AUTOMATED X-RAY

PHOTOELECTRON

SPECTROSCOPY

AXIS NOVA

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AXIS NOVA

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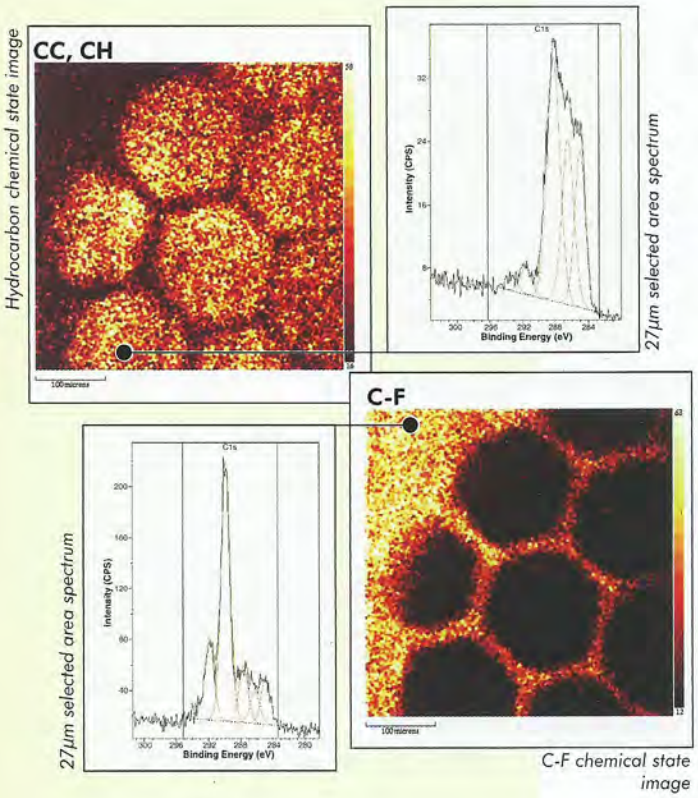


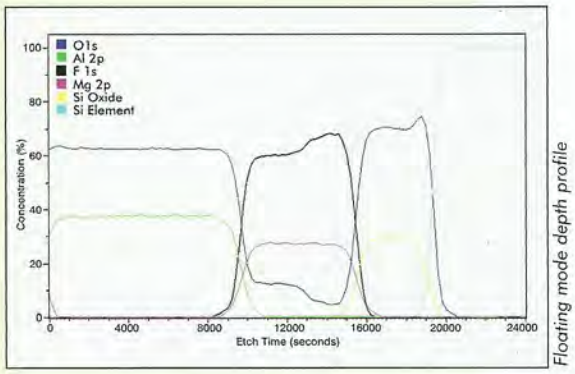
PARALLEL IMAGING AND SMALL AREA SPECTROSCOPY

The AXIS Nova provides unrivalled performance for fast elemental and chemical state imaging. The spectrometer uses a 165mm mean radius hemispherical analyser (HSA) for spectroscopy combined with the Kratos patented spherical mirror analyser (SMA) for parallel imaging.

In spectroscopy mode the large radius HSA ensures excellent photoelectron transmission guaranteeing high sensitivity and high energy resolution. Small spot spectroscopy is achieved by inserting an aperture into the electrostatic lens column forming a virtual probe at the surface as discussed further in technical note MO224. This approach to small area analysis removes problems of sample charging and X-ray induced degradation associated with micro-focussed X-ray sources. Small spot spectra can be acquired from areas as small as 10μm diameter from any point within the field of view of the lens. A unique electrostatic deflection system allows the position of the virtual probe to be deflected across the surface of the sample. The position for acquisition of small spot spectra can be defined by a simple mouse click from a parallel photoelectron image with the advantage that there is no need for translation of the sample. Alternatively the high resolution in-situ optical microscope image of the sample may be used to define the XPS analysis position directly.

In parallel imaging mode photoelectrons at a discrete kinetic energy (binding energy) are focussed on the two-dimensional photoelectron detector producing a direct image of the surface without scanning. The relative position of the photoelectrons is maintained as they pass through the SMA allowing acquisition of parallel images in a matter of seconds in contrast to the slower pixel by pixel acquisition of XPS mapping. One obvious advantage of fast parallel imaging is the reduced exposure of delicate samples to X-rays. Using a 256 x 256 pixel array, parallel XPS images of the sample surface with a spatial resolution of <3μm can be acquired. Elemental or chemical differences may be easily imaged even if they are not visible by optical inspection. The properties and design of the SMA mean that the parallel images have both high spatial and high energy resolution.

