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A comparison study on the uncertainty evaluation for the determination of total nitrogen, using ISO 11352 and other alternative approaches

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One of the important indicators of the water quality is the quantity of inorganic substances it contains. These can be evaluated using different analytical methods, such as Chemical Oxygen Demand (COD), Total Organic Carbon (TOC) and Total Nitrogen (TN). Since the determination of COD is the least environmental friendly, the combination of the TOC and TN are generally being accepted as the best methods.

To determine Total Nitrogen (TN) a sample is injected in the reactor at a temperature of 750-950 ºC in the presence of a catalyst where all the chemically connected nitrogen is converted into nitrite oxide NO. An air flux or oxygen conveys the oxidation products to the chemiluminescense detector where the nitrite oxide reacts with the nitrogen to form excited NO_2*. The rapid decay of NO_2* emits light which is in turn measured in photomultiplier tube. The electrical signal of the photomultiplier tube is then amplified and transmitted to a computer for data treatment.
The test also involves the determination of Nitrate + Nitrite from the injection of the sample into the reactor. These are converted into nitrite oxide, to be determined by chemiluminescense as in the total nitrogen procedure.

The procedure resumed above involves a number of intermediate calculations in order to determine the final quantities required. These quantities entail the estimation of an expanded uncertainty for the measurand, resulting from many sources of input uncertainties, during the overall process of determination. They include precision, reproducibility, reference materials, interlaboratory comparisons, among others.

This work aims to compare and discuss the different methods available to evaluate the measurement uncertainty associated with this typical test of water quality assessment. International standard ISO 11352:2012 exemplifies the estimation of measurement uncertainty based on (a) reference material; (b) proficiency tests; (c) quality control sample and recovery experiments. It will be attempted to establish a comparison between these approaches and the conventional ISO-GUM approach and a Monte Carlo method.

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[2] **Pereira, A.C., Reis, M.S., Saraiva, P.M., Marques, J.C. (2010),** *Anal. Chimica Acta,* **no. (659),** 93-101.

[3] **Pereira, A.C., Reis, M.S., Saraiva, P.M., Marques, J.C. (2011),** *Chemom. Intell. Lab. Syst.* **no. (105),** 43-55.