

## SHORT NOTES

### Crystallization of kaolinite at low temperature from an aluminosilicic gel

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KAOLIN mineral synthesis from amorphous aluminosilicates at low temperature and pressure has always encountered exceptional difficulties. In fact, no appreciable yield could be obtained below 250°C, though individual crystals were detected by electron diffraction (De Kimpe *et al.* 1961). More recent investigations have shown interesting methods of obtaining kaolinite from other sources. Poncelet and Brindley (1967) used montmorillonite containing aluminum hydroxy-polymers and observed that crystallization into kaolinite could be performed at 220°C in water, whereas more acid conditions were required with decreasing temperatures. De Kimpe and Fripiat (1968) studied the formation of kaolinite from zeolites which, in the first experiments, were hydrogen-exchanged. The subsequent treatment under

acid conditions during 10 days at 175°C gave rise to clay mineral formation.

In the present investigation, an aluminosilicic gel, obtained by simultaneous hydrolysis of ethyl orthosilicate and aluminum isopropoxide, has been used with an initial composition of  $Al_2O_3/Al_2O_3 + SiO_2 = 40$  per cent. The experiments were conducted in sealed glass tubes at 175°C in the presence of a 0.1 N NaOH solution and with continuous agitation. The presence of the gel in this solution very rapidly induced an acid reaction and after one day of ageing, the pH decreased to 6.3 and thereafter remained in the range 6.0-6.5 during the entire experiment. After 8 days of reaction, traces of kaolinite were already detectable by X-ray diffraction. Figure 1 shows the X-ray diffraction pattern (above) and the infrared

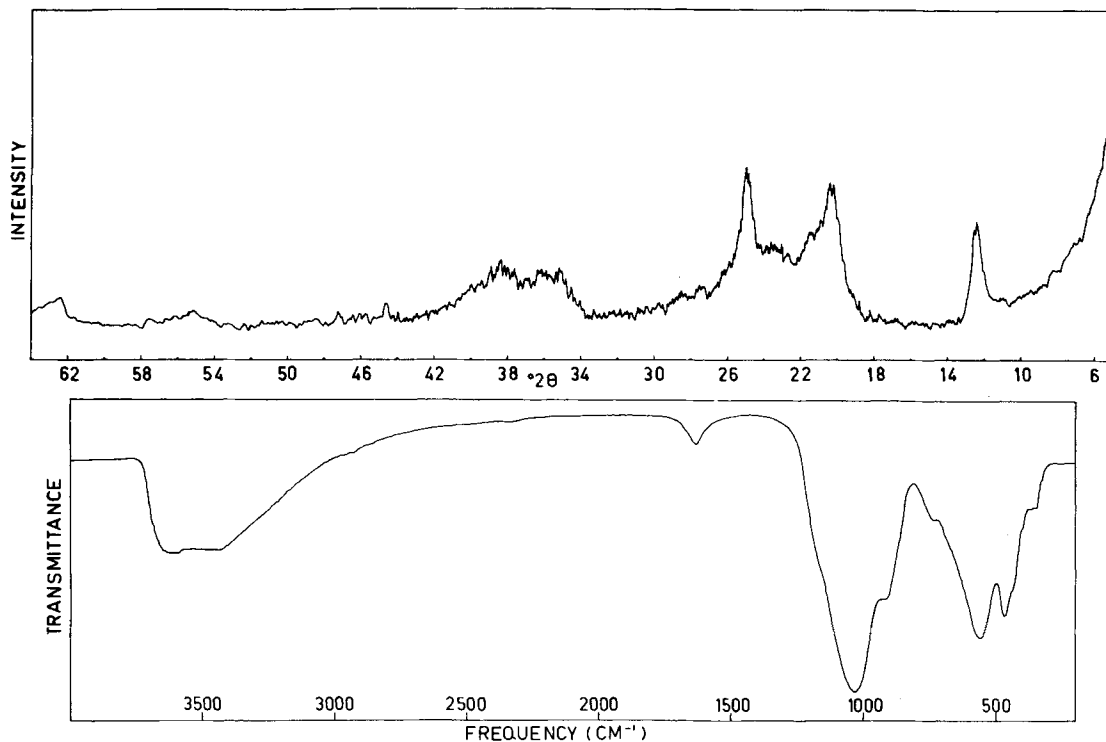


Fig. 1. X-ray diffraction pattern (above) and i.r. spectrum (below) of the kaolinite obtained after 16 days of ageing.

