SEPARATION OF ALUMINUM PHOSPHATE MINERALS FROM KAOLINITE BY SELECTIVE SOLUTION

By Irving May and Roberta Smith

Trace Elements Investigations Report 556

UNITED STATES DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY

Copy 1

IN REPLY REFER TO:

acc
em

UNITED STATES
DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY
WASHINGTON 25, D. C.

AEC-237/6 November 2, 1955

Mr. Robert D. Nininger, Assistant Director
Division of Raw Materials
U. S. Atomic Energy Commission
Washington 25, D. C.

Dear Bob:

Transmitted herewith are three copies of TEI-556, "Separation of aluminum phosphate minerals from kaolinite by selective solution," by Irving May and Roberta Smith, September 1955.

We are asking Mr. Hosted to approve our plan to submit this report for publication in the Journal of the Association of Official Agricultural Chemists.

Sincerely yours,

for John H. Eric
W. H. Bradley
Chief Geologist

Chemistry

This document consists of 8 pages.
Series A.

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

SEPARATION OF ALUMINUM PHOSPHATE MINERALS FROM KAOLINITE BY SELECTIVE SOLUTION*

By

Irving May and Roberta Smith

September 1955

Trace Elements Investigations Report 556

This preliminary report is distributed without editorial and technical review for conformity with official standards and nomenclature. It is not for public inspection or quotation.

*This report concerns work done on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

USGS - TEI-556

CHEMISTRY

Distribution (Series A)	No. of copies
Argonne National Laboratory	1
Atomic Energy Commission, Washington	2
Division of Raw Materials, Albuquerque	1
Division of Raw Materials, Butte	1
Division of Raw Materials, Casper	1
Division of Raw Materials, Denver	1
Division of Raw Materials, Hot Springs	1
Division of Raw Materials, Ishpeming	1
Division of Raw Materials, Phoenix	1
Division of Raw Materials, Plant City	1
Division of Raw Materials, Salt Lake City	1
Division of Raw Materials, St. George	1
Division of Raw Materials, Washington	3
Exploration Division, Grand Junction Operations Office	1
Grand Junction Operations Office	1
Technical Information Service, Oak Ridge	6
Tennessee Valley Authority, Wilson Dam	1
U. S. Geological Survey:	
Fuels Branch, Washington	1
Geochemistry and Petrology Branch, Washington	40
Geophysics Branch, Washington	1
Mineral Deposits Branch, Washington	2
E. H. Bailey, Menlo Park	1
A. L. Brokaw, Grand Junction	1
N. M. Denson, Denver	1
W. L. Emerick, Plant City	1
R. L. Griggs, Albuquerque	1
M. R. Klepper, Spokane	1
A. H. Koschmann, Denver	1
R. A. Laurence, Knoxville	1
J. D. Love, Laramie	1
L. R. Page, Washington	1
Q. D. Singewald, Beltsville	1
A. E. Weissenborn, Spokane	1
TEPCO, Denver	2
TEPCO, RPS, Washington, (including master)	2
	85

CONTENTS

ILLUSTRATION

TABLES

SEPARATION OF ALUMINUM PHOSPHATE MINERALS FROM KAOLINITE BY SELECTIVE SOLUTION

By Irving May and Roberta Smith

ABSTRACT

Wavellite, crandallite, and millisite are completely dissolved by boiling samples for 20 minutes with 1+1 hydrochloric acid; only 1 percent of the aluminum present in kaolinite is dissolved. This method of decomposition can therefore be used for differentiating the aluminum present in wavellite, crandallite, and millisite from that in kaolinite in samples from the aluminum phosphate zone. This method should prove valuable in prospecting and beneficiation studies.

INTRODUCTION

Solubility studies recently made in connection with the determination of aluminum in aluminum phosphate zone samples appear to be of sufficient general interest to report here, as part of a program conducted by the U. S. Geological Survey on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

The samples are from Florida, and aluminum is present as aluminum phosphates (wavellite, crandallite, and millisite) and as kaolinite. The problem was to determine the amount of aluminum present as phosphates but not to include any present in the kaolinite.

Previous experience with various solution procedures for the determination of uranium in such samples suggested that a brief digestion with 1+1 HCl would dissolve the phosphate minerals (Grimaldi and Guttag, 1954). We

therefore first studied the effect of heating kaolinite with 1+1 HCl.

