Technical Data Sheet

AZtecWave

WDS detection and **EDS** speed with **AZtec** accuracy and accessibility

AZtecWave combines the fully focussing Rowland Circle geometry Wave spectrometer with Ultim Max large area EDS detectors, resulting in:

- Fully integrated workflows designed to optimise combined EDS and WDS acquisition in the AZtec platform
- Outstanding spectral resolution to fully resolve the most closely spaced X ray lines
  - SiKα < 2eV
  - FeKα < 25eV
- Detection and measurement of trace elements down to levels of tens of ppm

Wave Spectrometer

AZtecWave utilises a fully focussing spectrometer using a 210mm Rowland circle with a 2θ range of 33° to 135°. Diffracting crystals are mounted on a six-position computer controlled turret, changeable at any position. The spectrometer is mounted at an inclined orientation for easy and fast sample positioning and includes a motorized gate valve as standard on the SEM chamber interface.

Wave spectrometer is available in two crystal configurations:

	Wave 500	**Wave 700**
Standard Crystals	4 standard crystals: LSM80N, TAP, PET, LiF (200)	5 standard crystals: LSM200, LSM60, TAP, PET, LiF (200)
Quantification Range	0.17 – 10.84 keV boron (B) to plutonium (Pu)	0.07 – 10.84 keV beryllium (Be) to plutonium (Pu)

EDS Integration

AZtecWave is fully integrated in the **AZtec** platform

- Optimised workflow for combined WDS and EDS data acquisition and quantitative analysis
- EDS and SEM parameters are used to calculate a synthesized WDS spectrum and automatically determine collection parameters for optimized data acquisition

AZtec Software

AZtecWave software includes Quant, Scan and Standardize navigators

- Setup spectrometer step for setting up counter gas, gate valve, validating spectrometer performance, and checking SEM beam current stability
- Acquisition and quantitative analysis using EDS and WDS or WDS data only
- Standardisation includes preloaded compositions and reference map for Oxford Instruments 42 and 56 element standard blocks

Quant Navigator

AZtecWave provides a guided workflow to ensure accurate, repeatable combined EDS and WDS measurements

- Describe Specimen
- Setup Spectrometer
- Preview EDS
- Acquire
- Review
- Calculate Composition

Describe Specimen

- Enter coating information
- Enter pre-defined elements for AutoID
- Functionality to register images for the purpose of specimen navigation and relocation

Setup Spectrometer

- Guided process for preparing the Wave spectrometer for analysis and validating its performance
- Proportional counter gas setup
- Gate valve operation
- SEM and sample setup
- Spectrometer performance test and validation report
- SEM beam stability check

Preview EDS

- EDS spectrum acquisition for confirming areas for analysis and automated analysis setup
- Spectrum AutoID, manual peak ID, and spectrum overlays
- Miniquant with EDS quant view

Acquire

- Collect EDS and WDS, WDS only, or Add WDS data to an existing acquisition
- Select elements for WDS acquisition
- Automatic set-up of acquisition parameters including:
  - Crystal
  - Analysis line
  - Background positions
  - Peak count time
  - Background count time
- Optional manual set up of acquisition parameters including:
  - Analysis line
  - Background positions
  - Peak count time
  - Background count time
- Manual optimisation of:
  - Beam current
  - Count time
  - Experiment uncertainty
- Synthesised WDS spectrum
- Standardisation selection
- Automatic EDS process time selection
- EDS detector position control with recommendation of detector position
- Self-populating acquisition timeline

Review

- Monitor live acquisition progress of WDS data including peak and background measurements
- EDS Spectrum display
- Miniquant with EDS/WDS quant view
- Acquisition timeline
- WDS Acquisition information table

Calculate Composition

- Review combined WDS and EDS quantification results
- Choice of 6 templates for different views of data:
  - Summary table (Single and multiple spectra)
  - Full results (customisable – single spectrum)
  - EDS Spectrum details
  - EDS Spectrum processing
  - Quant Result Details
- Quant results output in Weight%, Atomic% and Oxide%
- Toggle quantification results between WDS and EDS measurements
- Processing options (all elements, element by difference or oxygen by stoichiometry)
- Enable coating correction, automatic EDS line selection, normalisation, results thresholding
- Deconvolution elements for EDS processing

Scan Navigator

AZtecWave includes a guided workflow for acquiring qualitative WDS spectrum scans and comparing WDS scans and EDS spectrum

- Describe Specimen
- Setup Spectrometer
- Preview EDS
- Setup Scan
- Confirm Elements
- Compare

Describe Specimen

- Enter coating information
- Enter pre-defined elements for AutoID
- Functionality to register images for the purpose of specimen navigation and relocation

