TRACE

Pure Gas Analysis
General Gas Analysis

Extrel
Core Mass Spectrometers

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Trace level analysis of impurities in pure gases is a critical capability in the development and implementation of semi-conductor and other high technology industrial applications. New technologies demand a higher standard for bulk gas purity, improved performance from purification systems, and verification of bulk gas purity at the delivery point. Atmospheric Pressure Ionization Mass Spectrometry (API-MS) has developed into a critical tool for pure gas analysis, providing part-per-trillion (ppt) level detection of certain impurities in pure gases. For other impurities for which API is not a viable technique, electron impact ionization (EI) mass spectrometry can be used to supplement the analysis. In some cases, membrane introduction mass spectrometry (MIMS) with an EI source can provide sub part-per-billion (ppb) detection limits for some components. Extrel's VeraSpec Trace has the unique capability to combine these techniques in a single instrument.

	API	EI	MS/MS	MIMS
VeraSpec Trace	*Yes/No	*No/Yes	No	No
VeraSpec Trace DS	Yes	Yes	No	No
VeraSpec Trace DS-MIMS	Yes	Yes	No	Yes
VeraSpec Trace MS/MS	*Yes/No	*No/Yes	Yes	No
VeraSpec Trace DS MS/MS	Yes	Yes	Yes	No
VeraSpec Trace DS-MIMS MS/MS	Yes	Yes	Yes	Yes

Figure 1 *Note: The ionization system of the base system is selected by the customer. Either EI or API can be selected, but not both.

The **VeraSpec Trace** system provides the ideal ionization techniques for **pure gas** analysis in one system. The system can be configured for both ionization techniques, eliminating the need for two mass spectrometers and significantly reducing the overall expense. The base **VeraSpec Trace** system utilizes either Extrel's high performance closed source EI ionizer, or the proven, reliable DC Corona Discharge API source. The DC Corona Discharge Source is a chemical or soft ionization source that is ideal for monitoring molecules in the ppb and ppt range. The EI system is taken from Extrel's MAX300 Industrial mass spectrometers and has the same proven performance available on those systems with detection limits in the low ppb range.

The system utilizes a 19mm tri-filter quadrupole and is available in several mass ranges noted in Figure 2. The system is also offered in an MS/MS configuration, the **VeraSpec Trace MS/MS.**

The **VeraSpec Trace DS** is the dual source model with both an API and Extrel's decidedly capable closed source EI ionizer with a capillary inlet in the standard configuration. Source selection is accomplished using Extrel's large deflector for maximum flexibility and sensitivity. Adding a Membrane inlet to the EI system pushes the detection limits for applicable analytes into the sub-ppb range in the **VeraSpec Trace DS-MIMS** system.

System	Quadrupole Mass Filter	Operating Frequency	Mass Range	Relative Transmission	Resolution MI/M FWHM	General Sensitivity (mA/Torr)
MAX-4000HT	19 mm (3/4) inch tri-filter	440 kHz	4-4000	50%	1500	0.75
MAX-1000	19 mm (3/4) inch tri-filter	880 kHz	1-1000	50%	1800	1
MAX-500HT	19 mm (3/4) inch tri-filter	1.2 MHz	1-500	60%	2000	2
MAX-120	19 mm (3/4) inch tri-filter	2.1 MHz	1-120	65%	2500	3
MAX-60	19 mm (3/4) inch tri-filter	2.9 MHz	1-60	75%	3000	4

Figure 2

Dual Source Performance

One of the most innovative capabilities of the VeraSpec Trace is found in the Dual Source version. While API ionization allows for high ion currents resulting in low detection limits, the technique is limited to molecules whose ionization energy is less than that of the balance gas or any other component at a high concentration, and which have sufficient proton affinity to be ionized.

As a result of this limitation, every table of lower detection limits for pure gas analysis using API has gaps for components that cannot be measured. With the VeraSpec Trace DS, the gaps are filled in as seen in Figure 3. The addition of a high performance closed EI source delivers detection to ppb levels.

Bulk Gases \ Impurities	H2	H2O	N2	Ar	O2	CO2	CO	CH4	NH3
Hydrogen (H2)	■	10 ppt	*100 ppb	*10 ppb	*100 ppb	5 ppt	10 ppt	10 ppt	500 ppt
Helium (He)	*1ppm	10 ppt	*100 ppb	*5 ppb	10 ppt	5 ppt	10 ppt	10 ppt	500 ppt
Nitrogen (N2)	*1ppm	10 ppt	■	*5 ppb	10 ppt	5 ppt	*500 ppb	10 ppt	500 ppt
Argon (Ar)	*1ppm	10 ppt	*100 ppb	■	10 ppt	5 ppt	10 ppt	10 ppt	500 ppt
Oxygen (O2)	*1ppm	*2 ppm	*500 ppb	*10 ppb	■	*500 ppb	*100 ppb	*500 ppb	*200 ppb
Carbon Dioxide (CO2)	*1ppm	*2 ppm	N/A	*10 ppb	N/A	■	N/A	N/A	*250 ppb

Figure 3 Detection limits for some compounds in some bulk gas. Detection limits marked with * are based in EI ionization tri-filter.

