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Determination of impurities in phosphoric acid by INAA

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The increasing demand for phosphate fertilizers accounts for approximately 90 % of global phosphoric acid use. In the manufacture of phosphoric acid, thermal and wet processes are used. The thermal process has been abandoned for the production of phosphate fertilizers, because of the amount of energy which is needed. Decomposition of phosphate minerals with an acid, mostly sulphuric acid, is the only economic way to produce phosphoric acid. Phosphate ores, mostly in the form of apatite, are of two major geological types. The principal deposits are sedimentary rocks found, e.g., in Morocco, China, Algeria, Jordan, USA. Significant igneous deposits are found, e.g., in Russia, South Africa, Brazil. Whatever their origin apatites can be highly substituted and have variable compositions. More than 25 elements are known or proposed to substitute in the apatite structure [1]. The potentially hazardous elements found in phosphate rocks include As, Cd, Cr, Hg, Pb, Se, U, and V. Some cause difficulties in the reaction system, whereas others, soluble in phosphoric acid, may end up in the product acid. Therefore, there is a need for analytical method capable of accurate determination of the above and other elemental impurities in phosphoric acid. Instrumental neutron activation analysis (INAA) is a method of choice due to its multielemental capability and favourably low detection limits for most of the above elements. The problem is, however, that irradiation of liquid acids is prohibited in most nuclear reactors for safety reasons. Therefore, a sample preparation procedure is required that leads to "solidified" phosphoric acid. For this purpose, Silva et al. [2] employed precipitation with calcium hydroxide and evaporation for determination of uranium in phosphoric acid.

In this work three other procedures were tested, namely (i) deposition of 100- μ L amounts of phosphoric acid onto disks of chromatographic paper Whatman 1 and sealing in polyethylene (PE) irradiation capsule without drying, (ii) precipitation of $\text{Ca}_3(\text{PO}_4)_2$ with CaCO_3 in a beaker, (iii) precipitation of $(\text{NH}_4)_3\text{PO}_4$ by reaction with ammonia vapours in a dessicator (isothermal distillation of NH_4OH [3]) directly in the PE irradiation capsules. The first procedure failed, because of breaking the PE capsules and consequent losses of their content on irradiation. In the second procedure, the activities of ^{47}Ca - ^{47}Sc and ^{49}Ca increased background in the gamma-ray spectra that impaired detection limits of trace elements, as well as blank values of Sr and several other elements due to impurities added with CaCO_3 . Thus, the third procedure appeared superior due to its simplicity and contamination-free nature. INAA results obtained with the third procedure are presented for four brands of phosphoric acid differing in purity (pro analysis and technical grades) and concentrations (75 %-85 %).

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References

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