

RESEARCH GROUP: Analytical Chemistry

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Research lines:

1. ANALYTICAL ATOMIC SPECTROMETRY.

- 1.1. *Flow-Injection-Atomic-Spectrometry.*
- 1.2. *Sample Treatment by means of the use of microwave assisted procedures.*
- 1.3. *Study of alternative systems for sample introduction, including the use of*
 - i) slurries, ii) emulsions, iii) vapour phases.

Our group has a high background on all the different branches of analytical atomic spectrometry: flame emission, flame atomic absorption, electrothermal atomization atomic absorption spectrometry, ICP and DCP and, at present we have obtained also an adequate instrumentation to work in ICP-MS.

We have applied atomic spectrometry to solve real problems in different fields and we have developed new analytical procedures for different kind of samples: environmental, clinical, food and technological samples, from liver biopsies of less than 10 mg to sewage slugs, from waters to mineral oils and from urine to precious metals.

Our work has been focussed specially on the development of fast, accurate and precise methods for sample preparation (specially by means of the use of microwave ovens, having developed methods for the dry ashing in home mode microwave muffle furnaces and for the pressurized wet ashing, and also on-line digestion procedures for the analysis of solid samples) and also in the development of alternative methods for sample introduction.

In the study of alternative systems for the introduction of samples we have worked with slurries and with emulsions (which permits the direct determination of metallic elements in oil samples by using aqueous solutions for the preparation of standards) and also in the development of systems for the generation and introduction of samples as vapour phases.

At present, our work is carried out especially on the flow-injection-atomic spectrometry which permits the automatization of the analytical procedures, including not only the measurement steps but also the sample preparation steps.

Problems, such as the separation of contaminants, the hyphenation between separation techniques and atomic spectrometry, and the use of new preconcentration techniques, such as micellar enhanced ultrafiltration (MEUF) are the topics in due course in our group.

2. ANALYTICAL MOLECULAR SPECTROMETRY.

- 2.1. *Quantitative Infrared analysis, both in the NIR and FT-IR (middle).*
- 2.2. *On-line methods for infrared analysis.*
- 2.3. *Micellar enhanced fluorimetric procedures.*
- 2.4. *On-line derivatization procedures for the analysis of pollutants by diode-array UV/Vis spectrometry.*
- 2.5. *Derivative spectrometry (UV/Vis, NIR and IR ranges).*
- 2.6. *On-line catalytic photodegradation of pollutants.*
- 2.7. *Enzymatic analysis in non-aqueous media.*

In the field of Analytical Molecular Spectrometry, our job consists on the development of new procedures based on UV-Vis-NIR-FTIR, molecular fluorescence and phosphorescence.

In the field of NIR and FT-IR our group is one of the leader groups on the development of automatized procedures by means of the use of flow injection analysis and we have developed new-ideas which permit to enhance the accuracy, precision and sensitivity of infrared spectrometry and provide on-line quantitative procedures for the petroleum industry, pharmaceuticals, paints, environmental analysis and food control, and which can be applied to the simultaneous determination of several components in a same sample without requiring tedious procedures for sample preparation. At present we are working on HPLC by using IR spectrometry as detector.

The use of organized media is one of the topics in which our group has carried out a big number of contributions during the last 15 years. In this sense we have developed micellar enhanced procedures for the spectrophotometric and fluorimetric determination of dyes, drugs, pesticides and metallic ions and we have contributed to the development of the theoretical knowledge about the interaction of chemical systems with micelles by means of the development of a new procedure for the direct fluorimetric determination of binding constants at extreme pH values and for clarifying the parameters involved in the interaction between a series of molecules and different kinds of surfactants.

At present our work in this area includes the automatization of the micelles enhanced procedures and the development of new separation procedures based on MEUF and "clouding".

In the molecular UV/Vis spectrometry field we have developed new procedures for the derivative direct determination of pollutants in water samples and drugs in clinical samples, such as urine and serum, and also on the FIA-derivation spectrometry.

On the other subject of our common research on UV/Vis is the development of procedures for the simultaneous determination of several compounds in a same sample, based on the use of derivatization procedures in batch, in the stopped-flow mode or in the continuous flow analysis.

One of our major preoccupations consist of the development of clean analytical procedures, based on the on-line photodegradation of pollutant molecules after the measurement step and, in this field, we are studying the catalytic photodegradation of different pollutants by means of the use of TiO₂ slurries and UV light, which permits a fast degradation of linear and aromatic molecules in a few seconds.

In the field of FIA one of our last contribution is the development of analytical procedures based the enzymatic analysis in non-aqueous media which permits the development

of enzymatic reactions with a long life and low cost, which not requires the chemical immobilization of enzymes.

3. SUPERCRITICAL FLUID CHROMATOGRAPHY.

We have begun the research on this field recently. Our instrumentation is one of the firsts installed in Spain which permits to work with capillary columns but also with packed columns and we are working on SFC applied to the drug and pesticide analysis by using CO₂ and methanol carrier systems.

On the other hand we have also the instrumentation required for off-line and on-line supercritical fluid extraction and in the following months we expected to improve our research in this field.

From the results obtained in different research lines, our group has published until now more than 175 papers in journals of Analytical Chemistry and we include, at the end of this short notice a list of our publications. (omitted)

We consider that the following 5 articles are representative of our job:

1. Metal chelate fluorescence enhancement by nonionic micelles surfactant and auxiliary ligand nature influence on the Nb-lumogallion complex, A. Sanz Medel, M. Fernández-Pérez, M. de la Guardia and J. L. Carrión, *Anal. Chem.*, 58 (1986) 2161-2166.
2. Direct derivative spectrophotometric determination of nitrazepam and clonazepam in biological fluids, F. Rández-Gil, J. A. Darós, A. Salvador and M. de la Guardia, *J. Pharm. Biomed. Anal.* 9 (1991) 539-545.
3. On-line microwave assisted digestion of solid samples for their FAS analysis, M. de la Guardia, V. Carbonell, A. Morales-Rubio and A. Salvador, *Talanta*, 40 (1993) 1609-1617.
4. Enzymatic Flow Injection Analysis in Nonaqueous Media, L. Braco, J. A. Darós and M. de la Guardia, *Anal. Chem.*, 64 (1992) 129-133.
5. Simultaneous Flow Analysis FT-IR determination of Benzene, Toluene and MTBE in gasolines, M. Gallignani, S. Garrigues and M. de la Guardia, *Analyst*, 119 (1994) 653-657.

ps: I (Narusawa, Rikkyo University) received a letter dated 29th September 1994, from Prof. M. de la Guardia with a short notice about their research group. In this letter, there was a comment that Prof. Guardia hoped to have new possibilities to joint Japanese research groups in a near future. I introduce his short notice here. A list of 175 papers mentioned above is available from M. de la Guardia upon request.