Isotopx

Product Information

Phoenix Instrument Specifications

Geological Installation

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PRODUCT INFORMATION

Introduction

The Phoenix is an ultra high precision Thermal Ionization Mass Spectrometer (TIMS) featuring the highest sensitivity, best ultimate vacuum and most stable ion optical platform.

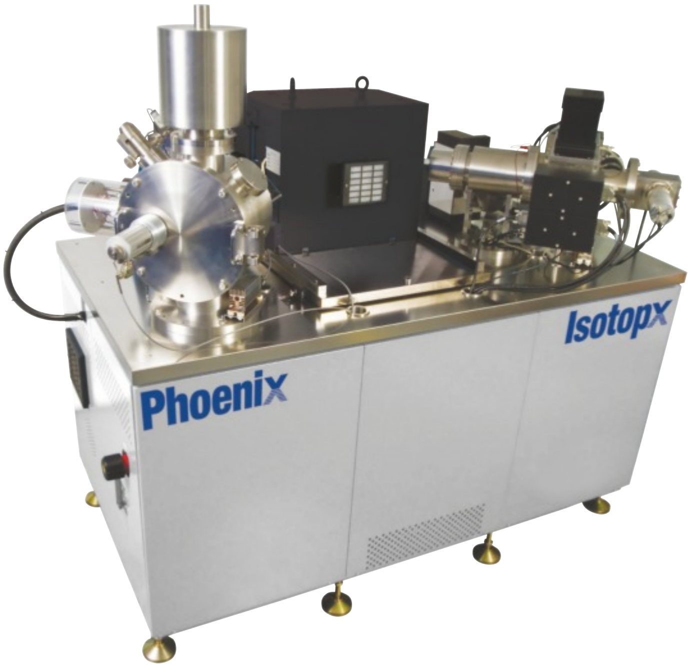
The Phoenix is a compact, single bench design. The vacuum envelope and magnet are positioned on a highly robust reinforced steel bench. All Phoenix electronics are housed below the bench with special attention being paid to ensuring adequate ventilation to the power supplies resulting in exceptional performance stability.

Ion source chamber - Highest vacuum and fastest pumping speed

- Highest grade Stainless Steel Construction designed to produce source vacuum of <1 x10^{-8} mbar in routine operation with liquid nitrogen cryopump, and <5 x10^{-8} mbar without cryopump.
- Largest capacity turbo-molecular pump available on commercial TIMS (700l/s), backed by an oil free scroll pump as standard. The turbo-molecular pump is situated immediately below the source housing and connected with a large diameter ISO160 flange providing maximum vacuum conductance between the turbo- molecular pump and the source chamber.
- Pump down to 2 x 10^{-7} mbar routinely achieved in 30 minutes, and 5 x 10^{-8} mbar in 1 hour.
- Standard liquid nitrogen cryopump, with capacity of 6 litres, sufficient for >24 hours operation with 1 fill.
- Ports for oxygen bleed, and preheat options.
- Hinged front door for easy unimpeded access to the sample magazine and ion focussing stack (collimator).
- Plug in collimator, designed to be easily removed without the need for rewiring the source.
- Differential pumping of 2 orders of magnitude between source and analyser vacuum.
- Automatic slide valve to isolate analyser vacuum from the source when turbo-molecular pump is vented.

Optional Pyrometer

The optional pyrometer is manufactured by Keller. It has a temperature range between 700 and 2400°C and the filament can be observed without removing the pyrometer from the source door. Filament temperature can be controlled through the software.



Optional Preheat

Used to increase sample throughput by pre-heating the next sample prior to its analysis. The pre-heat position is situated close to the source cold finger so that evolved material is effectively removed. Option includes a filament contact assembly, power supply plus feed-throughs and cabling.

Optional Gas Bleed

Typically used to introduce oxygen into the sample chamber to promote the production of oxides for negative ion operation. It consists of a sample reservoir and needle valve which can be opened and closed automatically through the software.

