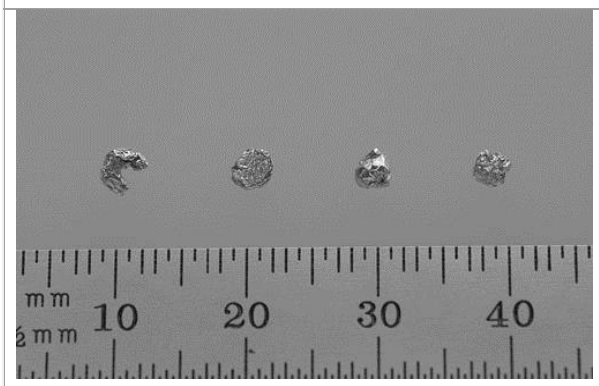
EXAMPLES: Standard sized samples (L to R)

- ~1mm tall X 5.5 mm diameter cylindrical sample
- ~ 4mm tall X 5.5 mm diameter cylindrical sample
- ~5mm spherical sample
- ~5mm cubical sample



EXAMPLES: Improperly shaped samples (L to R)

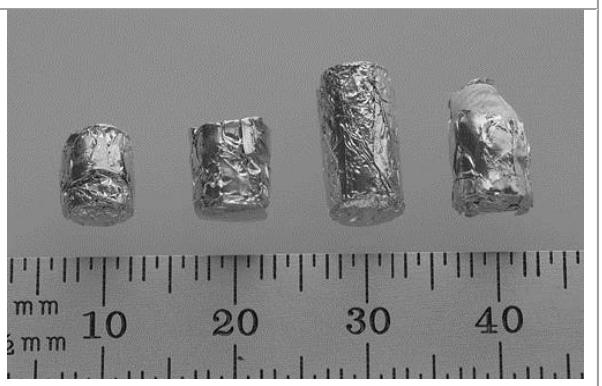
- Tube/cigar shaped sample >5mm long
- Very flat, flake- shaped sample, less than 1mm tall and over 5.5mm wide
- The opening of this capsule has only been pinched closed
- The opening of this capsule has only been folded over one time



EXAMPLES: Very small and compact samples (L to R)

These can slip between the gap manufactured into all 96-well trays. Use an index card to cover the tray before taping on the cover.

- ~1mm tall X 3mm wide crescent shaped sample
- ~1mm tall X 3mm wide cylindrical sample
- ~3mm spherical sample
- ~2.5 mm cubical sample



EXAMPLES: Very large samples (L to R)

- ~6mm tall X 5mm wide - An example of a good large sample
- ~6mm tall X 6mm wide - This sample is too wide. It will fill the well of a 96-well tray, making it hard to retrieve, and may jam the autosampler.
- ~12mm tall X 5.5mm wide - While this sample is narrow enough, the height of the sample will cause the auto-sampler to chop off the top portion, contaminating later samples and/or clogging the machine.
- Over-stuffed with filter, this sample has burst. This may occur as you are closing your samples, or later during shipping as filter tends to expand after being compressed. Trimming off excess filter will reduce volume.

Loading the capsule into trays

Place cube into clean tray.

It is highly recommended that you have the sample list with corresponding tray position with you during the weighing process for your own reference.

Before proceeding to the next sample, or if you have finished weighing samples for the day, wipe down the mirror, tweezers and spatulas. Clean the bench in front of the balance.

After weighing

Remove the weight storage device from the balance and upload the file onto a computer. The file will be in Excel form. Reconnect the device to the balance.

Clear the device ready for the next user: Press and hold CLEAR, when the screen flashes ALL, press SET.

It is your responsibility to retrieve your weights at the end of each weighing sessions. Any data stored will be cleared by the next user.

When a tray is complete, place a rubber band around the tray and tray lid. Place a hard copy of the sample submission form with each tray (between rubber band and tray). Email a soft copy to your lab contact. Place tray in desiccator 2.

Ensure both tray and tray lid are labelled to avoid confusion.

For more information and assistance

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