SOLUBILITY STUDIES ON KAOLINITE

One-gram portions of a finely ground standard sample of kaolinite 1/ were heated with 20-ml portions of 1+1 hydrochloric acid for varying periods. The digestions were made in resistant glass beakers on a hot plate. After addition of the acid to the clays, the acid was quickly brought to a boil and kept boiling for periods of 15, 30, 60, and 120 minutes. Immediately after the boiling period, the samples were diluted with water, filtered, and washed with 1+9 hydrochloric acid. Blanks were run in both glass and platinum to serve as checks on the reagents and the possibility of leaching aluminum from the glass vessels. Aluminum was determined on aliquots of the solutions spectrophotometrically with ferron reagent (Delevaux et al., 1954). The results of this experiment are shown in table 1.

For the time range studied, the extent of the leaching of aluminum from kaolinite by hot 1+1 hydrochloric acid is a linear function of the digestion time (fig. 1). A 20-minute boiling period would dissolve only 1.2 percent of the aluminum present in kaolinite. The data obtained for the blanks show that an insignificant amount of aluminum would dissolve from resistant glass in 20 minutes. This boiling period was therefore chosen for the decomposition of the aluminum phosphate minerals in the leached zone samples.

STUDIES ON ALUMINUM PHOSPHATE ZONE SAMPLES

Samples weighing 0.33 gram were boiled with 20 ml of 1+1 hydrochloric acid in resistant glass beakers for 20 minutes. The solutions were then diluted with water, filtered, and the residues washed with 1+9 hydrochloric

1/ API sample H-4, kaolinite from Macon, Ga.

Table 1.--Leaching of aluminum from kaolinite by 1+1 hydrochloric acid.

Boiling period (min)		Wt. Al_2O_3 dissolved (g)	Fraction Al_2O_3 dissolved 1/
15		0.0045	0.011
30		0.0068	0.017
60		0.016	0.040
120		0.029	0.073
60	(Blank in platinum)	0.00005	--
60	(Blank in glass)	0.00007	--

1/ Al_2O_3 content of kaolinite taken as 40 percent; therefore Al_2O_3 taken in 1 g sample = 0.40 g; fraction Al_2O_3 dissolved = $\frac{\text{Wt. } Al_2O_3 \text{ dissolved}}{0.40}$

acid. Aluminum was determined on an aliquot of the filtrate with ferron reagent.

In order to test whether the aluminum phosphate minerals had been completely decomposed, 8 out of 24 residues from the hydrochloric acid digestion were analyzed for phosphorus spectrophotometrically as the molybdovanadophosphate complex. The residue from the API kaolinite standard, which was run as a control, contained 0.2 mg of P_2O_5; all of the sample residues contained less than 0.6 mg of P_2O_5. As the P_2O_5 content of the original samples exceeded 10 percent, it may be concluded that there was a quantitative decomposition of the phosphate minerals. The analytical data obtained on these samples are summarized in table 2.

We wish to acknowledge the assistance of Zalman S. Altschuler on the mineralogical problems connected with this study.