Setup Spectrometer

- Guided process for preparing the Wave spectrometer for analysis and validating its performance
- Proportional counter gas setup
- Gate valve operation
- SEM and sample setup
- Spectrometer performance test and validation report
- SEM beam stability check

Preview EDS

- EDS spectrum acquisition for confirming areas for analysis and automated WDS scan setup
- Spectrum AutoID, manual peak ID, and spectrum overlays
- Miniquant with EDS quant view

Setup Scan

- Two tools for selecting an energy range for a WDS scan - candidate element-line and swipe region
- Automatic setup and optimisation of WDS scan collection settings, including:
  - Crystal
  - Slit size
  - Slit position
  - Dwell Time
- EDS spectrum and synthesised WDS theoretical scan in spectrum-scan viewer
- Updating synthesised WDS theoretical scan over entered energy range to show potential WDS scan with current settings and SEM beam current
- Dwell times between 5 and 50,000 ms
- Calculation of total scan time and average step size
- Manual adjustment options for WDS scan settings

Confirm Elements

- View and monitor acquisition of WDS scan
- Display of EDS spectrum and WDS scan
- Tools to identify element-lines
- Binning options for the acquired WDS scan data
- Markers for high order line identification

Compare

- Display multiple selected WDS scans and EDS spectra
- Normalize WDS scans and EDS spectra
- Adjust appearance of WDS scans and EDS spectra

Standardize Navigator

AZtecWave requires a database of WDS standardisations, this navigator guides users through acquisition of data from a standard and standardisation of that data

- Define Standards
- Image Registration
- Setup Spectrometer
- Acquire
- Review and Accept
- Manage Standardisations

Define Standards

- Select standard database, start a new database or delete a database
- Enter coating informationnce image of standard block

- Define standards and standard compositions
- Load reference image of standard block

Image Registration

- Steps to register a standard block reference image with SEM stage positions for easy navigation to different standards (and Faraday Cup, if present)

Setup Spectrometer

- Guided process for preparing the Wave spectrometer for analysis and validating its performance
- Proportional counter gas setup
- Gate valve operation
- SEM and sample setup
- Spectrometer performance test and validation report
- SEM beam stability check

Acquire

- Select acquisition settings for WDS reference spectrum
- Select standard block and element for WDS standard acquisition
- Automatic set-up of acquisition parameters including:
  - Crystal
  - Analysis line
  - Background positions
  - Peak count time
  - Background count time
- Optional manual set up of acquisition parameters including:
  - Analysis line
  - Background positions
  - Peak count time
  - Background count time
- Manual optimisation of:
  - Beam current
  - Count time
  - Experiment uncertainty
- Self-populating acquisition timeline

Review and Accept

- Observe WDS standard acquisition, confirm and save standardisation value into library
- Spectral scan for energy position and slit position optimisation
- Acquisition timeline with information table of acquisition results

Manage Standardisations

- Periodic table highlighting elements with saved WDS standardisation
- Comprehensive information table of data for each standardisation

Optimize Navigator

- Associate the beam current measurement by Faraday Cup for WDS and the pure element standard beam current measurement for EDS so combined EDS and WDS acquisition requires Faraday cup measurement only
- Association is made for the EDS detector in fully inserted and fully retracted positions

Additional AZtecWave Steps

Reporting

- Comprehensive selection of Word and Excel templates

Exporting

- All data generated including quantitative results can be copied to the clipboard

Sample Exchange

- Guided workflow for operating gate valve during SEM chamber venting and pumping

Spectrometer shutdown

- Guided workflow for safely shutting gate valve and turning off the proportional counter gas at the end of an analysis session

Spectrometer Specifications

Specification for both Wave 500 & Wave 700

- Reproducibility of wavelength position ±0.000014nm with the LiF(200) crystal
- Linearity of wavelength position ±0.0002nm with the LiF(200) crystal and operating environment at 22° ±3°C
- Software controlled crystal change with change motor mounted directly to crystal turret
- Crystal turret capacity of up to 6 crystals, additional crystals can be added to standard configurations
- Flow proportional counter and sealed proportional counter mounted in tandem
- Slit size motor for controlling width of receiving slit located in front of X-ray detectors
- Slit position motor for positioning slit and detectors to optimize X-ray collection for each spectral line
- SEM chamber interface including motorized gate valve

W1 Spectrometer control unit

The W1 contains all the motor drive electronics and high voltage power supplies.

- 400Mbit IEEE 1394 serial communication
- Motor drives for wavelength position, slit size, slit position, crystal, and gate valve
- Fully automatic detector calibration
- Pulse height analysis using multichannel analyser
- Amplifier gain programmable from 1x to 127x
- Multi channel analyser with programmable control of lower level and window from 0 to 9.99V in 0.01V steps
- Ratemeter, counter, and timer
- Specimen current meter for from 0.01nA to 1000nA
- Front panel LED indicators of communication and system health status

Crystal Specifications

In addition to the standard diffracting crystals, other crystals can be added up to a total of 6.

