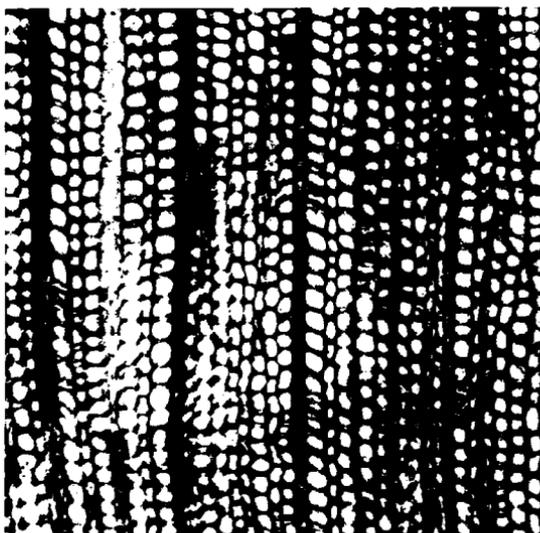


OKLAHOMA GEOLOGICAL SURVEY  
THE UNIVERSITY OF OKLAHOMA  
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# OKLAHOMA GEOLOGY NOTES



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## *Cover Picture*

### *Callixylon whiteanum*

The cover picture is a photomicrograph of a transverse section of petrified wood, *Callixylon whiteanum* Arnold, from the Woodford Formation in Atoka County (T. 3 N., R. 9 E.), southeast of Wapanucka. The section was taken from a large fragment of tree trunk, the size of which indicated that the living tree had a diameter of 4.5 feet.

The occurrence of *C. whiteanum* in Oklahoma was discussed by L. R. Wilson in 1958 (Okla. Geology Notes, vol. 18, p. 172-177) in a short paper in which the photomicrograph was originally published. Fragments of *Callixylon* are widely distributed over an area southeast of Ada. One specimen, found near Jesse, southeastern Pontotoc County, adorns the entrance to East Central State College at Ada.

The Woodford beds in which the *Callixylon* fragments are found are marine, and the abundance of these fossil trees suggests the nearness of a landmass. However, no other geologic evidence has yet been found to indicate the location of such a nearby landmass during Woodford time.

## Bibliography of Louise Jordan

Dr. Louise Jordan (1908-1966) was petroleum geologist of the Oklahoma Geological Survey for more than eleven years before her tragic death in November 1966. The following bibliography is based upon her own compilation, which she kept posted from year to year. To the original compilation have been added numerous short journalistic and review items that she contributed to *Oklahoma Geology Notes* but that she did not deem suitable for inclusion in her professional bibliography. The entries are listed chronologically by years and alphabetically by titles within years. Any errors or omissions brought to our attention will be greatly appreciated.

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## PICKERINGITE IN OKLAHOMA

RAYMOND L. KERNS, JR.

In October 1964, Carl C. Branson visited an abandoned coal mine in Cimarron County, Oklahoma, and there observed and collected a white, fibrous, crystalline mineral. The mineral occurred as encrustations and crystalline clusters on the floor of a drift driven into a hillside in NE $\frac{1}{4}$  sec. 19, T. 4 N., R. 1 ECM., on the Julius Burke property.

The specimen was submitted to the X-ray diffraction laboratory in the School of Geology at The University of Oklahoma. X-ray diffraction, differential thermal analysis, and chemical analyses by X-ray-fluorescence spectroscopy were utilized to identify and characterize the mineral.

### X-RAY DIFFRACTOMETRY

The sample was prepared for X-ray-diffraction analysis by grinding in an agate mortar and pestle to -115 mesh. The powder was then sprinkled onto a vaseline-coated glass slide and allowed to stand for 5 minutes until the powder was firmly bonded to the slide. The slide was then inverted and tapped gently on the reverse side and edge in order to remove excess material. This randomly oriented material was then subjected to X-ray-diffraction analysis, using a Siemens X-ray machine. The copper-target X-ray tube was operated at 35 kilovolts and 18 milliamps. A 1-mm collimating slit and a 0.1-mm receiving slit were utilized. A nickel filter was used to enhance the intensity of  $\text{CuK}\alpha$  with respect to  $\text{CuK}\beta$ .

The diffractometer was operated at a speed of 1 degree 2-theta per minute and the recording-chart drive was maintained at 1 cm per minute. A scintillation counter was used as the detector and  $\text{CuK}\alpha$  radiation was further enhanced with respect to background radiation and  $\text{CuK}\beta$  by selecting appropriate baseline and window settings in the pulse-height analyzing circuit. The diffractometer scan was made from 5 degrees 2-theta to 90 degrees 2-theta.

