MultiNeb®

Multiple inlet nebulizer for Spectrochemical analysis

Ref. Multineb – 11/2017

Summary

The MultiNeb® nebulizing device, developed by analytical chemistry experts and engineers, **simplifies** and makes more **cost-effective** all new **ICP-OES, MIP-OES** and **ICP-MS** analytical operations involving the **simultaneous nebulization of fluids** (i.e., samples, standard solutions, solvents, etc.).

Specially recommended, but not only, for **water** and **fuel innovative analysis,** for instance:

1) Simultaneous determination of **hydride and non-hydride forming elements in water or fuel samples.**
2) **Direct introduction of complex matrices** (e.g., high salt content, acid digested and organic samples) for elemental analysis.
3) Analysis of **biodiesel and oil samples** by on-line calibration.
4) **In-line redox reactions** for iodine determination.

Features

While designed and manufactured to be used on existing ICP and MIP equipment without any modification required, the MultiNeb is a keystone on innovative in-line analytical methodologies and potential new equipment development.

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Analytical procedure	MultiNeb® Innovation
Sample dilution	Samples can be directly diluted in place just getting the solvent into the nebulizer at the right flow rate, allowing the automatization of the dilution process
Internal standard calibration	Simultaneous introduction of the internal standard and calibration standards/samples allows the best available compensation for matrix effects.
Standard addition calibration	Simultaneous introduction of the standard calibration and samples has the advantage of combining the ease of use of external calibration with the accuracy provided by the standard addition calibration.
Isotopic dilution analysis	Samples can be spiked inside the nebulizer by introducing the spike solution to the nebulizer with the right flow rate, reducing the time of analysis. This application needs to be explored and proven yet.
Chemical vapor generation	Allowing chemical reactions to take place in aerosol phase inside the nebulizer between the analytes of the sample and calibration standards, and one or more reagents (e.g., on-line hydride generation, on-line cold vapor generation and on-line redox reactions).
Organic sample analysis	Allowing the analysis of organic samples with the plasma by emulsifying them with aqueous solutions. A high mixing efficiency is achieved and the formation of carbon deposits on the torch is substantially reduced.
Liquid-liquid extraction	Reducing the experimental time, analyte losses and sample contamination. In addition, large sample amounts, expensive organic reagents and/or toxic ones are not required. This application needs to be explored and proven yet.
Discrete samples/standards introduction	Directly introducing the analyte in a discrete way inside the nebulizer, removing diffusion problems.

Performance

Characteristic	Values*
Mean droplet size (D10)*	10.2 µm
Sauter mean diameter (D32)*	19.4 µm
Liquid flow rate	1.50 mL min^{-1}
Argon flow rate	0.75 L min^{-1}
Argon pressure	1740 mbar

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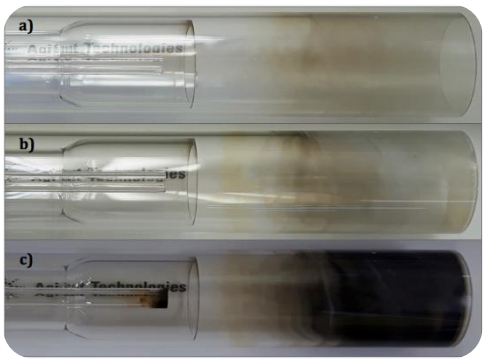
Materials	PEEK, Teflon, and other inert and resistant polymers

*as resulting from a bench testing nebulization with a two-dimensional phase Doppler particle analyzer (2D-PDPA, TSI Inc., Shoreview, MN, USA) equipped with data acquisition software (FlowSizer v. 2.0.1.0, TSI Inc., Shoreview, MN, USA). The primary aerosol was measured 3 mm from the nozzle along the centerline of the aerosol cone. In each PDPA acquisition experiment, approximately 10,000 droplets were measured. The parameters to be set in the PDPA system (i.e., detector voltage, burst threshold, band pass filter, downmix frequency, intensity validation, etc.) were optimized separately for each measurement, following strictly the producer's recommendations for this type of samples. Droplet size measurements were done in triplicate.

Simplicity and productivity

Multiple-inlet techniques have a clear advantage over conventional ones, since all or part of the sample preparation is performed by mixing the sample and reagents directly at the aerosol level, allowing the dosage of immiscible materials.



That way, most of the sample preparation can be performed in a quick, easy and efficient step.

There are additional benefits as a significant reduction of carbon deposits on the torch on the petrochemical analysis that allows: (i) to extend the life of the torch; (ii) to improve the accuracy of analysis; and (iii) more tests between cleanings could be done.

All this resulting in a significant increase of **productivity** and decrease the **cost** of analysis.

