Operation of the Olympus Delta Premium Handheld X-Ray Fluorescence Spectrometer

1 Introduction

The Olympus Delta Premium Handheld X-ray Fluorescence Spectrometer is a battery-operated, portable energy dispersive x-ray fluorescence spectrometer (EDXRF) used for non-destructive compositional analysis of solids ranging in atomic number from magnesium to uranium. The instrument operates in air.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy or anthropology examinations. The operation of the Olympus Delta Premium will follow procedures defined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by Energy Dispersive X-ray Fluorescence Spectrometry (EDXRF)* and the specific parameters described below.

3 Principle

The Olympus Delta Premium uses a Rh target x-ray tube (4W, 200μA maximum) to generate the incident x-ray beam and a silicon-drifted detector (SDD) to collect and measure the photons emitted by the sample. These components are contained in a portable housing. Light elements (such as Mg, Al, and Si) are measured by optimizing beam conditions, acquiring for extended times, compensating for atmospheric pressure (altitude), and measuring over a short, protected beam path.

Electronic control and analysis software is contained within the handheld instrument but is also accessible through integrated software that can be run externally from a personal computer. To protect personnel from emitted x-rays, the instrument has a proximity interlock that aborts a test if a sample is not in front of the measurement portal.

The instrument is configured in the “Alloy Plus” mode. This mode allows several beam options for measurement. The analysis software uses a fundamental parameters routine to calculate elemental compositions that are then compared to libraries of alloy grades. The software is designed to dynamically report alloy matching. Qualitative alloy class matches can be verified using appropriate CRMs. However, to use this matching functionality for quantitative analysis, relevant validation must be performed for specific elements of interest in the specific matrix of interest.

4 Specimens

Any solid sample can be analyzed on this instrument, provided a flat area of the sample can be presented to the measurement window of the analyzer. The measurement window is covered by a thin polymer film. Care should be taken so that protruding surfaces do not puncture this film. The entire portal does not have to be completely covered for qualitative measurement. If quantitative results are desired, it is necessary to prepare the samples so that they have a flat, smooth surface.

5 Equipment/Materials/Reagents

a. Olympus Delta Premium Handheld X-Ray Fluorescence Spectrometer

b. Calibration standard – AISI Grade 316 stainless steel (316 SS) standardization coupon

c. CRMs of alloys similar to the type(s) under analysis, if alloy class comparison or elemental quantitation is required

d. Delta docking (optional) – This station provides convenient storage and recharging for the instrument. It also houses a calibration standard, communication port and an extra charging station for a reserve battery.

6 Standards and Controls

Basic standardization of the instrument requires an AISI Grade 316 stainless steel standardization block. A standard is permanently mounted in the docking station, but any 316 SS CRM can be used to standardize the instrument. For quantitation, alloy class comparison or to establish the absence of an element, CRMs as similar as possible to the material under analysis are used.

Standardization is prompted daily when the instrument is powered up. This performance check is accomplished by repeated analysis of a 316 SS standardization disc. Initiation of the automated routine is described in 8.1 Basic Operation.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Basic Operation

The instrument can be used as a stand-alone unit or controlled by an external computer. For either control method:

a. Remove uninvolved personnel from the vicinity where the instrument will be operated (to beyond ~5m radius or behind walls).

b. When the CalCheck message is present, place the unit in the docking station or present a 316 SS standardization coupon to the measurement window. Select the green ✓ Cal icon to start the standardization routine. If the CalCheck is unsuccessful, the routine will be performed again. If repeated failure of the CalCheck occurs and the operator cannot correct the problem, the instrument must be serviced by an appropriately qualified person (see 14 Safety).

c. Select the measurement beam condition(s). The “Alloy Plus” configuration allows three different beam conditions to be used in four different combinations:
   i. Beam Conditions:
      Beam 1 (40kV excitation voltage)
      Beam 2 (13kV)
      Beam 3 (8kV)
   ii. Combinations:
      Single Beam with light element (LE) suppression – Beam 1 only
      Single Beam with LE – Beam 1 only; software infers LE presence
      Two Beams – Beam 1 followed by either Beam 2 or Beam 3
      SmartSort – Beam 1 always; adding Beam 2 or Beam 3 only as needed. Specific grades are set up to automatically extend testing time. Specific elements trigger the use of an additional beam condition.

d. Fill out the specimen identification field(s) prior to each test or maintain a log recording the auto-generated test number and the corresponding specimen identification. (Warning: Post-test editing of specimen information is not enabled on this system.)

e. Acquire data; label spectra; print results to .pdf files.

8.1.1 Stand-alone Operation

a. Insert a charged battery into the analyzer handle and turn on the instrument.

b. Read the radiation safety notice screen and acknowledge user certification.

c. Perform CalCheck.

d. Select beam conditions from “Test Condition” > “Mode”. Enter specimen ID via “Setup” > “Label Defaults”.

e. Acquire sample measurements by holding the instrument against the area of a specimen to be analyzed. Start a test from the green arrow on the touch screen or pull the trigger. The red x-ray warning light stops flashing when the test is complete.

f. Test session results are grouped by date. Innov-X software on an external computer can be used to import the spectra for analysis and printing reports.

