

**ISO - 9000
COMPLIANCE
STANDARDS FOR
MICRO-BEAM ANALYSIS**

Ladd Catalog Number: LUHV-ELx

Ultra High Vacuum Compatible

Reference Elements, Compounds, Alloys,

Glasses and Minerals for

Micro and Macro Analysis

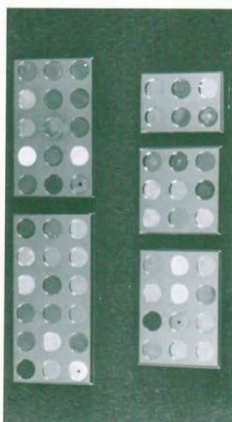
SEM/X-RAY, EPMA, XRF, SURFACE ANALYSIS (Auger, SIMS, XPS)



LUHV-EL37
Packaged for shipping under vacuum
in our 2" VACU-STORR



LUHV-EL37
Circular Retainer
Standard Configuration



LUHV-EL-X
Rectangular Retainer
Custom Configuration Only

Distributed By:

Ladd Research

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Geller
MicroAnalytical
Laboratory
offers several
unique products
and analytical
services. Devel-
oped out of the
need to support
our own labora-
tory and our
customers' needs.

SOME NOTES ABOUT OUR STANDARDS

After preparing the standards we check them using our Auger, SEM and electron microprobe instruments to verify that the proper material is in its designated position. We also examine the specimen surface for appropriate surface finish and use either Auger or x-ray analysis to insure the surface oxide is within acceptable limits and that there is an absolute minimum of embedded abrasives.

Some of the powdered standards that are embedded in either silver or tin have rather small grains. We get the largest grains available, but sometimes grains of only a few micrometers are available. In these cases it is important to position the electron beam on a polished grain surface to avoid exciting the mounting medium.

For XPS and other larger area excitation techniques not all of our standards are suitable for intensity measurements, since the area on the standard analyzed may be larger than the individual grains. They're generally adequate to measure binding energies.

To customize your set of RMs, or use our standard UHV-EL-37 configuration, please photocopy the back page indicating your choice of materials. If you have any questions at all, we encourage you to call and speak to one of our experienced staff members. In our experience very few standards are sold without discussion.

USE OF THE STANDARDS AT GELLER MICROANALYTICAL LABORATORY

These standards, and all of our products, were developed for use in our own commercial analytical service laboratory, where we provide Auger, Electron Microprobe, SEM/X-ray, XPS and metallographic services. Many of our customers require higher accuracy quantitative analysis than is commonly available. For instance, when we analyze carbon in SiC, TiC, WC, Mo₂C, and TaC we use our RMs as standards to determine sensitivity factors for Auger analysis or k-ratios for electron probe. Analyses that are often within a few % relative accuracy are common. It may take a little more time to be correct the first time, but we think the results are justified.

The enclosed list of standards is updated periodically. A continuously updated list can be found at our web site, <http://www.gellermicro.com>. If there is a standard you desire that is not listed, please call.

PRODUCTS

- **MRS-3™ Magnification Reference Standard and Stage Micrometer:** a NIST and NPL traceable standard for all types of microscopy (optical,

SEM, video, etc.).

- **Ion sputtering standards:** for calibrating ion etch rates. SiO₂ and Ta₂O₅ thin films.
- **Specimen Holders:** for JEOL™ microscopes, top referencing holder that accommodate two 1 1/4" specimens.
- **Counter-Rota-Cutter™:** pat. #4,949,605 for low speed diamond saws (Buehler ISOMET™, LECO™ VC-50, & Struer's™ Minitom) that allows you to cut faster, smoother, and thinner sections with less deformation.
- **Vacu-Storr™:** vacuum desiccators with holding times up to 5 years! Developed to protect our UHV-EL standards during shipping and while in storage.

COMPUTER CONTROL SYSTEMS

- **Energy-dispersive System upgrades:** Be window upgrades to ultra-thin for B, O and C detection. Replacement pulse processors, HV and PC based pulse height analysis boards with complete software.
- **dQant:** a replacement EPMA computer and software (Windows) system.
- **dPICT:** digital photo image collection tool, a PC based computer system (Windows) for automating analog SEMs. With its own scan generator up to 9 images can be digitized simultaneously.
- **Auger-32:** This is a Windows95 replacement computer system for the JEOL/KeveX computer control system for the JEOL JAMP-10(S) and 30 Auger microprobe analyzer.

SERVICES

Our staff takes pride in performing state-of-the-art analyses on difficult specimens and we do our utmost to go beyond just offering analytical data. We interpret the information, as well. As our satisfied repeat clients and publications reveal, we have developed several unique techniques for problem solving. We maintain your confidentiality with the utmost care as our small but capable company concentrates on providing you with the answers.