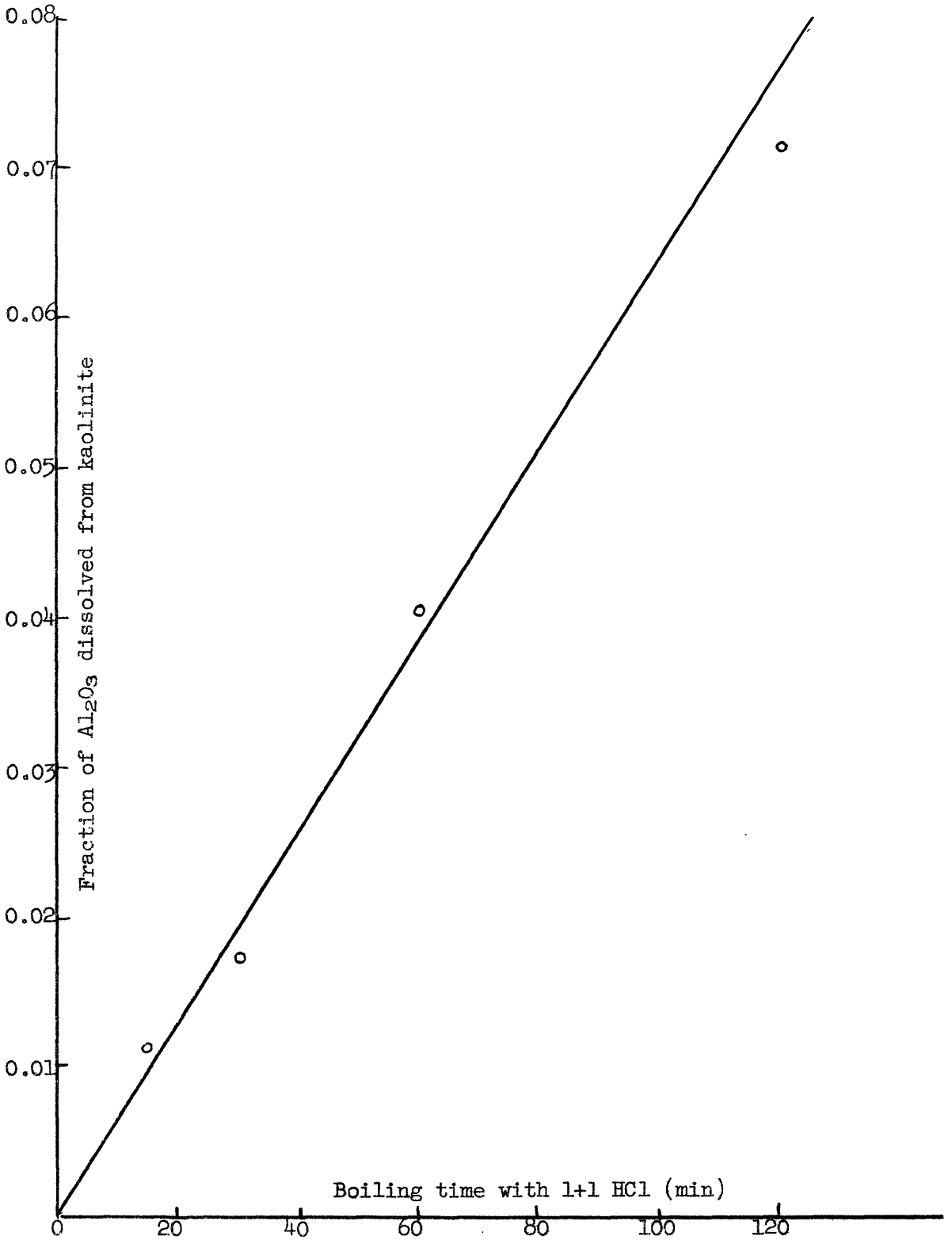

Figure 1.--Leaching of aluminum from kaolinite by 1+1 hydrochloric acid.

Table 2.--Analytical results on aluminum phosphate zone samples.

Sample	Percent Al_2O_3 (in entire sample)	Percent Al_2O_3 (leached by 1+1 HCl) 1/	Percent P_2O_5 (in residue from leaching with 1+1 HCl) 1/
147	15.6	11.1	0.16
150	18.3	14.0	.18
153	25.0	18.6	.18
159	15.6	12.9	.14
165	4.8	2.8	--
168	13.3	11.6	.10
174	10.5	9.3	--
177	23.0	20.4	.10
180	14.4	10.2	.08
186	21.7	18.2	.09
192	3.2	2.5	--
198	18.7	12.4	--
204	24.7	12.2	--
210	21.3	18.0	--
219	17.5	6.7	--
225	2.2	2.3	--
228	25.0	20.4	--
231	9.7	8.9	--
237	19.3	16.4	--
243	23.0	15.4	--
246	20.7	18.4	--
252	18.7	14.0	--
258	17.0	11.4	--

1/ Calculated as percentage of original sample.

LITERATURE CITED

Delevaux, Maryse, Smith, Roberta, and Grimaldi, F. S., 1954, The photometric determination of aluminum in phosphate materials with ferron: U. S. Geol. Survey TEI-450, issued by U. S. Atomic Energy Commission, Tech. Inf. Service, Oak Ridge, Tenn.

Grimaldi, F. S., and Guttag, Norma, 1954, Short routine direct method for the fluorimetric determination of uranium in phosphate rocks, in Grimaldi, F. S., et al., Collected papers on methods of analysis for uranium and thorium: U. S. Geol. Survey Bull. 1006, p. 105-109.