ANALYTICA специализирани семинари

20 TEAM

НАУЧНИ ИЗСЛЕДВАНИЯ 2012

Green
Analytical
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GAMA проект – БЪЛГАРО-ИСПАНСКО сътрудничество за развитие на зелени аналитични методи

ХИМИЧЕСКИ ФАКУЛТЕТ

В. Кметов, В. Стефанова, Д. Георгиева, К. Симитчиев, С. Начкова, С. Тенев, Е. Върбанова, Л. Георгиева, Л. Попов

A. Canals, Н. Ковачев, I. Roman, M. Angel

50 ГОДИНИ
ПЛОВДИВСКИ УНИВЕРСИТЕТ
•ПАИСИЙ ХИЛЕНДАРСКИ•

Prevention

Atom Economy

Less Hazardous Chemical

Designing Safer Chemicals

Safer Solvents

Energy Efficiency

Renewable Feed stocks

Reduce Derivatives

Catalysis

Design for Degradation

Real-time analysis

Accident Prevention

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ФОНД НАУЧНИ ИЗСЛЕДВАНИЯ

Министерство на образованието, младежта и науката

Национален Фонд „Научни изследвания"

ИНТЕГРИРАНИ НАУЧНИ ЦЕНТРОВЕ В УНИВЕРСИТЕТИТЕ

УНИВЕРСИТЕТСКИ ЦЕНТЪР ЗА РАЗВИТИЕ НА ЕКОЛОГИЧНО ЦЕЛЕСЪОБРАЗНИ МЕТОДИ ЗА СЛЕДОВИ АНАЛИЗ НА ОБЕКТИ ОТ ОКОЛНАТА СРЕДА

АУ

УХТ

MW | SPE | CPE | ASDI | MNPs

ANALYTICA специализирани семинари

НАУЧНИ ИЗСЛЕДВАНИЯ 2012

7700 ICP-MS

Agilent Technologies

Asian Chemistry Letters Vol. 15, No 1 (2011)

Microwave-Assisted Cloud Point Extraction in Combination with Air Segmented Discrete Sample Introduction as a Green Method for Flame Atomic Absorption Analysis of Zn, Cu, Pb and Cd*

Kiril Simitchiev [a,1], Elena Harizanova [b], Violeta Stefanova [a], Veselin Kmetov [a], Nikolay Kovachev [c], Antonio Canals [c]

[a] *Department of Analytical Chemistry and Computer Chemistry, University of Plovdiv "Paisii Hilendarski", 24 Tzar Assen Str., 4000 Plovdiv, Bulgaria*

[b] *Faculty of Chemistry, University of Sofia "St. Kliment Ohridski", 1 James Bourchier Blvd., 1164 Sofia, Bulgaria*

[c] *Department of Analytical Chemistry and Food Science, University of Alicante, P.O. Box 99, 03080 Alicante, Spain*

[1] *Corresponding author: T*

*This article is dedicated to

The utilization of non-hazard
chemistry has been applied for
to flame atomic absorption dete
(MW-CPE) using the non-toxic
parameters affecting the CPE w
optimized, being: i) pH of th
Triton X-100, 0.5 % and iv) 20
HNO_3 up to 2.5 g. Micro-sam
introduction (ASDI). The comb
Cd and Pb compared to the dire
The developed procedure was
method was validated by compa
ones obtained by ICP-MS analy
ware below the detection limit c

ПРИЛОЖЕНИЕ НА ХЕМОМЕТРИЧНИ ПОДХОДИ ЗА ИНТЕРПРЕТАЦИЯ НА ДАННИ ОТ ICP-MS АНАЛИЗ НА ПОВЪРХНОСТНИ ВОДИ

Ст. Начкова[а]*, В. Стефанова[а], К. Симитчиев[а], Д. Георгиева[а], В. Кметов[а], Ир. Караджова[б].

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Резюме

Обект на изследването са повърхностни води от територията на България, от гледна точка на елементния им състав. Изотопите в диапазона 7- 238 amu бяха измерени с ICP-MS модел Agilent 7700x в 120 проби води от пунктове за мониторинг, регламентирани в законодателството. За обработка на информацията и решаване на класификациони задачи, върху данните от анализа бяха приложени хемометрични методи – CA, PCA с FA, DA. Бяха потърсени елементни маркери за обособените региони с цел идентификация на произход на неизвестни водни проби.

Colloquium Spectroscopicum Internationale XXXVII

TU32

Comparison of ICP-MS and colorimetric determination of total and extractable phosphorous in soils

Krasimir Ivanov[a], Violeta Stefanova[b], Milena Petkova[a], Veselin Kmetov[b], Penka Zaprjanova[c], Deyana Georgieva[b] and Violina Angelova[a]
[a]*University of Agriculture, Dept. of Chemistry, Plovdiv, Bulgaria,*
[b]Plovdiv University "Paisii Hilendarski", Dept. of Anal. Chemistry, Plovdiv, Bulgaria,
[c]*Institute of Tobacco and Toba*
E-mail: kiva

50 ГОДИНИ ПЛОВДИВСКИ УНИВЕРСИТЕТ «ПАИСИЙ ХИЛЕНДАРСКИ»

9th Chemistry Conference and Workshop BioSupport
Plovdiv 14-16 October 2011

BioSupport

P-11

SIMULTANEOUS DETERMINATION OF P, S, K, OTHER ESSENTIAL AND TRACE ELEMENTS IN PLANT AND SOIL SAMPLES BY ICP-MS

ova[1], D. Georgieva[1], Kr. Ivanov[2], S. Petrova[3], L. Yurukova[4]
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ail: stefanova@uni-plovdiv.bg
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ersity of Plovdiv "Paisii Hilendarski", 24 Tzar Assen, Str., 4000 Plovdiv, Bulgaria
and Ecosystems Research, Bulgarian Academy of ria, Acad. G. Bonchev Str., Block 23, Sofia 1113, Bulgaria

DETERMINATION OF TRACE ELEMENTS IN COMMERCIAL BOTTELED BULGARIAN MINERAL WATERS BY MEANS OF ICP-MS AND TXRF

A. Detcheva[1], E. Ivanova[1], J. Harizanov[1], R. Georgieva[1], V. Stefanova[2], S. Nachkova[2], V. Kmetov[2]
[1] Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences, Acad. G. Bonchev Str. Bl. 11, 1113 Sofia, Bulgaria
[2] Plovdiv University "Paisii Hilendarski", Tsar Assen Str. 24, 4000 Plovdiv, Bulgaria

Universitat d'Alacant
Universidad de Alicante

Headspace single drop microextraction of organotin compounds followed by thermal desorption GCMS

L. Georgieva[1], I.P. Román[2], V. Stefanova[1], V. Kmetov[1], A. Canals[2]

[1] *Department of Analytical Chemistry and Computer Chemistry, University of Plovdiv "Paisii Hilendarski", 24 Tzar Assen, Str., 4000 Plovdiv, Bulgaria.*

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Introduction

In the past 30 years organotin compounds (OTs) have been widely used (e.g. Butyltin-BT and Phenyltin-PhT) as active ...

Conclusions

- A method for simultaneous determination of six organotin compounds was developed
- It was proved that the extraction with 1-octylimidazolium hexafluorophosphate gives greater yield than the others two ILs studied.
- Due to the special device for thermal desorption, created method allows the use of ILs , which are considered as environmentally friendly compounds
- The volume of IL used in the extraction procedure is 5µl, which is another conformity with the Green Chemistry principles

The aims

To develope green method for simultaneous determination of six organotin compounds: monobutyltin (MBT, dibutyltin DBT, tributyltin TBT, monophenyltin MPT, diphenyltin DPT, triphenyltin TPT).

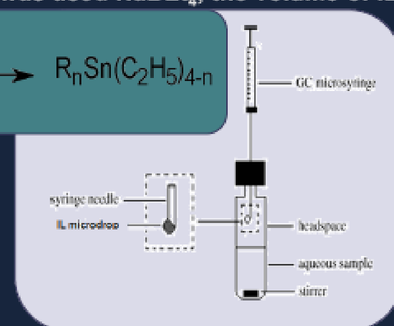
To evaluate the applicability of three different Ionic Liquids (ILs) as a collector phase:

3-Methyl-1-octylimidazolium hexafluorophosphate (Octyl); 1-Hexyl-3-methylimidazolium chloride (Hexyl) and 1-Butyl-3-methylimidazolium hexafluorophosphate (Butyl).

Experimental

Headspace single drop microextraction with in-situ derivatization followed by thermal desorption gas chromatography mass spectrometry [2] have been developed for simultaneous speciation analysis of six OTs, including butyl- and phenyltin compounds. ... n reagent was used $NaBEt_4$, the volume of IL was 5µl.

$\xrightarrow{NaBEt_4} R_nSn(C_2H_5)_{4-n}$



The headspace microextraction with in-situ derivatization was held at pH 5

...rption ... 240°... down ... flash heating 15°C/s up to 250°C

Temperature program for gas chromatographic separation: 50°C(1min),10°C/min, 300°C(4min)

A two-tubes concentrically disposed system made up of a Gerstel thermal desorption glass tube (187mm length, 4mmI.D., 6mm O.D.), a laboratory-cut glass Pyrex tube (20mm length, 3mm I.D., 4mm O.D.) from Corning Incorporated (Corning, NY, USA) and washed glass wool from Panreac (Barcelona, Spain) were used, which enabled the desorption of the compounds from the IL droplet, while preventing the IL from entering the GC system.

