



# Elemental analysis of two Egyptian phosphate rock mines by instrumental neutron activation analysis and atomic absorption spectrometry

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## ABSTRACT

Because of the increasing use of phosphate in industry world wide, it is interesting to investigate the elemental concentration in phosphate ores. The present work determines the elemental analysis of two different types of phosphate ores, containing different amounts of  $P_2O_5$  to check the level of the radioactive elements U and Th and of stable environmental pollutants like Cr, Zn in phosphate raw material. In addition, rare earth and other elements are analysed by the INAA method. This knowledge is found to be important in the assessment of possible radiological hazards to human health, since these materials may be used as building material or as phosphatic fertilizers and animal feed ingredients.

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## 1. Introduction

Nuclear analytical techniques, with their broad applicability to almost all matrix types and their exceptional sensitivity to many elements, are powerful tools for environmental research. Neutron activation analysis appears to be an attractive technique for determining heavy metals and rare earth elements present in environmental samples i.e. soils, sediments, plants, water, air particulates, etc. (Eissa et al., 1999; El Shershaby et al., 1999). Thermal neutron activation cannot be used for the  $\gamma$ -spectroscopic determination of phosphorus. The radioactive capture ( $n,\gamma$ ) reaction with the only isotope of P leads to the formation of  $^{32}P$  which is a pure  $\beta$  emitter (Alfassi, 1994). Therefore we used atomic absorption spectrometry to determine phosphorus together with other elements in phosphate samples from two Egyptian phosphate ores El-Sibaiya and El-Quasar. We used also the INAA technique to determine uranium and thorium, rare earth elements and other elements besides phosphorus.

## 2. Experimental

### 2.1. Samples preparation and irradiation

Ten phosphate ore samples collected from El-Sibaiya in the Nile Valley and El-Qusier at the Red Sea coast were prepared into finely ground homogenous material. They were crushed to a

diameter range of  $< 125 \mu m$  and  $> 63 \mu m$ . The crushed samples were dried at  $105^\circ C$  to constant weight. Polyethylene capsules filled with 100 mg of powder samples were then irradiated with the standard reference material with thermal neutrons at the university of Mainz Triga research reactor (100 KWth) for 6 hours with a flux of  $7 \times 10^{11} n/cm^2 s$ . The data were collected for various measurement and appropriate cooling times (see Table 1). To detect the elements with relatively short half-lives the samples were irradiated in the pneumatic system of the TRIGA Mainz research reactor in a neutron flux of  $1.6\text{--}1.8 \times 10^{12} n/cm^2 s$  for 1 and 5 min, respectively, and after cooling times of 4 min and 1 h the data were collected for 4 and 15 min, respectively (Kch-report-Triga Mainz, 1989; El-Taher, 2007).

### 2.2. Instrumentation

The low-level gamma-ray spectrometer used consists of a HPGe detector with its electronic circuit. The detector has the following specifications: energy resolution (FWHM) at 1.33 MeV Co-60 is 1.70 keV, Peak to Compton ratio Co-60 is 65.2, relative efficiency at 1.33 MeV Co-60 is 29.2%, energy resolution (FWHM) at 122 keV Co-57 is 686 eV, Operation bias voltage is +2000 V DC. The detector is connected to the following components: preamplifier, amplifier, ADC converter and MCA. The measurements were performed and analysed using the Intergamma Software produced by Intertechnique Deutschland GmbH, Mainz, Germany. Elemental contents were determined relative to certified reference materials which were irradiated and counted under the same conditions as those of the phosphate samples. The electronic dead time in all measurements was  $< 10\%$  and was automatically corrected by the Intergamma software.

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### 2.2.1. Atomic absorption spectrometry

Atomic absorption spectrometry (AAS) is a method for elemental analysis in solution (mainly). It is very sensitive, can detect different elements down to a few ppb or less. The analysis of the P can be carried out in the presence of many other elements. Atomic absorption spectrometry is one of the first modern commercial instruments for trace element analysis. Since its appearance, it has become the work horse of analytical laboratories. Atomic absorption spectrometry has many uses in different areas such as environmental analysis, industry, mining, clinical analysis and pharmaceuticals.

### 2.2.2. Basic principle

Atoms of different elements absorb characteristic wavelengths of light. Analysing a sample to see if it contains a particular element means using light from that element. For example, with lead, a lamp containing lead emits light from excited lead atoms that produce the right mix of wavelengths to be absorbed by any lead atoms from the sample. In AAS, the sample is atomized i.e. converted into ground state free atoms in the vapour state, and a beam of electromagnetic radiation emitted from excited lead atoms is passed through the vapourised sample. Some of the radiation is absorbed by the lead atoms in the sample. The amount of light absorbed is proportional to the number of lead atoms. During combustion, atoms of the element of interest in the sample are reduced to free, unexcited ground state atoms, which absorb light at characteristic wavelengths, as shown in Fig. 1.

