Frequently Asked Questions Utilizing Glow Discharge in Optical Emission Spectroscopy

Introduction

For over 70 years, industries around the world have trusted LECO Corporation to deliver technologically advanced solutions. Our state-of-theart research and development center includes teams of engineers working together to create and test ideas that will be incorporated into your next instrument—an instrument designed to meet your needs. At our in-house manufacturing facility, quality drives production. From stamped metal parts to complex electronic assemblies, LECO produces most of the components needed in our products, allowing strict control over the entire manufacturing process in accordance with the quality standards of ISO-9001:2000. As a final check before shipping, your instrument is thoroughly tested to assure quality and functionality.

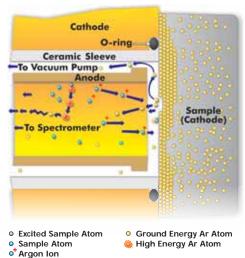
With dedicated support after the sale including highly trained service support personnel, customer training courses, SmartLine[®] Remote Diagnostics, preventive maintenance agreements, and a full line of consumables and spare parts, LECO stands by our commitment of helping you achieve the right results.



LECO World Headquarters—St. Joseph, MI

Technology & Source

- Q: What is Glow Discharge Optical Emission?
- A: Glow Discharge Spectroscopy (GDS) is an analytical technique used to measure the elemental concentrations of solid materials. A stream of argon ions mills material from the sample surface. The sputtered material is then excited in a low pressure plasma discharge and resulting light emission is used to characterize and quantify



the sample's composition. Glow Discharge offers an improved excitation source for fast, economical, accurate, and reliable sample turnaround. This source **uniformly** removes material from the sample surface which reduces the effects of metallurgical and chemical history inherent in all samples.

- **Q:** Is Glow Discharge a new technology?
- A: No, the Glow Discharge source has been known since the late 1800s. The Grimm-type lamp, originally developed in 1967, is typically employed to produce the analytical emission signal required for analysis. This style lamp, utilized in all LECO GDS spectrometers, allows for quick sample turnaround.
- Q: What advantages does a Glow Discharge spectrometer offer?
- A: Freedom from metallurgical history Analysis of difficult materials
 - Limited matrix effects
 - Linear working curves
 - Minimal spectral interferences
 - Excellent precision

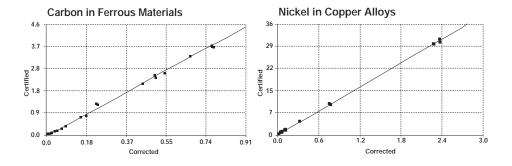
- Analysis of difficult materials (as-cast iron, low melting point alloys)
- Automatic cleaning between samples
- Low reference material and gas consumption
- **Q:** Where is the spectrometer manufactured and what type of support is available?
- A: All LECO GDS spectrometers are manufactured at the Corporation's World Headquarters in St. Joseph, MI, USA. This site also serves as the primary location for Research and Development. LECO's complete line of analytical instrumentation is supported by specialized sales, service, and applications staffs located in over 50 domestic and international offices.