Sample Magazine

- 20 sample magazine which can be fitted accurately and easily into the source chamber. Magazine change can be achieved in approximately 1 minute.
- Sample magazine rotates perpendicular to the focal plane ensuring ZERO chance of cross contamination between samples.
- Each bead block can be configured with single, double or triple filaments.
- Bead block exit slits are easily removed for cleaning or replacing between samples.

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Accelerating voltage

- HT is fully adjustable up to +/- 8kV.
- Ions are focussed in the X, Y and Z direction using a lens stack. The ion beam width is 0.3mm.
- The lens stack or collimator is a plug in design which is easily removed from the source for cleaning. There is no need to remove or replace wires, since the collimator uses spring loaded gold pins to make contact with a fixed, wired contact ring.
- All components of the collimator can be easily separated for cleaning.
- The front entrance slit of the collimator can be removed from the collimator with the lens stack still in the source for more frequent cleaning.
- It is advisable to have a spare lens stack for negative ion applications, particularly for osmium analysis.

Magnet

- The Phoenix uses a 27cm radius air cooled electromagnet, the largest magnet on any commercially available TIMS.
- Extended geometry providing a mass dispersion >540mm.
- The magnet uses a patented exit lens to rotate the focal plane. This ensures that the focal plane is perpendicular to the ion trajectories, so that ions consistently hit the bottom of the Faraday collectors.

Flight Tube

- The flight tube has a width of 80mm which allows ions to be detected at the analyser with a relative mass dispersion of 20%.
- The flight tube is pumped by two ion pumps with a pumping speed of 40l/s and 70l/s. The pumps are situated at the front and rear of the flight tube.
- The precision engineered bench provides a level platform to allow the ion optical path to be level to within 0.2mm from source to detector.

Multicollector

A mass spectrometer is only as good as its ion detector. The requirement is to collect ions with 100.0000% efficiency combined with the lowest noise detection electronics resulting in the best possible isotope ratio precisions and accuracies.

- Each detector is hand built and tested by Isotopx engineers to very exacting standards.
- The M20 housing holds 9 Faraday collectors; one axial and 8 off axis collectors.
- Each collector is individually motorized so that they can be precisely positioned to within 7 microns, to allow for different mass dispersions.
- The Faraday cups feature 'LIF' inserts that resist cup aging phenomena - a huge weakness with graphite collectors. The long life is demonstrated by the fact that not a single 'LIF' Faraday has been replaced due to premature aging since the design was introduced in 1996.
- Isotopx guarantees Faraday cup performance for 10 years. Compare this with graphite collectors which can degrade after less than 2 years of use.
- The wide flight tube and the design of the M20 permits the analysis of isotopes with a large mass range. For example ^{6}Li and ^{7}Li can be analyzed simultaneously.

*X*act Faraday Amplifiers

Xact amplifiers represent a breakthrough in Faraday amplifier performance. They offer improved stability and significantly faster response than previous generation amplifiers.

Amplifier boards are available with $1e^{10}\Omega$, $1e^{11}\Omega$ or $1e^{12}\Omega$ gain resistors offering an extremely large dynamic range for data collection on the Faradays. Boards are simply placed in the required channel(s) allowing the user to easily customize the amplifier configuration depending on the application.

Optional Multiple ion Counting

Up to 6 ion counting devices can be added to the main Faraday array in various configurations. The ion counters use a conversion dynode situated behind a 1mm defining slit. When ions strike the dynode, electrons are produced which are drawn into a multiplier (continuous dynode) which is situated at right angles to the incident ion beam. This geometry overcomes the spatial requirement for simultaneous measurement of U and Pb isotopes. The separation of the detectors required for such applications is ~2mm, and cannot be achieved if the multipliers are in direct line of sight with the ion beam.

In addition, by not having the ion beam directly incident on the multiplier, superior gain stability is achieved. Otherwise ions would deposit onto the active surface of the multiplier causing the gain to drift and a dramatic reduction in lifetime. This is avoided using the conversion dynode.

A variable voltage is used on the input to the multiplier to control the peak shape. Voltages applied to each multiplier can be controlled independently so that each multiplier can be operated in its plateau region, giving constant gain. An overload trip circuit is used to switch off all voltages to the multipliers if the count rate exceeds 10^{6} ions per second.

