



MultiNeb® - simultaneous sample introduction nebulizer

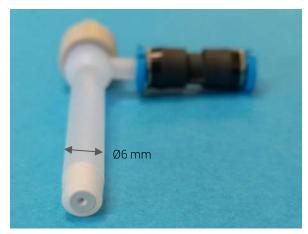


1. What is MultiNeb®?









- ❖ MultiNeb® is a very unique ICP OES/MS pneumatic nebulizer
- ❖ It allows the simultaneous atomization of two fluids (even immiscible ones)
- It provides sensitivity and precision as high as other high efficiency nebulizers like OneNeb® or SeaSpray®
- It is fully compatible with standard spray chambers. No hardware modifications are required.
- ❖ It has been developed and it is manufactured by Ingeniatrics, the creator and manufacturer of Agilent's OneNeb series, in collaboration with the University of Alicante.



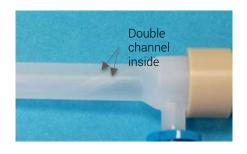
2. How does it work?

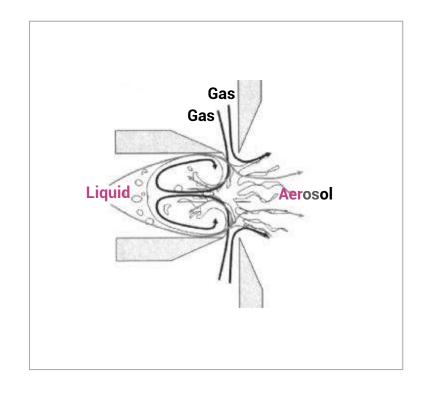


Microfluidics

Ingeniatrics' technology is based on Aerodynamics and Microfluidics and more specifically on the art of breaking a liquid stream into a specific targeted aerosol just by using a gas stream as the only breaking force.

MultiNeb® generates an aerosol suitable for ICP sample introduction just similar to what a regular nebulizer will do but in this specific case it will do it out of two liquid streams instead of just one, and still using the same Argon gas flowrate than a regular 1-channel nebulizer would use.







3. Examples of use



Examples of use	Main feature
1. Organic Matrix introduction	minimizes torch's cleaning
2. Internal standard calibration	minimizes solution preparations
3. Standard addition calibration	minimizes solution preparations
4. Standard dilution analysis	minimizes solution preparations
5. Chemical vapor generation	replaces complex set-up systems (e.g., FIA system, MSIS, etc)
6. Isotope dilution analysis	minimizes solution preparations

Examples of use

These are just some of the potential applications of the MultiNeb®

All of them are based on the competitive advantage of MultiNeb® allowing the simultaneous nebulization of two specific solutions

New applications are expected to be discovered since the "necessary tool" is now available





Example 1





Example 1. Organic Matrix Introduction

Fuel sample

Introducing organic matrix samples into ICP equipment using a regular 1 channel nebulizer will unavoidably produce Carbon deposits at the tip of the Injector tube and on the plasma torch tulip.

This will make necessary to stop the testing from time to time and remove the torch for cleaning, generating a great waste of resources: testing downtime + cleaning cost.

By simultaneously introducing the organic sample in one channel of the MultiNeb® and an aqueous solution on the other we introduce enough oxygen as to achieve a complete organic sample combustion, reducing carbon deposits. It allows a non-stop and cost effective testing operation.



Real pictures of the torch (a) initial state, (b) after 1 hour of continuous 1 μg g⁻¹ fuel sample introduction with multinebulizer-based system and (c) after 1 hour of continuous 1 μg g⁻¹ fuel sample introduction without multinebulizer-based system¹

1 M. García, M.A. Aguirre and A. Canals, Anal. Bioanal. Chem., 2017, 409, 5481-5490.





Aqueous blank

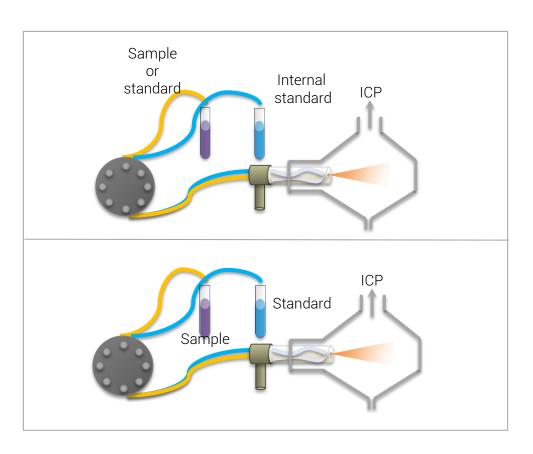


Examples 2, 3 and 4





Example 2 & 3. Online Internal Standard Calibrations and Online Standard Addition Calibrations



For on-line internal standard calibration:

One channel is used to sequentially introduce calibration standards and samples, and the other one is used to continuously nebulize the internal standard solution.

