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APPLICATION SPOTLIGHT

HOW TO ACHIEVE HIGH ACCURACY WITH DIFFICULT SAMPLES

The following report is the result of a collaboration between Glass Expansion and Inorganic Ventures

Introduction

It is very common for ICP labs to encounter samples with a varying degree of total dissolved solids (TDS). However samples with high TDS, 5 to 20%, can often pose many analytical challenges for the ICP analyst. These challenges include short torch lifetime and interrupted analyses due to clogged nebulizer or injector. Inorganic Ventures is a manufacturer of high-quality analytical inorganic and organic Certified Reference Material (CRM) standards and custom made CRMs for ICP-OES, ICP-MS, ion chromatography and numerous other testing modalities. Inorganic Ventures' certified values are obtained using two independent methods, with traceability to NIST SRMs, and certified uncertainties are calculated by means of error budgets that include both random and systematic errors.

Inorganic Ventures is accredited by A2LA to ISO Guide 34 and ISO Guide 17025, and registered to ISO 9001. Two of the ISO Guide 34 accreditation requirements are the assessment of homogeneity and stability of all products covered by this Guide. These assessments are designed and performed in accordance with ISO Guide 35 and are used to establish the uncertainty components contributed by these properties to the overall uncertainty of the CRM. The homogeneity assessment should be performed using the same detection principle that is the basis of the measurement technique by which the CRM is characterized, e.g. a CRM purposed for ICP analysis, should be assessed for homogeneity by ICP. An important criterion for homogeneity assessment is that the sample remains undiluted. Through dilution, heterogeneity effects may be masked or eliminated, e.g. dissolved gases or particulates could be re-dissolved by dilution of the sample. As a result of this requirement, Inorganic Ventures analyzes their reference materials undiluted to assess their homogeneity. This poses a difficult challenge for ICP-OES measurements, as the concentrations of analytes may range from 100 µg/mL to 10,000 µg/mL and higher concentrations. Under standard ICP-OES operating conditions, the injector and nebulizer can clog and prematurely shutdown the instrument. Analyzing high TDS samples under standard conditions can also lead to poor precision and accuracy, increased consumable costs, and down time for instrument maintenance. This report highlights a collaboration with Inorganic Ventures in which we describe how to optimize the sample introduction system and method parameters of the ICP to better handle high TDS samples. The performance achieved with a newly introduced fully ceramic D-Torch for the Spectro Arcos EOP (Axial) ICP-OES is also described.

Choosing the Optimum Nebulizer and Spray Chamber for High TDS

When dealing with any type of new sample you want to be sure to select a nebulizer capable of handling that particular matrix. A detailed report on nebulizer selection can be found in our Newsletter (1). When dealing with high TDS samples, Glass Expansion recommends using the SeaSpray or DuraMist nebulizer for non-HF containing and HF containing samples, respectively. The SeaSpray nebulizer is a glass concentric nebulizer designed to handle up to 20% TDS. The DuraMist is also a concentric nebulizer, but features a PEEK body with the ability to handle up to 5% hydrofluoric acid (HF) and up to 30% TDS(2). Both the SeaSpray and DuraMist nebulizer offer superior nebulizer efficiency for trace level analysis with the freedom from clogging due to "salting." Inorganic Ventures deals with aqueous samples and some that contain trace amounts of HF. For this reason our report will focus on the performance achieved with both the SeaSpray and DuraMist nebulizer.

In this report the Twister spray chamber was used for the non-HF work and the Tracey TFE spray chamber was used for the work with HF. The Twister cyclonic spray chamber includes a baffle which acts as a secondary particle separator, further reducing the mean droplet size entering the torch. This reduces the solvent load in the plasma without compromising detection limits, which is of great importance when dealing with high TDS. For HF applications, where a glass spray chamber is not suitable, it is necessary to use plastic. The Tracey TFE and PFA spray chambers are specially treated with Glass Expansion's proprietary StediFlow treatment to improve the wettability of the spray chamber's interior surface. The StediFlow treatment ensures efficient drainage and provides sensitivity and precision almost as good as what can be achieved with a glass cyclonic spray chamber. Figures 1a) and 1b), show photos of the glass and HF-resistant sample introduction systems described above.