- Different LSM crystals are optimised for particular elements, for example the LSM80E for nitrogen detection
- LiF220 crystal extends spectrometer range to 15.33 keV for analysis of higher energy lines

Crystal	2d (nm)	Energy Range (keV)	Element Range (K Line)	Type	Optimised Element
LSM200	19.7	0.07 – 0.22	Be to B	Johann	B
LSM80N	7.8	0.17 - 0.56	B to O	Johann	C
LSM80E	7.8	0.17 - 0.56	B to O	Johann	N
LSM60	6.0	0.22 – 0.73	C to F	Johann	O
TAP	2.575	0.52 – 1.70	O to Al	Johansson	
PET	0.8742	1.54 – 4.99	Si to Ti	Johansson	
LiF (200)	0.40267	3.33 – 10.84	Ca to Ge	Johansson	
LiF (220)	0.28473	4.712 – 15.33	V to Y	Johansson	

Table of guaranteed specifications for all crystals

Crystal	Spectral Line	Wavelength (nm)	Elemental Standard	kV, Detector Type	Resolution* (eV)	Peak Intensity (cps/µA)	P/B	Sensitivity (ppm)
LSM200	Be Ka	11.4	Be	10, FPC	8	2.4×10^4	40	335
LSM200	B Ka	6.76	B	10, FPC	15	9.0×10^5	30	63
LSM80N	B Ka	6.76	B	10, FPC	9	5.7×10^4	60	180
LSM80N	C Ka	4.47	Vitreous C	10, FPC	14	4.7×10^5	50	68
LSM80N	N Ka	3.16	BN	10, FPC	16	9.5×10^3	3	1950
LSM80N	O Ka	2.36	SiO_2	10, FPC	17	1.1×10^5	50	140
LSM80E	C Ka	4.47	Vitreous C	10, FPC	14	1.3×10^5	57	120
LSM80E	N Ka	3.16	BN	10, FPC	16	3.4×10^4	13	495
LSM60	C Ka	4.47	Vitreous C	10, FPC	12	3.2×10^4	70	220
LSM60	N Ka	3.16	BN	10, FPC	13	5.0×10^3	10	1500
LSM60	O Ka	2.36	SiO_2	10, FPC	15	1.0×10^5	65	130
TAP	O Ka	2.36	SiO_2	10, FPC	3	5.4×10^3	350	240
TAP	Al Ka	0.834	Al	20, FPC	9	2.7×10^6	800	7
PET	Si Ka	0.7126	Si	20, FPC	2	5.4×10^5	2600	9
PET	Ti Ka	0.2750	Ti	30, FPC/SPC	20	2.7×10^6	500	9
LiF(200)	Fe Ka	0.1937	Fe	30, FPC/SPC	25	1.0×10^6	525	15
LiF(200)	Cu Ka	0.1542	Cu	30, FPC/SPC	40	1.1×10^6	315	18
LiF(220)	Cu Ka	0.1542	Cu	30, FPC/SPC	35	3.0×10^5	400	30
LiF(220)	Ge Ka	0.1255	Ge	30, FPC/SPC	48	3.7×10^5	210	37

1. All data collected with specimen normal to the electron beam and X-ray take-off angle of 35°.
2. Sensitivities for standards were calculated using the following equation (Ziebold, 1967):

$C_{DL} \geq 3.29/(t \times P \times P/B)^{1/2}$
where t = total count time (1000 seconds)
P = peak count rate (0.1 µA specimen current)
P/B = peak to background ratio

Sensitivities (minimum detection limits) may not be attainable on all types of samples under all operating conditions and are therefore not guaranteed. Matrix effects, such as absorption and fluorescence, and sample coating may adversely affect sensitivity and cannot be predicted for all samples. If you have any questions regarding detection limits on specific sample types, please contact Oxford Instruments.
* Resolution values represent what is typically achieved.

Installation requirements

The following items and services are required for installation of the spectrometer and are the customer's responsibility.

- Power: 90-260 VAC, 50 or 60 Hz, 170 VA (must have ground with same potential as SEM power ground)
- Operating environment: 23°C ±5 ° with less than 65% relative humidity
- Vacuum pumping: vacuum system capable of maintaining at least 5×10^{-5} Torr inside spectrometer housing
- X-ray counting gas: cylinder of P10 gas (90% argon, 10% methane)
- Two-stage regulator: output range of 0 – 60 psig (0-4 bars) and one-eighth inch (3mm) hose barb output fitting

Visit nano.oxinst.com/AZtecWave

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