Data to the left show hydrocarbon and C-F chemical state images for a plasma patterned sample. The images were also used to define the analysis position for 27μm small spot spectra without translating the sample.

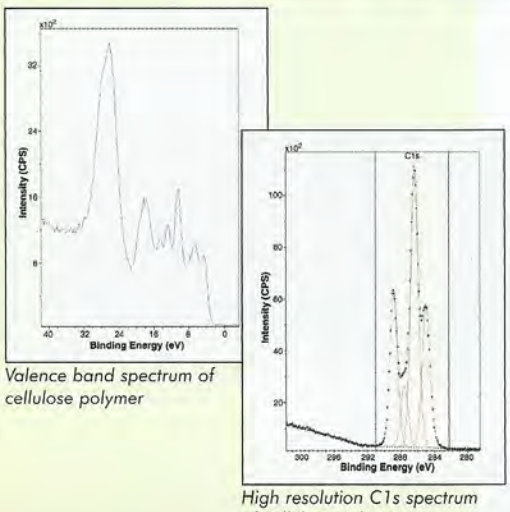


ION SOURCE

The Ar^+ ion gun of the AXIS Nova operates with continuously variable beam energies between 4kV and 50V. The precision ion column incorporates a bend for neutral suppression as well as the ability to operate in floating mode producing high current densities at low ion energies for improved interface resolution and fast etch rates even at low ion acceleration voltage. All control and status read-backs are displayed in the Vision acquisition software with pre-defined operating conditions provided in a look-up table. As expected for the level of automation of the AXIS Nova, the argon gas supply for the ion source can be turned on and off as required during unattended operation.

X-RAY SOURCE

The X-ray source on the AXIS Nova is based on a large 500mm Rowland circle geometry ensuring less energy dispersion in the focussed X-ray spot than systems based on smaller Rowland circles. This guarantees the highest energy resolution as demonstrated by a FWHM <0.48eV for Ag 3d. The Al Kα X-rays are monochromated by a single toroidal backplane for ease of alignment of the X-ray spot with the analysis position. The X-ray anode is engineered to allow indexed movement of the anode surface with respect to the cathode. Thus new anode surface may be exposed as required without breaking vacuum. The availability of 10 anode positions significantly increases the useful lifetime of the anode.

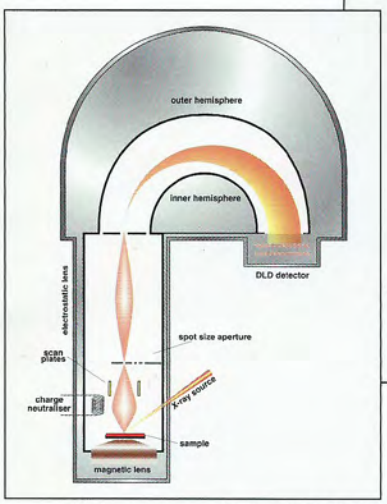


Valence band spectrum of cellulose polymer

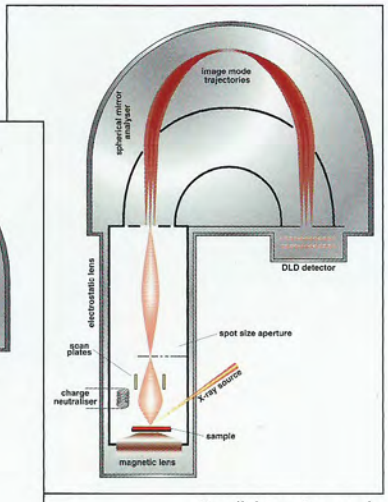
High resolution C1s spectrum of cellulose polymer