Features & Performance

- **Q:** My recent Glow Discharge demo showed notable improvements in precision over spark data. In addition, Glow Discharge showed significantly fewer inter-element and line overlap interferences. Why is this possible?
- A: Glow Discharge is a low pressure, non-thermal source that sputters the material uniformly from the sample surface. The excitation of primarily atom lines occurs away from the sample surface resulting in <u>reduced</u> <u>metallurgical effects and linear calibration curves.</u>
- **Q:** Foundries have used spark-type spectrometers for years; however, difficult materials like as-cast iron, leaded resulfurized steels, and low melting point alloys cannot be run successfully by spark. Why is Glow Discharge able to analyze these materials?
- A: GDS does not preferentially attack the sample surface. Iron, carbon, and oxide inclusions are sputtered <u>uniformly</u> across the sample surface. This is also true for elements which may not be in solid solution, like sulfur and lead. Since GDS is a non-thermal sampling process, low melting point alloys like solder, Pb, Zn, and Sn are successfully analyzed.
- Q: Our company runs six different matrices. Other spectrometer manufacturers have proposed as many as 38 analytical channels. Why are fewer channels required for your Glow Discharge spectrometer?
- A: Due to the excitation of primarily atom lines, LECO GDS spectrometers provide <u>linear calibrations</u> and <u>wide dynamic ranges</u>. For example, the same analytical line chosen for one material can easily be used as the matrix line for another material. Fewer lines are necessary to analyze a broad range of materials.
- **Q:** When we change matrices with our current spectrometer, we must change electrodes and clean the stand to avoid cross contamination. How is this problem handled with LECO's Glow Discharge spectrometers?
- A: The Grimm-type GDS lamp manufactured by LECO utilizes an automatic, software-controlled reamer to clean and eliminate cross contamination.
- **Q:** Does Glow Discharge have the same accuracy problems as spark spectrometers with carbon concentrations over 0.25%?
- A: No, problems inherent to spark-type spectrometers (i.e. vaporization temperatures) do not exist with LECO engineered GDS systems across this analytical range.
- **Q:** How can the LECO GDS-400A, with a 0.4 meter focal length, offer similar performance to competitive models with 0.75 meter and 1.0 meter focal lengths?
- A: Limited spectral interferences due to narrow emission lines and the excitation of primarily atom lines assure the spectra is simpler.

- **Q:** Can you show me some examples of linearity covering a wide concentration range?
- A: C in Ferrous-based Method

Ni in Copper-based Method



- Q: How do you analyze small samples?
- A: Wire samples, as small as 0.010 inches (0.25 mm) in diameter, have been successfully tested by "packing" them together. Thin sheet, down to 0.002 inches (0.05 mm) in thickness, has been run by the use of a backplate or optional cooling puck.
- Q: What is the Ar gas usage?
- A: Typically, total consumption during an analysis is less than 1 liter.
- Q: How much sample is removed during a bulk analysis?
- A: Material is <u>sputtered uniformly</u> to a depth of approximately 100 to 200 micrometers. In cast iron, about 100 to 150 micrograms are removed.
- Q: What sample preparation is required by Glow Discharge for bulk analysis?
- A: Most often, a 120 grit surface is easily prepared on a belt or disc grinder.
- Q: What is meant by multi-scan and how is it used?
- A: Multi-scan allows an operator to scan to either side of an analytical line creating the opportunity to measure additional wavelengths close to the existing analytical lines, while maintaining optical integrity. <u>True background correction</u> is an additional feature. Multi-scan and background correction are offered exclusively on the GDS850A model.

Surface Analysis & Non-conductive Materials

- **Q:** When we use Quantitative Depth Profile (QDP) Analysis, what resolution and precision can we achieve?
- A: Analyses start as low as 10 nm into the surface. Thickness of uniformly coated materials is routinely determined within 5% RSD.
- Q: How deep can the GDS sputter go for QDP?
- A: Depths from tens of nanometers to 100 micrometers are typical. This varies with the sample type.
- Q: How long does it take to do a multi-element depth analysis?
- A: Anywhere from 30 seconds to five minutes. This depends on your sample type. For example, electrogalvanized material typically ranges one to two minutes per analysis.
- Q: Can Glow Discharge analyze non-conductive materials?
- A: Yes, this is typically achieved with an RF source available on the LECO GDS850A and SA-2000 models. This is necessary for non-conductors.

LECO GDS Product Line

LECO GDS500A Bulk Analysis

Specifications

CD-based simultaneou

Optics: CCD-based, simultaneous vacuum spectrometer

Spectral Range: 165 to 460 nm complete spectral coverage

Source: 4 mm DC standard (2 mm optional)



LECO GDS850A Bulk and Depth Profile Analysis

Specifications

Optics: 0.75 meter simultaneous vacuum spectrometer; dual source compatible

Spectral Range: 120 to 800 nm

Resolution: < 0.025 nm over spectral range

Channels: 58 maximum with background correction and multi-scan capabilities

Source: 4 mm DC standard (2, 7 mm optional); 4, 2 mm RF optional

Grating: 1800, 2400, 3600 groove/mm

