

Isotop^x

The culmination of
40 years experience
in TIMS

Phoenix

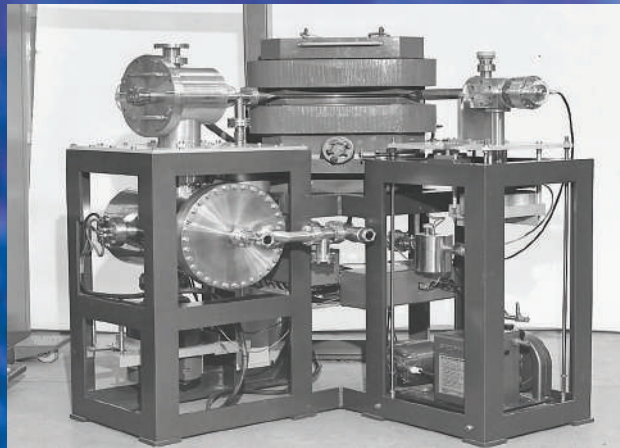
Thermal Ionization Mass Spectrometer



with **ATONA**

Excellence in mass spectrometry

World class heritage and knowledge



VG MM30 mass spectrometer, the worlds first multi-sample TIMS

Isotopx are the TIMS specialists.

Isotopx was formed in February 2008 as a Management buyout (MBO) from GV instruments. The MBO was in response to Thermo Corporation's purchase of GV instruments and the subsequent enquiry by the UK Competition Commission into the purchase.

We trace our heritage to the very first commercial TIMS, the MM30, launched by VG* Micromass in 1973. We are justifiably proud of this tradition, many of the subsequent instruments manufactured by VG Isotopes, VG Isotech, Micromass and GV instruments continue to be supported by Isotopx engineers even in some cases after more than 25 years of service.

With the latest Phoenix the tradition of excellence is maintained. Operating at the highest levels of stability, sensitivity and precision, the Phoenix provides the current state of the art in isotope ratio analysis, whatever the sample size. The Isotopx team continues to provide the finest customer support for the Phoenix and also for the many hundreds of legacy instruments.



VG Sector 54 1987-1996, a benchmark TIMS.

“it's just so much easier to generate high quality data”

Isotopx Customer

“it's the approach of the company that's important”

Isotopx Customer

* The VG Trademark is owned by Thermo Corporation

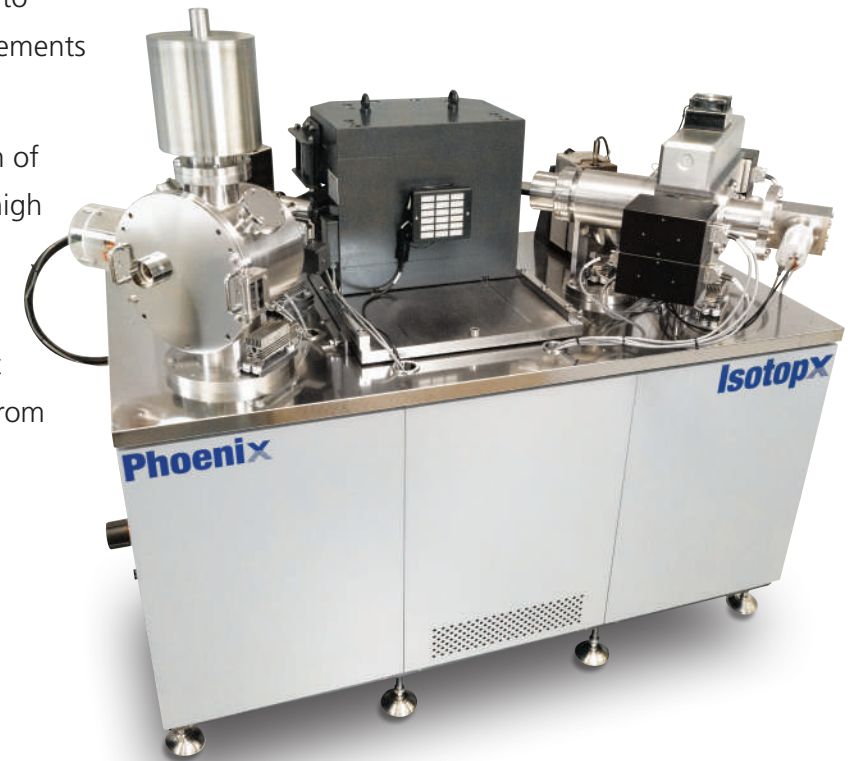
Phoenix TIMS

The Phoenix TIMS instrument is designed to measure isotope ratios of non-gaseous elements with the highest sensitivity and precision.

High sensitivity results from careful design of the ion extraction optics combined with high vacuum around the ionizing filament.

High precision is also the result of highly sensitive, low noise, stable detectors, that can measure extremely low ion currents from nanoamps (nA, 1×10^{-9} A) to single ion detection (1.6×10^{-19} A).

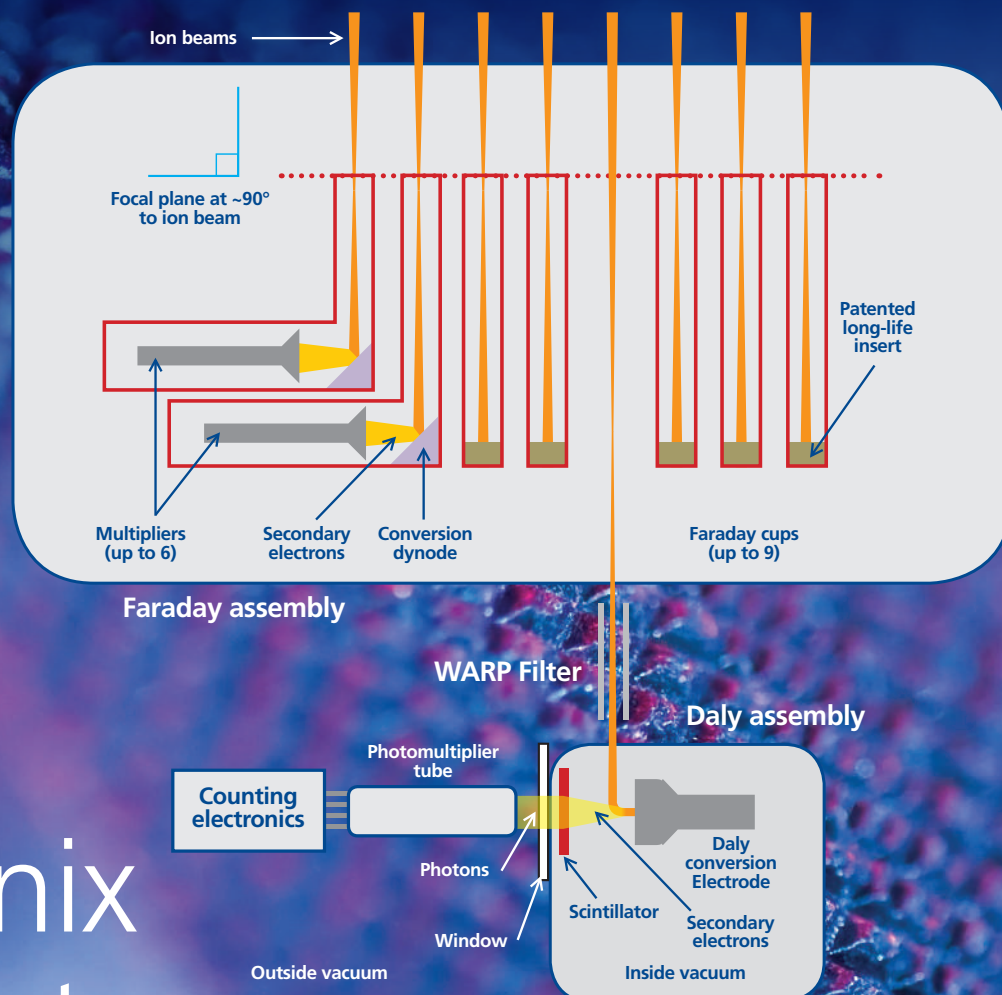
The Phoenix represents the ultimate combination of all these factors.