For on-line standard addition calibration:

The sample is continuously introduced through one channel, while calibration standards are sequentially nebulized through the other one.



Results for Online Standard Addition Calibration

This table shows the results obtained in the analysis of a 100% biodiesel reference material by on-line standard addition calibration with MultiNeb.

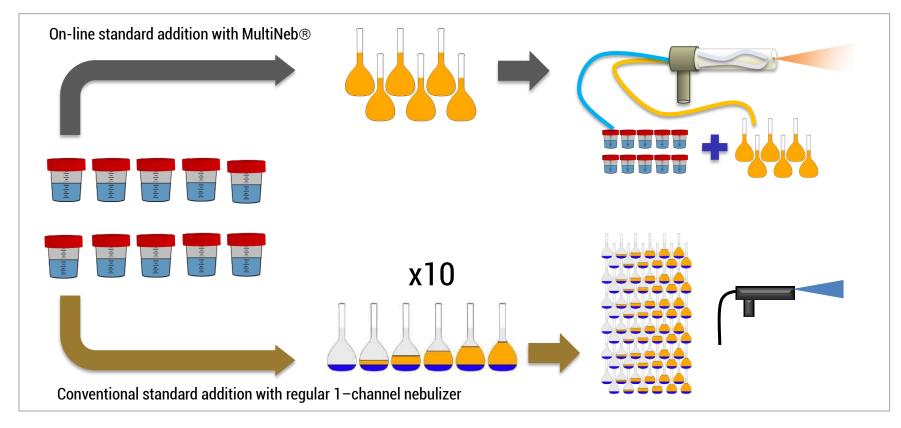
In this analysis, the 100% biodiesel reference material was directly introduced (i.e., without dilution) through one channel, while aqueous calibration standards were sequentially nebulized through the other one.



		On-line standard addition		
Analyte	Found value ^a	Recovery (%)	valueª	
К	9.6±0.6	96±6	10.00±0.10	
Na	9.6±0.6	96±6	10.00±0.10	
Ca	10.0±0.5	101±5	9.90±0.10	
Mg	10.3±0.4	104±4	9.90±0.10	

^aIn μg Kg⁻¹ ± confidence interval 95%.





Using MultiNeb® for online calibration methodologies reduces the amount of time, samples and reagents to be used.

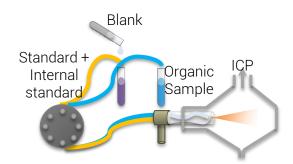
For instance, the analysis of 10 samples using 6 different calibration standards per sample on a conventional standard addition method would require 60 solutions to be prepared, but if we use the MultiNeb® On-line standard addition method we can avoid that. We would just need to directly use the 6 different standards for each of our 10 samples.

We can change the rules towards a much more cost effective operation.



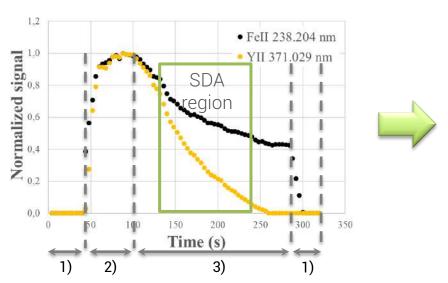


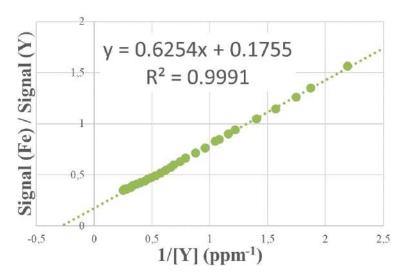
Example 4. On-line standard dilution analysis (SDA)



Three steps are required to apply the on-line SDA methodology using the MultiNeb® nebulizer: 1) the organic and the aqueous calibration blanks are simultaneously nebulized in order to rinse the system. 2) the organic sample and the aqueous calibration standard (+ internal standard, IS) are simultaneously introduced, achieving a stable maximum plateau. 3) the aqueous calibration blank is added to the aqueous calibration standard, which results in the continuous dilution of the aqueous calibration standard, that is continuously nebulized by the nebulizer.