VORTEX OR ULTRASOUND ASSISTED IN-SITU DERIVATIZATION AND LIQUID-LIQUID MICROEXTRACTION OF ORGANOTIN COMPOUNDS, FOLLOWED BY GCMS ANALYSIS

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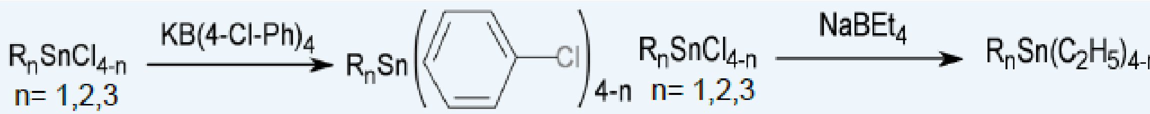
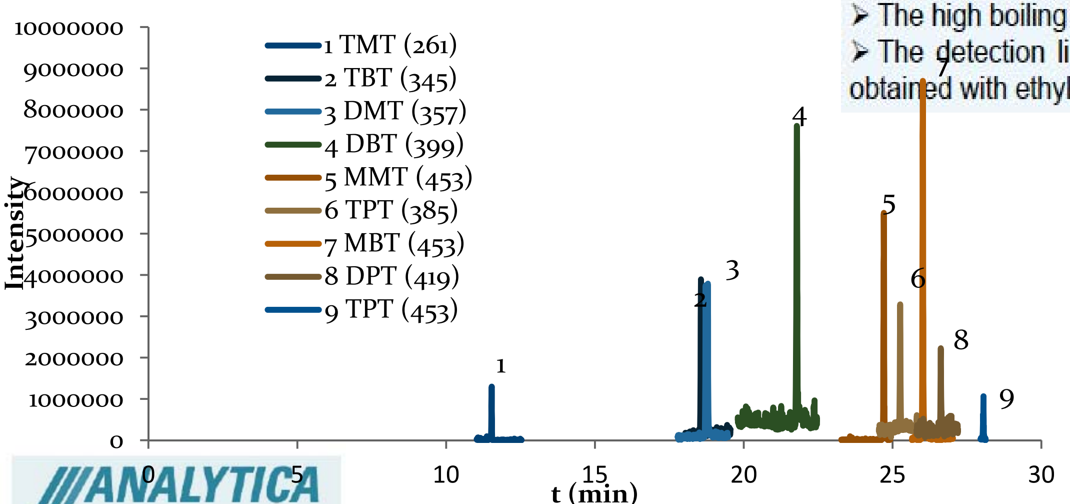


Potassium tetrakis(4-chlorophenyl) borate was selected as derivatization reagent because both volatile and semivolatile OTs can be analized simultaneously, without the need of two different extraction techniques. The obtained derivative compounds have not been reported before so their spectra were not included in the library of GC-MS instrument up to now. Their spectra were elucidated.

Conclusions

- Potassium tetrakis(4-chlorophenyl) borate allows both volatile and semivolatile OTs to be analyzed simultaneously, without the need of different extraction techniques.
- The high boiling points of chloro-phenyl-derivatives of all the OTs studied allow their determination.
- The detection limit obtained with chlorophenylation have been improved compared to the ones obtained with ethylation due to the higher bond-dissociation energy of Sn-aryl than those of Sn-alkyl.



P-5

DEVELOPMENT OF AN ULTRASOUND-ASSISTED LIQUID-LIQUID MICROEXTRACTION PROCEDURE FOR STEROID SEX HORMONES BEFORE LIQUID CHROMATOGRAPHIC ANALYSIS

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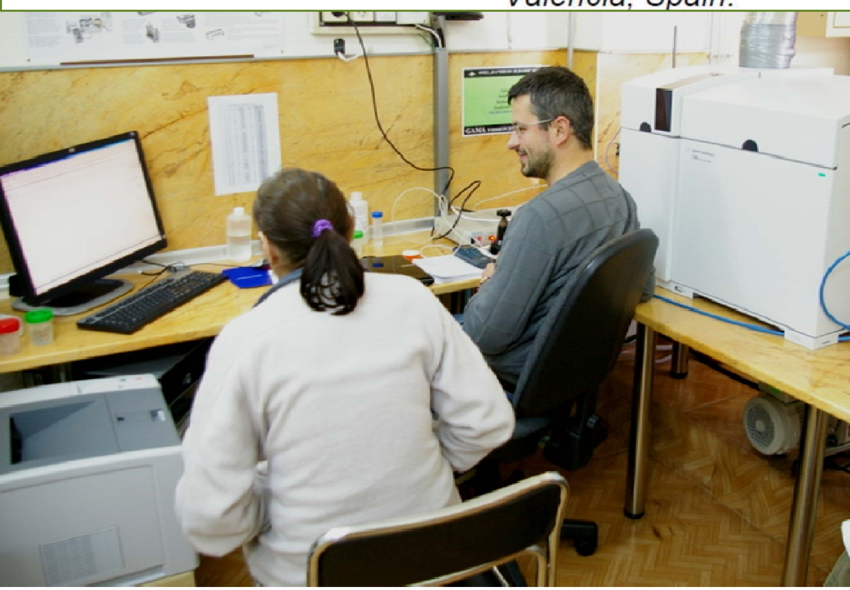

P-8

ELEMENTAL SPECIATION BY CAPILLARY ELECTROPHORESIS WITH INDUCTIVELY COUPLED PLASMA SPECTROMETRY: ENHANCEMENT BY FLOW FOCUSING® NEBULIZATION

N. Kovachev[a], M.A. Aguirre[a], M. Hidalgo[a], K. Simitchiev[b], V. Stefanova[b], V. Kmetov[b], A. Canals[a]

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JAAS
Journal of Analytical Atomic Spectrometry

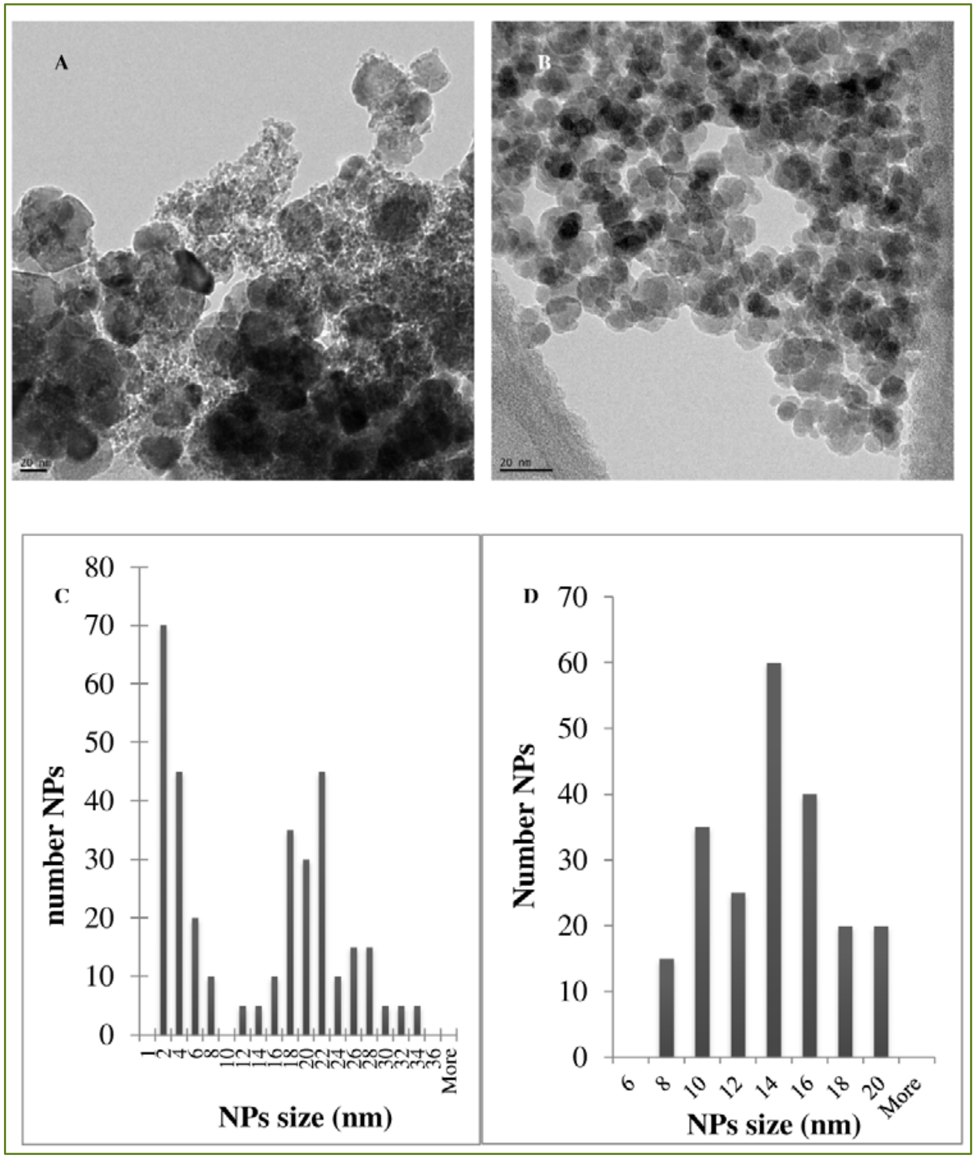
Manganese ferrite nanoparticles as a new sorbent for magnetic solid phase extraction of trace metals - APDC complexes followed by inductively coupled plasma mass spectrometry analysis

Violeta Stefanova[a], Deyana Georgieva*[a], Veselin Kmetov[a], Ivan Roman[b] and Antonio Canals[b]

