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ACTIVITY/PROJECT SPECIFIC PROCEDURE

SP 12-39
CALIBRATION, USE, AND MAINTENANCE OF THE MS6 TRANSMISSION IRON-57 MÖSSBAUER SPECTROMETER
Revision 0

Effective Date: November 20, 2020

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	(printed name)	(signature)	(date)

1.0 Purpose and Scope

This document describes the Sandia National Laboratories (SNL) Waste Isolation Pilot Plant (WIPP) procedure for use of the MS6 Transmission Iron-57 Mössbauer Spectrometer manufactured by SEE Co. Calibration and operation guidelines are included in the procedure. This SP is in support of activities described in several WIPP Test Plans (TPs) with the majority of the activities for the experimental work in support of WIPP-related studies. Both SNL and contractor personnel will use this SP. This document is not meant to substitute for the manufacturer instruction manuals for the instrument (see references).

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

1.1 Instrument Description

1. Lead box at the bottom: This box contains the following:
   - 1.1. Cobalt-57 gamma radiation source (less than 50 mCi)
   - 1.2. Detector
   - 1.3. Velocity transducer
   - 1.4. Sample holding tube extending from the cryostat below
2. Cryostat installed on top of the lead box above: This cryostat contains the following:
   - 2.1. Reservoir for liquid nitrogen (5-Liter capacity)
   - 2.2. Reservoir for liquid helium (6-Liter capacity)
   - 2.3. Sample holding tube extending into the lead box above
   - 2.4. Sample temperature controlling device
3. Pressurized helium gas cylinder
4. Vacuum pumps connected to the cryostat
5. Temperature controller and liquid helium level gauge connected to the cryostat
6. Operating computer

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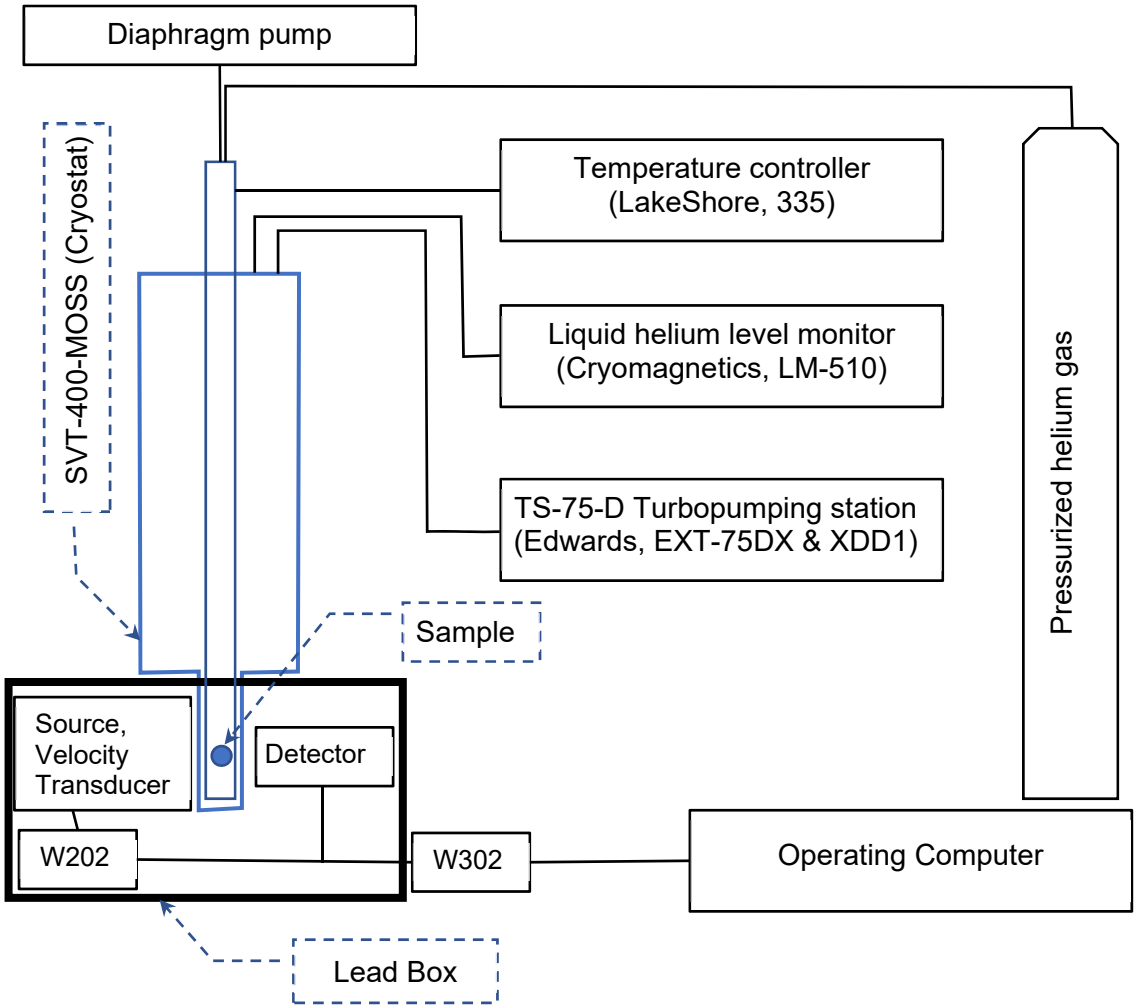