CHARGE NEUTRALISATION

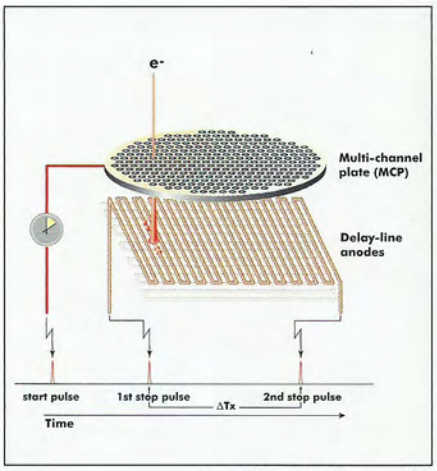
The AXIS Nova charge neutraliser is based upon proven market leading technology used in over 200 AXIS spectrometers. The use of a coaxial low energy electron source within the field of the magnetic lens ensures that the ultimate base pressure of the spectrometer is maintained during analysis of insulating samples in contrast to other neutralisation systems which require an additional source of Ar^+ ions. The advanced design of the Kratos neutraliser discussed in technical note MO222 ensures that the system is self-regulating and works effectively on all classes of insulating materials including fibres, powders and thin films. When used in conjunction with the monochromatic X-ray source, spectra with unrivalled energy resolution may be generated from electrically insulating materials as demonstrated by data showing FWHM of <0.68eV on the ester component in polyethylene terephthalate (PET).



