2. Selection of Method

2.1 General Approach

Dana Zimmer, Rhena Schumann

In nature, phosphorus mainly exists as phosphate or sometimes as phosphonate. Phosphate can form relatively stable (hardly soluble) compounds with numerous ions and organic compounds. For this reason digestion methods have to take into consideration the matrix of sample (Fig. 2.1-1). In some cases, interfering ions, such as arsenates, have to be eliminated before measurement, for example by precipitation. Often it is sufficient to adjust reaction conditions, that interfering ions cannot effect measurement.

With secondary ranking but not negligible is the selection of the analysis method. For example, the photometric P determination by molybdenum blue is much easier and more cost-effective than ICP (Inductively Coupled Plasma) as alternative. However, the measurement range of most photometric method is in the lower µMol-range, but sometimes a higher measurement range is necessary. The ICP has the advantage that almost each sample matrix can be measured and measurement range can, with some limits and in dependency of measurement of other elements, be adjusted. The molybdenum blue method does not tolerate high nitrate concentrations (nitric acid, *aqua regia*) in solution. Additionally, problems occur if the digestion solution is stored in PE-centrifuge tubes without neutralizing solution.

- Classification of matrix material to be analysed, according to
  - concentration of organic matter,
  - salinity and
  - interfering ions.
- Selection of method of digestion/extraction, according to
  - P binding form or sample preparation and
  - coordination with P analytical method.
- Coordinate with existing equipment for:
  - extraction/digestion,
  - P determination method,
  - available sample volume,

- measurement range of analytic method and
- potentially, auto analyser for high sample throughput.