Received (in XXX, XXX) Xth XXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX
DOI: 10.1039/b000000x

The applicability of $MnFe_2O_4$ nanoparticles as a new sorbent for group pre-concentration of V, Co, Ni, Cu, Zn, As, Se, Cd and Pb was investigated and compared with magnetite nanoparticles. A solid phase extraction (SPE) of target analytes based on sorption of their hydrophobic complexes with ammonium pyrrolidine dithiocarbamate (APDC) on the surface of non-modified magnetic nanoparticles (NPs) was optimized. Magnetic NPs with retained metal complexes were easily separated from the bulk solution by permanent magnet for 5 min. Analytes restoration in final solution was accomplished by heating with 0.5 mL of 7 mol L^{-1} nitric acid. The obtained solutions were suitable for continuous nebulization in ICP-MS. Matrix effects (spectral and non-spectral) for urine analysis were studied and adequate calibration strategies were suggested. Under optimized conditions the magnetic assisted SPE procedure enables enrichment of target analytes by factors between 7.4 – 10, with a linear dynamic range 1 – 100 µg L^{-1} for V, Co, Ni, Cd, Pb and 10-1000 µg L^{-1} for Zn, As, Se. The method detection limits (MLOD) of proposed SPE were improved by factor up to 20 compared to the direct analysis of diluted urine. The accuracy of magnetic NPs-SPE-ICP-MS method was evaluated analysing urine certified reference material Seronorm™ Trace Elements Urine 201205. For correct determination of As and Se in urine, a preliminary microwave sample treatment with a mixture HNO_3+H_2O_2 was needed, but it led to worsening of MLOD. The developed method was successfully applied for analysis of human urine samples.

Синтез на $MnFe_2O_4$магнитни нано-частички

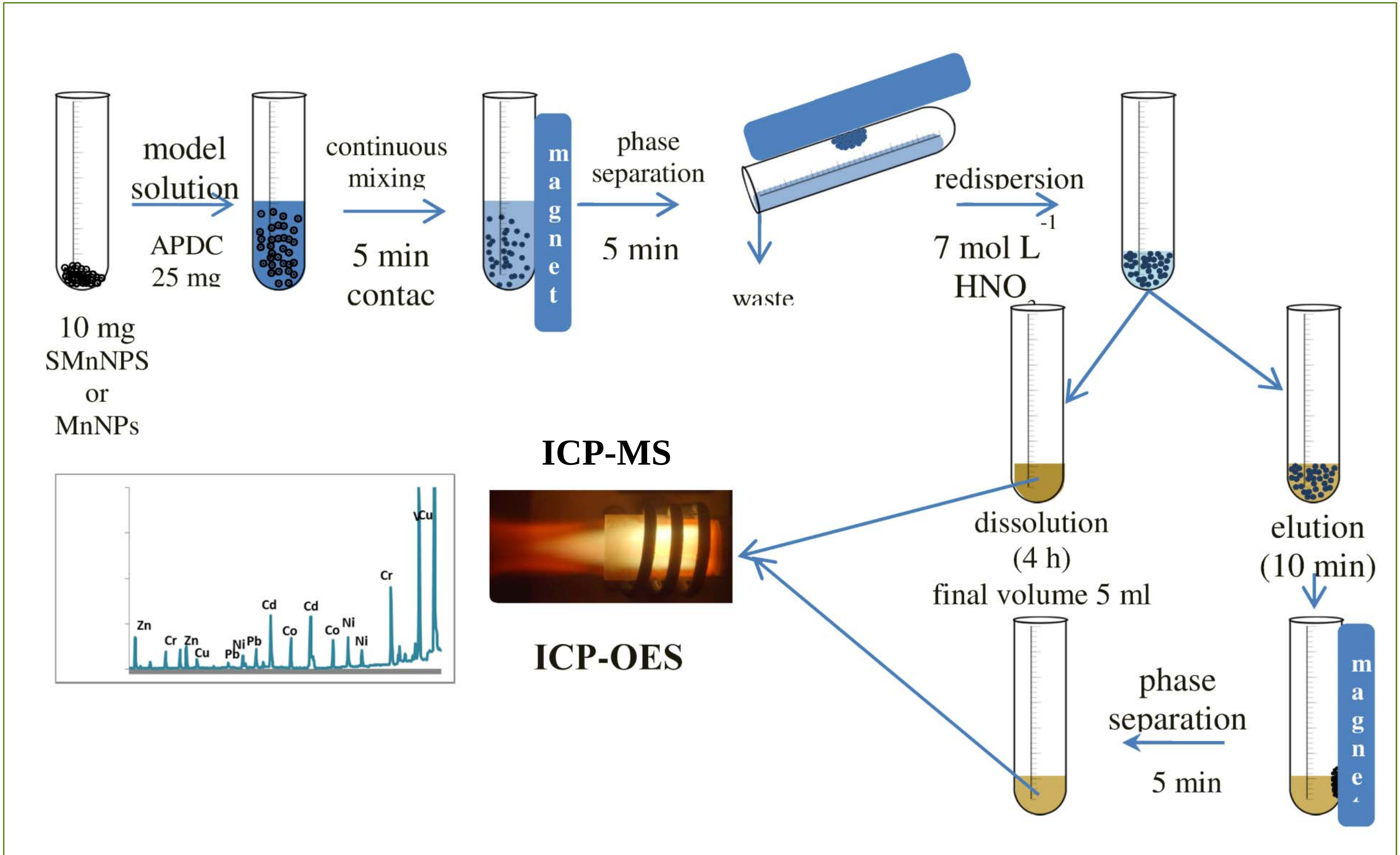


Съутаяване на смес от Me^{2+} йони (Me^{2+} = Mn^{2+} или Fe^{2+}) и Fe^{3+} в молно отношение = 1:2 (0.017 mol L^{-1} Me^{2+}: 0.034 mol L^{-1} Fe^{3+} в основна среда - NaOH внесена “наведнъж” в приготвения разтвор при 30^0C и загряване до 80^0C за 3 часа.

Магнитните наночастици се сепарират чрез постоянен магнит. Промиват се вода и с етанол ~50 mL .

HR-TEM снимки – за $MnFe_2O_4$ NPs показват две фракции ~2 nm и ~20 nm а тези от Fe_3O_4 – една ~14 nm

Твърдофазна микро-екстракция с МНЧ



Валидиране

Table 4. Validation of magnetic NPs-SPE-ICP-MS by analysis of urine certified reference materials **Seronorm™201205**

Element [isotope measured]	Assigned value µg L^{-1} ± U	Magnetic NPs-SPE treated with HNO_3 µg L^{-1} ± U[a]	Magnetic NPs-SPE treated with $HNO_3:H_2O_2$ (3:1) µg L^{-1} ± U[a]	Direct analysis µg L^{-1} ± U[a]	LOD µg L^{-1}
V [51]	25.2 ± 1.4	22 ± 1.8	25 ± 2.4		
Co [59]	10.0 ± 0.6	11.1 ± 0.9	8 ± 1.8		
Ni [60]	50.4 ± 3.2	52.1 ± 2.6	51 ± 3.3		
Cu [65]	78 ± 8[b]	70 ± 6	72 ± 7		
Zn [66]	1168 ± 92	1230 ± 30	1040 ± 33		
As [75]	142 ± 6	34 ± 1.1	121 ± 16		
Se [82]	58.6 ± 3.1	52 ± 5	59 ± 10		
Cd [111]	4.6 ± 0.4	5.1 ± 1	4.7 ± 2		
Pb [208]	40.3 ± 2.6	45 ± 4	44 ± 4		

[a] *expanded uncertainty is calculated using k=2*

[b] *Cu content in Seronorm™201205 is given as analyti corresponding SD*

[c] *measurements were performed with He as collision g*

Table 5. Concentration of the elements in µg L^{-1} with corresponding expanded uncertainty U (k=2), obtained by magnetic NPs-SPE-ICP-MS analysis of real urine sample, using $MnFe_2O_4$.

Element [isotope measured]	Urine sample µg L^{-1} ± U	Urine + spike added µg L^{-1}	Urine + spike measured µg L^{-1} ± U	Addition recovery %
V [51]	0.83 ± 0.09	10	10.6 ± 0.9	98
Co [59]	< 0.3	10	9.9 ± 0.7	99
Ni [60]	24.4 ± 1.2	10	33.9 ± 1.6	95
Cu [65]	27.1 ± 1.3	10	36.6 ± 1.9	95
Zn [66]	963 ± 20	100	1060 ± 50	96
Cd [111]	5.4 ± 0.6	10	15.2 ± 1.2	98
Pb [208]	46.9± 1.6	10	57.2 ± 1.6	103

Предимства на MNPs-SPE с $MnFe_2O_4$

- $MnFe_2O_4$ NPs притежават по-силни магнитни свойства и са по-ефективни при магнитно задържане (колекция).

- Те са по-стабилни в кисела среда и позволяват работа при по-ниско pH

- Показват по-слаба разтворимост при киселинното елуиране на аналитите и пробните разтвори са с по-лека матрица.

- Имат по добри качества за промиване и повторно използване

ICP- какво ново ?

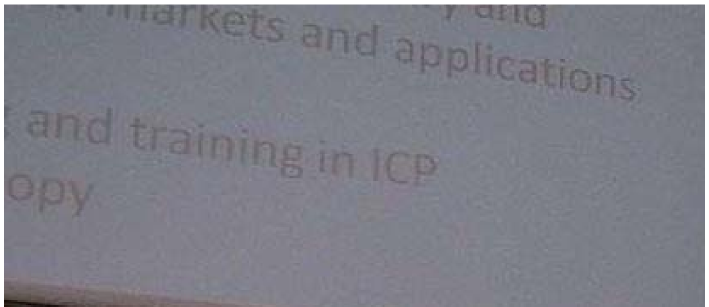
European Winter Conference on Plasma Spectrochemistry
ZARAGOZA 2011
Spain

Round Table

CURRENT STATUS AND FUTURE OF PLASMA-BASED ANALYTICAL INSTRUMENTATION

Moderator: Ryszard Lobinski. Université de Pau et des Pays de l'Adour. CNRS. France.

