

ELEMENTAL ANALYSIS SERVICE

Located: Room 4.22 School Of Chemistry Open: 8am – 4pm (Mon-Fri)

Staff: Martin Jennings, Anne Davies, Carole Webb

Contact: telephone internal 54595 email chem-microlab@manchester.ac.uk

Elemental Microanalysis is used as an aid to determine the composition and purity of compounds

TECHNIQUES AVAILABLE

Elemental Analysis

We measure %-level amounts of

CHN S Cl Br I Most Common Metals But NOT Oxygen

Thermal Analysis

TGA (Thermogravimetric Analysis) and DSC (Differential Scanning Calorimeter) are available.

SUBMITTING A SAMPLE

Obtain blank request forms from the lab. A separate form is required for each sample. Complete the form in full – most important are to list all the elements present and give expected % values for elements. Forms must be signed by your academic or lab supervisor, and COSHH hazards must be clearly indicated. Special storage conditions such as dessicator or freezer should be listed.

Before submitting for analysis samples should be:

Completely Dried (under vacuum if possible) to remove water or organic solvents.

Free from any extraneous matter (eg. filter paper fibres, silica dust from TLC plates).

Homogeneous (ie. Representative of the bulk material).

Ground to a fine powder for solid or crystalline materials.

BASICALLY AS CLEAN, PURE AND DRY AS POSSIBLE! ALWAYS SUBMIT THE NEAT MATERIAL – NEVER DISSOLVE SAMPLES IN ORGANIC SOLVENTS!

How much sample is required?

For CHN & S a minimum of 2mg is required.

For halide and metal analysis about 5 -10mg is needed.

For TGA or DSC a minimum of 10mg is required.

We must be able to easily extract the sample from the sample tube.

AS A GENERAL RULE THE LOWER THE % ELEMENT YOU WANT TO MEASURE THE MORE SAMPLE WE NEED.

Place the sample in a small sample tube (not RB flasks or other reaction vessels) and clearly label (using a stick on label not marker pen) with a SIMPLE REFERENCE NUMBER. This reference number should also be completed on the request form. **MAKE SURE THE TWO ARE IDENTICAL!**

Analysis usually takes around 1 – 2 weeks. The more elements you require the longer it will take. To get results back quickly it is best to submit first for just CHN. If this is good then resubmit for metals or halides etc. If the CHN is poor you may want to recalculate or re-purify the sample before submitting for further analysis.

All results are reported to an accuracy of +/- 0.3%. The green part of the request form is returned to you along with any unused sample.

If you require any advice then please ask before submitting samples!