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***SEQUENTIAL INJECTION SYSTEM FOR THE
SPECTROPHOTOMETRIC DETERMINATION OF AMMONIUM IN
PORTUGUESE ESTUARINE WATERS***

**Ricardo L. A. Segundo^a, Raquel B. R. Mesquita^{a,b}, M. Teresa S. O. B. Ferreira^a,
Catarina F. C. P. Teixeira^b, Adriano A. Bordalo^b and António O. S. S. Rangel^a**

^a *CBQF/ESB, Escola Superior Biotecnologia, Universidade Católica Portuguesa, Rua
Dr. António Bernardino de Almeida, 4200-072 Porto, Portugal*

^b *Laboratory of Hydrobiology, Institute of Biomedical Sciences Abel Salazar (ICBAS)
and Institute of Marine Research (CIIMAR), Universidade do Porto, Porto, Portugal*

Clean unpolluted water is essential not only for human health and well-being but also for sustain earth ecosystems. Plants and animals in lakes, rivers and seas react to changes in their environment caused by changes in chemical water quality. Almost all human activities can and do impact adversely upon the water. Water quality is influenced by both direct point source pollution (from sewage treatment and industrial discharge) and diffuse pollution (from farming) which come from urban and rural populations. For agriculture, the key pollutants include nutrients and pesticides. The European Environment Agency has set a list of potential pollution indicators to monitor coastal and transitional waters, such as ammonium. Large inputs of nitrogen (and phosphorous) to water bodies can lead to eutrophication causing ecological changes that result in loss of plant and animal species, and affect the use of water for human consumption and other purposes. In the form of ammonium, nitrogen is toxic to aquatic life at certain concentrations in relation to water temperature, salinity and pH. Furthermore, ammonium exerts a demand on oxygen in water due to its oxidation to nitrite and nitrate.

Within this context, a flow system for the determination of ammonium in transitional waters was developed. Therefore, the method should cope with a wide range of salinity. Samples were collected in three locations of four Portuguese rivers in the northern Portugal: Ave, Cávado, Douro and Lima. Other samples of interstitial water were also analyzed to evaluate the ammonium accumulation in the river bench.

The determination is based in the colorimetric change of bromothymol blue (BTB) indicator with the presence of ammonia. Ammonium is converted to ammonia by the

mixture with sodium hydroxide; afterwards ammonia diffuses through a gas diffusion unit causing a pH change in the BTB indicator. The pH change of the BTB in the acceptor channel causes the colour change, measured at 620 nm. No BTB solution is consumed as this solution is recirculated back to its container. No interference from different saline levels of the samples was observed.

With the same manifold configuration, two dynamic ranges were achieved just by altering the number of introduced plugs. Some figures of merit of the developed system are summarized in the table below.

Dynamic concentration range (mg/L)	Calibration curve	LOD (mg/L)	LOQ (mg/L)
0.1 – 1	$A = 0.156 (\pm 0.003) [\text{NH}_4^+] - 0.082 (\pm 0.016); R^2 = 0.993 \pm 0.002$	0.03	0.09
1 – 5	$A = 0.0863 (\pm 0.0098) [\text{NH}_4^+] + 0.0775 (\pm 0.0187); R^2 = 0.999 \pm 0.001$	-	0.14

The developed system was applied to the determination of ammonium in estuarine waters with direct introduction of the sample, without any previous treatment. Other samples were also analyzed with the developed method such as water from wells and interstitial water from the bench of the studied rivers.

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