Comparative advantages

- Even with immiscible liquids, MultiNeb® obtains stable emulsions during the short period of time in which the droplets are transported to the plasma torch.
- MultiNeb is versatile since it works properly as a conventional nebulizer on "Single Mode" by feeding just one of the inlets or on "Multiple Mode" by simultaneously feeding the different inlets.
- Less manual operations are needed, reducing analysis time, reproducibility issues and potential errors or accidents.
- The samples/standards and reagents volumes are reduced significantly.
- Some mixing processes are intensified due to high speeds and pressures at the mixing zone inside the nebulizer.
- The nebulizing gas flow is not divided between different exit orifices, so the working conditions are equal to the optimum working conditions of the spectrometer. In this way, the quantity of liquid input capillaries can be adjusted to the specific application of the nebulizer.

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- MultiNeb is built on the right dimensions to allow the connection to any commercial spray chamber (for all manufacturers and models of spectrometers based on plasma).

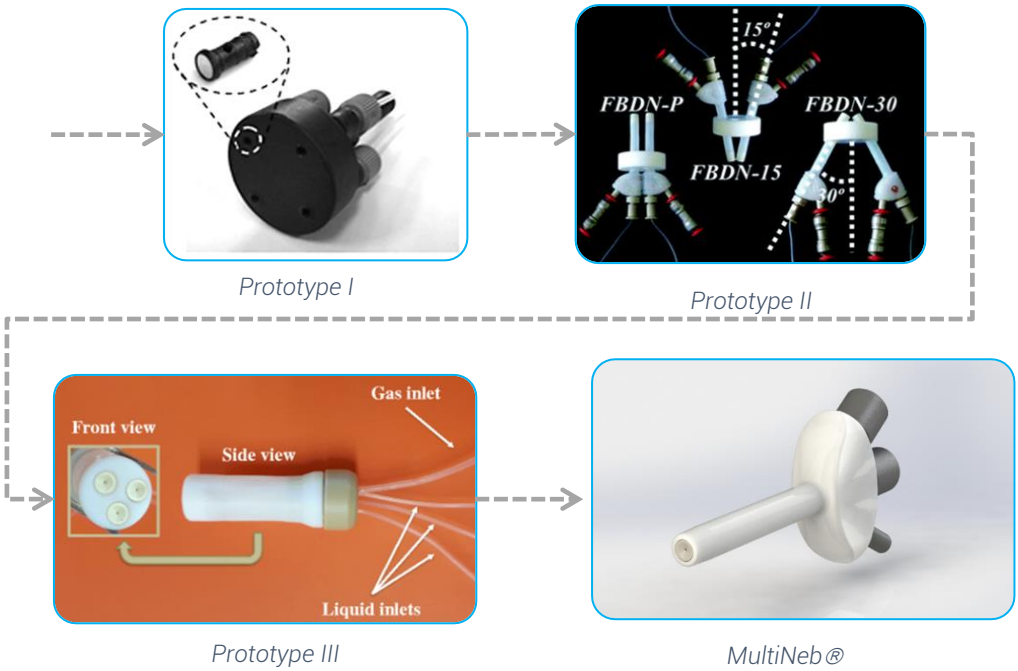
Before the invention of the MultiNeb®, many alternatives have been tried by scientist and some equipment manufacturing companies. We found evidence of at least three main different types of multi-nebulizer systems with different levels of success:

Type 1) Systems that incorporate several spray chambers, each one being equipped with a conventional nebulizer. These systems are complex to handle and a low mixing efficiency is achieved. There are not commercial products of this type in the market.

Type 2) Systems that incorporate several liquid introduction inlets using one or several conventional nebulizers in a modified spray chamber. These systems are easier to handle in comparison with the preceding ones. However, the mixing efficiency critically depends on the nebulizer position in the spray chamber.

Type 3) Systems that incorporate one nebulizer with several aerosol outlet orifices and several independent liquid input capillaries (multinebulizers). In these systems, the greater proximity between the aerosol outlet orifices could increase the mixing efficiency. There are not commercial application products currently available.

MultiNeb could be understood as the evolution of the Type 3, through the following sequence of prototypes:



Prototype I *Prototype II*

Prototype III *MultiNeb®*

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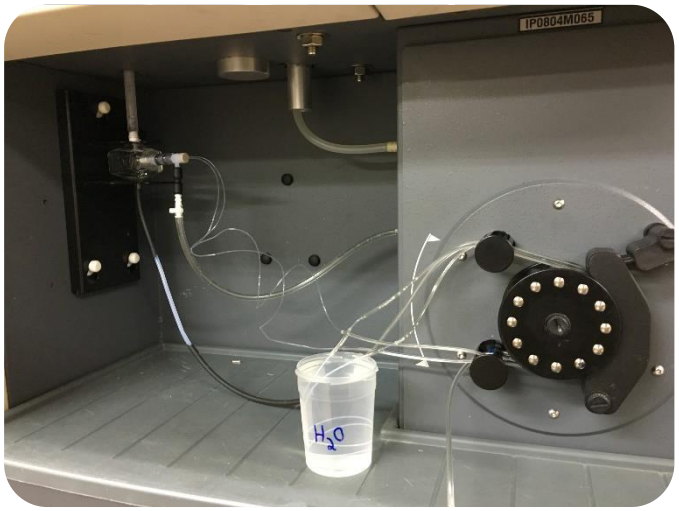
A common unresolved issue with the abovementioned Type 1, 2 and 3 systems, until the invention of MultiNeb, is the fact that the total nebulizing gas flow rate is divided among the various nebulizers or exit orifices of the nebulizer. Therefore, they usually work under non-optimal nebulizing gas flow rate conditions. This fact adversely impacts on the aerosol transport to the plasma. Moreover, most of these systems have additional problems, such as frequent clogging and memory effects what make them undesirable from an analytical point of view because they do not allow the analysis of complex real samples (e.g., sea water, wastewater, etc.).