The X-ray-diffraction data are listed in table I, in which the  $d$  values of the sample are compared with the  $d$  values of pickeringite  $[\text{MgAl}_2(\text{SO}_4)_2 \cdot 22\text{H}_2\text{O}]$ , halotrichite  $[\text{Fe}''\text{Al}_2(\text{SO}_4)_2 \cdot 22\text{H}_2\text{O}]$ , and alunogenite  $[\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}]$ , as given by the ASTM powder diffraction file (Smith and others, 1966). The X-ray diffractogram is illustrated in figure 1,  $d$  values and Miller indices of only the more prominent diffraction peaks being labeled.

Pickeringite proves to be the best match for the diffraction data obtained for the sample. Upon the basis of X-ray diffractometry, positive identification of the sample as pickeringite is only suggested, but not definitely established.

### CHEMICAL DATA

Chemical analysis by X-ray-fluorescence spectrometry provided quantitative information on the percentages of the oxides of magnesium,

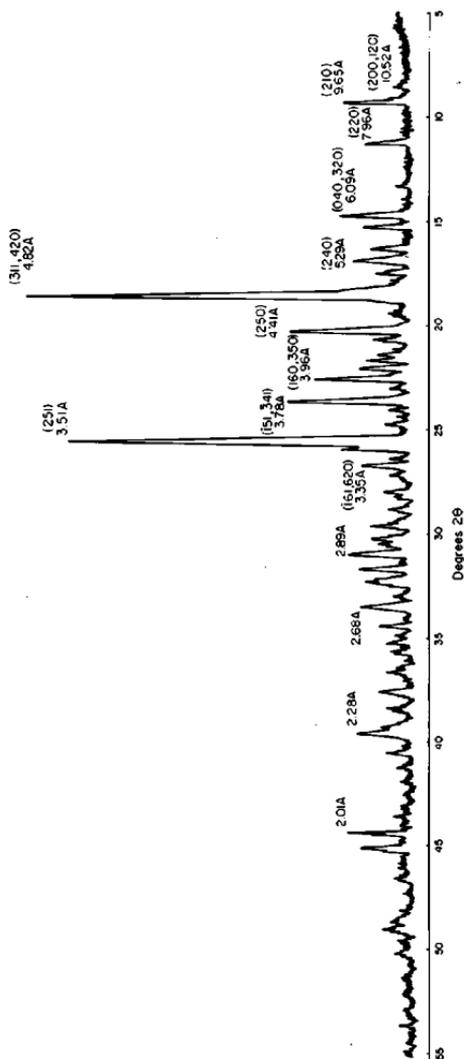


Figure 1. X-ray-diffractogram ( $5^{\circ}$  to  $55^{\circ}$  2-theta) of mineral identified as pickeringite, Cimarron County, Oklahoma. Specimen was -115-mesh powder, randomly oriented on vaseline-coated glass slide. The d values and Miller indices of only the more prominent diffraction peaks are labeled. CuK $\alpha$  diffraction line monochromatized by a nickle filter and pulse-amplitude discrimination.

Diffractometer speed:  $1^{\circ}$  2-theta/min      Scale-factor setting:  $1 \times 10^6$   
 Recording-chart speed: 1 cm/min      Statistical-error setting: 0.75%