- **Scanning Electron Microscopy:** JEOL 840 with EDS and WDS, and LaB₆ source.
- **Electron Probe Microanalysis:** JEOL 733 with 4 WDS, EDS, full computer control.
- **Auger Electron Spectroscopy:** JEOL JAMP-10S with EDS and full computer control.
- **X-ray Photoelectron Spectroscopy:** XPS (ESCA) SSI-X100 small spot analysis.
- **Other services:** Profilometry (Dektak 3030), Metallography, Optical Microscopy, Particle Size Counting, Microhardness testing, Critical Dimensional Measurements.

Our current list of reference materials should be included in this brochure. An up-to-date list can be found at our web site. If you can't find what you are looking for we may be able to supply other Reference Materials to suit your needs or custom prepare your own materials for the UHV-EL.

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ISO-9000 Compliance Standards

Suitable for:

- Energy and wavelength dispersive EPMA x-ray analysis
- X-ray Fluorescence
- X-ray Photoelectron Spectroscopy
- Secondary Ion Mass Spectroscopy
- Auger Electron Spectroscopy

ISO -9000 COMPLIANCE INFORMATION ON HIGH PURITY REFERENCE MATERIALS FOR MICRO AND MACRO ANALYSIS.

To be in compliance with ISO-9000 requires the calibration of instruments if those instruments are used to make measurements for research or production purposes. The accuracy of those measurements dictate what type of analytical equipment is required, and to what degree of calibration (accuracy, precision, and frequency of calibration) is needed. Reference standards are necessary for energy scale and intensity calibration of electron, mass, and x-ray spectrometers. ISO-9000 (per ISO Guide 25) requires laboratory instruments be calibrated using standards from the following sources. The listing is from most to least desirable.

- Certified Reference Materials: those from a national standards laboratory such as: NIST (National Institute of Standards and Technology), and the NPL (National Physical Laboratory, England)
 - Standards traceable to a national laboratory (such as some of those from Geller MicroAnalytical Laboratory)
 - Standards that have been the subject of an inter-company or inter-laboratory round-robin
 - Internal standards that have permanence
- There are **very few reference materials (RMs) available from national laboratories** for microbeam analysis techniques. Many RMs are available for applications such as x-ray fluorescence, but these are not guaranteed homogeneous to the micrometer scale.

The UHV-EL STANDARDS OFFER

- Appropriate for spectral and intensity references in the following spectroscopies; Auger, X-ray Photoelectron Spectroscopy, SEM/X-ray, Electron Probe Microanalysis, etc...
- Ultra high vacuum (UHV) compatible (10^{-10} torr) construction. No epoxy or other high vapor pressure contaminating materials used. Retainers machined from SS304.
- Custom mounting bases for various instruments including; AMRAY, Cambridge (LEO), Zeiss, Philips, PHI, VG, Kratos, etc.
- Each RM is individually and separately:
 - prepared from bulk or powdered materials.
 - polished individually to prevent cross-contamination.
- removable and re-insertable into the retainer.
- Vacuum desiccator (VUS-2) for shipment.

WHY USE STANDARDS?

- **SEM/X-ray**
Spectroscopists working with electron microprobe (EPMA) analyzers routinely use standards to determine the proper Bragg diffraction angles for their spectrometer, peak shapes, and reference intensities for quantitative analysis. The accuracy of their analyses are routinely within 1-2% (relative). Quantitative analysis based upon standardless techniques, which may normalize the concentrations to 100%, give no indication that some elements may not be accounted for or represent an oxide phase. In our experience, errors with standardless analysis can

range from a few % for common metal alloys (such as stainless steels) to as much as 200% for oxides. Errors can be predicted by analyzing standards as unknowns.

• SURFACE ANALYSIS

Standards for surface analysis have been more difficult to obtain since surface cleanliness, finish, oxidation state, cross contamination, and general material availability have been limited. Commonly available (epoxy mounted) x-ray analysis standards should not be used in UHV systems due to mounting material outgassing, and lack of conductivity when carbon coatings are sputtered removed.

Spectroscopists have commonly used sensitivity factors from published reference data (books and technical papers). This practice inherently limits the accuracy of their analysis since these factors should be determined on one's own instrument since every spectrometer has its own transmission and resolution function.

RMs supplied with the UHV-EL, which contain only UHV compatible components, can be used to characterize electron energy spectrometer transmission, determine peak shapes, binding energies and sensitivity factors for quantitative analysis.