Figure 1a: Aqueous sample introduction system, SeaSpray Nebulizer and Twister spray chamber for Spectro Arcos EOP ICP-OES.

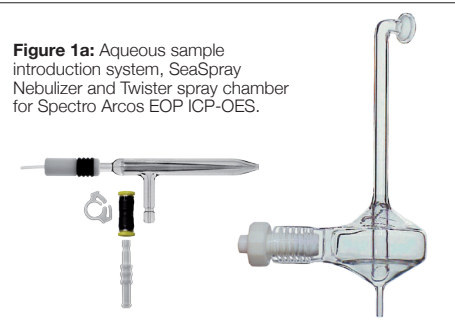
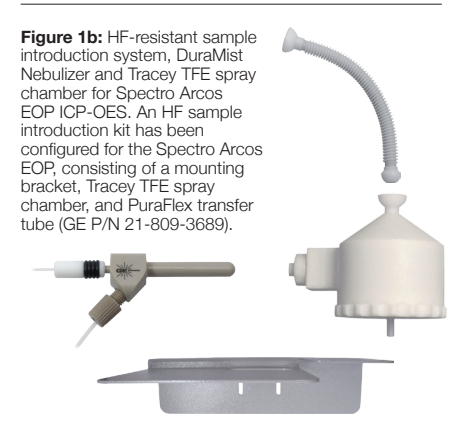


Figure 1b: HF-resistant sample introduction system, DuraMist Nebulizer and Tracey TFE spray chamber for Spectro Arcos EOP ICP-OES. An HF sample introduction kit has been configured for the Spectro Arcos EOP, consisting of a mounting bracket, Tracey TFE spray chamber, and PuraFlex transfer tube (GE P/N 21-809-3689).



Optimizing Instrument Parameters for High TDS

When dealing with high TDS it is recommended that the nebulizer sample flow rate is reduced as much as the required detection limits allow. This will help prolong the lifetime of the torch by introducing less sample into the plasma. Since the injector tip is also susceptible to clogging, utilizing a large bore injector, e.g. 2.4mm or 3.0mm, is a good idea. We also recommend increasing the auxiliary gas flow to lift the plasma further away from the injector to help slow the build-up of salt at the injector tip. Adding a long rinse with deionized water (DIW) or very dilute acid in between each sample of course helps the analyst to run longer without interruption and maintain optimum performance. Utilizing the high TDS instrument parameters listed in Table 1 has allowed Inorganic Ventures to drastically reduce the amount of sample introduced into the plasma and cut down on interrupted runs on their Spectro Arcos EOP (Axial) ICP-OES. Previously, samples with as much as 2% TDS would deposit salt at the injector tip, resulting in poor signal stability and eventually instrument shutdown. Now, analyses involving 10,000 µg/mL and higher can be run without interruption. Although not utilized in this work, an argon humidifier, like the Capricorn can be added to the sample introduction system. The added moisture in the gas from the humidifier helps to prevent salt build up at the nebulizer and injector tips. This option should be explored if runs are still being interrupted after optimizing all other parameters.

[Click here for more information on the Capricorn.](#)

In addition to the changes listed in Table 1, Inorganic Ventures has also utilized a smaller i.d. peristaltic pump tubing (0.38mm), nebulizer capillary (0.5mm), and autosampler probe (0.5mm) to reduce sample flush times. Even with sample flow rates as low as 0.24 mL/min, the sample flush time is only 100 seconds (70 seconds at 0.6mL/min fast pump and 30 at 0.24mL/min). Another alternative is to add an enhanced productivity accessory like the Niagara Rapid Rinse or Niagara Plus. See www.geicp.com for more information. This has been shown to reduce rinse and stabilization times to several seconds (3).

