VALIDATION OF A SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF TOTAL PHOSPHORUS IN SOILS AND SEDIMENTS USING MICROWAVE-ASSISTED DIGESTION

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Phosphorus (P) is an important macronutrient and its accurate determination in environmental matrices such as soils is essential for understanding the biogeochemical cycling of the element, studying its role in ecosystem health and monitoring compliance with legislation. There have been many methods developed to extract and analyze total P in soil for assessing the environmental risk of P leaching and surface runoff, but they are not designed for routine analysis and monitoring aims. The current methods apply different digestion procedures. Two of the more commonly used methods of P extraction are sodium carbonate fusion and acid digestion. It seems there is not a tendency of unifying these methods because of the enormous range of soils and climatic conditions. The wet digestion procedures using conventional heating and strong acid mixtures are very prolonged and there is potential possibility of phosphorus species lost. The quality assurance of the methods used is not usually discussed.

In our laboratory a microwave-assisted digestion procedures with aqua regia were developed and validated in order to determine the heavy metals content in environmental samples. These procedures (with power control and temperature control of the microwave systems and closed vessels) were adapted to the soil and sediment samples in order to determine the total phosphorus content by the spectrophotometric molybdate method, which is commonly used for soils. In this way the time of the microwave digestion step was limited to 20 minutes instead of several hours needed for the conventional digestion. These procedures were validated using the certified reference materials NIST 2709, BCR 684, LKSD 4. The analytical characteristics of the method, presented in this work, suggest the reliability of the method: MDL -0.006 % P, repeatability -2.5 %, reproducibility -2%, accuracy -8 %, expanded uncertainty -10 %.

1. DIN 38414 Bestimmung von Phosphor in Schlammen and Sedimenten.
2. EN 13346 Characterization of sludges - Determination of trace elements and phosphorus-Aqua regia extraction methods.