Figure 1. Schematic diagram showing the configuration of Mössbauer spectrometer and the supporting peripheral devices.

2.0 Implementation Actions

The user is responsible for reading and understanding the appropriate manuals (see section "References").

2.1 Responsibility, Safety, and Training

The technical staff, or designee, whose activities warrant the use of this procedure, are responsible for implementing the requirements of this procedure and shall conform with SNL Environmental Safety and Health programs (ES&H). Laboratory ES&H procedures and specific instrument hazards are described in the ES&H Standard Operating Procedure, *Activities in the Sandia National Laboratories/Carlsbad Program Group Laboratory* and laboratory-specific TWDs and JSAs.

In addition, the technical staff or designee is responsible for performing the measurements, following the requirements of this procedure, documenting the results, and assuring that the use of the latest revision of the document is followed.

Required trainings are RAD210, RAD218, RAD219, PRS115, PRS150, PRS250.

2.2 Switching On the System

- Make sure all the cables are securely connected.
- Turn on the operating computer, detector, velocity transducer in this sequence (Refer to Figure 1 and references, i.e., SEE Co. (2012, 2015)).

2.3 Calibration Standard

Calibration standard for the Mössbauer spectrometer is iron foil provided by the manufacturer. The iron foil is embedded in a plastic cup also provided by the manufacturer.

- A 0.001 inch (25 μm) pure iron foil is the normal working standard. Such foil can be purchased at ESPI Metals (https://espimetals.com/shop/iron-foil-001-3n.html).

2.4 Calibration

- Interval for calibration:
  - Once every other month for the purpose of regular monitoring of the instrument.
  - Prior to data collection if user decides to verify current calibration.
- Temperature for calibration: Calibration is performed at ambient room temperature.
- Put the iron foil through the sample tube using the sample loading stick.
  - Caution: Be careful not to damage the wires along the sample loading stick.
- Follow instructions in the manuals, i.e., SEE Co. (2012; 2015) in section “References”. The manuals illustrate the followings:
  - How to select and stabilize the 14.4 keV gamma radiation peak at near channel 100. Inserted below is an example screenshot to guide the readers to the manual (SEE Co., 2015), which contains full explanation on how to select the 14.4 keV gamma ray. This SP is not intended to replace the manual. (See Figure 2)
  - What to click to begin counting.
  - How to save the collected counts.
- Collect counts for a half-day or longer.
  - Count collection time varies, depending on the current source activity, density of iron in the sample
  - Usually, ~100,000 counts at the background give acceptable spectrum analysis
- Save the counts as a text file with a file extension “.t”.