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Optional Ion counting Daly detector

The Daly ion counting detector is the benchmark ion counting detector for positive ion isotope ratio applications. The Daly is unlikely to need replacement during the lifetime of the instrument. It is warranted for 10 years.

The Daly comprises a conversion dynode (approx. -25kV; fully adjustable) that attracts ions onto a highly polished surface. This results in the conversion of one ion into approximately 6 electrons. Electrons are repelled onto a high speed scintillator, producing photons, which are detected by a photomultiplier that is held outside the instrument's vacuum system.

The Daly has a very high dynamic range (>$3x10^6$), excellent linearity, low noise, exceptional gain stability, long lifetime and flat topped peaks. It can be positioned immediately behind the M20 collector, or behind a WARP filter. All operating parameters are controlled from the data system.

Optional Secondary Electron Multiplier (SEM)

The SEM can be used instead of the Daly detector or can be positioned behind the Daly as an additional ion counter. The SEM has both positive ion and negative ion detection capability and can also be used in combination with a WARP filter.

Optional WARP Filter

The WARP (Wide Aperture Retarding Potential filter) provides a potential barrier, preventing ions of lower energy (produced by collisions with residual gas molecules in the analyzer vacuum) from passing through to the ion counting detectors behind the main Faraday array. Thus abundance sensitivity, as measured using Daly Detector or ETP detector, is improved by two orders of magnitude to 10ppb at 237 with respect to ^{238}U without compromising ion transmission.

Electronics

The Phoenix electronics have a modular design so facilitating configuration for all applications. The core of the electronic system is the embedded PC that links the data system to the main electronic elements of the instrument via a fibre-optic link. The electronics are designed such that the instrument will return to a safe condition on loss of power.

- For the ultimate in isotope ratio precision and reproducibility the Faraday electronics are housed in an evacuated head amplifier with temperature regulation maintained at 16°C ± 0.02°C by a Peltier device.
- The amplifier housing is continuously pumped with a dedicated oil free diaphragm pump.
- Noise levels on the Faraday cups using $1x10^{11}$ ohm amplifier boards are close to the theoretical limit predicted for 16^oC.
- For a 10 second integration the noise is ~$1 x 10^{-16}$ A or 10mV RMS. Noise drift is typically ~$3 x 10^{-17}$ A over an hour.

Ion Vantage Software

The Ion Vantage software is a comprehensive ion beam control, and data acquisition package. It has been customized to produce the ultimate in isotope ratio acquisition, using the very flexible and comprehensive M20 multicollector. Examples of the software functionality are shown here.

- Adjustment of focus potentials, automatically or manually through the data system
- Control of HT and magnetic field
- Peak centre through HT or field control
- Turret position
- Ionization and evaporation filament control
- Optimization of ion beam using focusing, filament current or temperature
- Peak and baseline scanning for all collectors
- Real time monitoring of ion intensities for all collectors
- Automatic focusing and peak centre during analysis
- Static, multidynamic and single collector analysis on Faradays, ion counting or combinations
- Individual, high precision motor control of each Faraday and ion counting detector
- Independent control of ion multiplier voltage, discriminator, and deflection voltage
- Baseline subtraction
- Total evaporation
- Total ion charge accumulation analysis
- Customizable analysis routines using Microsoft EXCELTM spreadsheets, e.g. isotope dilution, oxide de-convolution
- Automatic interference and exponential mass bias correction
- Customizable mass fractionation corrections
- All analysis data and system information is stored for each analysis
- Post processing of analysis data
- Full QC control is built in
- Instrument calibrations
- Collector gains
- Resistor decay (Tau)
- Noise tests
- Mass/Field Calibration
- Source and analyzer vacuum monitoring
- Analyzer vacuum protection in case of power failure
- Control of bake-out temperature and duration

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Basic System Specifications

(demonstrated during installation)