Finally, when a stable minimum is obtained, calibration blanks are introduced again (step 1) to rinse the system and ready it for the next run. Both analyte and internal standard signals are continuously and simultaneously measured during all the analysis. Analyte concentration is obtained from linear regression of signal (analyte) / signal (IS) ratio vs 1/[IS].







Results for Online Standard dilution analysis (SDA)

	Unused motor lubricant oil			
	Found value	Found value	Recovery	
All	SA (μg g⁻¹)	On-line SDA (μg g ⁻¹)	(%)	
Cd	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	
Cr	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	
Cu	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	
Fe	0.44 ± 0.04	0.452 ± 0.015	103 ± 10	
Mn	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	
Ni 🚺 📗	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	
Pb	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	

		Used lubricant oil (1 year/20,000 km)			
Analyte	/	Found value	Found value	Recovery	
Analyte	1	SA (μg g ⁻¹)	On-line SDA (μg g ⁻¹)	(%)	
Cd	Ų.	<l0q< th=""><th><l0q< th=""><th>-</th></l0q<></th></l0q<>	<l0q< th=""><th>-</th></l0q<>	-	
Cr 🚺		0.38 ± 0.02	0.36 ± 0.02	95 ± 7	
Cu	M.	2.40 ± 0.15	2.56 ± 0.17	107 ± 10	
Fe		17.5 ± 0.6	16.9± 0.3	97 ± 4	
Mn	Ad	0.25 ± 0.02	0.23 ± 0.02	92 ± 10	
Ni 🅖		<l0q< th=""><th><loq< th=""><th>-</th></loq<></th></l0q<>	<loq< th=""><th>-</th></loq<>	-	
Pb 🖊		1.05 ± 0.07	1.08 ± 0.03	103 ± 7	

These two tables show the results obtained in the analysis of unused and used lubricating oil by online SDA with MultiNeb® and by conventional standard addition calibration (SA).

The results obtained with both methodologies are in good agreement.

The found concentration values for unused oil are below the LOQ, with the exception of iron. The test shows iron concentration values higher for used lubricating oil than for unused oil which indicates possible wear of a number of components such as piston rings, ball/roller bearings or gears inside the engine. This could be a good methodology for engine wearing analysis and SDA (with MultiNeb) that can be performed continuously.

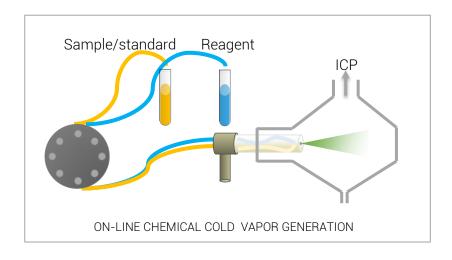


Example 5





Example 5. On-line chemical vapor generation. In-place



For on-line hydride generation and on-line cold vapor generation:

e.g. One channel is used to sequentially introduce calibration standards and samples, and the other one is used to continuously nebulize the sodium borohydride solution.

$$As^{3+} + 5BH_4^- + 2H^+ + 15H_2O \rightarrow AsH_3 + 5H_3BO_3 + 17H_2$$

$$Hg^{2+} + 4BH_4^- + 2H^+ + 12H_2O \rightarrow Hg^0 + 4H_3BO_3 + 15H_2$$

For on-line **reduction/oxidation** vapor generation:

One channel is used to sequentially introduce calibration standards and samples, and the other one is used to continuously aspirate the reductant or oxidant.

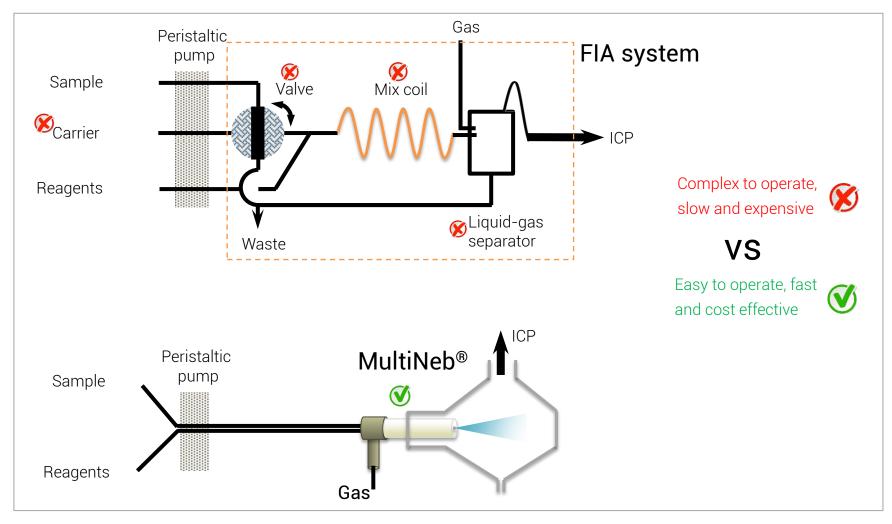
$$2 IO_3^- + 3 BH_4^- + 5 H^+ + 3 H_2O \rightarrow I_2 + 3 H_3BO_3 + 7 H_2$$
 (reduction)

$$2 I^{-} + H_{2}O_{2} + 2 H^{+} \rightarrow I_{2} + 2 H_{2}O$$
 (oxidation)