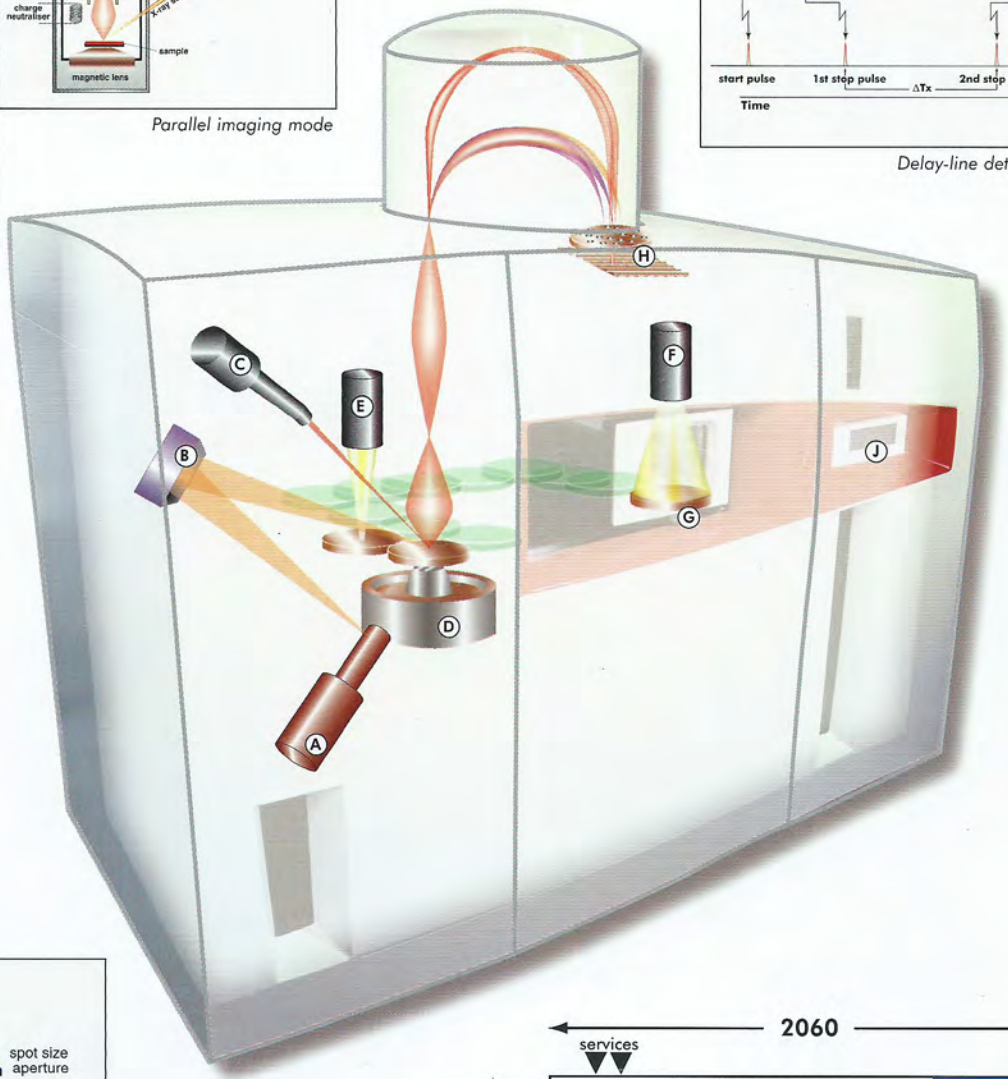
Spectroscopy mode



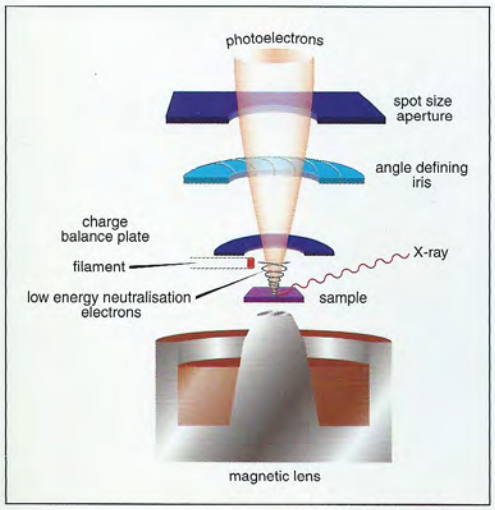
Parallel imaging mode



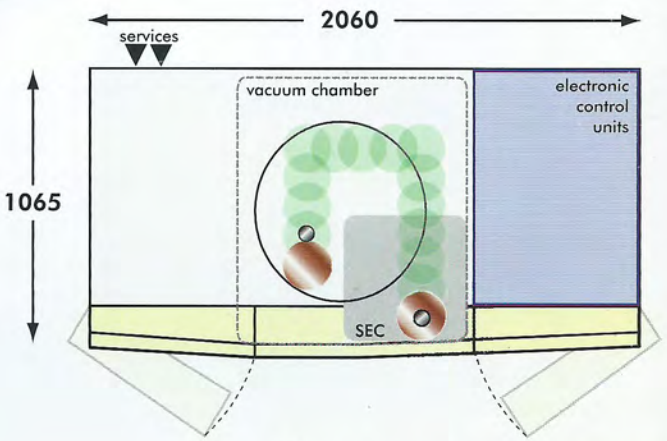
Delay-line detector assembly



- (A) X-ray source
- (B) Monochromator
- (C) Ion gun
- (D) Magnetic lens
- (E) SAC camera
- (F) SEC camera
- (G) Platen movement
- (H) Delay-line detector
- (J) Control units



Patented AXIS technology



Schematic plan view including platen movement

DELAY-LINE DETECTOR

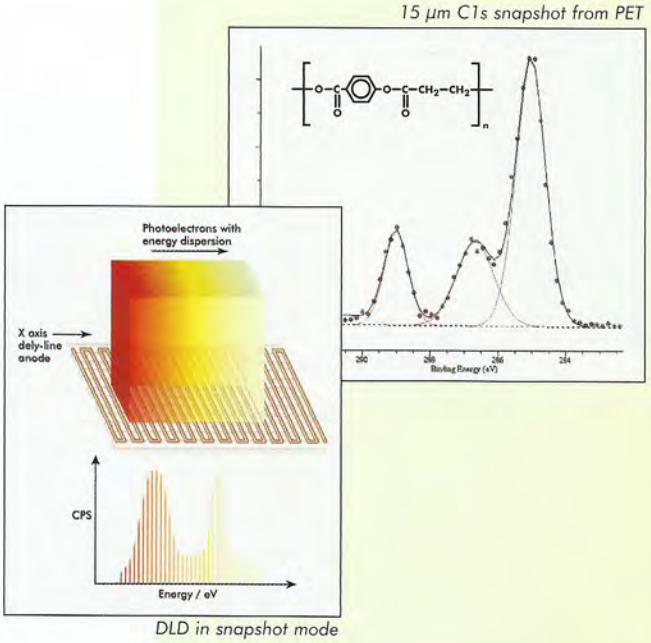
Kratos Analytical continues to lead in the development of imaging X-ray photoelectron spectrometers with the introduction of the delay-line detector (DLD). Representing the next generation of photoelectron detectors, the DLD provides a single pulse counting detector for both spectroscopy and imaging experiments. Comprising a multi-channel plate stack above two orthogonal delay-line anodes and associated electronic control units, the DLD is at the heart of the AXIS Nova.