	Original Settings	High TDS Settings	High TDS Settings (with HF)	High TDS Settings (with HF)
RF Power (W)	1400	1400	1400	1400
Plasma gas flow (L/min)	15	15	15	15
Auxiliary gas flow (L/min)	0.8	1.5	1.5	1.5
Nebulizer gas flow (L/min)	0.65	0.65	0.75	0.75
Nebulizer flow rate (mL/min)	1.2	0.24	0.54	0.54
Torch	Single piece	Single piece	Semi-demountable	Ceramic D-Torch
Injector i.d. (mm)	1.8	1.8	2.4	2.4
Nebulizer	SeaSpray (P/N ARG-07-USS2)	SeaSpray (P/N ARG-07-USS2)	DuraMist (P/N ARG-07-DM1)	DuraMist (P/N ARG-07-DM1)
Spray Chamber	Twister (P/N 20-809-0243HE)	Twister (P/N 20-809-0243HE)	Tracey TFE (P/N 20-809-2506)	Tracey TFE (P/N 20-809-2506)

Table 1: Instrument parameters, Spectro Arcos EOP ICP-OES

Results

Each day, Inorganic Ventures performs an ICP optimization check verifying the conditions for the ICP-OES. The data presented in Table 2 shows the optimization data collected on one day for each sample introduction setup utilized. When reducing the sample flow rate from 1.2 mL/min down to 0.24 mL/min (80% reduction) there was only a sensitivity decrease of 42% to 57%. The transport efficiency at a sample flow rate of 0.24 mL/min is greatly improved and allows for longer runs with high TDS. The data in Table 2 also confirms that there is no significant loss of sensitivity or increase in RSD when the introduction systems are interchanged. The “High TDS” setup uses a single piece torch with a 1.8mm i.d. injector, thus the reason for the lower counts compared to the systems utilizing a 2.4mm i.d. injector. Table 3 shows a comparison of the fully ceramic D-Torch run at an auxiliary gas flow of 0.8 L/min versus 1.0 L/min, proving that there is no significant change in the results when increasing the auxiliary flow to better handle high TDS.

Sample introduction system	Mn 257		As 189		Pb 220	
	Intensity (cps)	RSD (%)	Intensity (cps)	RSD (%)	Intensity (cps)	RSD (%)
Original Setup	6,126,000	≤ 0.5	165,000	≤ 0.5	277,000	≤ 0.5
High TDS	3,518,000	0.35	71,226	0.83	148,760	0.30
High TDS with HF (SDT)	4,948,970	0.35	112,346	0.57	188,877	0.31
High TDS with HF (D-Torch/quartz outer tube)	5,283,250	0.27	139,672	0.51	211,841	0.28
High TDS with HF (D-Torch/ceramic outer tube)	4,679,360	0.15	105,734	0.38	170,613	0.41

* All data collected at 0.8 L/min auxiliary gas flow.

Table 2: Optimization data for each sample introduction system.

Auxiliary Gas Flow	Mn 257		As 189		Pb 220	
	Intensity (cps)	RSD (%)	Intensity (cps)	RSD (%)	Intensity (cps)	RSD (%)
0.8 L/min	4,679,360	0.15	105,734	0.38	170,613	0.41
1.0 L/min	4,587,320	0.44	119,154	0.48	184,956	0.28

Table 3: Comparison of optimization data for ceramic D-Torch at 0.8 L/min and 1.5 L/min auxiliary gas flow.

A Fully Ceramic Demountable Torch

Even after optimizing the sample introduction system for high TDS, a combination of high temperature and salt deposit causes a quartz torch to devitrify. Higher concentrations of salt in the samples lead to more rapid devitrification. The disadvantage of a single-piece torch is that it is a relatively high cost consumable item that requires regular maintenance and replacement, particularly with more demanding samples, such as HF, organic solvents or high TDS.

The Glass Expansion D-Torch is a revolutionary new torch design, providing the benefits of a demountable torch at a significantly lower cost without sacrificing performance. The D-Torch incorporates a ceramic intermediate tube for greater robustness and provides the analyst with an outer tube (quartz or ceramic) which can be replaced when it fails rather than replacing the entire torch. A ceramic outer tube is of particular benefit for the analysis of difficult sample matrices such as high TDS and HF. The D-Torch also features an interchangeable injector, allowing the analyst to have a specific injector for each application whether aqueous, organics, high TDS or HF. Having a demountable torch also allows for easy maintenance and cleaning.