MultiNeb® dramatically simplifies the generation and use of chemical vapors in ICP testing

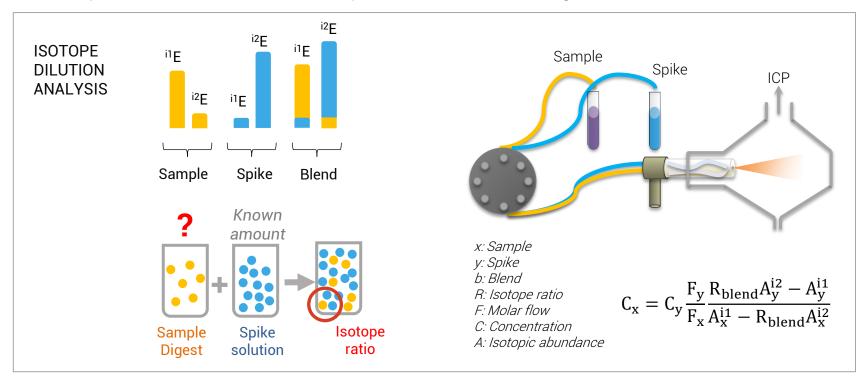


Example 6





Example 6. On-line isotope dilution analysis



One channel is used to introduce the sample, while the other one is used to nebulize the spike. Adding an isotopically altered standard (i.e., spike) to the sample changes the natural isotopic composition of the analyte. By measuring the resulting isotopic composition (i.e., isotope ratio) it is possible to calculate the concentration of the analyte in the sample.

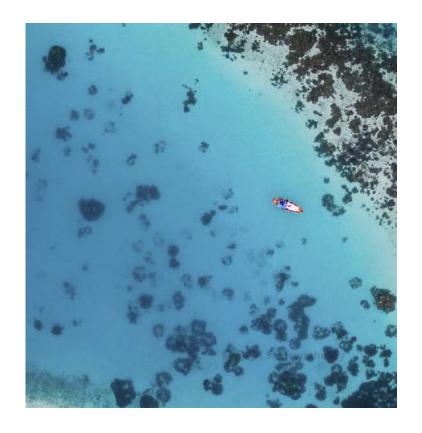


On-line isotope dilution

	Isotope Dilu (batch)	tion	On-line Isotope Dilution using MultiNeb®		
Analyt e	Mass fraction (ng g ⁻¹)	RSD (%)	Mass fraction (ng g ⁻¹)	RSD (%)	Recovery (%)
Cu	2281 ± 76	3.3	2155 ± 81	3.8	94 ± 5
Ni	4856 ± 123	2.5	4538 ± 112	2.5	93 ± 3

 $^{\mathrm{a}}$ Mass fraction value \pm expanded uncertainty ($\mathrm{U_{c}}$ = $\mathrm{k\cdot u_{c}}$; $\mathrm{k=2}$; 95% level of confidence). Combined standard uncertainties were obtained by propagating individual uncertainty components

As can be observed, the concentrations obtained with the evaluated methodologies are in good agreement, achieving recovery values close to 100 %.







4. Conclusions



Conclusions

- The MultiNeb® has been presented as an innovative and powerful device for elemental spectrochemical analysis.
- It is a compact, versatile, inert, durable, efficient, easy to handle and low-maintenance operation nebulizer.
- It has been proved that the use of aqueous calibration standards overcomes practically all drawbacks in the analysis of organic matrices.
- The main advantages using on-line calibration methodologies by the new MultiNeb® are: handling simplicity, significant reduction in the total analysis time, reagent and sample consumption and number of solutions to be prepared.
- It simplifies the experimental set-up on chemical vapor generation, increasing the sensitivity of ICP-based techniques.
- Finally, on-line isotope dilution provide excellent results in a simple way, avoiding the tedious and time consuming conventional isotope dilution analytical procedures.





P.I. Parque Plata.

C/ Camino Mozárabe, 41

41900 Camas, Seville (Spain)

T.(+34)954081214

E.info@ingeniatrics.com





