



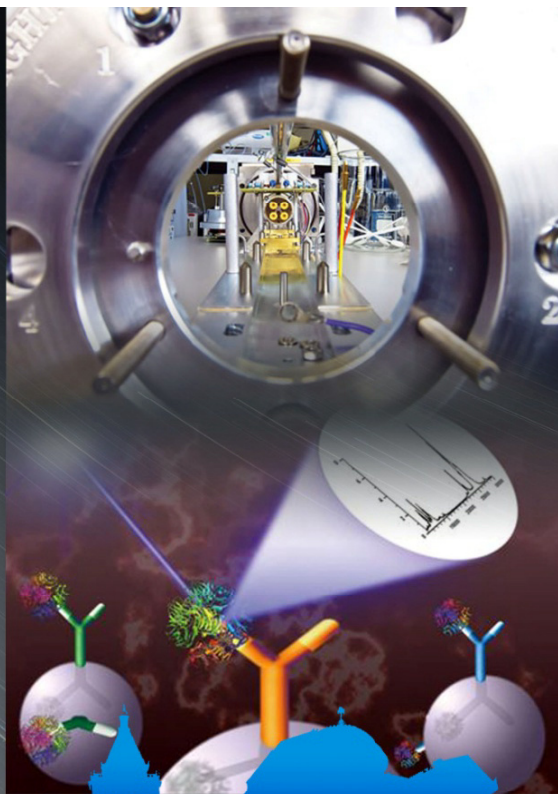
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2013, May 5-8

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BOOK OF ABSTRACTS

Concept and Graphic Design C&M Unipa
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MoOr16

**NEW INSIGHTS ON SUPRAMOLECULAR AGGREGATES OF SODIUM
DOCUSATE (AOTNa) BY ION MOBILITY MASS SPECTROMETRY**

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Keywords: *Ion Mobility, Surfactants, micelles, cross section*

Background: The ability of some surfactants to form charged aggregates in gas phase has been proven experimentally by electrospray ionization (ESI) spectrometry [1- 5].

However, in the early investigations, interesting questions concerning the structural organization of these aggregates in gas phase and if they reflect their typical self-assembling in the starting liquid solutions were not addressed. Subsequently, independently of their aggregation motive in liquid phase and of the charge state in the gas phase, some hints suggesting that surfactant molecules are organized in gas phase as reverse micelle-like aggregates have been reported

Objective: The supramolecular charged aggregates formed by ESI of AOTNa solutions have been studied through ion mobility experiments in order to gain more informations as concer their size, and possibly, shape.

Method: AOTNa (99%) was purchased from Sigma-Aldrich-Fluka (Milan, Italy).

For ion mobility experiments, a 2.4 mg/ml AOTNa solution in a 1:1 water/acetonitrile mixture was prepared and directly infused at a flow rate 10 μ l/min in positive mode electrospray.

The ESI mass spectra are recorded under following operative conditions: source temperature 120 °C, capillary voltage 3 kV, cone voltage 45 V, extraction cone 4.0 V, Desolvation Temperature 350 °C, Cone Gas Flow 10.0 L/Hr, Desolvation Gas Flow 800.0 L/Hr. The ion mobility cell contained nitrogen at a flow rate of 90 ml/min. The travelling wave height and velocity were 6 V and 311 m/s respectively.

Under the same experimental conditions, solutions of polyalanine (Sigma, nominal 1000-5000 mol wt range; 5.6*10⁻⁵ to 2.8*10⁻⁴ M in a 49:49:2 water/acetonitrile/acetic acid mixture) were also infused.

The samples were analyzed by a Synapt G2 HDMS (Waters UK Ltd, Manchester, UK) and data were acquired by MassLynxTM version 4.1 in high-definition mass spectrometry (HDMS) mode and handled by DriftScope.

Results and conclusion: Positively charged clusters with several charge states, ranging from +1 to +4, have been investigated. Despite of previous investigations (Siuzdak et al. 1995, Nohara et al. 1998), suggesting random aggregation of charged surfactant aggregates, specific ionic clusters with peculiar cross sections are observed. Indeed the occurrence at each aggregation number and extra charge of an unique value of cross section points toward aggregates whose conformations do not show discernible shape change in the experiment time scale. It is worth noting that calculated cross-sections (mobcal EHSS algorithm, considering reversed micelle structures) and experimental ones (through ion mobility mass spectrometry using Polyalanine at various charge states as reference) are in fair agreement. This supports once again that reverse micelle is the most likely structure that characterize such aggregates. Further this allows evaluating of the charge state effects on the shape of AOTNa clusters.

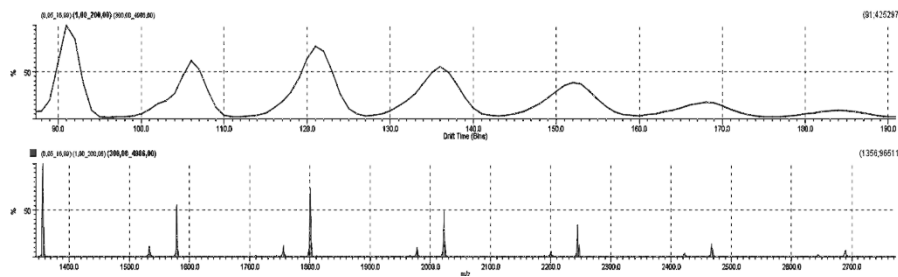


Figure 1: Ion Mobility graph of doubly charged AOTNa clusters

In particular, both theoretical calculations and experimental CCS suggest an increase of the size of the whole aggregate on increasing the charge states.

Finally, the regular cross section dependence on the aggregation number of AOTNa clusters at the same charge state makes this surfactant an ideal candidate as calibration standard for CCS measurements.

References

1. D. Nohara et al. "The possibility of the direct measurement of micelle weight by electrospray ionization mass spectrometry", *Rapid Commun. Mass Spectrom.* 12: 1933 (1998)
2. G. Siuzdak, B. Bathner, "Gas-Phase Micelles", *Angew. Chem. Int. Ed.* 34: 2053 (1995)

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Presentation type: oral