On the opposite, MultiNeb® enables the sample preparation in aerosol phase, having a low blocking risk, low memory effect, high chemical and mechanical robustness, optimum use of nebulizing gas, easy to handle and having a high analyte conveyance efficiency allowing universal application to any complex real samples.



The Makers

Ingeniatrics is a privately owned company founded in 2001 that develops innovative technologies on microfluidics fundamentals. It currently operates on two fields of application: Industrial Innovations and Nebulizer Manufacturing for Chemical Analysis apparatus. Throughout the recent years Ingeniatrics has developed innovative microfluidics applications in the Analytical Chemistry field.

MultiNeb® has been developed in partnership with the University of Alicante in Spain.

The University of Alicante (UA) houses 231 research groups in Social and Legal Sciences, Experimental Sciences, Technological Sciences, Human Sciences, Education and Health Sciences and 18 Research Institutes. Thus, the UA employs over 3.500 researchers/professors and has a

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complex management/administration structure of 1.300 people, which involves an annual budget of 200 million Euros.

The school of Chemistry is among the world top 300 institutions on chemistry area (2017 Academic Ranking of World Universities, http://www.shanghairanking.com/Shanghairanking-Subject-Rankings/chemistry.html.

The research team led by Dr. Canals has a long experience on elemental analysis by means of spectrochemical techniques and development of faster and environmentally respectful sample preparation methodologies http://web.ua.es/en/qace/presentation.html. One of the main goals is focused on the characterization and development of new and more efficient nebulizers for spectrochemical analysis.

Publications

1 Determination of As, Se, and Hg in fuel samples by in-chamber chemical vapor generation ICP OES using a Flow Blurring® multinebulizer.
M. Garcia, M. A. Aguirre and A. Canals, Anal. Bioanal. Chem, Accepted paper. DOI: https://doi.org/10.1007/s00216-017-0484-6.

2 Vortex-assisted dispersive liquid–liquid microextraction for the determination of molybdenum in plants by inductively coupled plasma optical emission spectrometry.
J. A. V. A. Barros, M. A. Aguirre, N. Kovachev, A. Canals and J. A. Nóbrega, Anal. Methods, 2016, 8, 810-815.

3 Compensation of inorganic acid interferences in ICP-OES and ICP-MS using a Flow Blurring® multinebulizer.
M. A. Aguirre, L. L. Fialho, J. A. Nóbrega, M. Hidalgo and A. Canals, J. Anal. At. Spectrom, 2014, 29, 1218-1227.

4 Analysis of metals and phosphorus in biodiesel B100 from different feedstock using a Flow Blurring® multinebulizer in inductively coupled plasma-optical emission spectrometry.
F. D. Avila Orozco, N. Kovachev, M. A. Aguirre, C. E. Domini, B. S. Fernández Band and A. Canals, Anal. Chim. Acta, 2014, 827, 15-21.

5 Aerosol generation of As and Se hydrides using a new Flow Blurring® multiple nebulizer for sample introduction in Inductively Coupled Plasma Optical Emission Spectrometry.
C. D. Pereira, M. A. Aguirre, J. A. Nóbrega, M. Hidalgo and A. Canals, Microchem. J, 2014, 112, 82-86.

6 Correction of matrix effects for As and Se in ICP OES using a Flow Blurring® multiple nebulizer.
C. D. Pereira, M. A. Aguirre, J. A. Nóbrega, M. Hidalgo and A. Canals, J. Anal. At. Spectrom., 2012, 27, 2132-2137.

7 Analysis of biodiesel and oil samples by on-line calibration using a Flow Blurring® multinebulizer in ICP OES without oxygen addition.
M. A. Aguirre, N. Kovachev , M. Hidalgo and A. Canals, J. Anal. At. Spectrom., 2012, 27, 2102-2110.

8 Compensation for matrix effects on ICP-OES by on-line calibration methods using a new multi-nebulizer based of Flow Blurring® technology.
M. A. Aguirre, N. Kovachev, B. Almagro, M. Hidalgo and A. Canals, J. Anal. At. Spectrom., 2010, 25, 1724-1732.

9 Development and characterization of a Flow Focusing multi nebulization system for sample introduction in ICP-based spectrometric techniques.
N. Kovachev, B. Almagro, M. A. Aguirre, M. Hidalgo, A. M. Gañan-Calvo and A. Canals, J. Anal. At. Spectrom., 2009, 24, 1213–1221.

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