With a large active area the DLD possesses 128 channels for photoelectron detection in spectroscopy mode. The high number of channels means that, as well as collecting photoelectrons in usual energy scanning mode, the spectrometer can also be used to collect spectra with 128 data points without scanning the analyser, also known as 'snapshot' spectroscopy. This mode allows an individual photoelectron peak to be collected in less than 1 second with obvious advantages for data acquisition during depth profiling or dynamic surface characterisations.

When used in parallel imaging mode photoelectrons with 2D spatial distribution are incident on the DLD. The fast DLD control electronics convert the electron arrival position on each of the two delay-line anodes into an x, y co-ordinate for every electron event. Integrating the signals over several seconds allows the generation of truly quantitative elemental or chemical images of the surface over a 65,500 pixel array. In the highest magnification imaging mode the spatial resolution at the surface is <3μm.

As further discussed in technical note MO223, a significant advantage of the digital DLD technology is that the system has inherent low noise characteristics with spike immunity assured.

15 μm C1s snapshot from PET



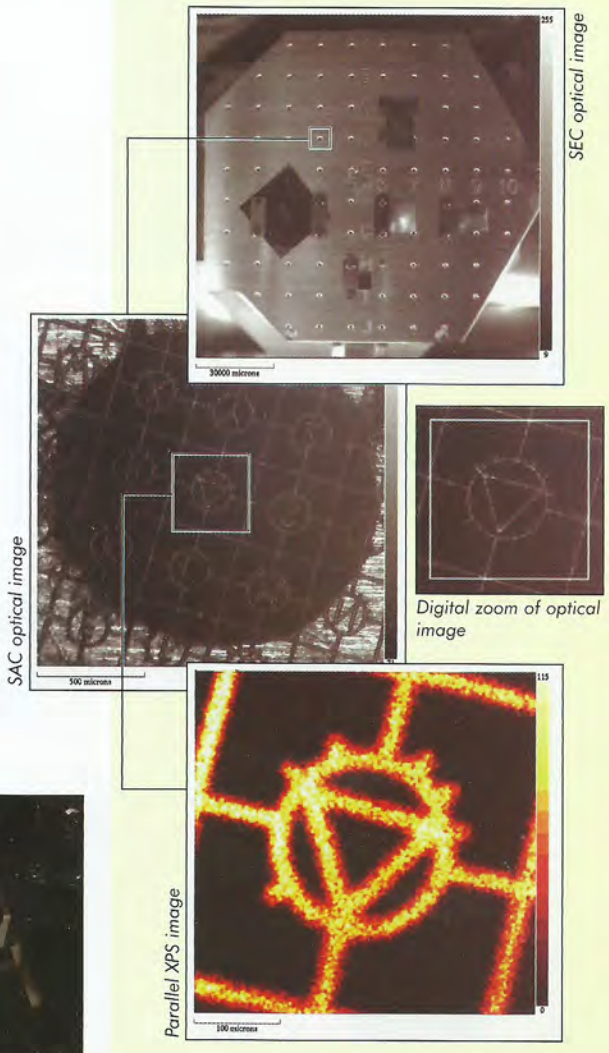
DLD in snapshot mode

SAMPLE HANDLING

The AXIS Nova sample handling and platen navigation system integrates in-situ optical microscopes with the Vision acquisition software for easy, rapid definition of sample analysis positions. Both full platen and 2mm high resolution optical images can be saved into a dataset and recalled for sample positioning. A large viewport allows the user to see the platen in the analysis chamber giving complete confidence in sample location.

An orthogonal colour camera in the sample entry chamber (SEC) provides a digital image of the entire 110mm platen allowing XPS analysis positions to be defined with precision. For large homogeneous samples spectra can be acquired directly from the defined points using an automated sample height definition routine. Alternatively the chosen position can be translated to the high resolution orthogonal microscope in the analysis chamber. The in-situ optics ensure maximum flexibility, avoiding the need for separate external alignment systems so that samples may be re-examined during analysis for greatest analytical flexibility. The high magnification optical image can also be used to directly define the analysis position for selected area spectroscopy.