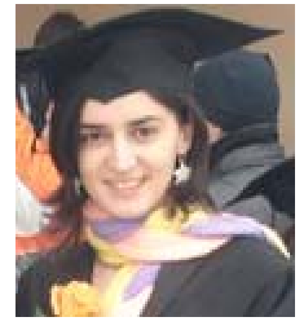
Participants:
S. Wilbur. Agilent Technologies.
A. Ryan. Bruker.
C. Schneider. Perkin Elmer.
D. Ardelt. Spectro Analytica Instruments GmbH.
P. Neal. Thermo Scientific



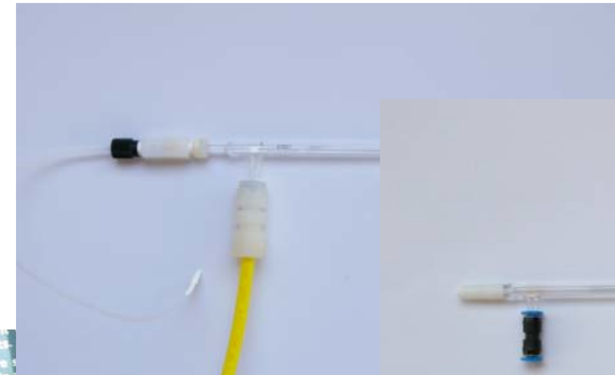
- Проблемът е в BL, а **не** в DL
- **Ще има прогрес в системите за пробовъвеждане**
- Ще се върви към миниатюризация на апаратите
- Ще се развиват “hyphenated” техниките

Охарактеризиране на пулверизатори за директно въвеждане на водни и етанолни аерозоли в ICP

Недялка Р. Чолакова



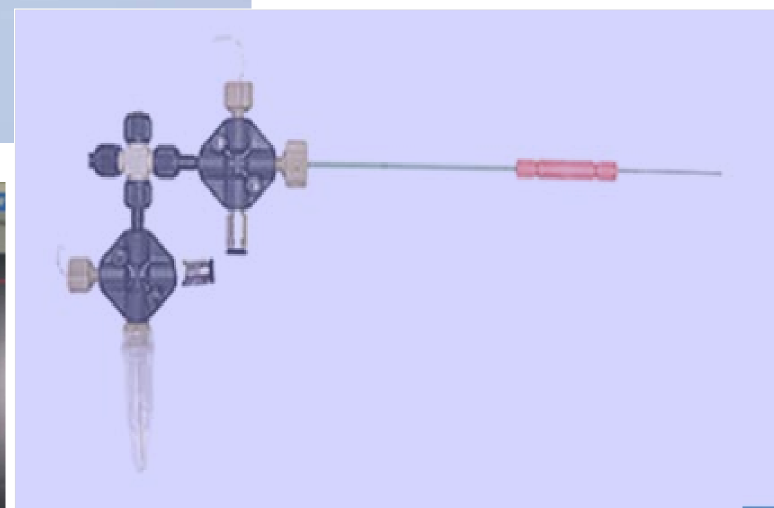
LB – DIHEN, Meinhard Inc.

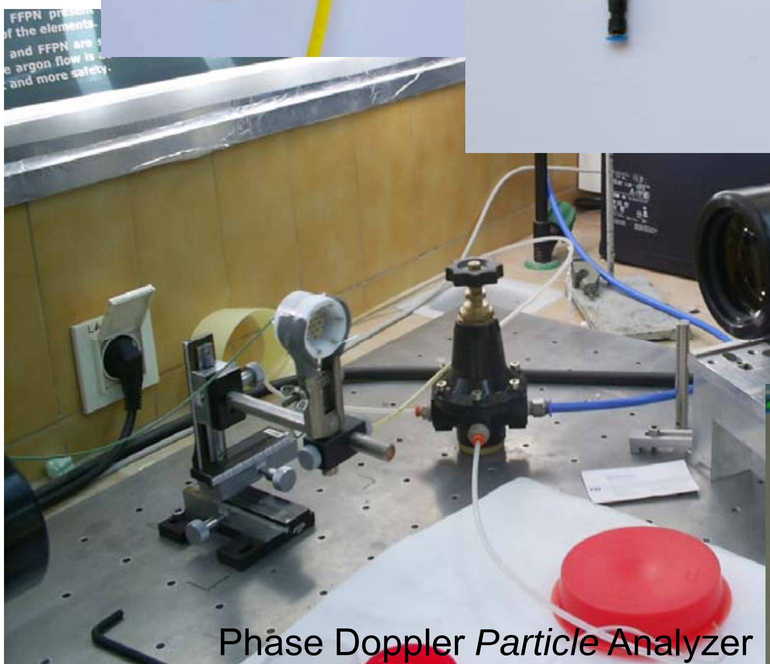


Vulkan, Glass-Expansion

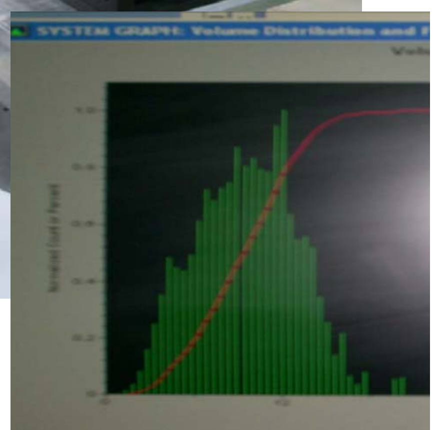
FFDIN, Ingeniatrics

EFFDIN, Ingeniatrics



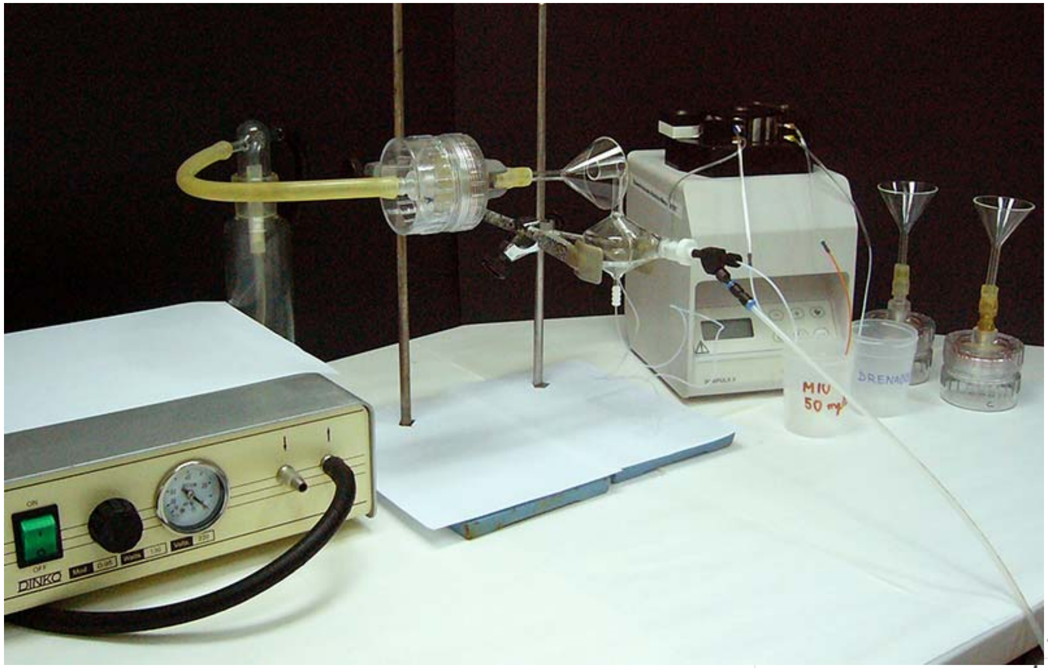


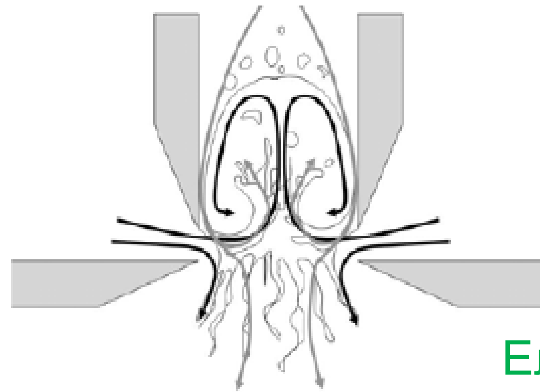
Phase Doppler *Particle* Analyzer



ERASMUS
STUDENT CHARTER

Ефективност на пренос при различни типове пулверизатори за въвеждане на водни и органични разтвори в ICP-OES





Елисавета Ганчева

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Flow-Blurring Multiple Nebulizer **FBMN**

Universitat d'Alacant
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OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Miguel Ángel Aguirre Pastor, Nikolay Kovachev, Beatriz Almagro Fernández, Montserrat Hidalgo Núñez, Alfonso M. Gañán Calvo and Antonio Canals Hernández