8.1.2 Computer Acquisition

a. Turn on the computer first, letting it become fully functional. Connect a USB cable between the computer and the handheld unit. Turn on the handheld unit. The Windows Mobile application will start. Choose “Connect without setting up device”.

b. Open the Innov-X control software on the computer. Select “Close Device App”. Select “Import to PC”. Select “Start”.

c. Perform CalCheck. Select beam conditions from “Test Conditions” > “Mode”. Enter specimen ID via “Setup” > “Results Test Info”.

d. Acquire sample measurements by holding the instrument against the area of a specimen to be analyzed. Start a test from the green arrow on the computer screen or pull the trigger. The red x-ray warning light stops flashing when the test is complete.

8.2 Qualitative Analysis

a. Verify the peak identification performed by the instrument by inspecting the spectrum. Annotate the spectrum using the interactive periodic table. (Refer to *Compositional Analysis by EDXRF* for more details regarding peak identification.)

b. Set report parameters on the "Print" tab under "Test Conditions" >"Setup". (The multi-print function is not enabled.) From the "View Data" page, save each result as a .pdf using the "Print" icon.

8.3 Quantitative Analysis

Although the Olympus Delta Premium is configured to output quantitative compositions, at best these can be considered approximations unless specific alloy classes have been validated.

9 Instrumental Conditions

The instrument offers limited options for operational parameters. The Alloy Plus two-beam mode provides adequate results for qualitative alloy classification and comparison.

10 Decision Criteria

General decision criteria for peak identification, spectral comparison and quantitative EDXRF analysis are described in *Compositional Analysis by EDXRF*. Options specific to this instrument include:

a. Spectra are automatically analyzed and quantified by the Olympus Delta Premium. Results are presented in table format. These compositional results should be considered as qualitative information to determine element components within the measured specimen, unless a quantitative validation has been performed for the specific matrix. To verify the accuracy of the peak identification, the graphical spectrum can be examined by the analyst. Peak labels can be generated by selecting the instrument-identified elements from the interactive periodic table chart. The software does not present marker lines for escape peaks or sum peaks. This requires the analyst to calculate and identify these peaks which are usually associated with the major constituents of a sample.

b. The nominal chemistry functionality looks for "invisible" elements based on grade identification, including Al in Beam 1 or elements like Be or C that are not detectable by XRF. These inferred elements can be used by an analyst to direct investigation (e.g., to seek an alternate identification method) but should not be used for reporting qualitative or quantitative results.

11 Calculations

Not applicable.

12 Measurement Uncertainty

Typically, the Olympus Delta Premium is not used for quantitative analysis. Should it be required, the measurement uncertainty will be estimated in accordance with *Chemistry Unit Procedures for Estimating Measurement Uncertainty* in the CU Quality Assurance and Operations Manual.

13 Limitations

Compositional Analysis by EDXRF contains general limitations of compositional measurement by x-ray fluorescence spectrometry. The Olympus Delta Premium is restricted to operating in air, thus it cannot detect fluoresced x-rays from any element lower in atomic number than Mg.

Flat samples of adequate size are required for optimal results. Where only smaller samples are available, the use of a micro x-ray fluorescence spectrometer is recommended.

14 Safety

a. Only trained operators may use this instrument. A sign stating “TO BE OPERATED ONLY BY TRAINED PERSONNEL” will be stored with the device.

b. The Olympus Delta Premium produces x-rays that may propagate many meters in open air. Operate the handheld XRF spectrometer with respect for the direction of the emitted x-ray beam. Assure no personnel are present in the region in front of the instrument in the direction of the emitted beam when operating.

c. The unit is equipped with a proximity sensor that shuts off the x-ray tube if a sample is not in place in front of the measurement window. Also, a software trigger lock will engage if five minutes pass between tests. Both situations require user intervention to unlock/restart testing. All personnel operating the spectrometer routinely are monitored via personal radiation monitors (dosimeters), administered at the unit level and tracked by the Health and Safety Group.

d. Internal components within the handheld device contain beryllium windows. These are extremely delicate and, if damaged, the beryllium dust created could pose an acute health hazard. If this occurs, isolate the instrument and seek assistance from the Laboratory Health and Safety Group.

e. The Prolene window covering the measurement window can be exchanged by the instrument operator following the instructions provided by the manufacturer. Never open the main housing of the instrument. Only a qualified manufacturer's representative or radiological technician should attempt to service this instrument.

15 References

Jenkins, R., *X-ray Fluorescence Spectroscopy*, Wiley Interscience, New York 1988

Buhrke, V.E., Jenkins, R., Smith, D.K., *Preparation of Specimens for X-ray Fluorescence and X-ray Diffraction Analysis*, Wiley-VCH, New York 1998

Tertian, R. and Claisse, F., *Principles of Quantitative X-Ray Fluorescence Analysis*, John Wiley & Sons, Inc. West Sussex, England 1982

Margui, E. and Van Grieken, R., *X-Ray Fluorescence Spectrometry and Related Techniques*, Momentum Press LLC, New York 2013

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Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), CU Metallurgy SOP Manual, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
0	08/18/2014	Original issue.
1	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 26 and is now designated Metal 503. Added personnel to section 2. Made minor editorial corrections throughout document. Incorporated section 7 into section 6 and renumbered subsequent sections. Added requirement for sampling plan retention in section 7. Updated safety requirements in section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

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