Phoenix TIMS Design Features

- New ATONA amplifiers allow for largest dynamic range Faraday cup detection up to 1nA (100V equivalent $1e11\Omega$ resistor), with the fastest response time, surpassing the theoretical noise of a $1e13\Omega$ resistor after 10 seconds of integration.
- Largest radius electromagnet on commercial TIMS for optimal transmission, resolution, and stability
- Positive and negative ion capability as standard
- Highly stable high voltage and magnet field control such that mass drift is $<20\text{ppm}$ of mass over 40 minutes.
- Rotated ion focal plane such that the collector focal plane is perpendicular to the ion trajectory, ensuring optimal peak flat irrespective of collector position across the focal plane
- Extended ion optics to allow for simultaneous measurement of UO_2^+
- Long life Faraday cups that carry a 10 year guarantee of never having to be replaced
- Largest pumping capacity on commercial TIMS for the fastest pump down from atmosphere leading to improved productivity and best ultimate vacuum
- Stainless steel source chamber with accessory ports for sample pre-heat, cold trap and oxygen bleed
- 20 position sample carousel that rotates perpendicular to the ion trajectory, ensuring zero possibility of sample cross contamination
- Hinged door with a viewing window that a pyrometer can be mounted onto
- Highest capacity analyser ion pumps on commercial TIMS for best ultimate vacuum and abundance sensitivity
- Highest sensitivity due to largest ion extraction slits and optimal focussing
- Largest dynamic range ion counter. (Daly detector)
- Options for Secondary Electron Multiplier
- Option for multiple ion counting with conversion dynode technology
- Option for Retarding filter to improve abundance sensitivity on axial Daly or SEM to $<1 \times 10^{-8}$ at 237 with respect to ^{238}U
- All detectors except for axial Daly and SEM are independently and individually motorized. No other TIMS offers this flexibility
- Powerful and flexible software for tailor made applications as well as repetitive analyses
- Fully networkable to permit remote control of the instrument

Phoenix Collector



Ion Counting Daly

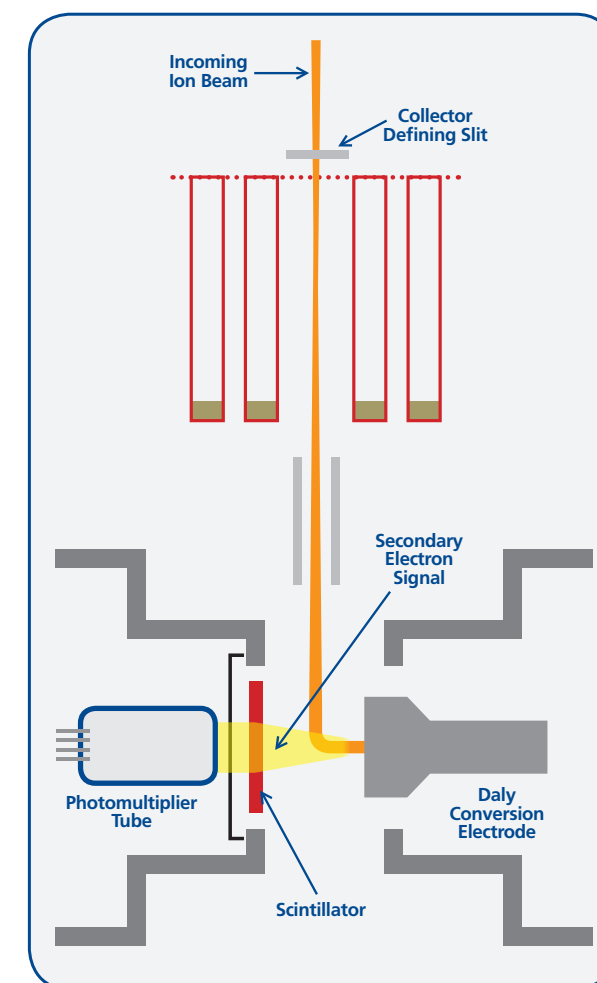
The ion counting Daly is unique to Phoenix. It is positioned behind the axial Faraday and can be used by moving the axial Faraday to the low mass side of the collector.

A photomultiplier that is external to the vacuum is used to detect photons generated from a scintillator which is positioned opposite the Daly knob. The knob is biased at approximately 25KV and generates electrons from ion impacts on its surface. The Daly is used for positive ion detection only.

The features of the Daly are:

- Peak flat >300ppm of mass, this is almost as large a flat as the Faradays, making it ideal for peak jumping analyses in combination with the Faraday cups
- Linearity <0.1% up to 3×10^6 cps, there is an effective dynamic range overlap of almost 3 orders of magnitude with the ATONA
- Noise <10cpm
- Efficiency >95% relative to the Faradays for Uranium
- Gain stability relative to the Faraday cup of <0.1%/hr
- The Daly is guaranteed for 10 years of use

The Daly has become the benchmark ion counter for high precision isotope ratio measurements on very small samples e.g. single zircon crystals, or for measuring very low abundance isotopes e.g. ^{236}U and ^{230}Th . What makes the Daly so good is its extreme linearity from a few cps to >3 million cps. The isotope ratio stays the same irrespective of the count rate.



M20 Collector

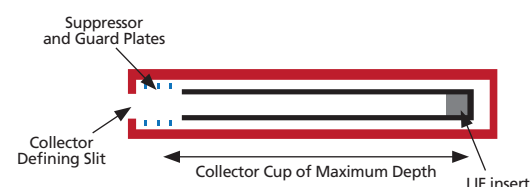
The M20 multi-collector sets the Phoenix apart.