The D-Torch is currently available for a range of ICP-OES and ICP-MS instruments. In this report we feature the release of the D-Torch for the Spectro Arcos EOP (Axial) ICP-OES, shown in Figure 2. Inorganic Ventures has found the benefits of a fully ceramic D-Torch to also include greater signal stability. The example shown in Figure 3 shows a dramatic increase in the signal stability obtained from a homogeneity assessment of a custom blend environmental standard when using the fully ceramic D-Torch compared to a semi-demountable torch with quartz outer tube. The ceramic outer tube of the D-Torch is made from sialon, which is a ceramic material derived from silicon nitride. Sialon is one of the most durable and robust ceramic materials known and maintains its properties at high temperatures. The ceramic outer tube of the D-Torch does not devitrify and is not affected by salt deposits. It also runs hotter than quartz which slows the build-up of material on the outer tube and results in a more robust plasma, a result which is especially important in the presence of high TDS samples.



Figure 2: D-Torch for Spectro Arcos EOP

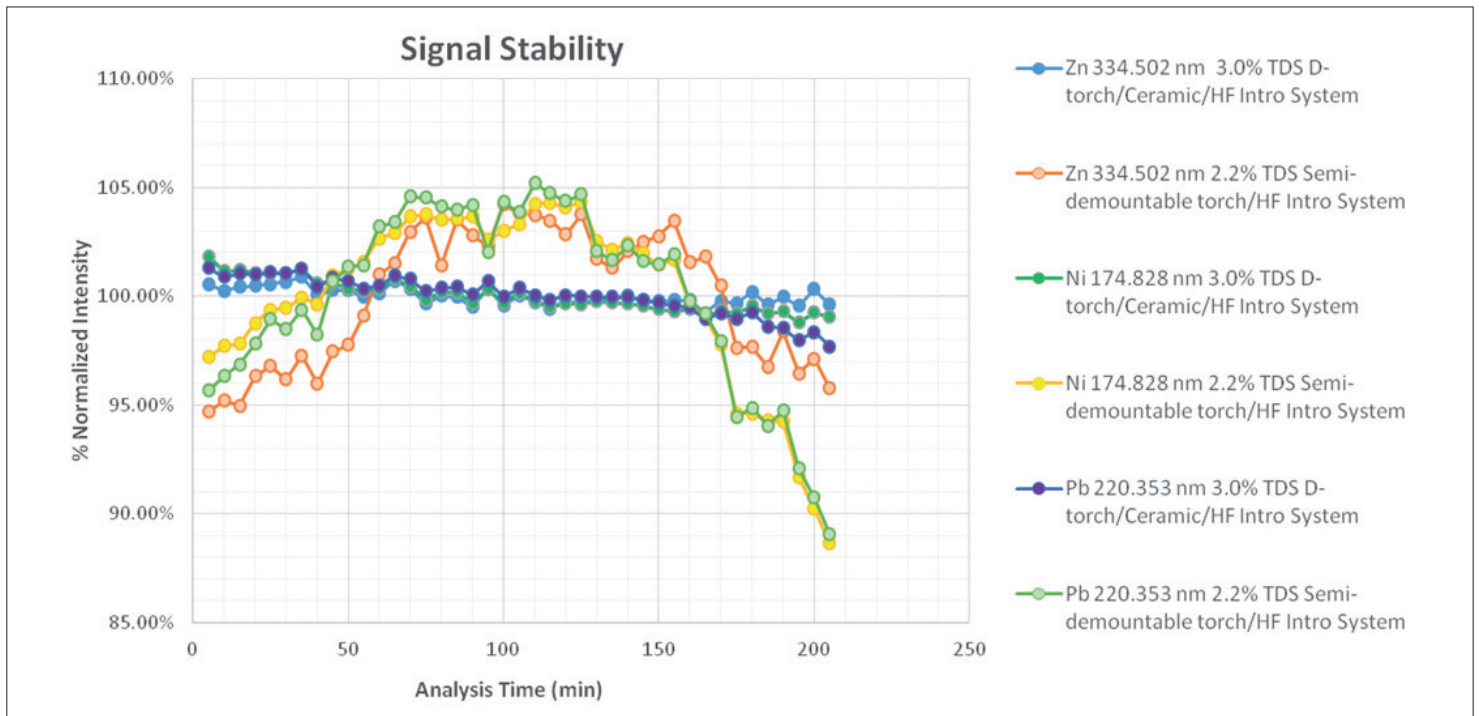


Figure 3

Element	Ubb D-Torch 3% TDS	Ubb SDT 2.2% TDS	Improvement in Ubb with D-Torch
Zn 334	0.001710	0.003025	43%
Pb 220	0.001136	0.001879	40%