To provide element specific wavelengths, a light beam from a lamp whose cathode is made of the element being determined is

passed through the flame. A device such as a photomultiplier can detect the amount of reduction of the light intensity due to absorption by the analyser, and this can be directly related to the amount of the element in the sample.

Phosphorus belongs to the most important nutrients, needed for the proper physiological development and growth of plants. The achievable economic benefit of agricultural production depends largely on the properly applied doses of artificial fertilizers during the growth period. It is important therefore to know the proper quantities of available phosphorus and other nutrients both in the soil and in the fertilizers. The average phosphorus content of the earth's crust is  $\approx 0.12\%$ . The determination is usually performed by chemical separation followed by spectrophotometry or gravimetry (Kolthoff and Sandell, 1950). Due to the complex matrix of phosphorites it was necessary to prepare and certify a reference material for comparison and to determine the accuracy for analytical quantity assurance of the measurements.

### 2.3. Methods

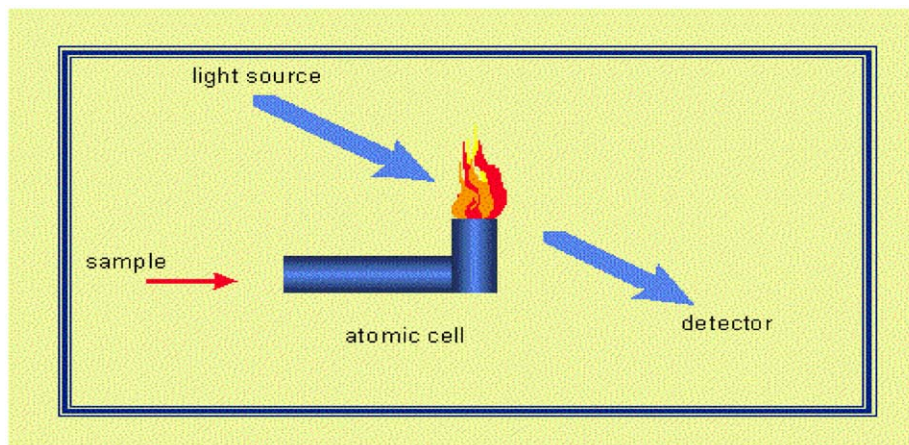
Two grams of the sample were digested with aqua regia (21 ml HCl conc.+7 ml HNO<sub>3</sub> conc.; both from Merck, p.a.) and refluxed for 2 h. After cooling the aqua regia solution was transferred into a graduated 100 ml flask, the flask was filled with water to the mark. Measurement of trace heavy metals was performed by atomic absorption analysis (Perkin Elmer AAS 2100). The elements Pb, Cd, Cu, Ni and Zn were determined with flame analysis, C<sub>2</sub>H<sub>2</sub>/Air. Ca, Mg and Cr were determined with flame analysis, C<sub>2</sub>H<sub>2</sub>/N<sub>2</sub>O. Hg was analysed with cold vapour AAS (reduction with (Sn Cl<sub>2</sub>) and K with flame photometry, C<sub>2</sub>H<sub>2</sub>/Air.

#### 2.3.1. Phosphorous photometric analysis

The digested samples were diluted by 1:100 with distilled water. Two ml of the diluted sample were pipetted into a 100-ml flask and the flask was filled to the mark with distilled water. Two ml ascorbic acid solution (10 g/100 ml Merck p.a) were added and the flask was shaken. Then 4 ml molybdate reagent (70 ml water, 250 ml sulphuric acid, 13 g hexammonium molybdate and a solution of 0.35 g potassium antimony (III) oxidetartrate) were added and mixed again. After 10 min the samples were measured at 880 nm (Photometer Perkin-Elmer Lambda 2).

**Table 1**  
Irradiation cycles.

Irradiation time	Cooling time	Counting time	Elements determined
1 min	5 min	4 min	Mg, Ca, V, Ti
5 min	1 h	15 min	Na, Mn, Sr, Ba, Eu
6 h	2 h	1 h	Na, K, Ga, As, La, Sm, U
6 h	14 d	8 h	Sc, Cr, Fe, Co, Zn, Rb, Zr, Nb, Sn, Ba, Cs, Ce, Nd, Eu, Yb, Lu, Hf, Th



**Fig. 1.** Operation principle of an atomic absorption spectrometer.

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