It is the culmination of over 30 years of experience in ion detection hardware and electronics. Up to 9 individually motorized Faraday collectors as standard, in addition, up to 6 multiple ion counting detectors, all individually moveable and motorized, can be added to the Faraday array. All of these collectors are on the same focal plane, that is rotated to provide optimal peak shape across the collector.

Behind the focal plane, a secondary electron multiplier and/or ion counting Daly can be positioned. Both of these detectors can operate behind a high abundance sensitivity retarding filter (WARP).

Long Life Faraday's

Phoenix Faraday collectors are extremely robust. No collector has had to be replaced in the field since 1996; they therefore carry a 10 year warranty.



M20 Collector in detail

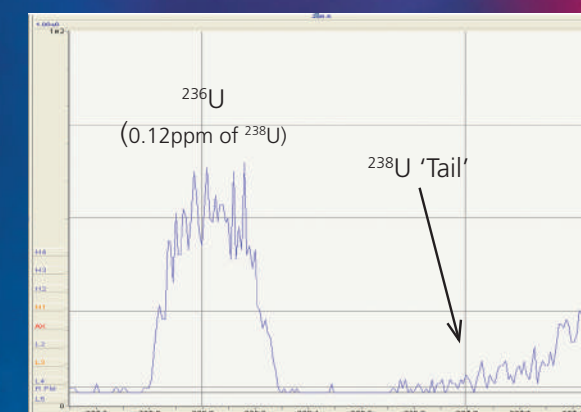
Below is a plan view of the M20 multicollector, showing the main multicollector. On top of the multicollector is the amplifier housing that contains the detector electronics in an evacuated and peltier cooled environment. Behind the collector is the WARP filter, and below that, at the bottom of the picture the Daly detector.



Wide Aperture Retarding Potential Filter (WARP)

The optional WARP filter is positioned immediately behind the Faraday array.

It acts as a potential barrier only allowing those ions that have 8000V potential through. The small fraction (<0.001%), of ions that have lost their energy (due to collisions with gas molecules in the analyser vacuum), are not admitted through to the collector which dramatically improves the abundance sensitivity.

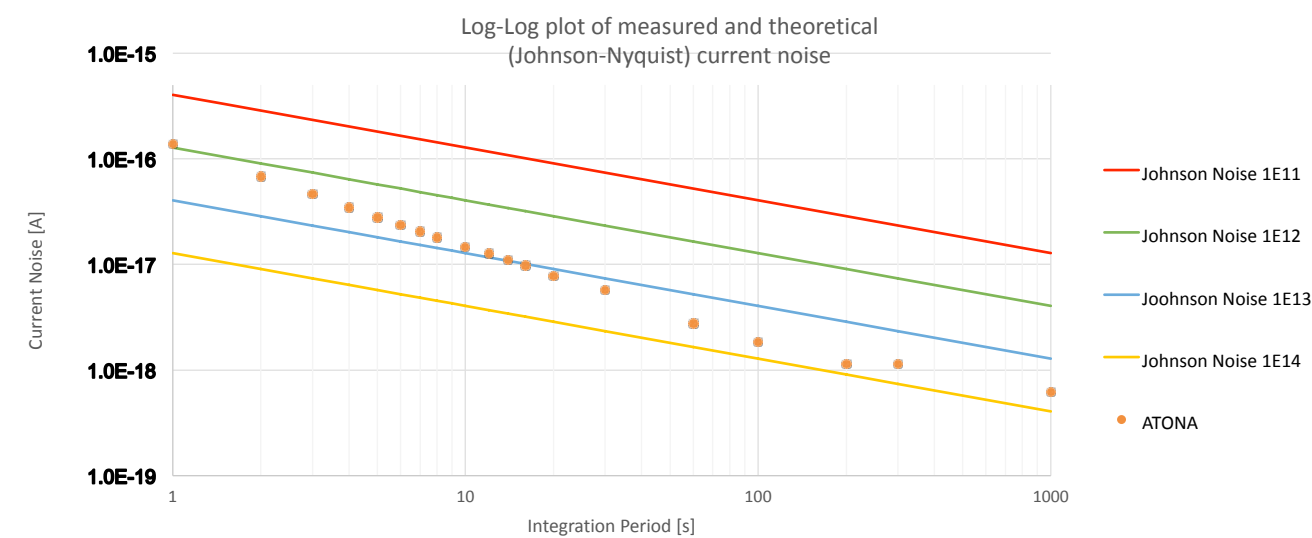


ATONA

TIMS

Introduction

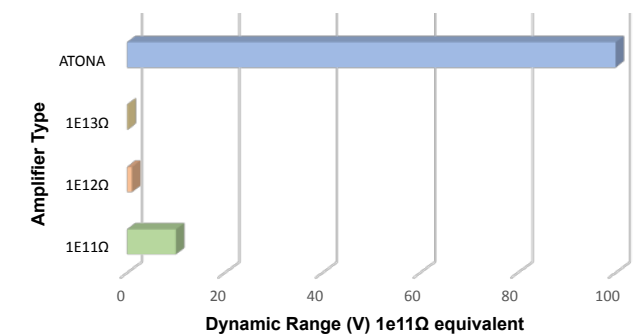
The newly developed ATONA (aA to nA) amplification technology from Isotopx has eliminated the need for a “feedback resistor”. The outcome is a significant reduction in amplifier noise, a dramatic increase in dynamic range, rapid amplifier decay, and improved baseline and calibration stability. In practice, this means analysis of signal sizes from 10’s cps up to 1nA (equivalent to 100V on $1\text{e}^{11}\Omega$ resistor amplifier), with lower noise than any resistor-based amplifier, <0.2 seconds amplifier decay time and ultimate baseline stability of <10 cps 1SD over 10 hours of analysis time. In addition, gain calibration of the amplifiers is stable to ~1ppm with no apparent drift over time.



Log-Log plot of measured ATONA noise [A] against theoretical (Johnson-Nyquist) noise across different integration times [s].

Dynamic Range

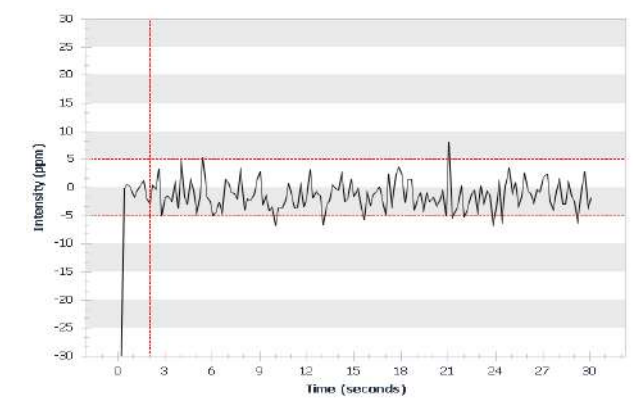
In contrast to conventional resistor-based technology, ATONA amplification can detect signal sizes from 10’s cps to 1nA (100V in reference to $1\text{e}^{11}\Omega$ resistor amplifier) without any electronic or software switching throughout the entire dynamic range. This is of particular use in applications where large isotopic abundance ratios are present (e.g., Ca, Pb, U). Furthermore, the extreme dynamic range, in conjunction with low noise, enables measurement of unknowns with optimal precision regardless of isotopic composition.



