

[CONTRIBUTION FROM THE CHEMICAL LABORATORY, UNIVERSITY OF MISSOURI, AND THE NEW MEXICO NORMAL UNIVERSITY]

## "Glorieta" Monazite

By O. B. MUENCH

Several years ago, Dr. A. C. Lane secured a large and almost perfect crystal of monazite from P. Krieger, who had obtained it from someone near Glorieta, New Mexico, and was under the impression that originally it had come from a deposit in that neighborhood. A small fragment of this crystal was sent by Lane to Dr. F. Hecht in Vienna. The microanalysis made by F. Hecht and Edith Kroupa is reported on page 44 of "The Report of the Committee on the Measurement of Geologic Time," of the National Research Council, April, 1935.

The Committee on the Measurement of Geologic Time is interested in obtaining a quantity of "thorium-lead," free from "uranium-lead" and common lead, for an atomic weight determination and other experimental purposes. Mr. Rufus C. Little of Albuquerque, who was in possession of twenty to thirty pounds of the monazite, kindly agreed to furnish specimens of the mineral which he had personally collected from the old Cribbenville Mica Mine near Petaca, New Mexico. It is quite possible that the crystal which Krieger had obtained came from the same deposit.

Monazite is suitable material to use for an age determination by the lead-uranium method. This particular monazite sample consisted of large pieces (crystal fragments, etc.) and besides smaller lumps, some fines. The fines were separated and not used in this analysis. The largest piece weighed 252 g. and all the better pieces used in this determination weighed over 100 g. The material was fresh, tan to reddish brown in color, most of it clear of any other adhering mineral and showing hardly any alteration.

The larger pieces were broken in a clean iron mortar and then finely powdered in a clean agate mortar to pass through an 80-mesh sieve. Precautions were taken to avoid contamination, especially by lead, not only during the preparation of the sample, but throughout the entire analysis.

### Analysis

Fenner<sup>1</sup> has worked out a detailed method for the analysis of monazite. His method with several modifications was used in the decomposi-

tion of the sample and the lead determination. The details of analysis will not be included in this paper. Hecht<sup>2</sup> makes the statement, "By solution of monazite in sulfuric acid, one would find too little lead." A. C. Lane adds to this the following, "Possibly such an error has crept into many analyses heretofore."

The solution of the monazite was accomplished according to the method of Fenner by heating the powdered sample in a platinum dish for about six hours with a mixture of sulfuric and hydrofluoric acids. This treatment usually disintegrates the sample, but occasionally it was found that all was not decomposed completely. This residue was given further treatment till all of it was decomposed.

In the lead determinations, care was taken to test all residues and filtrates for traces of lead and add these when necessary. This is important, for in the hydrofluoric acid method of decomposition, much of the lead may be found in the residue after hydrofluoric and sulfuric acid treatment and even after the extraction with ammonium acetate. There is very little possibility of any lead being lost in this determination. All reagents used in this analysis were tested and found lead free. The lead was determined as the sulfate.

The iodate method as described with references in previous papers<sup>3</sup> was followed for the thorium determinations.

The uranium was determined as the pyrophosphate,<sup>3</sup> also described in detail in previous papers.

### RESULTS OF ANALYSES

Sample, g.	Lead	
	PbSO <sub>4</sub> , g.	Lead, %
30.0000	0.1475	0.336
30.0000	.1502	.342
30.0000	.1498	.341
30.0000	.1490	.339
	Average	.339
Sample, g.	Thorium	
	ThO <sub>2</sub> , g.	Thorium, %
2.0035	0.1707	7.49
2.0024	.1720	7.54
3.0044	.2615	7.55

(2) Report of the Committee on the Measurement of Geologic Time, Natl. Research Council, p. 45, April 27, 1935.

(3) Muench, THIS JOURNAL, 59, 2269 (1937).

(1) Fenner, *Am. J. Sci.*, 16, 369 (1928).

Thorium (Concluded)		
Sample, g.	ThO <sub>2</sub> , g.	Thorium, %
2.5011	.2131	7.49
2.0015	.1700	7.46
2.0550	.1762	7.52
	Average	7.50

  

Uranium		
Sample, g.	(UO <sub>2</sub> ) <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	Uranium, %
3.0044	0.0050	0.109
2.5011	.0038	.101
2.0015	.0033	.109
	Average	.106

  

Loss on ignition	0.838%
Loss at 110°, three hours	.14%

There were no indications of ordinary lead mineral associated with the sample. If we assume the absence of ordinary lead and make use of the conversion factor 0.36 for thorium, the lead-uranium ratio is

$$\frac{0.339}{0.106 + 0.36 \times 7.50} = 0.1208$$

The approximate age of the mineral is

$$\frac{0.121 \times 1.15 \text{ million years}}{1.57 \times 10^{-4}} = 886 \text{ million years}$$

With so old a mineral, the more exact or "logarithmic" formula for calculating age gives a materially different result from that found by the above "approximate" formula, and should be used in calculating the age of this material. Age, based on formula

$$\frac{\log(U + 0.36\text{Th} + 1.155\text{Pb}) - \log(U + 0.36\text{Th})}{6.6 \times 10^{-6}}$$

million years = 858 million years

As a pilot analysis for this work, Hecht made a complete microanalysis of a small piece from the crystal in Dr. Lane's possession. His results show, 9.36% thorium, 0.392% lead and give no figures for the uranium. Hecht obtains an age of 800 million years from his determination.

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### Summary

This monazite is not a suitable source for pure "thorium-lead," as the analysis shows evidence of 0.106% uranium. The other analytical results are, 7.50% thorium and 0.339% lead. The age of the monazite is 858 million years, placing it in the Pre-Cambrian Age. Hecht, from his pilot analysis, obtains an age of 800 million years. It is probable but by no means certain that this material and the material Hecht used in his analysis came from the same deposit.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY, LYNCHBURG COLLEGE]

## The Effect of the Triple Bond on the Rate of Reaction of $\omega$ -Chlorides with Potassium Iodide in Absolute Acetone

BY M. J. MURRAY

Conant and his students<sup>1</sup> have studied the rates of reaction of a variety of organic chlorides with potassium iodide in absolute acetone. The only triple bond compound they reported on, however, was CNCH<sub>2</sub>Cl. Truchet<sup>2</sup> has shown qualitatively that chlorine attached directly to a triple bond carbon is very unreactive toward sodium iodide in acetone.

The present investigation was made because no study has been reported showing the effect of the acetylenic linkage on the reactivity of chlorine

in the  $\omega$ -position. The series selected was C<sub>6</sub>H<sub>5</sub>C≡C(CH<sub>2</sub>)<sub>n</sub>Cl, all members of which were prepared from phenylacetylene. Cinnamyl chloride was available for comparison of the effect of the ethylenic linkage.

The method of measuring the velocity of the reaction and of calculating the constant for the reaction was essentially that given in detail by Conant and students.

The results are summarized in Table I. It is seen that in this series of acetylenic chlorides there is a definite alternation of reactivity, the order with respect to the triple bond being  $\alpha > \gamma > \beta$ . This order of reactivity is the same as that found

(1) (a) Conant and Kirner, *THIS JOURNAL*, **46**, 232 (1924); (b) Conant and Hussey, *ibid.*, **47**, 476 (1925); (c) Conant, Kirner and Hussey, *ibid.*, **47**, 488 (1925).

(2) Truchet, *Ann. chim.*, [10] **16**, 309 (1931).