A new design for automated CO$_2$ extraction and cleaning for $\Delta^{47}$C measurements

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Measurements of clumped isotopes are time consuming and generally require the continual, or at least frequent, presence of an operator. To reduce required person-hours while maximizing information gathered during sample processing, we designed an automated extraction line that maximizes the time between human interventions while delivering a clean, unfractionated CO$_2$ separate. This line can be configured either as an online version (attached to the mass spectrometer) or as a sample port version that traps gas for later measurement. The major departure from other common acid bath autolines that rely on automated lifters and large open vessels of various fluids is a new design of the gas traps and chromatographic column that rely on heat exchange from closed loop pumped fluids of varying temperatures.

The slush traps and cryogenic focus traps were designed to minimize the number of moving parts, improve efficiency of consumables such as helium and liquid nitrogen and reduce human input. These traps are based around the principle of heat exchange, where pipes and a heater wire are nested together. In both trap types, a 1” diameter copper pipe forms the backbone, with a 1/8” diameter 316 stainless steel tube wrapped around this backbone. In the cryogenic focus traps, a ¼” copper tube is also wrapped around this backbone, forming a nested helix with the stainless steel tubing; heater wire is then wrapped around this helix. These elements are all coated with a highly thermally conductive epoxy (Duralco 132P) to insure they act as a single thermal mass. The sample will traverse the 1/8” 316 stainless steel tube and thus be subject to the temperature of the thermal block; temperatures can be changed by forcing a fluid through one of the other tubes or the main pipe, or by increasing the temperature of the heater wire. We plan to use the backbone pipe to pump ca. -80°C ethanol, and the ¼” copper tube to suck liquid nitrogen (similar to the VG SIRA and OPTIMA cold fingers). This should allow the thermal block to reach liquid nitrogen temperatures when the vacuum pump is pulling; these temperatures will be ca. -80°C when the ethanol is flowing, and above 20°C via the heater wire. To further clean samples for clumped isotope analysis, we will use a 2-stage Peltier cooler to chill a small, insulated box (similar to a GC oven) containing a column packed with Poropak. The box will be heated with a resistance heater when the chromatographic column is not in use.

Computer control and automation will be attained with an Arduino Mega microcontroller board. This controller will also be used to log temperature, vacuum pressure, sample yield, and gas composition data (from an attached residual gas analyzer) for all samples. The controller will also monitor for problems, such as insufficient sample size, and take appropriate action. If problems requiring human intervention are encountered, such as a vacuum leak or insufficient liquid nitrogen in the system, the controller can notify the appropriate person.