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Impurities	VOCs with MIMS in Air	Impurities	VOCs with MIMS in Air
Carbon Disulfide	45 ppt	**Toluene**	10 ppt
Dimethyl Sulfide	100 ppt	**Pyrrole**	15 ppt
Dimethyl Disulfide	90 ppt	**Xylenes, Ethylbenzene**	15 ppt
Benzene	6 ppt	**VCM**	28 ppt

Figure 4 Based on EI with a MIMS inlet.

With the membrane inlet, analytes, examples of which are shown in Figure 4, sub-ppb levels can be achieved.

The VeraSpec Trace DS draws from the strength of both Extrel's Research products and Industrial experience. The result is an instrument with exceptional detection limits for all required components with the stability and reliability required in an Industrial setting. In addition, it has unprecedented flexibility and capability for trace analysis in one instrument. The following data gives some examples of the instrument's broad potential. All of the data in Figures 5 and 6 below was taken using a VeraSpec Trace DS MS/MS.

Lower Detection Limits in Pure Gas

Trace levels of carbon dioxide and oxygen in dry nitrogen were measured in the VeraSpec Trace at a series of levels below 2000 ppt. A multi-point calibration curve was constructed by collecting one spectrum every 60 seconds (30 seconds / mass) while scanning m/z 31.5 - 32.5 and m/z 43.5 - 44.5. The detection limits were calculated at a t value of 3 (a 99.5% confidence interval), and the calibration curves had R^2 value of > 0.9. The calibration curve was used to calculate detection limits for CO2 of 0.9 ppt, and for O2 of 4.1 ppt.

CO2 Data Table

Conc (ppt)	0	44	158	317	475	937	1875
Mean (int CPS)	326	12532	55872	97376	152246	271527	478743
Stdev	79	688	1865	2700	3950	5524	9519
RSD	24.2%	5.5%	3.3%	2.8%	2.6%	2.0%	2.0%

Figure 5

O2 Data Table

Conc (ppt)	0	125	158	317	475	937	1875
Mean (int CPS)	357	9455	21355	32939	54823	93790	157983
Stdev	102	596	1024	1086	1244	2664	3853
RSD	28.6%	6.3%	4.8%	3.3%	2.3%	2.8%	2.4%

Figure 6

Freon trace measurements with identification by MS/MS

Figure 7 is an MS/MS product ion spectrum taken of a 500 ppt sample mixture of R11 and R12 and 2.25 ppm Methane. This spectrum qualitatively confirms the presence of R11 (Trichlorofluoro-methane) in the presence of Methane and R12 (Dichlorodifluoromethane). m/z 101 is the parent ion and is a fragment ion of both R11 and R12. Due to the differing structure of R11 and R12, R11 allows the loss of the additional Chlorine atom to form the m/z 66 fragment. Thus, the strong signal at m/z 66 indicates the presence of R11.

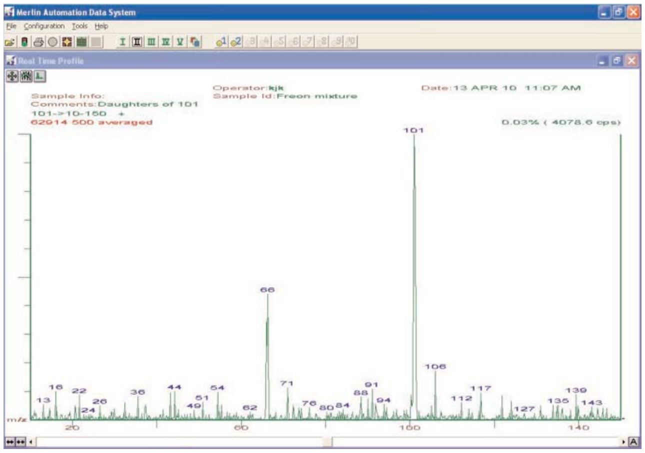

Figure 7

Figure 8 is analysis of room air taken in negative ion mode. The spectrum is zoomed in to see the very low concentrations of SOx, NOx, and COx, which are environmentally significant components.

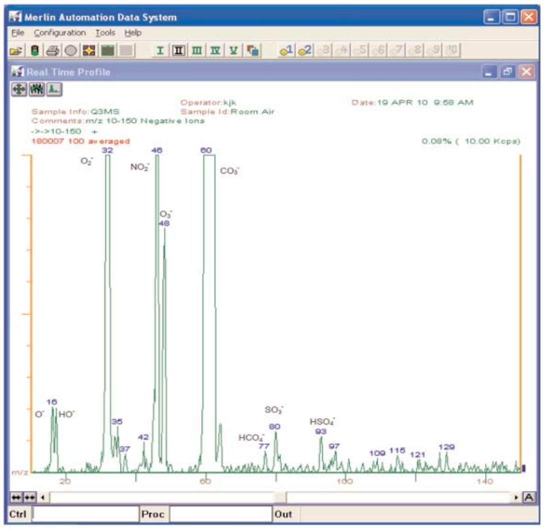

Figure 8

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