Storage for three 110mm diameter platens is provided in the SEC giving a total of 285cm^2 accessible analysis area. Automated multi-platen exchange allows platen transfer from the SEC to the analysis position without user intervention.



SEC and sample entry door

Large 110mm diameter sample platen

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THE AXIS NOVA INCORPORATES MARKET LEADING AXIS TECHNOLOGY INCLUDING COAXIAL CHARGE NEUTRALISATION, MAGNETIC IMMERSION LENS AND DELAY-LINE PHOTOELECTRON DETECTION WITH A NEW LEVEL OF AUTOMATED SAMPLE HANDLING IN A COMPACT INSTRUMENT. THE AXIS NOVA IS IDEAL FOR AUTOMATED QA AND PROBLEM SOLVING WITHOUT COMPROMISING THE ULTIMATE SPECTROSCOPIC AND IMAGING PERFORMANCE REQUIRED FOR DEMANDING RESEARCH APPLICATIONS.

Services and Interlocks

Services required for the AXIS Nova which include N_2(g), Ar(g), compressed air and cooling water are input via a discrete 'fluids panel' at the rear of the spectrometer. Thermostatically controlled bake-out heaters and insulating covers are integrated into the design of the spectrometer for easy instrument bake-out. A comprehensive set of hardware and software interlocks protect the AXIS Nova from operation under incorrect conditions.

Compact Footprint

The spectrometer is designed with a compact footprint measuring only 2060mm x 1065mm which incorporates all vacuum pumps. Additional space for a small closed loop water chiller and computer desk is also required. Easy access to the electronic control units and X-ray source is provided via doors at the front of the instrument.

Sample Analysis Chamber (SAC)

A clear view of the sample platen in the analysis chamber is provided by a 140mm diameter window and internal illumination. An in-situ optical microscope produces a high resolution digital image with a 2mm field of view equivalent to 2μm per pixel. The microscope is integrated with the acquisition software and is used for easy sample positioning.

Sample Entry Chamber (SEC)

Sample platens are introduced into the spectrometer via a large hinged door. The 250l/sec turbo pumped SEC has an elevator system with positions for introduction and storage of three sample platens simultaneously. The contents of the SEC can be easily viewed through a large window in the SEC entry door. An orthogonal optical camera in the SEC provides an image of the entire platen for sample identification and analysis position definition.

AXIS NOVA

AUTOMATED X-RAY PHOTOELECTRON SPECTROSCOPY

SYSTEM SUMMARY

VACUUM SYSTEM

Sample entry chamber (SEC)
- Sample elevator (3 platen storage positions)
- Turbomolecular pump (250l/sec) (oil free option)
- Cold cathode wide range pressure gauge
- 236mm ID gate valve

Sample analysis chamber (SAC)
- Stainless steel with Mu metal screening
- Ion pump with cryoshield
- Auxiliary pumping by titanium sublimation pump
- Cold cathode wide range pressure gauge

X-RAY SOURCE

High power Al monochromator
- 500mm Rowland circle
- Single quartz toroidal backplane
- Indexed moveable anode face
- Full computer control with read-back and interlocks

ELECTRON ENERGY ANALYSER

180° hemispherical analyser
- 165mm mean radius

Spherical mirror analyser
- Fixed analyser transmission (FAT) mode

PHOTOELECTRON DETECTOR

MCP stack and delay-line detector
- Scanned and snapshot spectroscopy modes
- 2D imaging mode

CHARGE NEUTRALISATION

Coaxial low energy electron source
- Fully automatic

SAMPLE PLATENS

Standard 110mm diameter plane surface
ARXPS platen for 0 - 85° sample rotation
Compucentric rotation platen
- Continuous 360° rotation about the vertical axis
- Compucentric rotation about a point off central axis of platen

AUTOMATION AND SAMPLE VIEWING

5 axis software control of sample stage (platen dependent)
Software control of platen exchange
Automated small area selection
Optical microscope in SEC (110mm field of view)
High resolution optical microscope in SAC (2mm field of view)

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Kratos Analytical has a policy of continuous product improvement and therefore reserves the right to make alterations to specifications without notice. While Kratos Analytical believes this document to be true and accurate, Kratos Analytical cannot be held responsible for any errors and omissions contained herein.

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