Comparison of $1\text{e}^{11}\Omega$, $1\text{e}^{12}\Omega$, theoretical $1\text{e}^{13}\Omega$ and ATONA dynamic range reference to $1\text{e}^{11}\Omega$ amplifier.

Amplifier Decay

The time for an amplifier to return to baseline following measurement of a signal has always presented a significant challenge to resistor-based designs. Lengthy amplifier decay reduces “on-peak” measurement time, ultimately leading to fewer ions being collected and lower analytical precision. Higher resistance amplifiers are characterized by prolonged amplifier decay times, i.e., for $1\text{e}^{13}\Omega$ resistor amplifier the time would be several seconds, making any peak hopping methodology difficult. With ATONA amplification, amplifier decay time is <0.2 seconds, faster than any commercially available resistor amplification, and requiring no artificial correction. Amplifier decay is therefore not a limiting analytical factor using ATONA.



25 x 200ms integrations of on peak 8V signal measured, magnet moved 0.5 AMU and baseline measurements taken for 30 seconds of 200ms integrations.

Noise

Amplifier noise directly impacts on the precision of a measurement. The noise performance of a resistor-based amplifier improves with increased integration time, but is theoretically limited by the Johnson-Nyquist noise of the specified resistor. By eliminating the “feedback resistor”, Isotopx ATONA amplifier surpasses the theoretical Johnson-Nyquist noise of a $1\text{e}^{13}\Omega$ resistor after 10 seconds of integration. At 100 seconds of integration the noise approaches the theoretical limit of $1\text{e}^{14}\Omega$ resistors. Unlike other amplification systems, these noise properties are applicable across the entire dynamic range and improve linearly with integration time.

Baseline Stability

Long term baseline stability has been measured at significantly less than 10 cps 1SD using 1000 second integrations over 72 hours. This results in a quantification limit (defined as $5 \times 2\text{SD}$) less than 100 cps. This emphasizes the low crossover point between ion counting and ATONA amplification. This stability in combination with low noise offers the possibility of static Faraday measurement with ATONA yielding comparable precision to peak hopping on an ion counter in the 100’s cps range.

Inter-channel Gain stability

Long term inter-channel gain stability for ATONA amplification is exceptional. Using a completely novel gain calibration technique, amplifier gain stability is ~1ppm over 2 days. The data displayed no long-term drift or trends during the measurement period. Routine amplifier calibration therefore becomes unnecessary.

	Mean	1RSD (PPM)
L5	1.0078	0.92
L4	1.0188	0.75
L3	1.0029	0.76
L2	1.0025	0.88
Ax	0.9944	0.57
H1	0.9985	1.05
H2	0.9945	0.74
H3	1.0016	1.04
H4	1.0144	0.93

48 hours of gain calibration based on 1 hour integration time.

48 hours of gain calibration based on 1 hour integration time.

Multiple Ion Counting

Conversion Dynode MIC

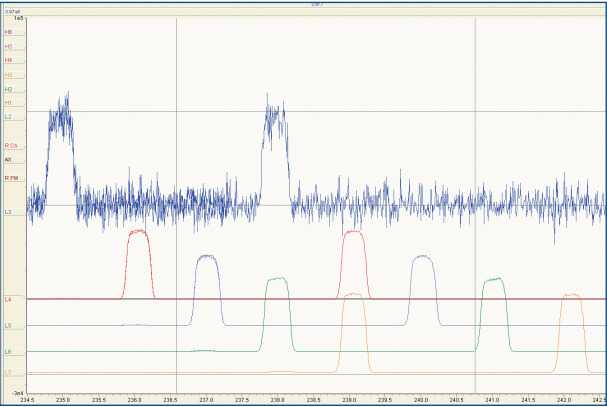
For extremely small amounts of material e.g. at the femtogram level, where ion beams might be small and transient, it may not be possible to sequentially measure isotopes using a single ion counter. For these small samples the ion beam may be unstable, further; isotopes of interest may only be detected for a fraction of the time. As with Faraday collection, using several ion counters together can overcome this problem since each isotope is measured by its own dedicated detector.

Phoenix can be fitted with an array of up to six conversion dynode ion counting detectors positioned alongside the Faraday array. The ion beam passes through an entrance slit which has the same dimensions as the Faradays and passes down a “chimney” before striking a conversion dynode. At the conversion dynode the ions are converted into electrons which are directed towards an electron multiplier sitting at right angles to the ion beam (see diagram to the right). This configuration allows Phoenix MIC to use full size continuous dynode electron multipliers.

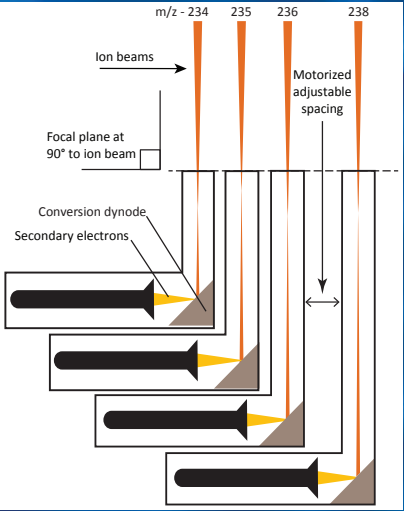
All of Phoenix’s MIC’s are independently moveable, each with its own motor allowing for multiple isotope systems to be measured on a single set of MIC’s simply by moving ion counters via software control (see table above right). No other multiple ion counting system offers this level of versatility.

L7	L6	L5	L4	L3
MIC	MIC	MIC	MIC	Faraday
²⁰⁴ Pb	²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁸ Pb	
²³³ U	²³⁴ U	²³⁵ U	²³⁶ U	²³⁸ U
²³⁴ U	²³⁵ U	²³⁶ U	²³⁸ U	
²³⁹ Pu	²⁴⁰ Pu	²⁴¹ Pu	²⁴² Pu	
¹⁸⁵ ReO ₃	¹⁸⁷ OsO ₃	¹⁸⁸ OsO ₃	¹⁸⁹ OsO ₃	

Isotope systems that can be measured on a single set of 4 ion counters



Mass scan of NBS U500 across 4 ion counters with a Faraday for reference. The count rate is 25,000cps ²³⁸U. The noise level on the Faraday is clearly higher. The scan shows the ion counters are set at uranium unit mass separation with respect to each other and to the Faraday cup.



Schematic showing conversion dynode MIC set at uranium spacing. All individual ion counters can be moved via software control, and each has its own motor. This allows a single set of ion counters to be used for a range of applications and as the ion counters move independently of the Faradays it is straightforward to use Faradays and MIC’s together within an analysis protocol.

