

4. Digestions

4.4 Neutralisation

4.4.1 Neutralisation for increased iron concentrations

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When there are increased iron concentrations in soil and sediment samples the sample turns red after ashing, since the present yellow-brownish ferrihydrite resp. goethite dehydrates between 500 and 600 °C and converts to red hematite (Derie et al. 1976, Prasad et al. 2006, Schwertmann 1959). If, after sample digestion with acid persulfate or HCl, the sample is alkalized by ammonia (colour change of nitrophenol from colourless to yellow, subsequently neutralised with HCl, colour change to colourless), the iron precipitates as yellow flakes (probably ferrihydrite) after addition of ammonia (Fig. 4.4.1, Schwertmann et al. 2000, p. 73 ff.). Normally, these flakes dissolve after addition of HCl. Nevertheless, sample extracts have to be checked for small flakes after reaching the point of colour change (from yellow to colourless). If necessary, 1 or 2 drops of HCl have to be added to dissolve flakes completely. It must be ensured that no flake exists any longer, since iron is a strong sorbent for phosphorus. For this reason, phosphate concentrations could be underestimated by photometric P determination if P is precipitated and sedimented with Fe flakes. Furthermore, such flakes would block the hose system of the ICP-OES.



Fig. 4.4.1-1 Precipitation of the ferrihydrite after the digestion of a ferrous samples

References

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