spectrum (below) of the product obtained after 16 days of ageing. That the reaction is still not complete is indicated by the broad band around  $22^\circ 2\theta$  on the X-ray trace, ( $\text{CuK}\alpha$  radiation). This appears to be the first time that the crystallization of kaolinite is realized at such a low temperature from an amorphous material and in an almost neutral solution. It is thought that the transformation occurs through an undetectable "zeolitic" phase, which readily forms at high pH and facilitates the subsequent crystallization into kaolinite under neutral conditions. The direct transformation from the gel into a clay mineral using an acid solution or water is not feasible as it has been shown (De Kimpe, 1967) that under these conditions, the final products are pseudo-boehmite and an amorphous aluminosilicate with a different composition.

Further experiments are in progress to investigate in more detail this reaction, and a complete report will be given later.

Part of this study has been performed at the Laboratoire de Physico-Chimie Minérale, University of Louvain, Belgium, where the author was working as an assistant under the Ministère de l'Éducation Nationale.

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## Boron contamination from platinum crucibles\*

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### INTRODUCTION

THE PAST few decades have seen the accumulation of a voluminous literature dealing with boron concentration in rock and soil materials. These numerous studies, which have been both experimental and empirical in nature, have been largely concerned with the role of boron as a nutrient in soils and the role of boron as a paleosalinity index in argillaceous sediments. The literature of the past few years suggests that the number of laboratories making such investigations continues to increase. While the analytical techniques used in these investigations vary considerably, colorimetric methods employing sample fusion in platinum crucibles appear to be quite common.

While platinum is widely recognized as a nearly ideal "inert" metal for various types of reaction vessels, it is known to be slightly soluble in extreme acid and alkaline environments. Thus, it is to be expected that trace quantities of impurities can be freed from the metal during alkaline fusion. However, to discover that enough boron can be leached from a new platinum crucible during sample fusion to make even semi-quantitative analysis for trace quantities of the element impossible, may be somewhat surprising. That such contamination is possible was recently discovered in our laboratory.

A few years ago, with the purchase of new platinum crucibles for boron analysis of rock samples, a contami-

nation was observed which appeared to have come from the platinum (A. A. Levinson, unpublished report). Recently, when additional new crucibles were purchased, it was decided to make a deliberate effort to determine the nature and amount of such contamination. The results of that study are presented here in the hope that it may alert other analysts using fusion techniques of a possible source of analytical error.

Since our analytical method requires fusion of 1 g  $\text{Na}_2\text{CO}_3$  with 20-50 mg of sample, this study involved blank fusion of that amount of  $\text{Na}_2\text{CO}_3$  in the new crucibles. Six of the new crucibles were chosen randomly from the set and the melt from each analyzed for boron after various fusions. Melts from two old, reliable crucibles were used for control blanks with each analysis.

### RESULTS

The amount of boron leached from each of the six crucibles during various fusions is shown in Table 1. During the first fusion, all the crucibles yielded more boron than can be accurately measured by our method (maximum about  $10\mu\text{g}$ ). Comparison with prepared standards indicated 20-30  $\mu\text{g}$  B was present in each melt. All the crucibles showed an order of magnitude decrease in the amount of boron leached during the second and successive fusions.

The data of Table 1 are more meaningful when it is noted that, for the sample size used in our analysis (20-50 mg),  $1\mu\text{g}$  B leached from the platinum would represent a 20-50 ppm error in analysis. Such error

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