Table 4: Uncertainty measurements: ceramic D-Torch vs. Semi-Demountable Quartz Torch

With the exception of the torch, all other components of the sample introduction system were the same: DuraMist nebulizer, Tracey TFE spray chamber and a 2.4mm i.d. alumina injector. The duration of each analytical run was approximately 3.4 hours. Due to this improvement in signal stability with the fully ceramic D-Torch, the calculated uncertainties for Zn and Pb showed a significant improvement over the semi-demountable torch. The uncertainties calculated in Table 4 are bottle to bottle uncertainties (ubb) and were determined using a single factor analysis of variance algorithm.

Conclusions

In summary this application spotlight shows the importance of selecting the proper sample introduction system to best fit your application needs. By optimizing their ICP-OES for high TDS measurements, Inorganic Ventures has drastically reduced the amount of sample introduced into the plasma and cut down on interrupted runs. Both sample introduction setups, SeaSpray/Twister and DuraMist/Tracey TFE, provided excellent sensitivity and precision in the presence of varying concentrations of high TDS. A fully ceramic D-Torch helps to reduce consumable cost and increase plasma stability in the presence of difficult sample matrices. In the next phase of this collaboration Inorganic Ventures will look to further improve their ICP-OES and ICP-MS measurement accuracy by incorporating the IsoMist temperature controlled spray chamber and the Assist syringe driven sample introduction accessory. The use of the IsoMist significantly improves long-term signal stability by holding the spray chamber at a constant temperature. The Assist will cut sample analysis time in half and improve precision by eliminating the signal pulsations which occur with the peristaltic pump delivery system.

References

1. Glass Expansion Newsletter, June 2013.
2. Glass Expansion Newsletter, October 2011.
3. Glass Expansion Newsletter, February 2013.

NEW PRODUCTS

New Control Module for Niagara and Assist products

The Niagara and Assist range of products dramatically improve the speed and accuracy of ICP analyses. These products have now been upgraded by the release of a dedicated Control Module. The benefits are:

- Faster communication, resulting in even higher sample throughput.
- The option of running unattended without any connection to the instrument PC once the method has been loaded.
- Simplified installation.

The **Niagara Plus CM** utilises flow injection technology to halve your operating costs and your analysis time.



The **Assist CM** syringe-driven sample introduction system improves throughput, accuracy and precision.



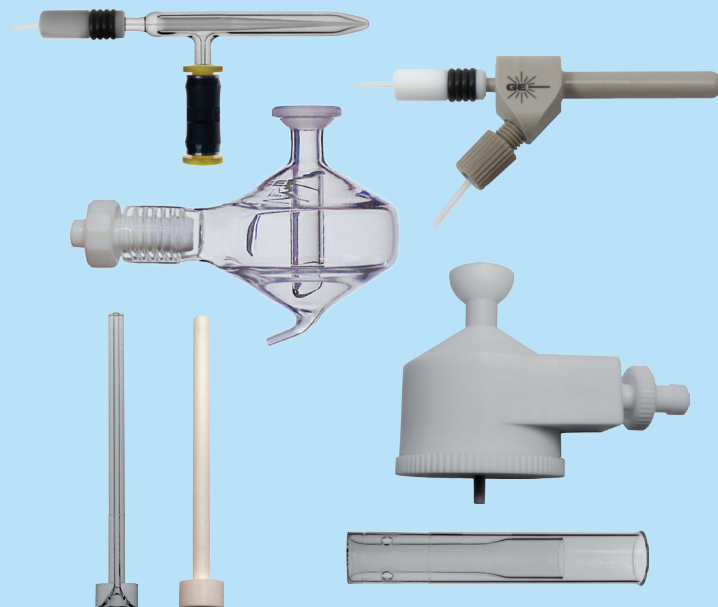
Consumables for Agilent 4100 MP-AES

We can now supply a range of nebulizers and spray chambers for this model. [Click here to see the available range.](#)



Consumables for SPECTROBLUE ICP-OES

We can now supply a full range of nebulizers spray chambers and torches for this model. For products to suit your application, [click here for SPECTROBLUE EOP](#) or [here for SPECTROBLUE SOP](#). For the full range of products, [click here for the EOP](#) and [here for the SOP](#).