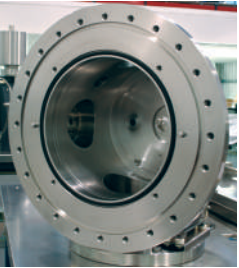
The schematic shows four ion counters, Phoenix can accept up to 6 channels of MIC.

Nuclear

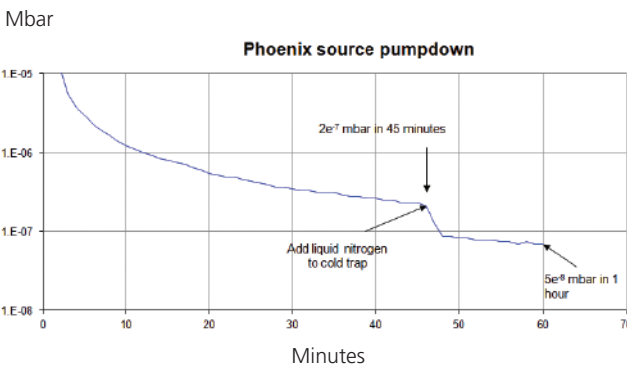
TIMS mass spectrometers are used in a variety of nuclear applications. Each application has its own exacting challenges but Phoenix is designed to meet every one.

Nuclear Fuel assay

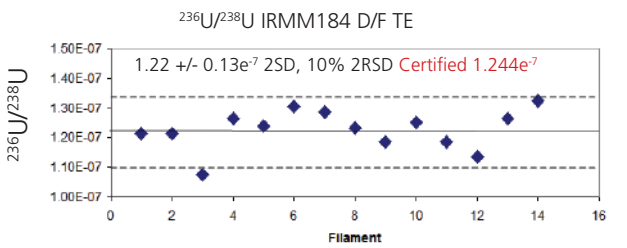
Glove Box Adaptation - For the analysis of Pu and highly enriched U a glove box is required. The Phoenix has a convenient glove box adaptor flange that can be customized to the users requirements.



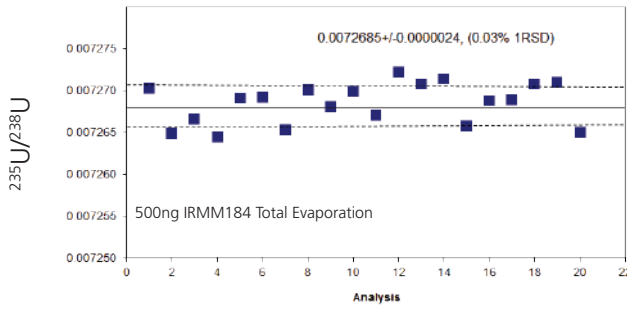
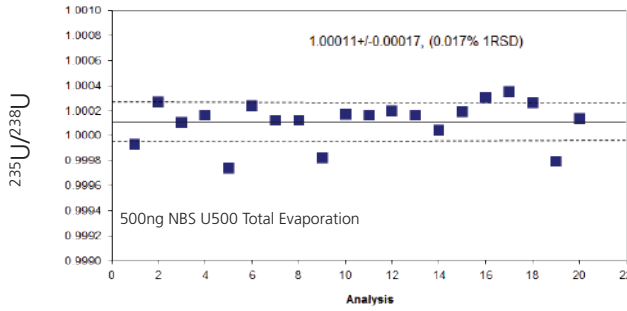
Productivity - Phoenix can measure 20 samples in one turret completely automatically. The extremely rapid source pumpdown is provided by the largest turbomolecular pump available on a TIMS. A potential of 40 samples can be measured comfortably in 24 hours. The plot below shows a typical pumpdown performance.



Abundance Sensitivity - Large capacity analyser ion pumps combined with the large source turbo-molecular pump ensure analyses at the highest possible vacuum. Peak tail corrections are minimized. For high precision measurement of minor isotopes at high abundance sensitivity, the Daly with a WARP filter is an unbeatable combination. The plot below shows replicate total evaporation measurements of ²³⁶U/²³⁸U in IRMM 184 using a combination of Faraday and Daly detector.



Accuracy and Precision - Phoenix supports traditional external mass fractionation correction and also total evaporation analysis. Reproducibility and Precision are ensured with data presented as isotope ratio and atom and weight percent. All data is stored in Excel spreadsheets and is completely traceable.



The 2 plots above show the result of total evaporation measurements of 500ng of NBS U500 and IRMM 184. High precision can be obtained irrespective of the isotope ratio. Accuracy relative to certified value are 0.04% and 0.06% respectively.

Nuclear Safeguards

The SEM and Daly behind the WARP filter provide ion counting analysis at high abundance sensitivity. The flexible multiple ion counting array can be used for ultra small samples.

Isotope Geochemistry

Phoenix is ideally suited for isotope geochemistry. It combines the highest sensitivity with the highest isotope ratio precision and the most versatile detector platform on the market.

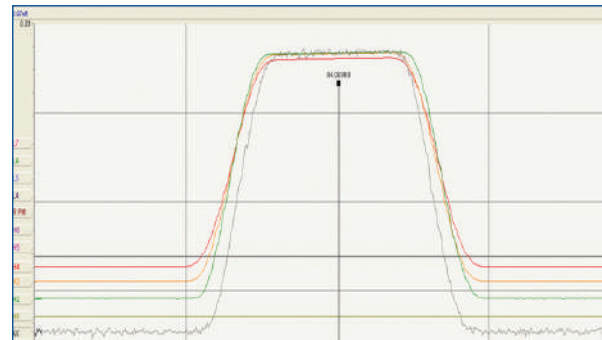
For decades isotope geochemists have been pushing the frontiers in our understanding of how the earth, moon, and cosmos developed. At the forefront of this endeavour has been TIMS mass spectrometers. Their ability to determine precise and accurate isotope ratios on the smallest samples has been the key application of this technology. The Phoenix continues this tradition.

Strontium and Neodymium

High precision requires large peak flats, this figure shows the coincidence of all 4 strontium isotopes. The Phoenix produces exceptional isotope ratios, for example reproducibility of 3ppm 1RSD can be routinely achieved on as little as 200ng of Sr using a 5 Volt ion beam (below), and 3ppm 1RSD on 400ng of Nd using a 4 Volt ^{144}Nd ion beam.

For these sample sizes the detection efficiency (ions measured/atoms loaded) is typically 2.5% for Sr and 2% for Nd.

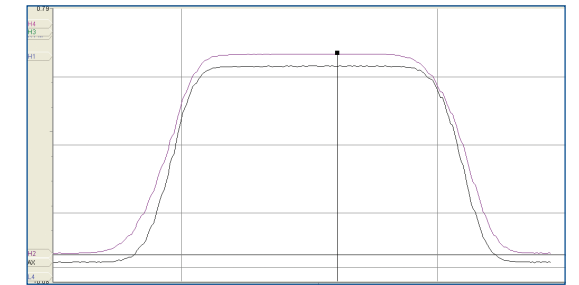
For sub-nanogram sample loads the detection efficiency increases to over 20% for Sr and to 17% for NdO^+ .