TABLE I.—X-RAY-DIFFRACTION DATA FOR SELECTED HYDRATED SULFATES

SAMPLE			PICKERINGITE <sup>1</sup>			HALOTRICHITE <sup>2</sup>			ALUNOGENITE <sup>3</sup>		
d(A)	I/I <sub>0</sub>	hkl	d(A)	I/I <sub>0</sub>	hkl	d(A)	I/I <sub>0</sub>	hkl	d(A)	I/I <sub>0</sub>	hkl
—	—	—	—	—	—	—	—	—	18.7	—	—
10.52	4	—	10.6	5	200,120	10.4	15	—	—	—	—
9.65	24	210	9.7	18	210	9.5	13	200,120	—	—	—
7.96	16	220	8.0	12	220	7.82	10	210	—	—	—
—	—	—	7.12	4	—	—	—	220	7.2	—	—
6.66	3	—	6.73	4	310	—	—	—	—	—	—
6.09	20	040,320	6.08	20	040,320	6.02	30	—	—	—	—
5.86	13	140	5.87	10	140	—	—	040,001	—	—	—
5.49	8	—	—	—	—	—	—	—	—	—	—
5.29	12	240	5.31	14	240	5.24	15	240	—	—	—
5.14	3	—	—	—	—	—	—	—	—	—	—
4.97	13	—	4.97	20	211	—	—	—	4.95	—	—
4.82	100	311,420	4.82	100	311,420	4.77	100	150	—	—	—
4.69	6	131,221	4.69	10	131,221	—	—	—	—	—	—
4.62	3	231	4.62	14	231	4.62	30	231,321	—	—	—
4.41	9	—	4.40	14	250	—	—	—	4.42	—	—
4.32	27	141,311	4.32	35	141,311	4.29	55	141,331	—	—	—
4.18	13	141,401	4.18	20	141,401	—	—	—	—	—	—
4.13	17	241,321	4.12	30	241,321	4.09	45	141,311	—	—	—
3.96	22	160,350	3.97	20	160,350	3.95	35	160,350	3.95	—	—
3.78	27	151,341	3.79	30	151,341	—	—	—	—	—	—
3.72	3	—	—	—	—	3.75	40	241,260	—	—	—
3.67	3	251	3.67	6	251	—	—	—	3.67	—	—
3.62	9	—	—	—	—	—	—	—	—	—	—
3.51	78	251	3.51	90	251	3.48	100	070,360	—	—	—



iron, manganese, aluminum, titanium, silicon, calcium, and potassium. Trace amounts of cobalt, nickel, and zinc were also detected in the analysis.

Standards were prepared by mixing accurately weighed quantities of reagent-grade oxides of aluminum, iron, and manganese to a matrix of  $MgSO_4 \cdot 3H_2O$  of reagent-grade purity. Analyses for the minor amounts of  $SiO_2$ ,  $TiO_2$ ,  $CaO$ , and  $K_2O$  were made using silicate standards from a variety of sources. Although it is necessary, for best results, to use standards that closely match the matrix of the sample being analyzed, it was felt that an estimate of the relative percentages of these minor and primarily nonessential components was adequate for this study.

In order more closely to match the chemical composition of the standard, the sample being analyzed was first dried at  $150^\circ C$  to remove the water of crystallization ( $\sim 22H_2O$ ). This process reduced the unknown sample basically to a sulfate matrix. Samples and standards were prepared by pressing -115-mesh powders, without a binding agent, at 30 tons pressure into a polyvinyl alcohol substrate, using a Buehler die and a Paul Weber press.

The X-ray-fluorescence unit was a Siemens constant-potential X-ray generator, mounted with a chromium-target X-ray tube as the excitation source. A gas-flow proportional counter, using P-10 gas (90% argon, 10% methane), was used as the detector. Pulse-amplitude discrimination to monochromatize the  $K_\alpha$  spectral lines was facilitated by using a pulse spectroscopy. An automatic step scanner and an automatic print-out device were also utilized.

After making the proper counter-voltage, attenuation, baseline, and window settings to monochromatize the  $K_\alpha$  spectral line for the particular element, the 2-theta position for the spectral line was pre-

TABLE II.—CHEMICAL DATA FOR SELECTED HYDRATED SULFATES

	SAMPLE	PICKERINGITE*	HALOTRICHITE*	APJOHNITE*	ALUNOGENITE*
MgO	4.03	4.69	—	—	—
FeO	0.45	—	8.07	—	—
MnO	0.21	—	—	7.97	—
$Al_2O_3$	12.63	11.87	11.45	11.47	15.30
$SO_4^1$	36.39	37.28	35.96	36.00	36.04
$H_2O^2$	44.52	46.16	44.52	44.56	48.66
$SiO_2$	0.90	—	—	—	—
$TiO_2$	0.47	—	—	—	—
CaO	0.22	—	—	—	—
CoO	Tr.	—	—	—	—
NiO	Tr.	—	—	—	—
ZnO	Tr.	—	—	—	—
$K_2O$	0.11	—	—	—	—
Total	99.93	100.00	100.00	100.00	100.00

<sup>1</sup> $SO_4$  estimated as (Wt. loss at  $1,800^\circ C$ ) - (Wt. loss at  $150^\circ C$ ).

<sup>2</sup> $H_2O$  estimated as (Wt. loss at  $150^\circ C$ ).

\*Based on ideal compositions of pure end members.