NEW PRODUCTS

D-Torch for Horiba Jobin Yvon and Spectro EOP models

The D-Torch is a new demountable torch design that provides the benefits of a fully demountable torch at a significantly lower cost. We have previously released the D-Torch for several ICP-OES and ICP-MS models. D-Torches are now also available for the Horiba Jobin Yvon Radial and Spectro EOP models.

The D-Torch is a cost-effective alternative to the standard fixed torch or semi-demountable torch. It will save money for any laboratory with a moderate workload. In most cases, when the torch wears, you will only need to replace the outer tube instead of replacing the entire torch. You will realize a saving after replacing the outer tube three to five times.

The optional ceramic outer tube is much more robust than quartz and provides extended torch life, particularly with samples that cause rapid deterioration of a quartz torch, such as fusions, high salt solutions or oils.

Part No.	Description
30-808-2882	D-Torch for HJY Radial
31-808-2893	Alumina Injector 3.0mm for HJY D-Torch
30-808-3371	D-Torch for Spectro EOP
31-808-3425	Ceramic Outer Tube and Fibre Optic for Spectro EOP D-Torch
31-808-2974	Tapered Quartz Injector 1.8mm for Spectro D-Torch
31-808-3388	Tapered Alumina Injector 2.4mm for Spectro D-Torch

[Click here to see the full D-Torch range.](#)



Expanded range of peristaltic pump tubing

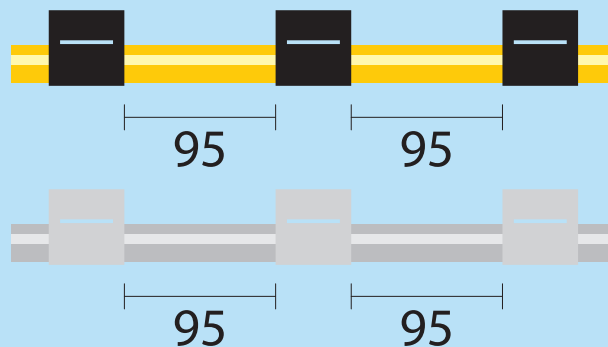
Glass Expansion offers an extensive range of peristaltic pump tubing covering all of the most common sizes (from 0.13mm ID to 3.17mm ID) and materials (including PVC, Solva, Viton and Tygon MH). We have recently added 3 tag Solva tubing to our range. The Solva material is specifically recommended for petroleum products and the 3 tag tubing is suitable for the Agilent ICP-MS products as well as the Thermo Iris series.

The newer Thermo iCAP Q ICP-MS and iCAP 7400/7600 ICP-OES have a different sized peristaltic pump which requires tubing with 95mm between tags. We now have 3 tag tubing with 95mm between tags in both the standard PVC and the Solva material.

To simplify the connection between the nebulizer capillary tubing and the pump tubing, all of the above tubing is available in the Contour Flared End configuration.

[Click here to see the benefits of the Contour tubing.](#)

[Click here to see the full range of peristaltic pump tubing.](#)



INSTRUMENT NEWS

From Agilent Technologies – New Revision of ICP-MS MassHunter, Agilent Compliance Software Options, and USP<232>

An updated version of ICP-MS MassHunter software for the 7700 Series ICP-MS and 8800 ICP-QQQ has been released. The major developments in the new B.01.03 revision are:

- Adds support for OpenLAB Data Store compliance software
- Enhanced functionality with SDA and OpenLAB ECM compliance software
- New Preset Method for USP<232>/<233> Elemental Impurities in pharma products
- New data output and report storage options
- Support for GC 7890B
- Support for Injector Program for LC autosamplers

The addition of support for Agilent's Data Store compliance software means that Agilent ICP-MS users now have a complete range of compliance solutions to choose from, depending on their lab type, number of ICP-MS instruments, data volume, and security requirements.

