expensive to make than the leaf-spring type but the advantages gained in the ease and speed of operation will soon offset the original cost.

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THE CHEMICAL FORMULA OF CLARKEITE1

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The mineral clarkeite in pegmatites from Spruce Pine, N. C., when first described by Ross, Henderson, and Posnjak (1931) was given the formula $RO \cdot 3UO_3 \cdot 3H_2O$. In their paper (p. 214) they state: "Alkalies are the essential RO constituents and sodium predominates over potassium. Analysis No. 1 is closely similar to No. 2, the only essential difference being a slight excess of calcium in the former. The quantity of lead and alkalies is nearly the same in the two analyses and the uranium contents agree very closely. Both analyses contain only small quantities of iron and aluminum oxides but there is a difference in the water content." They also give a diagram of an x-ray picture which agrees with one obtained by Frondel of the same material. A film (Table 1) obtained by us from a sample received from the Trace Elements Laboratory of the U. S. Geological Survey which is labelled TE-1334 also agrees within reasonable limits.

The author was able to synthesize clarkeite in a number of experiments (Gruner, 1952) from uranyl nitrate solutions and CaCO₃ or CaO. While the x-ray patterns unquestionably agreed with the natural material there was reason to suspect judging by the orange buff color that the synthetic clarkeite was mechanically mixed with uranyl hydroxides and possibly uranyl silicates and carbonates. Therefore, no analyses were made of these synthetics. It is evident, however, that no Pb, Na, or other alkalies are contaminants in them, as they were absent in the experiments.

Among our older chemical reagents we found a container with orange buff $Na_2U_2O_7$ (Uransaures Natrium) made by A. C. F. Kahlbaum, Berlin. This material gave a very faint x-ray pattern of clarkeite. But on heating, the pattern improved and reached near-perfection after heating the sample five hours over the Bunsen burner at low redness. Table 1 gives the spacings and intensities of its lines compared with those of the natural and synthetic products. From the similarity of them and the

¹ Research done in connection with a contract of Division of Raw Materials of the U. S. Atomic Energy Commission.

Table 1. X-Ray Data for Clarkette and Na₂U₂O₇ Unfiltered Fe-radiation, Camera Radius=57.3 mm.

Clarkeite U.S.G.S. TE 1334		Clarkeite Synthetic JO-155		Na ₂ U ₂ O ₇ Heated to Low Redness A-105a	
d	I	d	I	d	I
5.75	5	5.76	5	5.80	7
3.66	f	3.66	f	3.66	1
3.48	f	3.46	1	3.49	1
3.339	6	3.337	6	3.323	8
3.156	10	3.138	10	3.156	10
2.920	2	2.912	4	2.945	3
2.683	5	2.662	5	2.689	4
2.435	1	2.423	4	2.444	2
2,164	1	2.152	1	2.165	1
		2.012	1	2.029	2b
1.960	6	1.942	7	1.965	7
1.845	5b	1.838	8	1.856	8
				1.694	2
1.673	1	1.674	1		
		1.650	1	1.668	2
1.633	4	1.619	3	1.635	6
1.550	f	1.556	2b	1.557	2b
1.526	f	1.517	1	1.535	2
				1.484	1
		1.443	1	1.458	1
				1.415	1
1.386	1	1.379	2	1.393	3
1.350	1	1.344	1	1.352	1
1.275	2b	1.259	1	1.273	2 b

b=broad line.

fact that shrinkage on heating of these compounds is almost negligible, it is evident that they have essentially the same structures and that H₂O is not required in them though always present in the material unless heated to a relatively high temperature. As there was the possibility that the Na compound might be Na₂UO₄, an analysis for Na₂O was made on the heated sample in the Rock Analysis Laboratory of the University of Minnesota. It gave 8.8%. K₂O was about 0.1%. Theoretically, the percentages of Na₂O are:

in
$$Na_2U_2O_7 = 10.36$$

in $Na_2UO_4 = 18.78$

It is obvious that the analysis agrees fairly well with Na₂U₂O₇. Complete agreement had not been expected in a complex compound of U prepared more than 30 years ago. The corresponding Ca synthetic uran-

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ate is CaU₂O₇·xH₂O. The natural mineral is essentially a Na-Ca uranate. K and Pb are considered "impurities" in the sense that their ions are too large to occupy Ca or Na positions. They could, however, be in the H₂O positions if these are more or less fixed. The formula would be (Na, K)_{2-2x}(Ca, Pb)_xU₂O₇·yH₂O. The material of Ross, Henderson, and Posnjak indicates that they probably had a mixture of U-minerals. Also, the dark brown color of their material as compared with the lighter colored synthetics suggests this.² Such mixtures are almost the rule instead of the exception in U-minerals, as more recent work has indicated. Ross, Henderson, and Posnjak state that clarkeite is of hydrothermal origin, that is, of a late stage in pegmatite formation. Our synthesis at 258° C certainly supports this claim. In many experiments which we have made below 100° C we have never obtained the mineral.

The following recent paper dealing with synthetic diuranates has come to the writer's attention too late to be included in the discussion (Hoekstra, Henry R., and Katz, Joseph J., Studies on the alkaline earth diuranates: *Jour. Am. Chem. Soc.*, 74, 1683 (1952)).

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² As the minerals are extremely fine grained, indices are difficult to obtain. Ca-clarkeite after heating has a mean index above 2.00 while that of Na-clarkeite is around 1.84.

THE INDIRECT DETERMINATION OF β AND 2V

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The following method is suggested for the indirect determination, using the polarizing microscope, of β and 2V for crystals capable of being mounted so that they can be rotated around the β direction. β and 2V are determined concurrently with the indirect determination of α and γ by the method of Wood and Ayliffe (*Phil. Mag.* (1936) (7) 21, 324). Hippuric acid is cited as an example.

The apparatus used was a rotation apparatus in which the crystal, 0.5-2 mm. long, mounted on a needle with β lying in the direction of the needle, could be rotated through 360° while immersed in a liquid held in a glass cell (see Hantshorne and Stuart, Crystals and the Polarising Microscope, p. 214). The crystal was observed through an 8 mm, objective