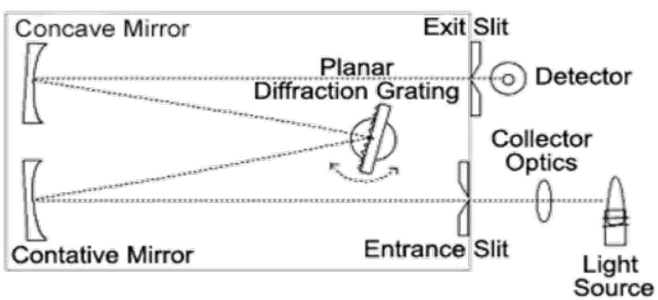
30 June 2012, Sofia, Bulgaria

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

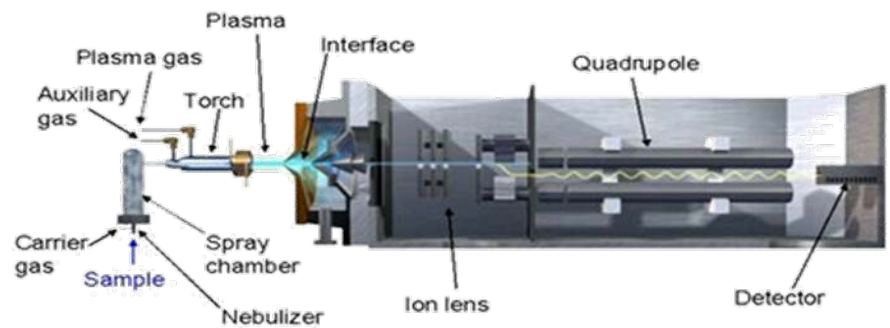
ICP - based spectrometry

ICP spectrometry:

- Wide element coverage
- Excellent detection limits
- High precision



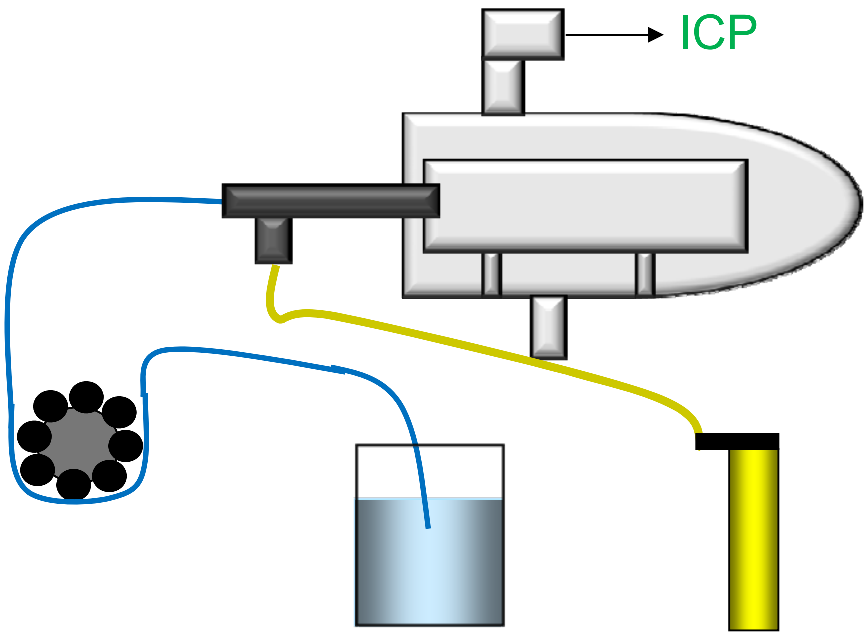
ICP-OES



ICP-MS

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Sample introduction in ICP - based spectrometry



The “ideal” primary aerosol:

☺ Small and monodisperse drop size

☺ “Low” and uniform velocity

Conventional nebulizers drawbacks:

☹ Produce polydisperse aerosols – both in size and velocity

☹ Large sample uptakes are needed to achieve sufficient aerosol transport to the ICP

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

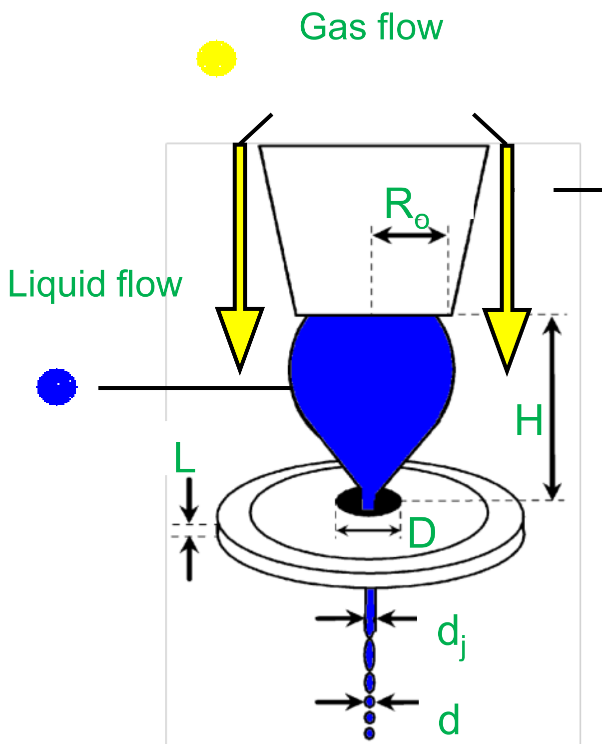
ICP - based spectrometry

Some enhanced nebulizer designs:

- High efficiency nebulizer (HEN)
- Microconcentric nebulizer (MCN)
- Hydraulic high pressure pneumatic nebulizers (HHPN)
- Single-bore high-pressure pneumatic nebulizer (SBHPPN)
- Oscillating capillary nebulizer (OCN)
- Sonic-spray nebulizer (SSN)
- Direct injection nebulizers (DIN)

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Flow Focusing® nebulization principle



R_o: Feeding tube radius

H: Feeding tube-exit orifice distance

L: Plate thickness

D: Exit orifice diameter

d_j: Jet diameter at the exit hole

d: Droplet diameter

Schematic representation of Flow Focusing® liquid breakup

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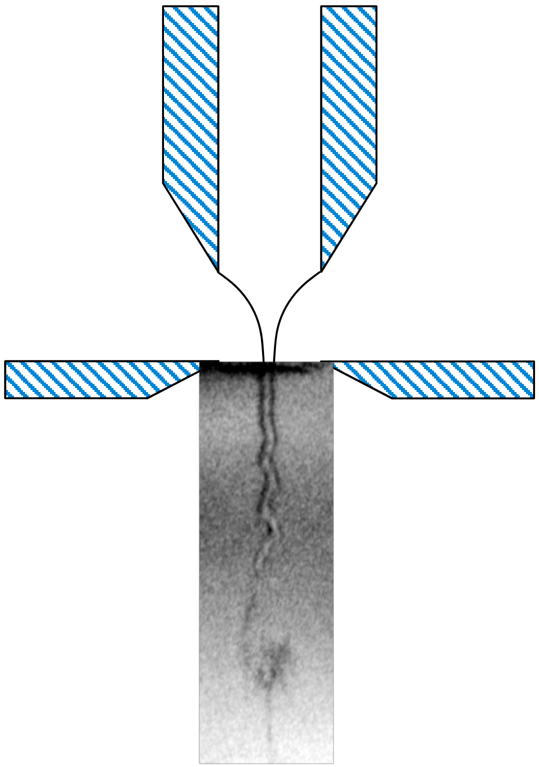
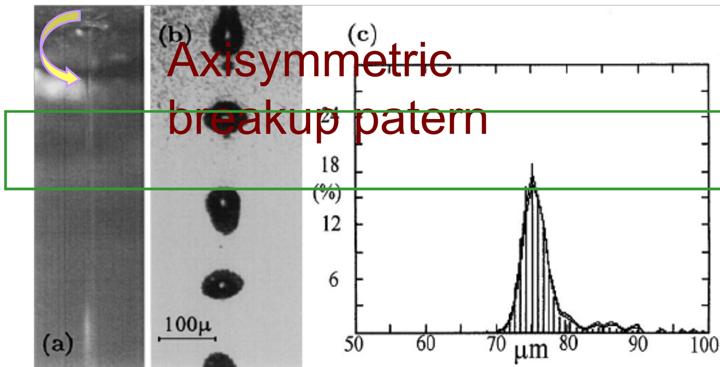