The new B.01.03 revision of ICP-MS MassHunter ships as standard with all new 7700 Series ICP-MS and 8800 ICP-QQQ instruments from August 2013.

From SPECTRO – SPECTROBLUE ICP-OES analyzer now available in three versions: Axial, Radial or the combination of Axial and Radial observation

SPECTRO Analytical Instruments now offers three versions of its SPECTROBLUE ICP-OES. These models set new benchmarks for compact, mid-range analyzers characterized by high performance, simple operation, low maintenance, and great affordability.

SPECTROBLUE EOP, the unique axial interface version, provides high sensitivity and detection limits for superior analysis of industrial and environmental trace elements.

SPECTROBLUE SOP radial interface version offers precise performance at higher sample concentrations, exhibiting excellent tolerance for high saline and organic fractions plus superb analysis of suspensions and slurries.

SPECTROBLUE TI twin-interface version automatically performs both axial and radial viewing of the plasma. This optimizes linearity and dynamic range, while enabling high-sensitivity measurement of toxic elements. "This model is especially suitable for environmental analyses," says Olaf Schulz, ICP-OES Product Manager. "It can accurately determine levels of alkali/earth alkali elements in complex alkali matrices. So the user gets superior analysis of sodium, potassium, or calcium in wastewater or soil matrices." SPECTROBLUE TI minimizes the easily ionizable element (EIE) effect that can decrease linearity. It also cuts cost and contamination by eliminating ionization buffers. Contamination, and thus cleaning and maintenance, also is greatly reduced by offsetting the analyzer's switchable three-mirror periscope at a 45° angle. Finally, this version features a revolutionary design that minimizes the number of optical surfaces, maximizing light throughput and optimizing performance. The new SPECTROBLUE'S (Model No. FMX26) flexible design allows field conversion of a SPECTROBLUE EOP model to full TI version functionality.

All three versions of the SPECTROBLUE spectrometer are available for shipment from SPECTRO dealers worldwide. For more information please visit www.spectro.com/blue.



From Teledyne Technologies – Teledyne Completes Acquisition of CETAC Technologies

Teledyne Technologies Incorporated has announced that its subsidiary, Teledyne Instruments, Inc., has completed the acquisition of assets of SD Acquisition, Inc. (d/b/a CETAC Technologies). Headquartered in Omaha, Neb., CETAC Technologies is a leading designer and manufacturer of automated sample handling and sample introduction equipment for laboratory instrumentation. Terms of the transaction were not disclosed.

CETAC manufactures rugged automated sample introduction equipment for a range of elemental and chemical analysis systems including inductively coupled plasma mass spectrometry (ICP-MS) and high-performance liquid chromatography (HPLC). In addition, CETAC provides unique sample preparation devices, including nebulizers and laser ablation systems, for trace elemental analysis, as well as complete mercury analysis systems.

Teledyne Technologies is a leading provider of sophisticated instrumentation, digital imaging products and software, aerospace and defense electronics, and engineered systems. Teledyne Technologies' operations are primarily located in the United States, Canada, the United Kingdom and Mexico. For more information, visit Teledyne Technologies' website at www.teledyne.com.

NEW GLASS EXPANSION D-TORCH*

Reduce your ICP running costs
with our revolutionary new
demountable torch.



- Why replace the entire torch when just the outer tube wears? With the D-Torch, you need only replace the outer tube. And most operators will be saving money after only two replacements.
- Interchangeable quartz and ceramic outer tubes.
 - Quartz is ideal for most aqueous samples.
 - Ceramic lasts much longer for samples with high dissolved solids or for organics applications such as wear metals in engine oils.
 - Ceramic provides low background for Si determinations.
- Interchangeable injectors for aqueous, organics, high dissolved solids or HF applications.

The D-Torch is also much lower cost than other demountable torches. Please contact us for information on a D-Torch to suit your ICP model.

* Patent Pending