Preparation and measurement of ultra-small radiocarbon samples at ANSTO

A.M. Smith[1], Bin Yang[1] and Quan Hua[1]

[1] *Australian Nuclear Science and Technology Organisation, Lucas Heights, Australia*

At ANSTO, we use the Bosch reaction to produce targets for radiocarbon Accelerator Mass Spectrometry (AMS). Carbon dioxide sample gas and hydrogen are introduced into the reaction volume, which contains approximately one milligram of an iron catalyst, and the reaction proceeds when this is heated to about 600°C. Filamentous graphite is precipitated out on the iron powder which is then loaded into aluminum target holders, ready for insertion into an ion source.

At ANSTO we refer to an externally-heated reaction vessel as a 'conventional' furnace: we use electrically-heated 'tube furnaces' inside which the reaction volume is placed. ANSTO operates a bank of 24 conventional furnaces that utilise a modified Swagelok 'UltraTorr' cross to provide for gas and vacuum connections. There are a number of differently-sized cold fingers permitting operation with a range of CO_2 sample sizes from 10 µg to ~3 mg of C. As the speed and efficiency of the graphitisation process are pressure-dependent, the reaction volume must be minimized to optimize the preparation of small samples. ANSTO has developed miniaturised 'Laser Heated Furnaces' (LHF, mark-I and mark-II) where the heating power is provided by a focused infrared laser. Additionally, we have developed Micro Conventional Furnaces (MCF). In both the LHF and MCF the internal volume has been minimised by machining the gas manifold from stainless steel, which incorporates integrated valves, and by fitting a miniature pressure transducer (Saiying Electronic Technology Development, MCT-190 1 Barr range). Thus they are ideal for the graphitisation of extremely small CO_2 samples.

Samples as small as ~ 5 µg of carbon (µgC) can be prepared in the LHF and MCF with essentially complete conversion of CO_2 to C in approximately 30 minutes. The LHF have a smaller reaction volume and heated area than the MCF and add approximately half as much extraneous carbon, however they are expensive and their use is restricted to sample sizes of less than ~100 µgC. The MCF is a cost-effective solution for producing graphite from carbon dioxide sample gas. There are two type of MCF: the type 1 furnace (C1 and C2), with an integral stainless steel cold finger and the type 2 furnace (C3 and C4), with changeable glass tube cold fingers. All three LHF have integral stainless steel cold fingers. We are currently investigating operation of the LHF and MCF at pressures up to 3 Barr which will triple the maximum samples size for graphitisation reactions.

We give an overview of the graphitisation furnaces, special techniques used to produce microgram-sized targets and AMS analysis of such small samples at ANSTO.

[1] Bin Yang, A.M. Smith and Quan Hua, *A cold finger cooling system for the efficient graphitisation of microgram-sized carbon samples*, Nuclear Instruments and Methods in Physics Research B **294**, 262–265 (2013).

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