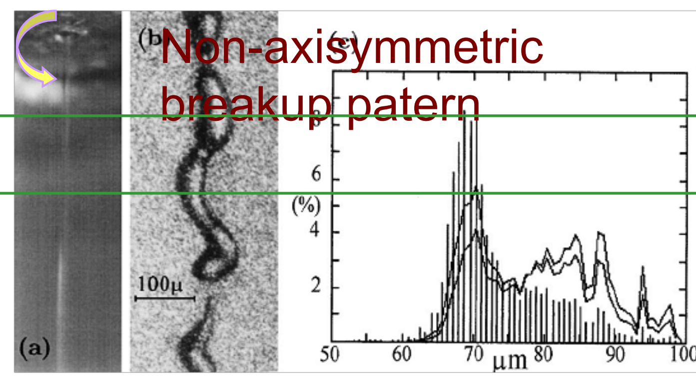
We < We$_c$ **We$_c$~40**



Axisymmetric breakup patern

We = ——— >> 1
σ

We > We$_c$ **We$_c$~40**



Non-axisymmetric breakup patern

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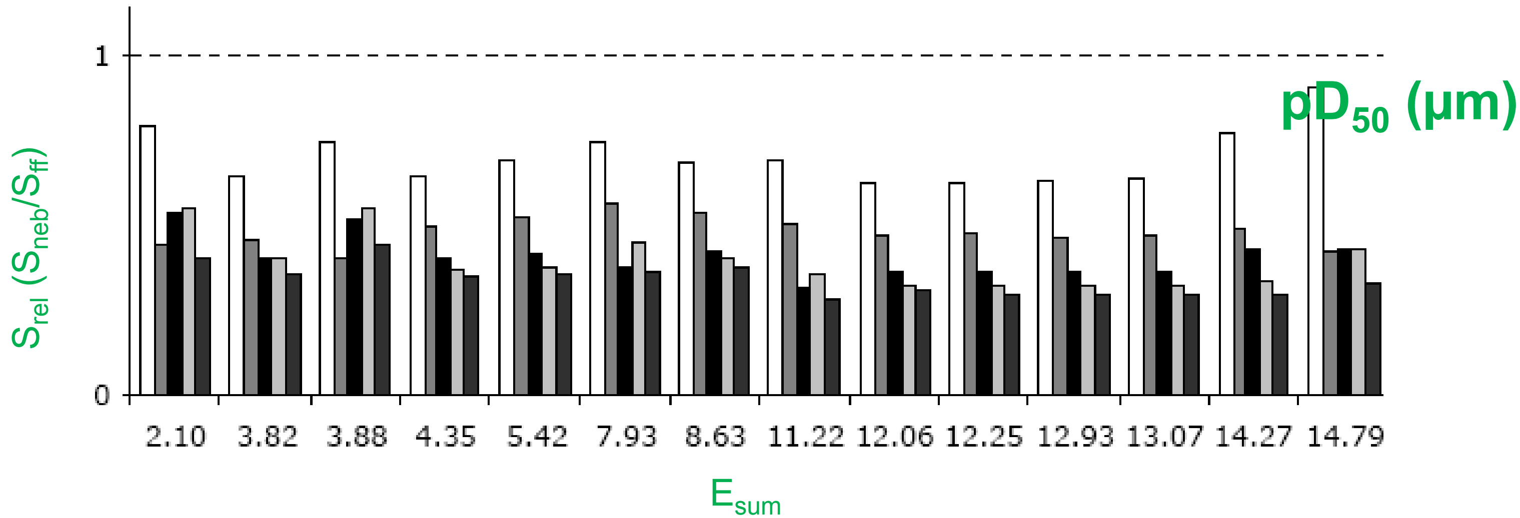
Flow Focusing® based nebulizer

ICP OES comparison with various conventional nebulizers:



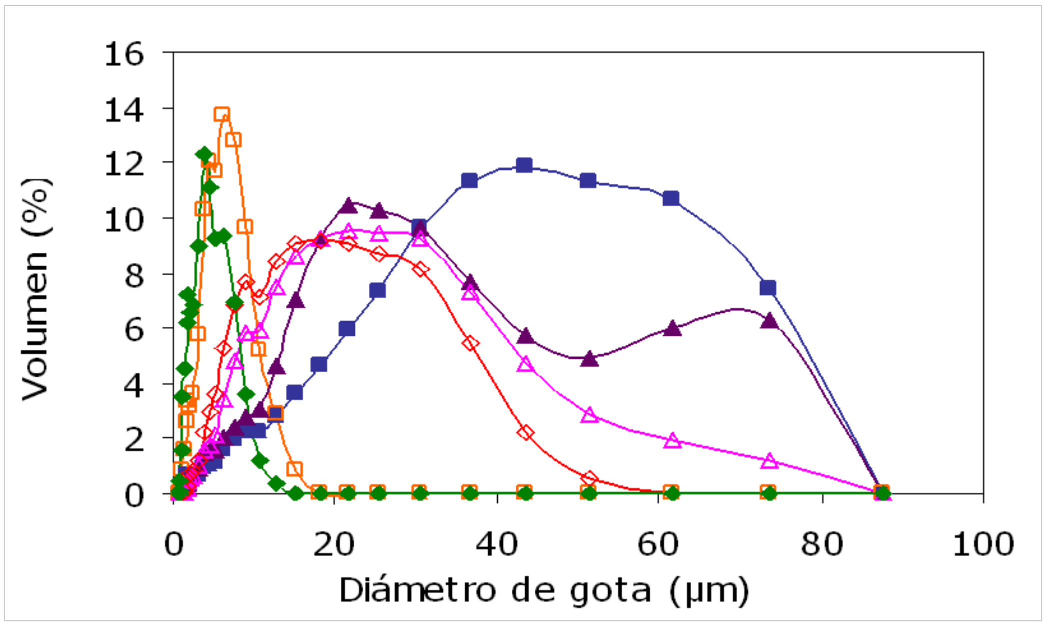
Q_g = 0.70 L min^{-1}; Q_l = 0.20 mL min^{-1}

B. Almagro, A.M. Gañán-Calvo, M. Hidalgo and A. Canals, *J. Anal. Atom. Spectrom.*, 2006, **21**, 770-777

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Flow Focusing® based nebulizer

Droplet size distribution comparison with various nebulizers:



	pD_{50} (µm)
□ HEN:	4.92
■ MM:	31.75*
▲ PFA:	21.71*
△ AM:	16.84
◇ MiM:	13.13
◆ FF:	3.25

Q_g = 0.70 L min^{-1}; Q_l = 0.20 mL min^{-1}

B. Almagro, A.M. Gañán-Calvo, M. Hidalgo and A. Canals, *J. Anal. Atom. Spectrom.*, 2006, **21**, 770-777

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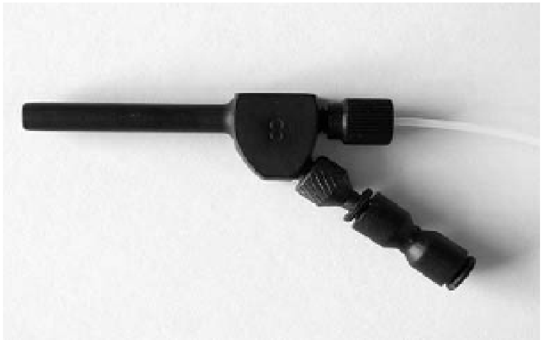
Flow Focusing® nebulizer evolution

2000

2001

2004



2005

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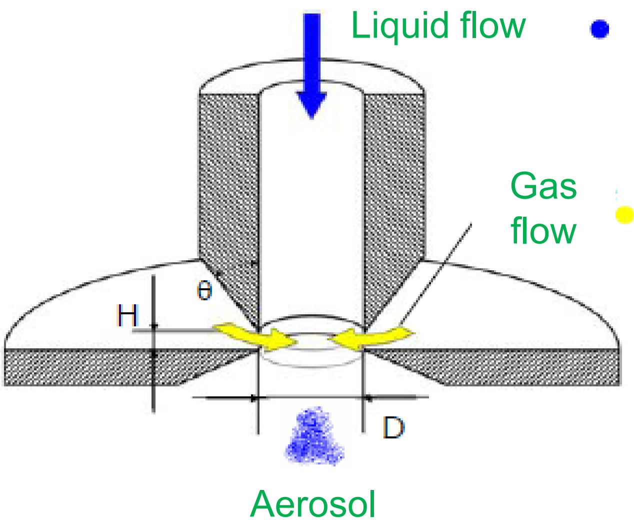
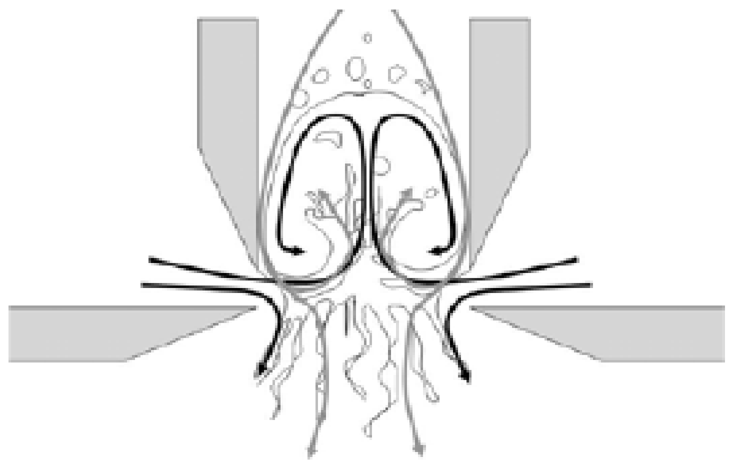
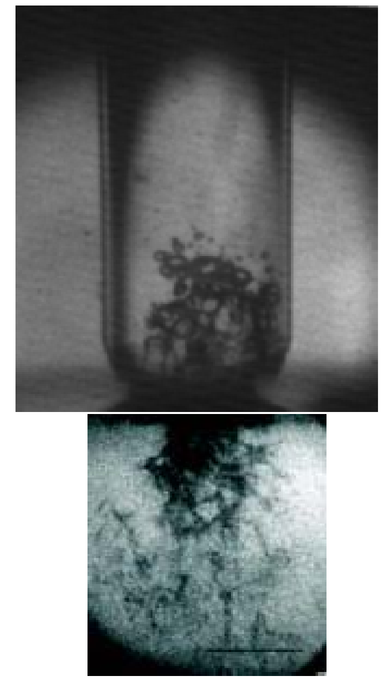
Flow Blurring® Nebulization