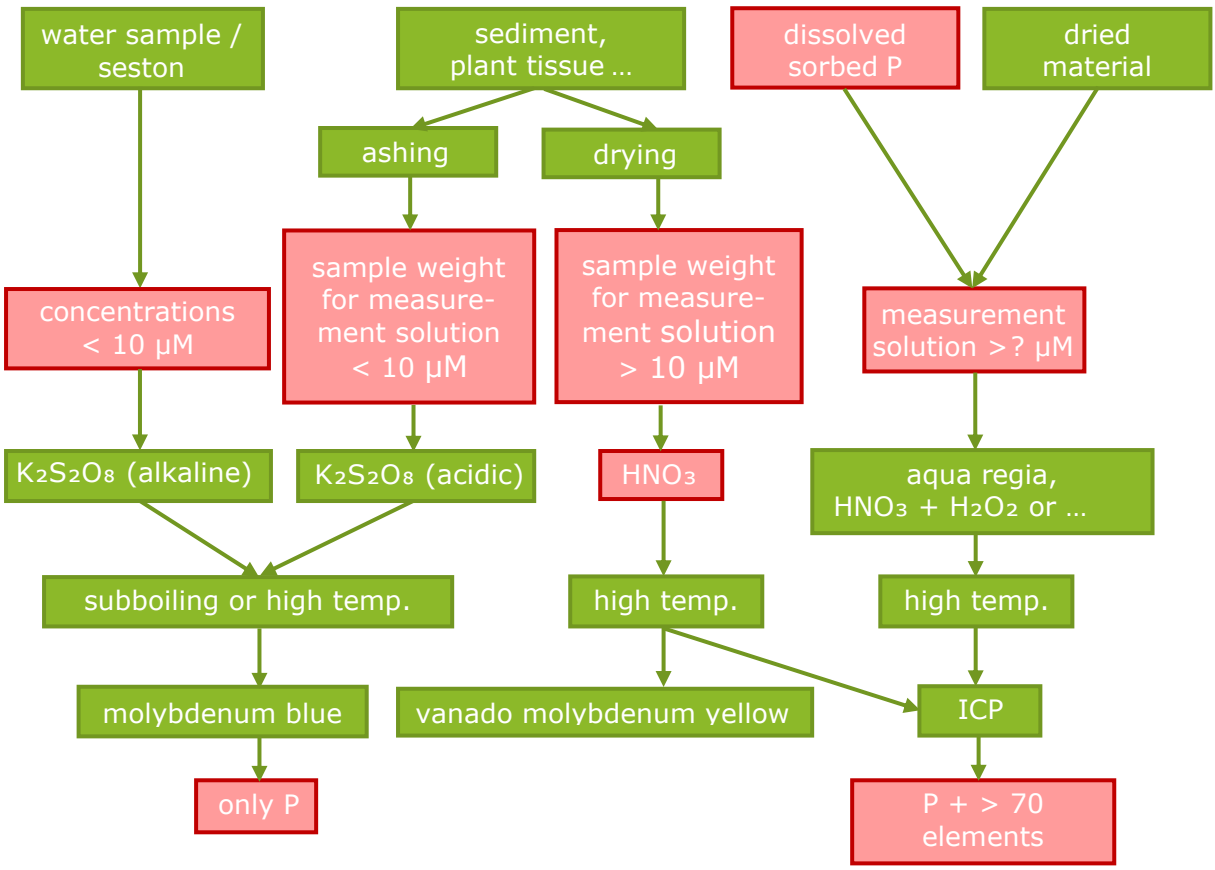

Figure 2.1-1 visualisation of the most common methods of digestion and matching analytical method (red: important decision criteria)

All material can be frozen (Fig. 2.1-2). Material with low water content is usually dried and stored by room temperature. High concentrations of organic matter of biomass can be the elimination criterion for persulfate digestion. Under these circumstances, incinerated material can be used for persulfate digestion.

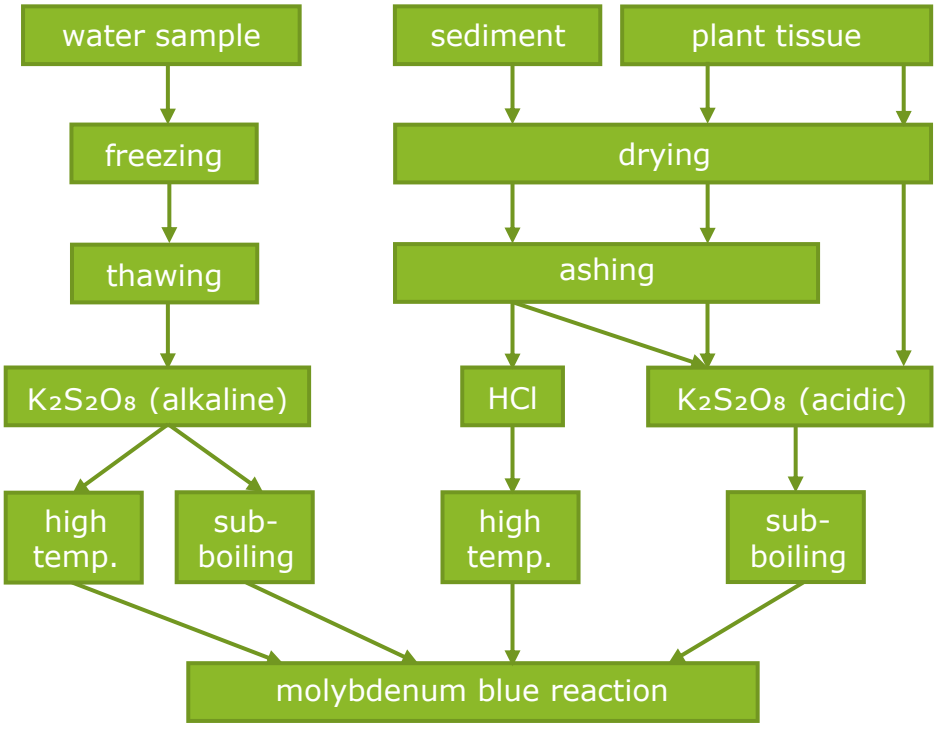

Figure 2.1-2 Sample preparation and digestion methods for photometric P determination

For citation: Zimmer D, Schumann R (*year of download*) Chapter 2.1 General Approach (Version 1.0) in Zimmer D, Baumann K, Berthold M, Schumann R: Handbook on the Selection of Methods for Digestion and Determination of Total Phosphorus in Environmental Samples. DOI: 10.12754/misc-2020-0001