Strontium isotope coincidence, showing the excellent peak flat and shape of the Phoenix.

Negative ion Capability

Some elements are extremely difficult or impossible to ionize as a positive ion due to their high ionization potential. They can be analysed as a negative ion species either in combination with oxygen e.g. OsO_3^- , BO_2^- or by themselves e.g. Cl^- , Br^- . Phoenix uses small Al-Ni-Co magnets in the bead blocks to deflect electrons away from the ion extraction optics. Resulting in excellent peak shapes, for example BO_2^- as shown opposite.



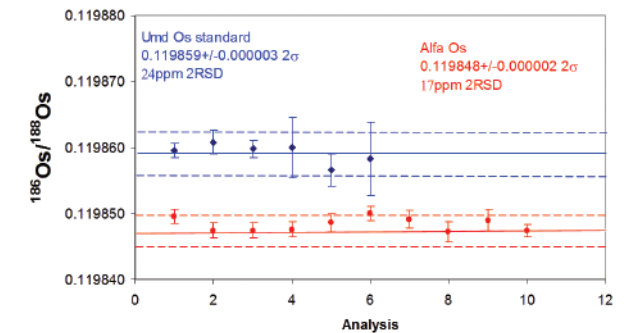
Mass scan showing BO_2^- masses 42 and 43 in coincidence. Sample is 1 microlitre of seawater.

Osmium Analysis

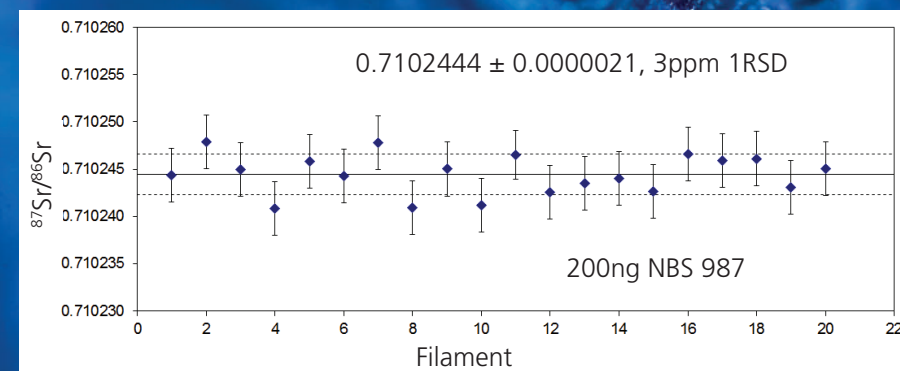
For osmium isotope ratio measurements an oxygen bleed system is put onto the source chamber and oxygen is introduced into the source area.

All oxygen and fractionation corrections are made automatically during the analysis by the IonVantage software.

The Figure shows results of high precision $^{186}\text{Os}/^{188}\text{Os}$ on two Osmium samples. Reproducibility of better than 30ppm 2RSD can be achieved with ^{186}Os ion signals of 200mv.



Sr and Nd Isotope Ratio Performance

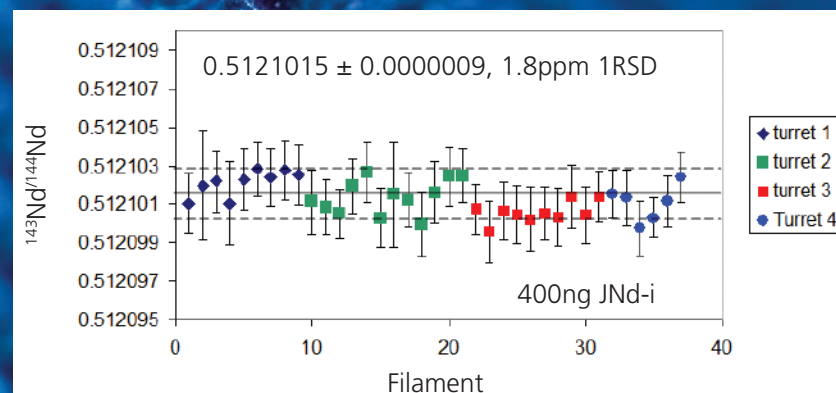


Strontium

Replicate measurements of 200ng NBS987 Strontium. Error bars are 2SE.

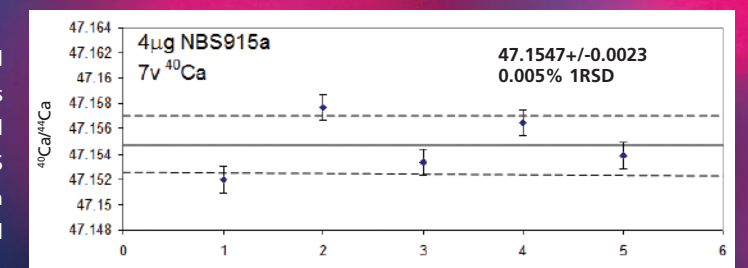
Neodymium

Replicate measurements of 400ng JNd-I Nd standard on different turrets over a 2 week period. Error bars are 2SE.



Non-Traditional Stable Isotopes

In recent years there has been an increasing demand for isotope ratios as a stable isotope tracer. This has been largely fuelled by the high precision obtained using ICP-MC-MS. However, unlike ICP-MS, the TIMS source does not produce isobaric interferences that can cause inaccuracies. Elements such as Ca, Cr, Se, Fe are all potential areas where Phoenix can provide the answers.



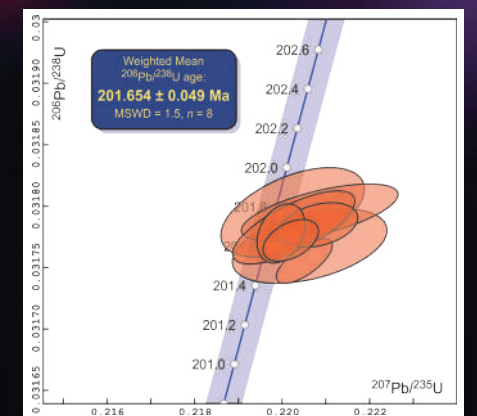
Replicate measurements of $^{40}\text{Ca}/^{44}\text{Ca}$ in NBS 915a.

U-Pb Geochronology

Phoenix is rapidly becoming the instrument of choice for high precision geochronology because of the exceptional stability and linearity of the Daly detector.

The X62 variant of the Phoenix has been designed in response to a request from Prof. Sam Bowring at MIT to allow the measurement of uranium isotopes with a double spike to correct for mass fractionation. The uranium must be measured as an oxide and so a slight increase in the mass dispersion of the Phoenix was required to enable simultaneous collection of all the uranium oxides on all 9 collectors.

This development combined with the new large dynamic range Daly detector has resulted in unprecedented levels of precision in single zircon studies.