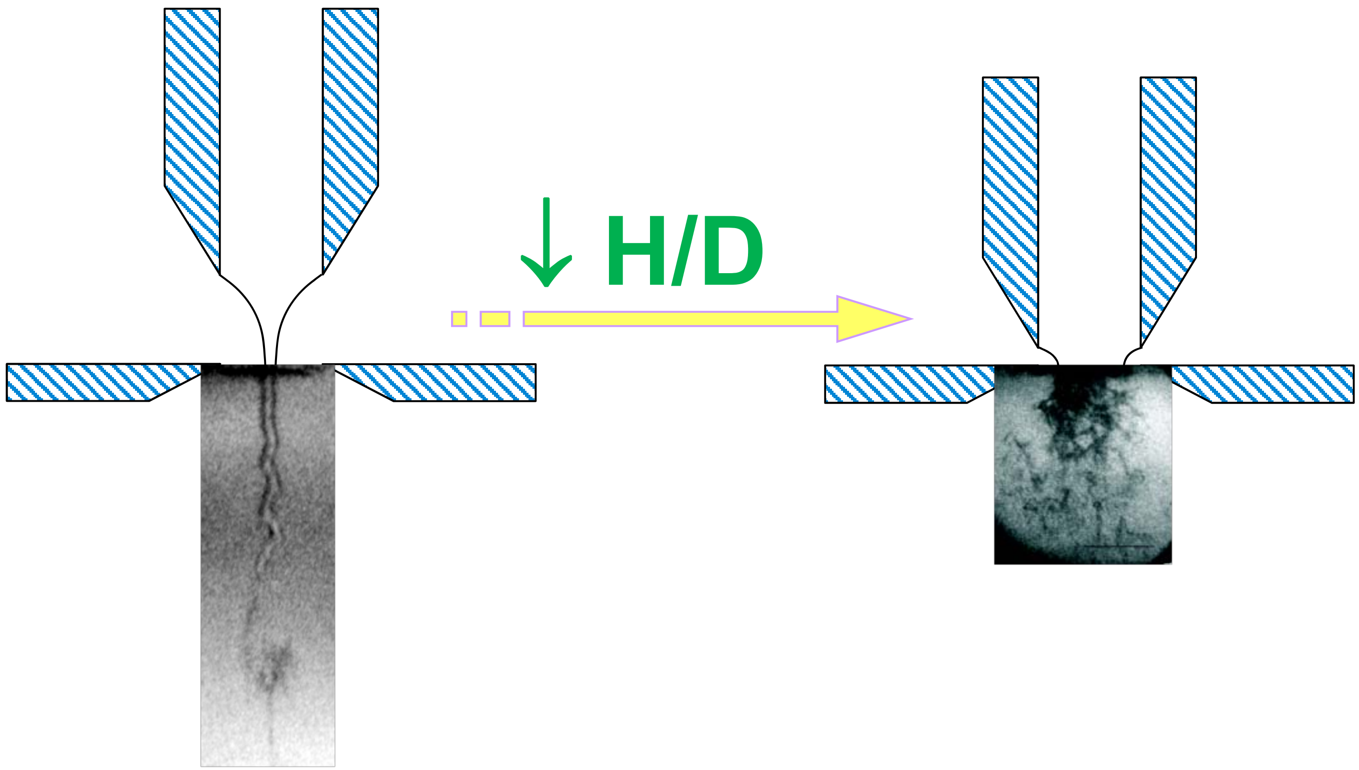


Condition
H/D < 0.25

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Flow Focusing® and Flow Blurring®

Flow Focusing® **Flow Blurring®**



H/D = 1 **H/D < 0.25**

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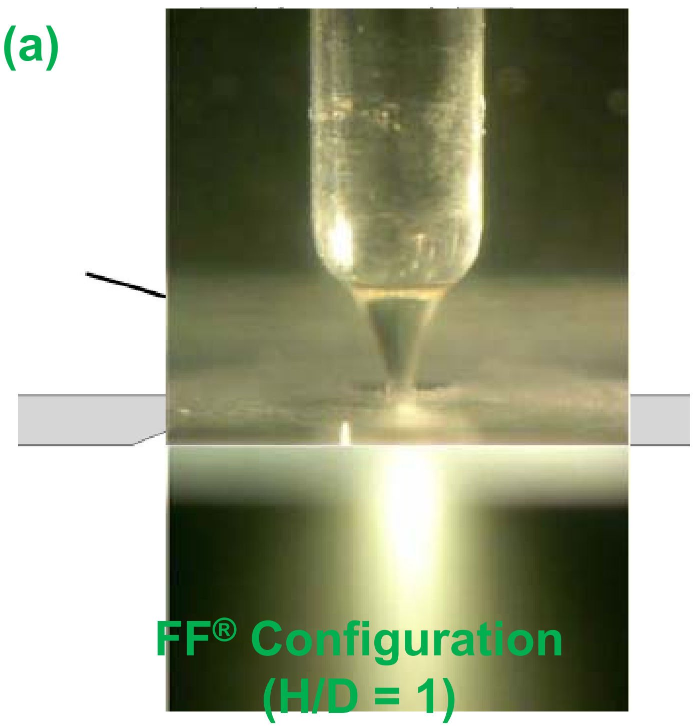
Flow Focusing® and Flow Blurring®

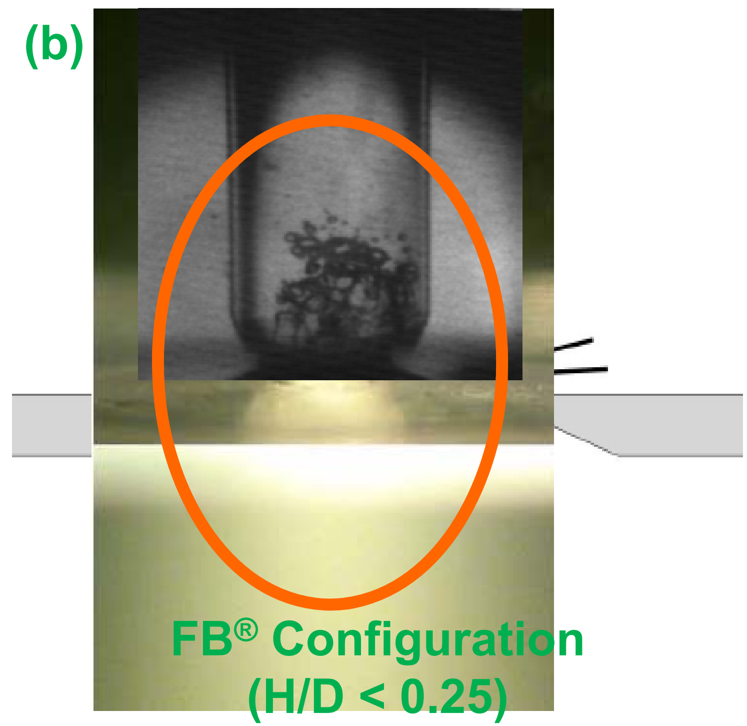
Flow Focusing® – Flow Blurring®

(a)



(a) FF® Configuration (H/D = 1)

(b)



(b) FB® Configuration (H/D < 0.25)

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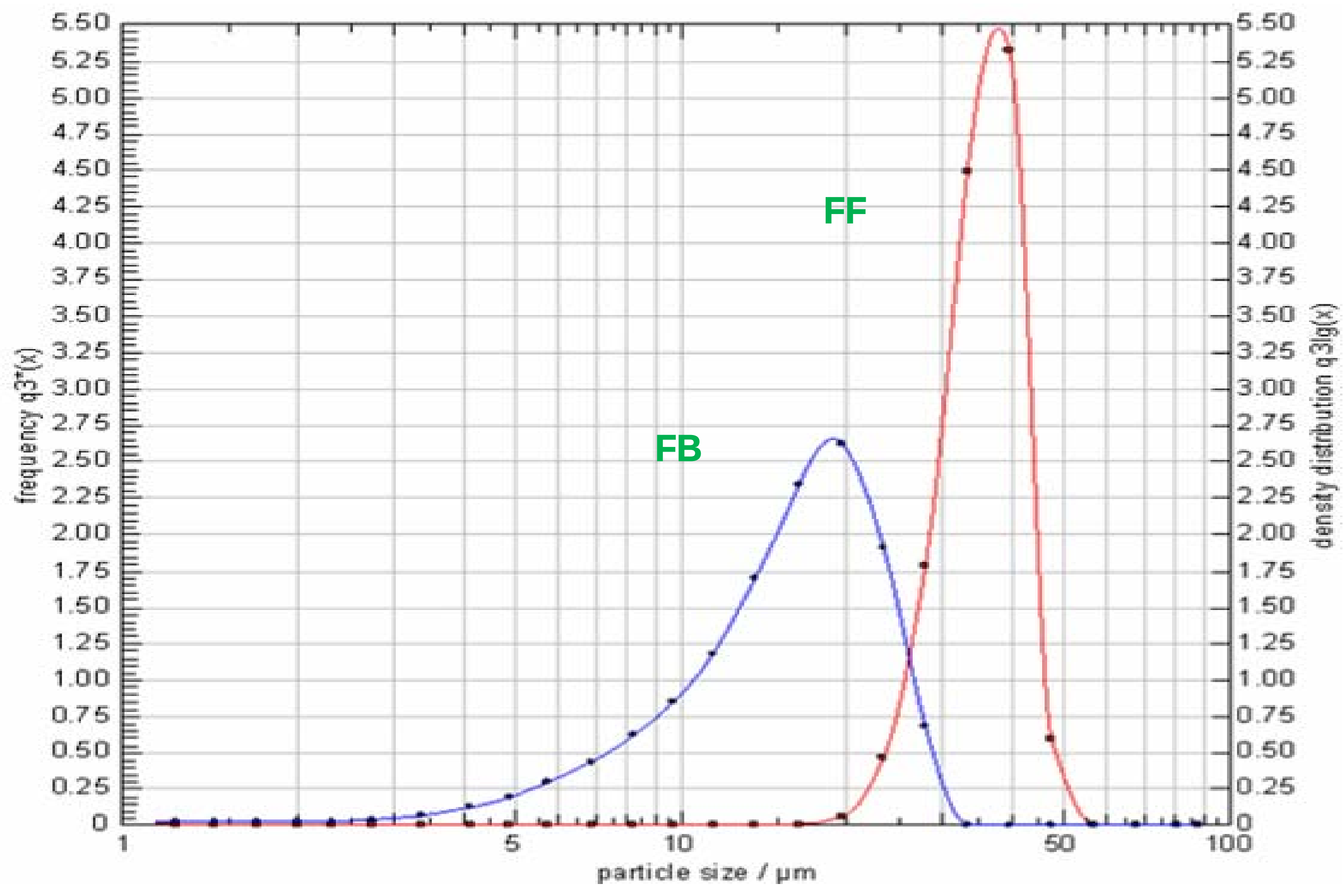
Flow Focusing® and Flow Blurring®