CONSTRUCTION & STANDARD PREPARATION

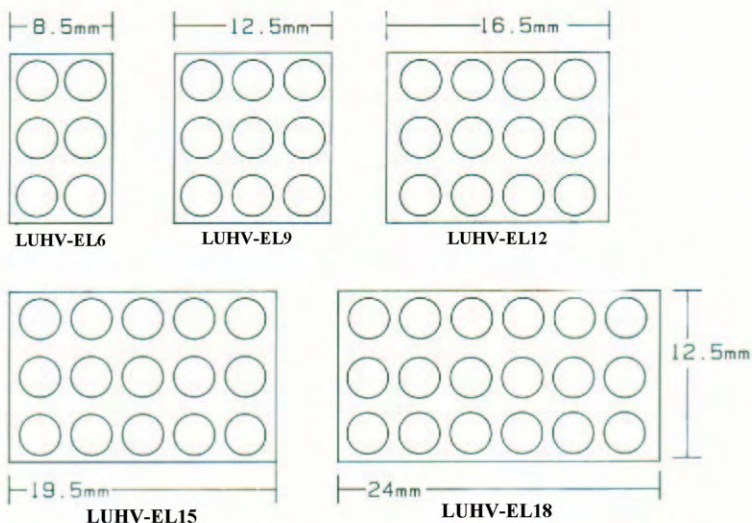
The SS304 retainer is precision machined by numerically controlled tools. The UHV-EL-37 circular retainer is laser engraved to help locate the desired RM. The standards are loaded from the back of the mount against a reference lip. All of the RMs are precisely referenced within 0.13mm of the mounts top surface. This recess helps protect the RM's surfaces from contamination and scratching. The recess also minimizes the possibility of x-ray fluorescence and ion sputter re-deposition. The RMs (which vary in thickness) are retained by SS304 clips which are inserted from the bottom of the mount. If an RM surface gets roughened from excessive ion beam sputtering or is too heavily oxidized, it can easily be removed and replaced by you. Tools for removal and insertion of new RMs, as well as extra retaining clips are provided.

The high purity materials we offer are among the best available. We size and polish each according to the properties of that material. You will find an absolute minimum of embedded abrasives. Standard sets from other manufacturers, which are polished as an entirety, suffer from poor surface finish on some materials. The polishing technique used in this case represents a compromise where you will often find embedded abrasive and smearing of the softer materials onto harder ones. Our way takes longer. We believe it is better.

Each of our RMs is a full 3mm in diameter, with the entire surface usable as a standard. If bulk materials are not available we obtain the largest crystalline chunks or powder grains available. These materials are mixed with silver flake, pressed, and then polished. What results is an analyte (the reference material) surrounded by an excellent heat absorbing and electrically conductive media. The silver can also be used as a standard. For some sulfides, which react with silver, we use tin flake as a substitute.

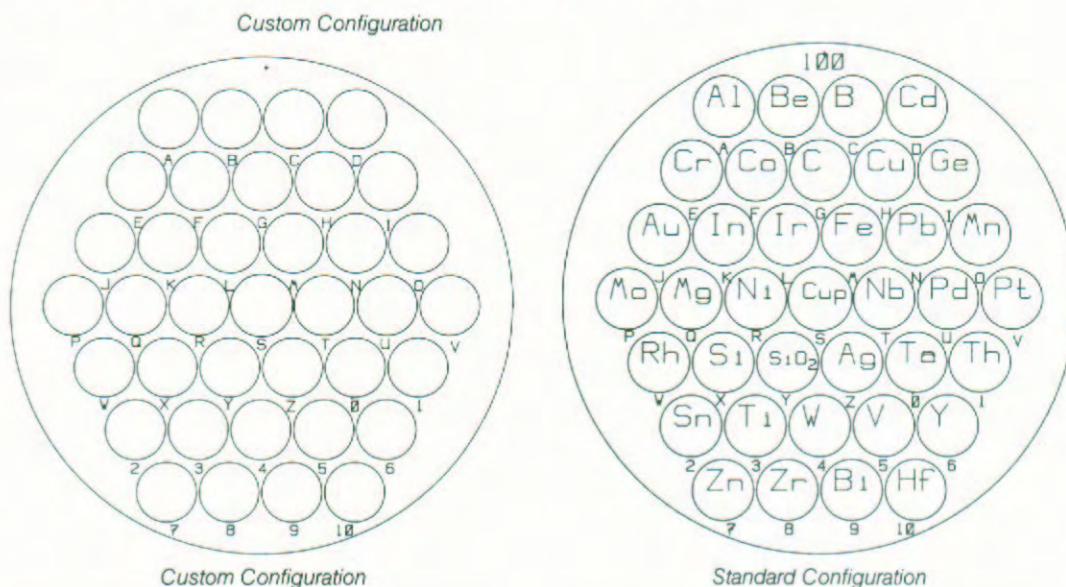
ORDERING INFORMATION

Rectangular Retainer



Note: Thickness is 3mm. Dimension accuracy: $\pm 0.5\text{mm}$

LUHV-EL37 Circular Retainer



For Our Information

Instrument Mfg _____
 Model _____
 Technique (AES, XPS, etc.) _____
 RM Requested (UHV-EL-?) _____
 Carbon Coat Insulators? _____

Your Name _____
 Company _____
 Department _____
 Address _____
 City _____ State _____ Zip _____
 Phone _____ FAX _____

Instructions

Please photocopy this page, indicating your choice of retainer and Reference Materials from the previous page. If you wish to include any materials not listed, please contact us directly for availability. Again, we are prepared to mount and polish your own special materials in our Metallographic Laboratory.

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