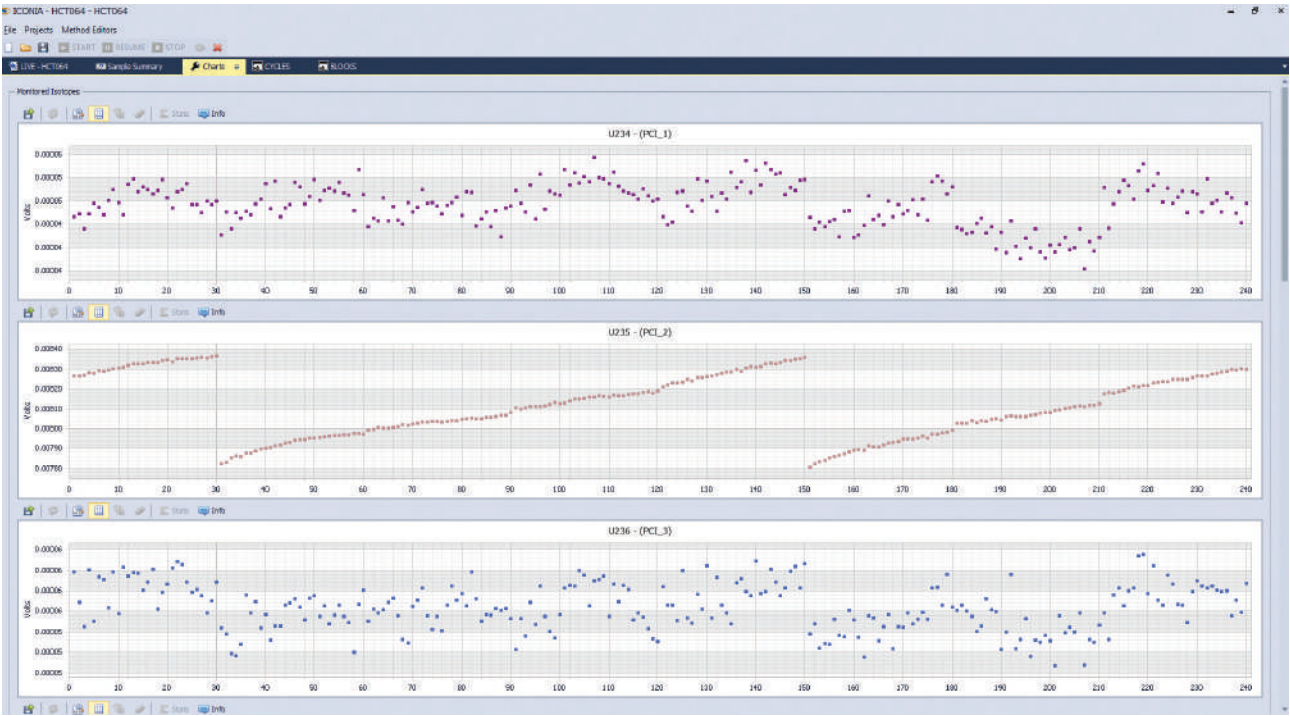
U-Pb isochron diagram courtesy of MIT, showing excellent concordant dates with an age of $201.65 \pm 0.05 \text{ Ma}$.

NEW Software

Isotopx NEW software suite, a fresh take on traditional mass spectrometry software. Clearly laid out and intuitive, it has been designed to make instrument control and data acquisition as simple and productive as possible.

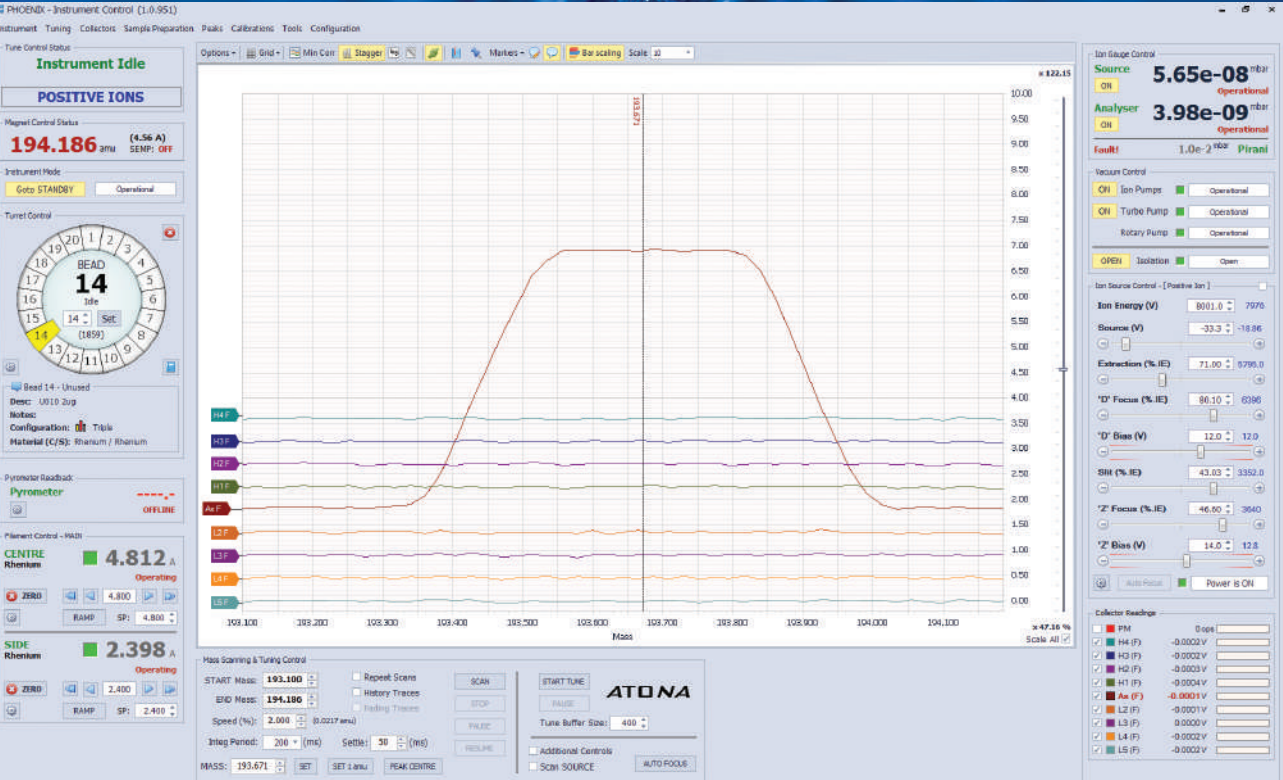
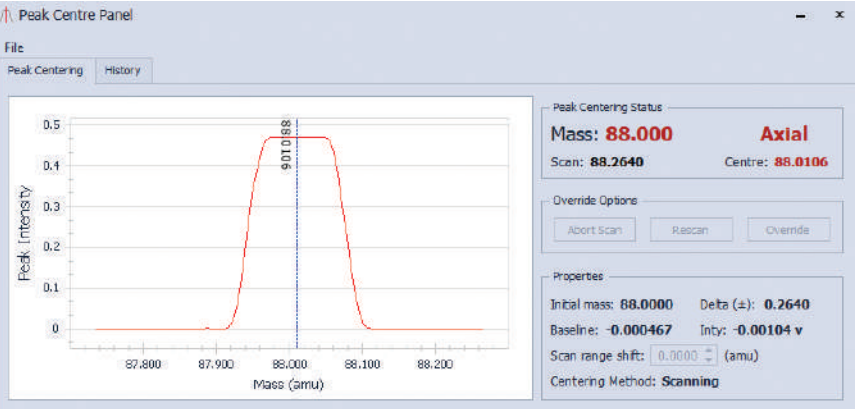
Software features:

- Built from the ground up using the latest developer technologies & fully compatible with newest version of Windows.
- Single PC instrument operation (no embedded PC).
- Improved sample management tools including: dedicated name editor, filament type, bead status indicator and information import via 'QR' barcode reader.
- Advanced instrument control with: mass scanning/time acquisition modes, mass/intensity markers, IsoMarkers, variable zoom feature, calibration/profiling tools, safety features, and ion-mode reversal assistant.
- New peak centre control panel. Peak scanning method utilising highly advanced algorithms to detect peaks at the 1mV level – with real-time manual over-ride option.
- USB Pyrometer support to 0.1 C resolution.
- Intuitive analysis method construction.
- Dynamic task-based sample preparation methodology. Easy configuration and real-time parameter modification for fully automated analysis runs.
- Significantly improved beam management with flexible filament control and powerful Total Flash Evaporation (TFE) facility.
- Comprehensive sample run event logging.
- Real-time and Post processing of analysis data.
- Raw data stored in open source SQLite database files. Export of raw and processed data in machine readable ASCII files.

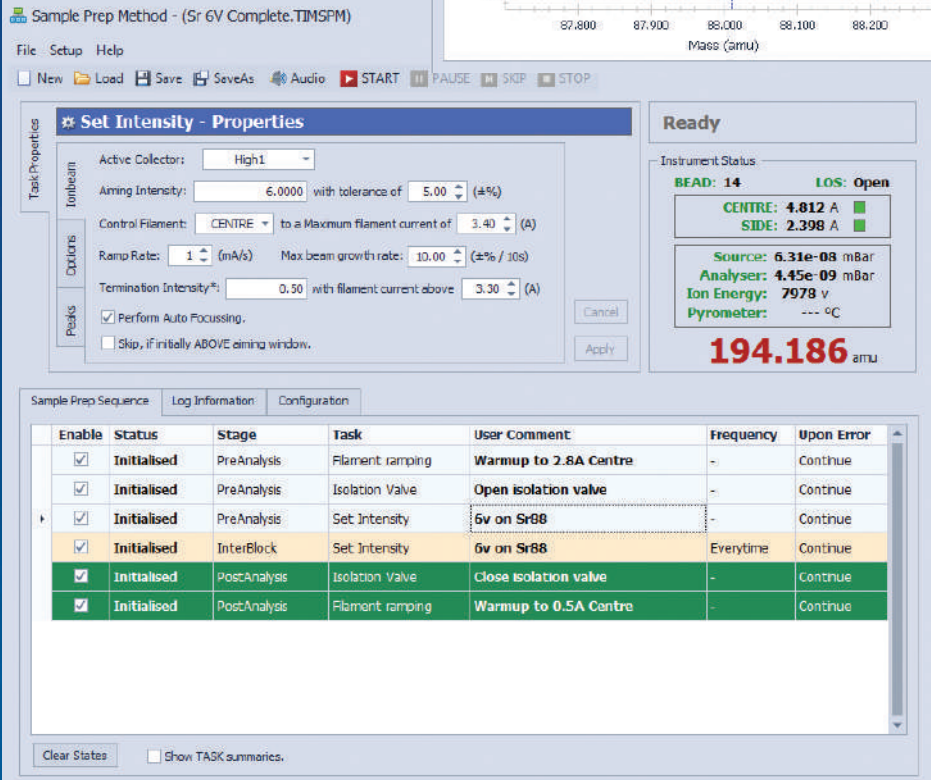


Above. New live fully customisable/ dockable data processing - charts view.

Right. New peak scanning peak centre panel.



Above. New TIMS tune page with all necessary information easily accessible.



Above. New sample preparation window.

DG60

Isotopx DG60, setting new standards for automation and reproducibility in filament degassing.

Fully automated degassing of up to 60 filaments per cycle.

Floor standing unit that is controlled by a laptop minimizing footprint.

Universal power supply, 110V – 240V at 50-60Hz.

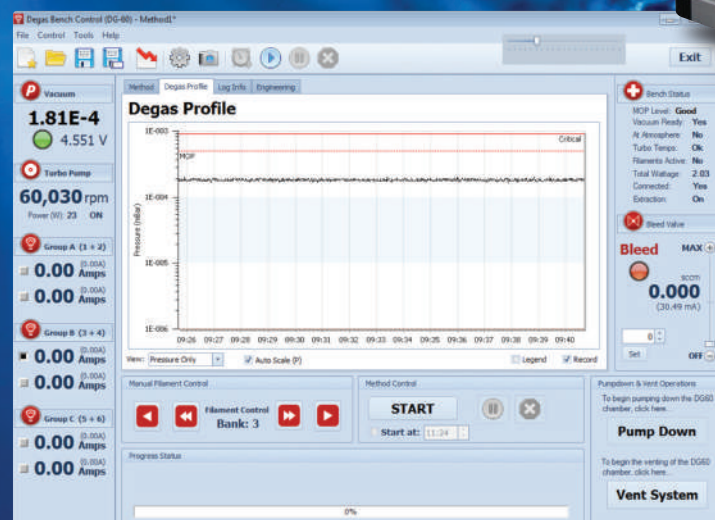
Oil-free pumping system consisting of an air-cooled 300 l/s Turbo pump backed by a dry Scroll pump; ensures clean degassing environment and rapid pump-down.

Stainless steel vacuum enclosure with sight glass enables an ultimate vacuum of <5x10⁻⁸mbar.

An automated gas bleed option with vacuum pressure feedback control facilitates consistent filament carburisation.

Suitable for Isotopx Phoenix, Isoprobe-T and VG Sector 54 filaments. Please ask about filaments for other TIMS systems.

Tool free filament loading and unloading for ease of use.



Newly developed, highly advanced, software suite allows for an unparalleled degree of automation.

Each degas cycle produces an archived event log, this permits the user to monitor the degassing process over time.

Fully networkable.

Customer Service

Isotopx is very proud of its ability to service not only current instruments but also instruments which are up to 25 years old. This is due to the robustness of the instrument designs but also due to Isotopx being able to retain skilled and dedicated engineers who have the essential knowledge to support our instruments.

We can supply a variety of post warranty service plans, which can vary from simple email, and telephone support to complete extended warranty support with preventative maintenance contracts.

Isotopx provides a unique service for a unique instrument.

Isotopx

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