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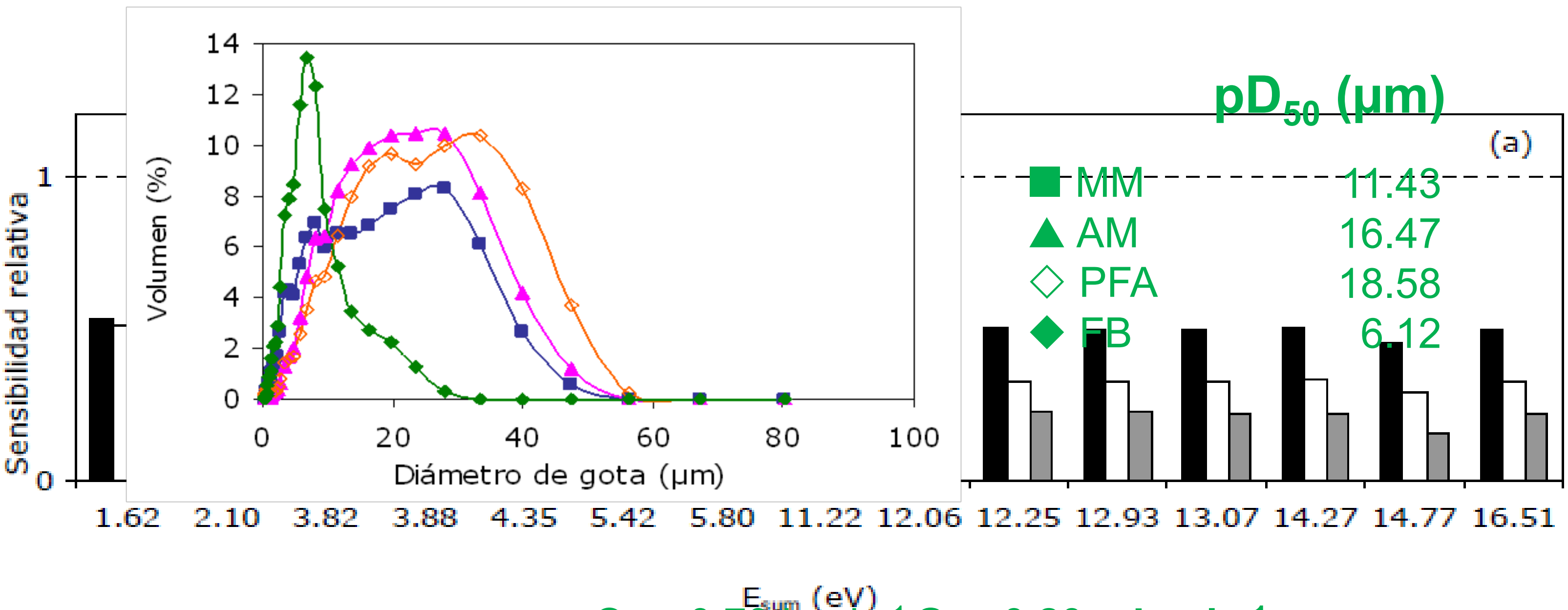
Flow Blurring® based nebulizer

ICP OES comparison with various micronebulizers:

Droplet size distribution comparison with various nebulizers:



	pD$_{50}$ (μm)
■ MM	11.43
▲ AM	16.47
◇ PFA	18.58
◆ FB	6.12

Q_g = 0.70 L min^{-1} **Q_l = 0.20 mL min^{-1}**

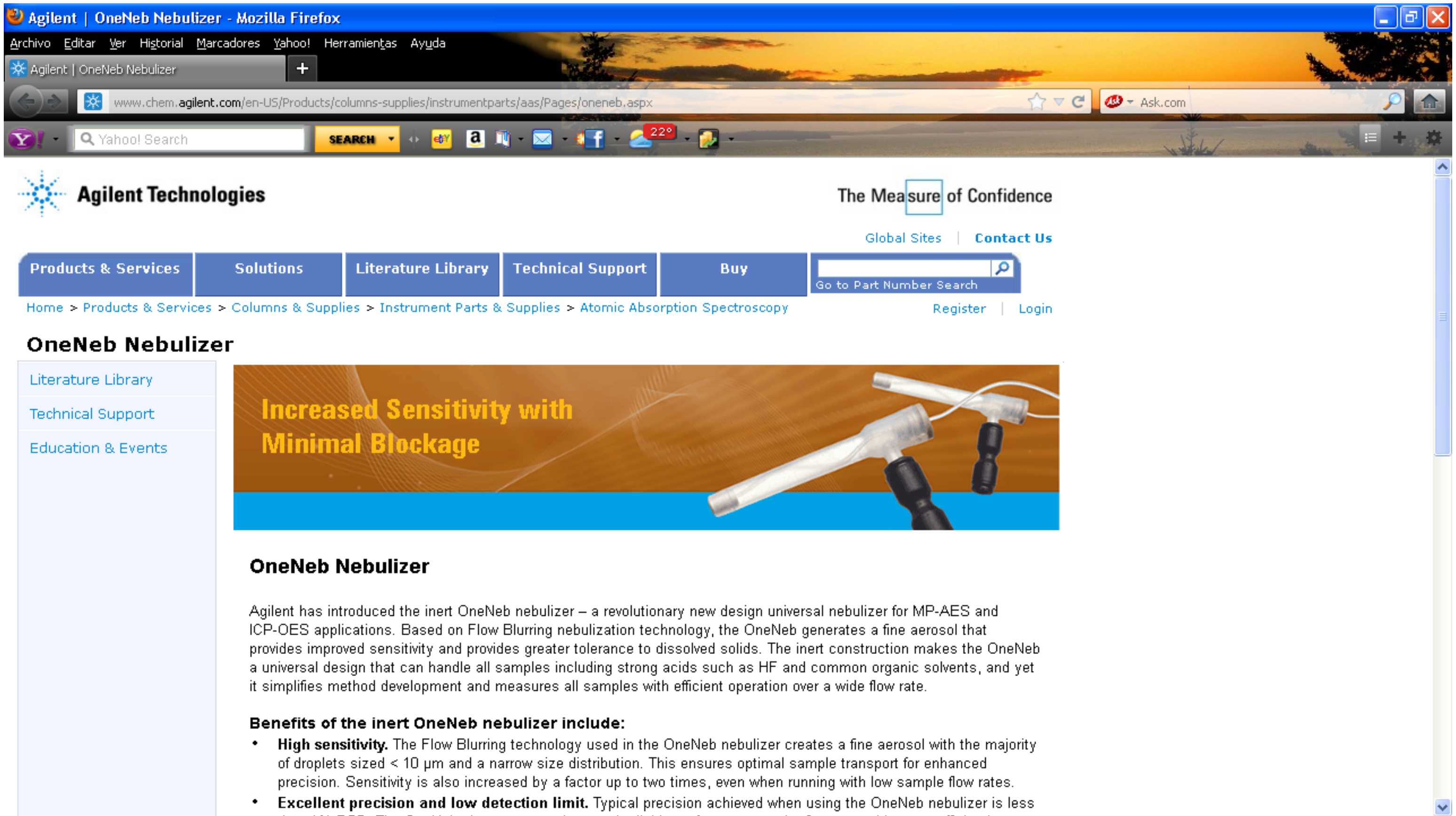
OneNeb®: A new generation of (micro)nebulizer for elemental analysis

OneNeb® Nebulizer

http://www.oneneb.com

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

OneNeb® Nebulizer



http://www.chem.agilent.com/en-US/Products/columns-supplies/instrumentparts/aas/Pages/oneneb.aspx

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Conclusions

Flow Focusing® (FF®) and Flow Blurring® (FB®) nebulization principles:

Very efficient energy transfer from the nebulizing gas to the liquid sample stream
Fine and monodisperse (in the case of FF®) aerosols
High aerosol transport rate through the spray chamber
Simple and robust nebulizer design, no clogging risks

FF® and FB® nebulizers for sample introduction in ICP OES:

- Micronebulizer based on FF® principle
- (Micro)nebulizer based on FB® principle – OneNeb®
- Multiple nebulizer based on FF® and FB® principles
- Dedicated nebulizer for coupling with capillary electrophoresis based on FF® principle

OneNeb®: A new generation of (micro)nebulizer for elemental analysis

Thank you for your attention!

ANALYTICA специализирани семинари

30 June 2012, Sofia, Bulgaria

НАУЧНИ ИЗСЛЕДВАНИЯ 2012