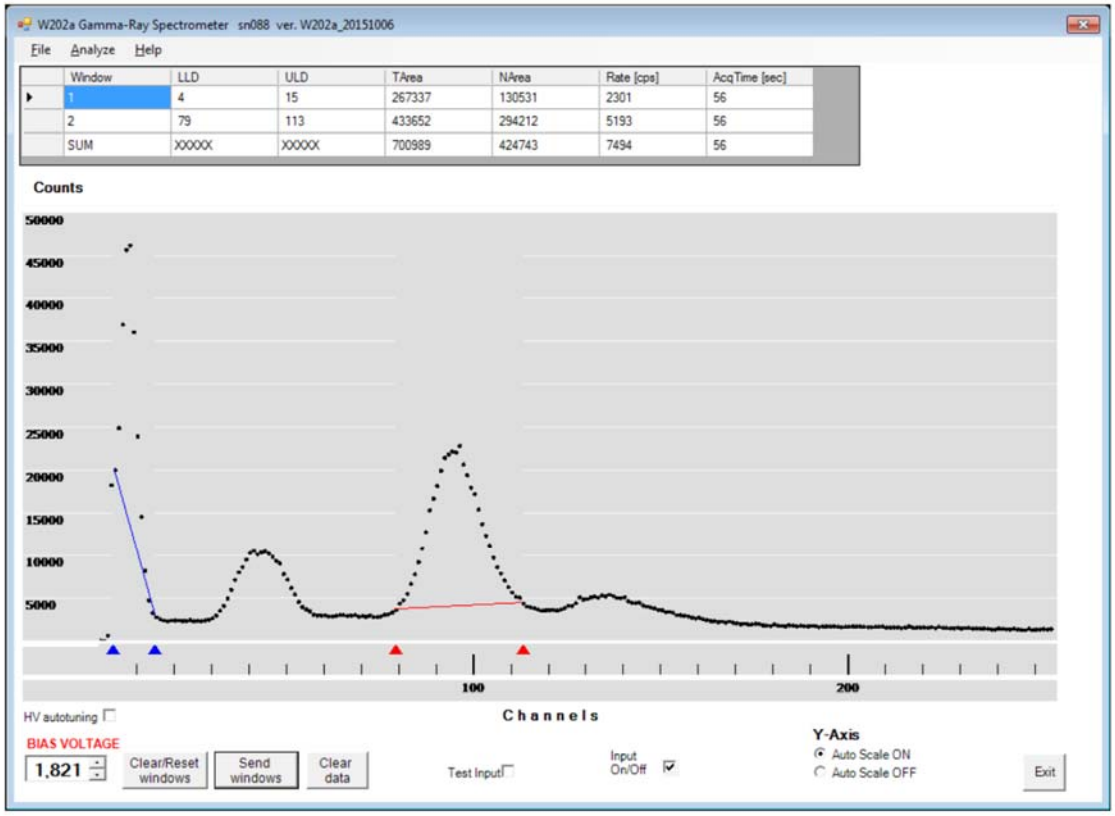

Figure 2. Selection of channels to select 14.4 keV gamma ray. See manuals (SEE Co., 2012; 2015)

- Refer to a spectrum analysis software manual, e.g., WMOSS.ORG and SEE Co. (2013) in section “References”.
  - For a new source, a thin Iron foil, e.g. 6 μm thickness, would have widths on the innermost lines < 0.23 mm/s FWHM (Full Width at Half Maximum). The theoretical minimum width is 0.20 mm/s FWHM.
  - A 25 μm = 0.001 inch foil would have innermost lines ~ 0.015 mm/s wider due to thickness broadening.
  - As the source ages, the observed line widths will increase slightly due to resonant absorption by the increasing amount of Fe-57 in the source.
  - Also, when the signal-to-noise of the gamma counting is good, the peak absorption amplitude of the innermost lines of a 25 μm Iron foil spectrum would be > 12%. This is a good way to check the counting efficiency of your system.
  - The Integral non-Linearity of the velocity scan should be 0.1% or less. The lines of the iron metal spectrum span a velocity range of 10 mm/s. So, the residuals of the linear fit to the velocity waveform (used in the WMOSS Data Load/Fold/Iron Cal procedure) should be less than 0.01 mm/s.
  - Figure 3 illustrates how the calibration spectrum typically looks like. Refer to the software manual (WMOSS.ORG and SEE Co., 2013) to convert the channel numbers to mm/s scale.
- Calibrations will be noted in the Mössbauer Logbook and documented in the scientific notebook.

