AMMONIUM DETERMINATION FROM MUNICIPAL WASTEWATERS. METHOD VALIDATION AND UNCERTAINTY ESTIMATION

Camelia DRAGHICI[1]*, Domnica COMAN[2],
Mihaela SICA[2], Carmen DIMA[2]

Abstract.
The analytical method for ammonium ion measurements in aquatic media was subject to validation procedure, based on the standard method. The following method performance criteria were investigated: selectivity, linearity domain, precision, accuracy, limit of detection, limit of quantification and robustness. The sources of uncertainties were identified, and the extended standard uncertainty was estimated. The method was demonstrated to be suitable for ammonium determination and was further used to determine ammonium content in domestic and industrial wastewaters.

Key words: chemiluminescence, chromatography, method selectivity, validation.

Introduction

Ammonium in wastewaters is mostly produced by the decomposition of organic compounds containing nitrogen, or by urea hydrolysis, being one of the primary forms of nitrogen in wastewater. When high concentrations of nitrogen compounds are released in running waters and lakes, eutrophication (excessive plants growth) can occur. Due to the restrictive nitrogen presence in water, a good control of ammonium concentration is imposed. To fulfil this aim, reliable analytical results are necessary.

In accredited laboratories the method validation is compulsory for the quality control system, but the international scientific community recognises more and more the method validation as a meaningful tool to demonstrate that the results are trustful and reliable. Thus, the method performance criteria like specificity/selectivity, precision, trueness, linearity range, limit of detection (LOD), limit of quantification (LOQ), and robustness are required for methods validation (EURACHEM Guide, 1998; Taverniers et al., 2004). Moreover, even if not yet imposed by all standard methods, measurement uncertainty is a statistical parameter, which describes the possible dispersion of the values reasonably attributed to the measurand, that is determined by the addition of

---

[1] Transilvania University of Brasov, Department of Product Design, Mechatronics and Environment, Brasov, Romania, c.draghici@unitbv.ro
[2] Compania Apa Brasov (Water Company), Wastewater Laboratory, Brasov, Romania