DOE-ID NEPA CX DETERMINATION
Idaho National Laboratory

CX Posting No.: DOE-ID-INL-16-026

SECTION A. Project Title: Accelerator Mass Spectroscope Purchase, Lab Remodel, Installation, and Operation - R1: Carbon Sample Preparation

SECTION B. Project Description and Purpose:

Rev 1
This Environmental Checklist (EC) revises EC 14-010 (Overarching [OA] 18), Accelerator Mass Spectroscope Purchase, Lab Remodel, Installation, and Operation. This work is part of the overall operation of the Accelerator Mass Spectrometer. The Project Description, Environmental Aspects, Work Activities, Conditions and Instructions in this revision are specific to this revision. The original EC remains fully valid for the work described therein.

This continues work under the laboratory-directed research and development (LDRD) 14C Analyses by Accelerator Mass Spectrometry.

This research involves the chemical techniques required to convert samples into graphite prior to analysis by mass spectrometry. The overall process steps vary according to the initial sample matrix and include digestions, leaching, heating in furnaces, gas phase transfers and cryogenic trapping in a gas manifold system. Activities are to be located in Research and Education Campus (REC)-603 lab A7 and REC-611 labs 101 and 102. This project is an LDRD that is currently funded for Fiscal Year (FY) 16 and FY17; research is intended to result in subsequent programmatic research activities that could continue for periods > 5 years from the current date. Project is funded at the $175K level for FY16.

More specifically, this work covers the sample preparation steps required to convert organic and inorganic samples into graphite prior to analyses by Accelerator Mass Spectrometry (AMS). The overall process steps vary according to the initial sample matrix and include digestions, leaching, heating in furnaces, and gas phase transfers and cryogenic trapping in a gas manifold system. In general, the process typically consists of 2 main steps: 1) Sample conversion to CO2 and 2) CO2 reduction to graphite.

1. Sample Conversion to CO2
The first step is to oxidize carbon containing samples to form CO2 where carbon containing samples are reacted with a catalyst to form CO2 and water. The exact chemical steps taken to enact this process depend on the initial matrix, but generally fall under three main categories: 1) acidification (where microliter to milliliter quantities of acid are added to samples in order to oxidize carbon to CO2); 2) gaseous oxidation (for air samples, where air samples may be slowly passed through a heated gas manifold line containing a catalyst); and 3) combustion.

The primary route to converting carbon in solid samples to CO2 is through combustion. During combustion, mg quantities of sample are carefully weighed into quartz tubes containing a catalyst (often CuO). Quartz tubes are connected to a vacuum system and evacuated. Tubes are then flame sealed using an oxygen/propane torch. Upon sealing, samples are placed inside ceramic tube “sleeves” and placed into a muffle furnace where they are baked for several hours at up to 950 degrees C to complete the combustion process.

2. CO2 Reduction to Graphite
Once samples are converted to CO2 the CO2 must be purified from H2O vapor and other trace gases prior to graphitization. Purification is typically performed by passing the CO2 gas through a gas manifold system containing two or more sequential cold traps. These traps typically consist of passing gases through a tube that is externally cooled by either a dry ice/methanol slurry (to condense H2O vapor) or liquid nitrogen (to condense solid CO2). Depending on the specific sample matrix, multiple traps may be required in order to obtain sufficiently pure CO2.

Introduction of samples to the CO2 gaseous purification manifold varies slightly as a function of the initial sample type. For combustion samples, quartz tubes are placed within a metal sleeve attached to a vacuum system, and tubes are cracked open (either by scoring with a glass scoring tool and using a flexible bellows system, or by crushing the tube by partially closing a gate valve on top of the tube). For samples containing liquids (such as those reacted with acids to produce CO2), introduction into the manifold can be accomplished by sealing sample vials with a septum type lid and introducing gases into the manifold via attaching a syringe to the manifold and piercing the septum. For air samples, air sample containers can be attached directly to the manifold (ensuring appropriate pressure controls on the introduction system are maintained for the specific air sample type and container via appropriate pressure regulators and pressure relief systems).

Once CO2 has been isolated it can be reduced to form graphite. As the quantity of carbon in most samples is too small to physically handle Fe, or a similar metal, is added as a catalyst (where during the reduction process graphite microcrystals form on the surface of the Fe powder, thus enabling more convenient handling of the purified carbon). In order to reduce CO2 to graphite, a chemical reductant such as hydrogen gas is typically needed.