1. **Resolution:** M/ΔM > 450
2. **Peak Flat Faradays:** ±100ppm over 480ppm of mass
3. **Resistor decay:** <5ppm of the peak in 2 seconds
4. **Peak side stability:** <±25ppm of mass in 30 minutes
5. **Faraday noise:** <2 x 10^{-16} A for 30 x 5 sec integrations
6. **Gain stability:** <5ppm over 5 hours
7. **Peak Flat Daly:** ±1000ppm over 300ppm of mass
8. **Daly Dark noise:** <10 counts per minute
9. **Daly linearity:** <0.15% between $1x10^3$ and $2x10^3$ cps
10. **Faraday/Daly gain stability:** <0.1% 1 RSD over 30 min
11. **Abundance sensitivity:** <2ppm at m/z 237 with respect to ^{238}U.
12. **Abundance sensitivity (WARP):** <0.02ppm (20ppb) at m/z 237 with respect to ^{238}U

Analytical Specifications

Strontium isotope precision - 200ng NBS 987
Internal precision: ^{87}Sr/^{86}Sr <5ppm 1 RSE
External precision: ^{87}Sr/^{86}Sr <5ppm 1 RSD
Accuracy: Within 30ppm of 0.710248
8 out of 10 filaments

Neodymium isotope precision - 400ng JNd-I
Internal precision: ^{143}Nd/^{144}Nd <5ppm 1 RSE
External precision: ^{143}Nd/^{144}Nd <5ppm 1 RSD
Accuracy: Within 20ppm of 0.512104
8 out of 10 filaments.

Specification for optional Daly

Faraday/Daly - 2 µg IRMM184 natural Uranium
External precision ^{234}U/^{238}U ≤ 0.15% 1RSD
8 out of 10 filaments

Notes

1. **Resolution:** M/ΔM where ΔM is the peak width in AMU at 10% peak height.
2. **Peak Flat Faradays:** Measured using a Sr ion beam with collectors at 1 AMU spacing (Sr). The peak centre is located and the intensity measured. The intensity at -240ppm, -160ppm, -80ppm, centre, +80ppm, +160ppm and +240ppm relative mass positions are measured. Each of the measured intensities will be identical to the centre to within 100ppm.
3. **Resistor decay:** This is a measurement of how quickly the Faraday detection electronics respond to a rapidly changing ion signal which is essential for a multidynamic analysis. The measurement involves measuring an ion beam of ~$5e^{-11}$ amps, then switching the mass to a baseline. The intensity shall drop to ±5ppm of the baseline within 2 seconds. For the $6e^{-11}$ amps example this is equivalent to $6e^{-11}$ amps x $2e^{-6}$ = $1.2e^{-16}$ amps. This is essentially equivalent to the baseline noise, meaning there is no residual signal when a second peak is measured.
4. **Peak side stability:** The mass position at 50% of the peak height will not move by more than ± 25ppm of relative mass over 30 minutes. This shows the stability of both magnet and HT. Requires a temperature stabilized environment.
5. **Faraday noise:** Baselines are measured for a minimum of thirty, 5 second integrations. The standard deviation is the noise on the baseline.
6. **Gain stability:** High and low reference voltages are switched between each of the resistor/ amplifier circuits and the relative standard deviation over the course of 5 hours is determined.
7. **Peak Flat Daly:** The peak flat on the optional ion counting Daly is determined in the same way as test 1, except that the measurement points are -150ppm, -100ppm, -50ppm, centre, +50ppm, +100ppm and +150ppm. The specification is the same both with and without a WARP filter.
8. **Daly Dark noise:** Measured at ~90% efficiency relative to Faradays.
9. **Daly linearity:** Measured using NBS U500, or NBS 982. Ratios normalized to ^{235}U/^{238}U or ^{208}Pb/^{206}Pb. Below 1000 cps the errors expand due to counting statistics.
10. **Faraday/Daly gain stability:** Measured using a stable ion beam of approximately $1e^6$ cps or by isotope ratio.
11. **Abundance sensitivity:** Measured using natural Uranium, or NBS U010. The ratio 237/^{236}U is multiplied by ^{236}U/^{238}U.
12. **Abundance sensitivity (WARP):** Same as #11. Zone refined Re is usually used to ensure no filament background beneath the 237.