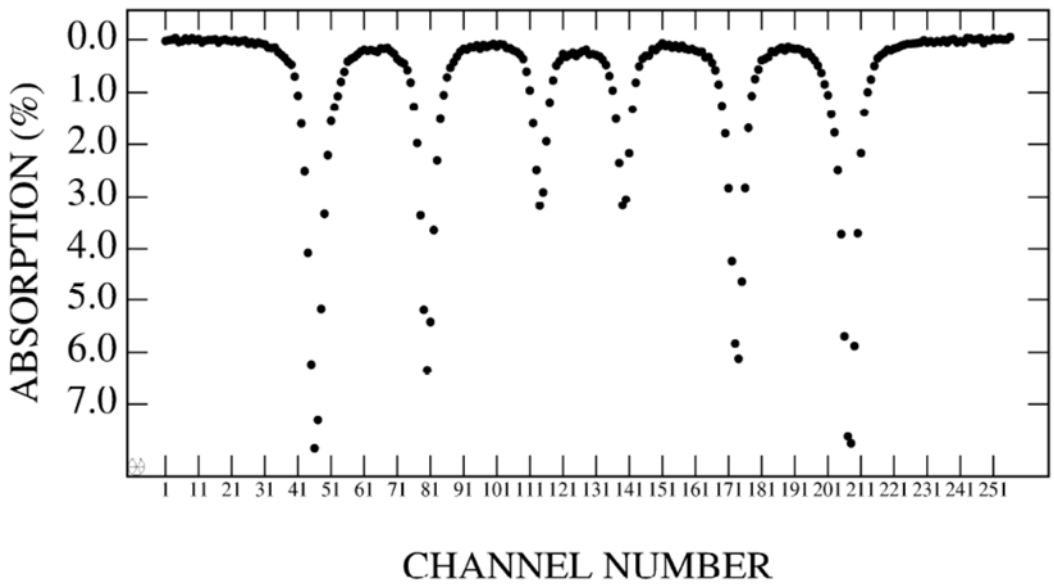

Figure 3. Example of iron foil spectrum. Refer to manual (WMOSS.ORG and SEE Co., 2013)

2.5 Data Collection

- Put sample into the plastic sample cup (Figure 4). Larger one is the cup, and the smaller one is a plug to seal the cup. Less than 0.1 gram of sample is needed. The assembly of the cup and the plug is placed into the hole of the gold-colored mount.

Figure 4. Photo of a plastic sample cup, plug, and sample mount. This mount is located at the position of blue circle in Figure 1.

- Replace the calibration standard with sample.
- Collect counts for a half-day or longer
  - Count collection time varies, depending on the current source activity, density of iron in the sample
  - Usually, ~100,000 counts at the background give acceptable spectrum analysis
- Save the counts as a text file with a file extension ".t".
- Refer to the software manual (WMOSS.ORG and SEE Co., 2013) for spectrum analysis.
- Data collection will be noted in the Mössbauer Logbook and documented in the scientific notebook.

2.6 Data Collection at Temperatures down to 80K

- No calibration shall be performed when the cryostat is filled with cryogens due to current radiation safety protocol set for the instrument.
  - Calibration, if needed for the two-months criteria or user's decision to verify the calibration, should be performed prior to putting the cryogens into the cryostat.
- Evacuate the vacuum insulation using the pump.
  - Overnight is recommended.
- Evacuate the sample tube and fill it with helium gas.
- Slowly fill the liquid nitrogen reservoirs per the cryostat manual (Janis Research Company, LLC. in section "References").
  - The cryostat has two cryogen reservoirs inside: outer, and inner.
  - Outer one (5-Liter capacity) is for liquid nitrogen
  - Inner one (6-Liter capacity) is for both liquid nitrogen and liquid helium. For the operation at 80K, this inner one is also filled with liquid nitrogen.
  - Make sure all vents are unplugged or loosely plugged.
  - Fill the outer one before the inner one.
- Refer to the cryostat manual (Janis Research Company, LLC. in section "References") to control the temperature at the sample mount (Figure 4).
- Data collection will be noted in the Mössbauer Logbook and documented in the scientific notebook.

2.7 Maintenance

No parts are user-serviceable. Until the Mössbauer Spectrometer can meet the acceptance criteria, the Mössbauer Spectrometer will be labeled accordingly.

The Mössbauer Spectrometer shall be stored with the lead box closed at all times, so as (i) to avoid unnecessary exposure to the radiation, and (ii) to minimize the exposure to dusty, corrosive, excessively humid environments, and mechanical shock and vibration. If the Mössbauer Spectrometer calibration cannot meet the acceptance criteria (see Section 2.4), contact the manufacturer (SEE Co.) under the service contract for the troubleshooting.

2.8 References

Janis Research Company, LLC., undated. Operating Instructions for the Janis Research Model SVT-400 Research Cryostat.

SEE Co., 2012. SEE Co. Model W302 Resonant Gamma-ray Spectrometer User's Manual, Version 1.0.

SEE Co., 2015. Users Manual for the Model W202A Gamma-ray Spectrometer.

WMOSS.ORG and SEE Co., 2013. Manual for "WMOSS4, Version F: Mössbauer Spectral Analysis Software". Copyright © 2011-2013 by Ion Prisecaru (wmoss.org); Copyright © 1991 to 2011 by Thomas A. Kent (seeco.us).

3.0 Records

The following records generated through implementation of this procedure shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1, "Records".

QA Record

- Scientific Notebook

Non Quality (NQ) Record

- Mössbauer Logbook

4.0 Appendices

Not Applicable.

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