There are several approaches that can be utilized to add hydrogen into the system. First, hydrogen can be added directly as H2 gas through the manifold system into the reaction cell. During this process, cells are evacuated, then an appropriate quantity of H2 gas is added and cells are heated to ~550°C.

A second, preferred approach is to add hydrogen in a solid form (such as TiH), where the solid decomposes upon heating to produce H2 gas in situ (and thus bypass the need for external H2 gas). For this approach, CO2 containing samples are cryogenically frozen into glass tubes containing mg quantities of the solid hydrogen containing reagent. After cryogenic collection of CO2, tubes are sealed (typically using an oxygen/propane torch) and tubes are placed in a furnace at up to 650°C for several hours. Upon cooling, samples may be delivered for mass spectrometry pressing and/or final sample preparation.

SECTION C. Environmental Aspects or Potential Sources of Impact:

Air Emissions

Expected air emissions such as Argon and Hydrogen are not regulated.

Generating and Managing Waste

Small quantities of industrial and hazardous waste are expected. Industrial waste includes common lab personal protective equipment (PPE), broken glass, and routine trash. Lab washwater may be discharged to the sewer system in accordance with city sewer regulations and the Idaho National Laboratory Research Center (IRC) permit. Hazardous waste, such as ignitables and corrosives may be generated. Elementary neutralization may be performed. All Solid Waste will be managed by Waste Generator Services (WGS).

Releasing Contaminants

Lab washwater may be discharged to the sewer system in accordance with city sewer regulations and the IRC permit.

Using, Reusing, and Conserving Natural Resources

All applicable waste will be diverted from disposal in the landfill when possible. Project personnel will use every opportunity to recycle, reuse, and recover materials and divert waste from the landfill when possible. The project will practice sustainable acquisition, as appropriate and practicable, by procuring construction materials that are energy efficient, water efficient, are bio-based in content, environmentally preferable, non-ozone depleting, have recycled content, or are non-toxic or less-toxic alternatives. New equipment will meet either the Energy Star or SNAP requirements as appropriate (see http://www.sftool.gov/GreenProcurement/ProductCategory/14).

SECTION D. Determine Recommended Level of Environmental Review, Identify Reference(s), and State Justification: Identify the applicable categorical exclusion from 10 Code of Federal Regulation (CFR) 1021, Appendix B, give the appropriate justification, and the approval date.

For Categorical Exclusions (CXs), the proposed action must not: (1) threaten a violation of applicable statutory, regulatory, or permit requirements for environmental, safety, and health, or similar requirements of Department of Energy (DOE) or Executive Orders; (2) require siting and construction or major expansion of waste storage, disposal, recovery, or treatment or facilities; (3) disturb hazardous substances, pollutants, contaminants, or Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)-excluded petroleum and natural gas products that pre-exist in the environment such that there would be uncontrolled or unpermitted releases; (4) have the potential to cause significant impacts on environmentally sensitive resources (see 10 CFR 1021). In addition, no extraordinary circumstances related to the proposal exist that would affect the significance of the action. In addition, the action is not “connected” to other action actions (40 CFR 1508.25(a)(1) and is not related to other actions with individually insignificant but cumulatively significant impacts (40 CFR 1608.27(b)(7)).

References: National Environmental Policy Act (NEPA) Implementing Procedures, Final Rule, 10 CFR 1021, Appendix B to Subpart D, Categorical Exclusion B1.31 "Installation or relocation of machinery and equipment."

Justification: The proposed activities are consistent with CX B1.31 "Installation or relocation and operation of machinery and equipment (including, but not limited to, laboratory equipment, electronic hardware, manufacturing machinery, maintenance equipment, and health and safety equipment), provided that uses of the installed or relocated items are consistent with the general missions of the receiving structure. Covered actions include modifications to an existing building, within or contiguous to a previously disturbed or developed area, that are necessary for equipment installation and relocation. Such modifications would not appreciably increase the footprint or height of the existing building or have the potential to cause significant changes to the type and magnitude of environmental impacts."

Is the project funded by the American Recovery and Reinvestment Act of 2009 (Recovery Act) ☐ Yes ☒ No

Approved by Jack Depperschmidt, DOE-ID NEPA Compliance Officer on: 3/15/2016