

Preparation and Shipping Instructions for Solid Phase Organic Synthesis (SPOS) Samples for Raman Analysis

Sample Preparation Instructions

1. SPOS samples must be lightly ground to remove any clumps present.
2. Samples are placed in special 5-mm wide, 3-inch long NMR tubes to a minimum depth of 9 mm. [If tubes are not filled to the 9 mm depth the laser beam will not make contact with the sample and there will be no signal.]

Tubes are ordered from:

Wilmad/LabGlass
1002 Harding Hwy
Buena, NJ 08310
800-220-5171
www.wilmad.com

Cat # WG-5MM-ECONOMY-3

DO NOT SUBSTITUTE

This number is not in the online catalog but is active.

3. If sample quantity is limited, an equal amount of FTIR-grade KBr may be mixed with the sample. Alternatively, two or three 3-mm glass beads may be placed in the tube prior to adding sample. Gently tap the tube to evenly distribute the sample around and on top of the beads. [Please note that either approach will result in a slightly lower Raman signal.] Beads are available on request via email to dmsteffen@purdue.edu or from ChemGlass Cat # CG-1101-02.
4. Each tube MUST be labeled with a unique sample identification code. Use an institution abbreviation in the identification code e.g. UIC for University of Illinois Chicago or BSU for Ball State. Ensure that the label is placed at the top of the tube otherwise the tube will not fit in the autosampler slot.

Shipping Materials

Corrugated cardboard; provided by Instrumentation Lab at request

Screwcap mailing tube (3 1/8" x 7 1/2"); provided by Instrumentation Lab at request

Raman sample list

Tape

Packing material

Shipping Instructions

1. The corrugated cardboard has slits to accommodate 15 3" NMR tubes. Label the slits 2 through 16 (Note the photos only go up to 15). Each slit represents a position in the autosampler. Sample #1 is always a sulfur standard that is loaded by the CASPiE lab and is available to all students. Mark the smooth side of the cardboard sheet with the institution and date.



2. Assign each sample a position in the corrugated cardboard sheet, insert it into the sheet and record the sample information on a sample list. If you have labeled the tubes with paper labels, make sure that the labels are flush with the tube and do not resemble flags. Once all the samples are loaded in the cardboard, check the caps on the NMR tubes to insure they are not loose.



3. Fold the corrugated cardboard in half, roll the samples keeping the numbers on the inside of the roll, and secure the roll with tape. Place in the roll in a plastic bag and seal.



5. Place a small amount of packing material in the bottom of the mailing tube. Insert the cardboard roll and a copy of the sample list (keep the original as this is how you will know which sample is in each autosampler position when you collect data). Fill the remainder of the tube with packing material. Seal and secure the lid.
6. Mail samples to:

CASPiE Instrumentation Laboratory
c/o Debora Steffen
Purdue University
Department of Chemistry
560 Oval Drive
West Lafayette, IN 47907-2084
7. SPOS samples are considered non-hazardous according to Department of Transportation regulations and do not require additional special handling or labeling.
8. Samples and tubes will NOT be returned to the originator; they will be discarded after the end of a given semester.