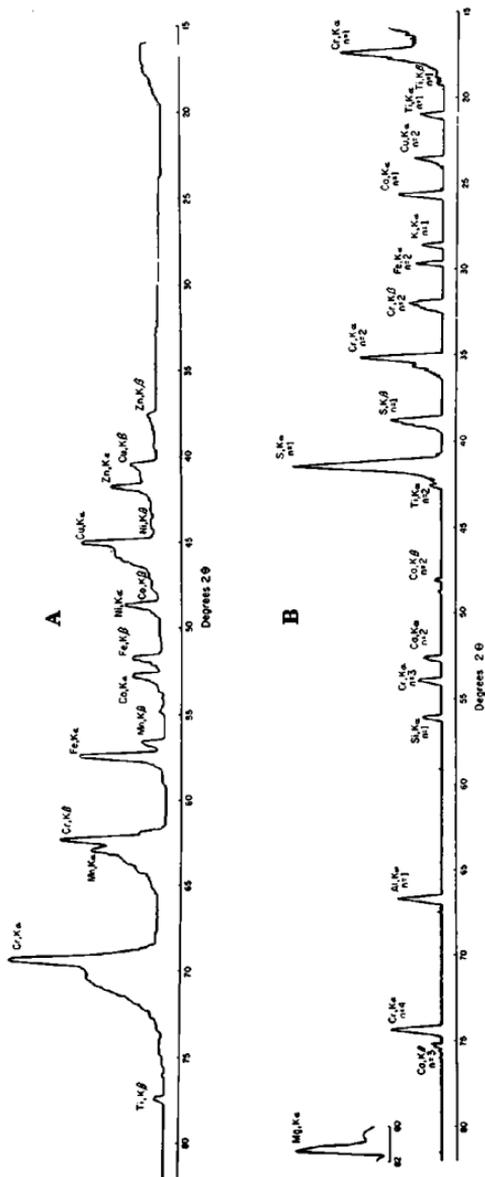


Figure 2. X-ray-fluorescence spectrograms ( $16^{\circ}$  to  $82^{\circ}$   $2\text{-theta}$ ) of mineral identified as pickeringite, Cimarron County, Oklahoma. Excitation source: chromium-target X-ray tube, operated at 35 microvolts and 50 milliamperes. Logarithmic scale factor, baseline 5 volts, window open (integral). Vacuum path maintained at 0.1 torr. Detector: flow-proportional counter, P-10 gas at 0.06 cu ft/hr flow rate.

A With LiF analyzing crystal. Counter voltage: 1,925 volts. Attenuation:  $2 \times 10$ .

B With gypsum analyzing crystal. Counter voltage: 2,100 volts. Attenuation:  $5 \times 10$ .  
MgK $\alpha$  spectral line retraced on  $2 \times 10^4$  linear scale and statistical-error setting of 7.5%.

cisely located by step scanning across the peak range at 0.005 degrees 2-theta intervals and taking 4-minute-duration fixed counts at each position. With the peak properly located, each standard sample and the unknown were rotated into position and 4-minute-duration fixed counts were made to establish peak-plus-background intensities. A 4-minute fixed count at an appropriate position was also made to establish background intensities. The differences between the two sets of counts yielded the  $K_{\alpha}$  peak intensities.

The routine was repeated three times and average values were used to establish analytical curves for each element. The chemical components were then established by interpolation of intensities to percentages from this set of curves.

The results of the chemical analysis are listed in table II. Ideal compositions were calculated for pickeringite, halotrichite, apjohnite [ $MnAl_2(SO_4)_2 \cdot 22H_2O$ ], and alunogenite. The more significant components are  $MgO$ ,  $FeO$ ,  $MnO$ , and  $Al_2O_3$ . The weight percent of crystalline water is estimated by weight loss determinations at  $150^{\circ}C$ . The weight percent of  $SO_4$  is based on the difference in weight loss at  $1,800^{\circ}C$  and  $150^{\circ}C$ .

Chemical data are also provided in the form of X-ray-fluorescence spectrometer scans in figure 2.  $LiF$  (fig. 2-A) and gypsum (fig. 2-B) analyzing crystals were used, with a logarithmic scale factor setting in each case. Chromium spectral lines are due to scatter of primary radiation from the X-ray tube within the sample chamber. Copper spectral lines are from the brass sample chamber.

#### DIFFERENTIAL THERMAL ANALYSIS

Differential thermal analysis was performed on a Robert L. Stone dynamic-gas DTA unit. The sample was in the form of -115-mesh powder, with alpha alumina as the inert standard and nitrogen as the purging gas. The mercury manometer in the gas system was maintained at a relative value of 2.0. The heating rate was set at  $10^{\circ}C/min$ , and the heating run was made from room temperature to  $1020^{\circ}C$ . Calibration was set at 1, and the microvolt selector at 500. The DTA pattern is illustrated in figure 3. No definable exothermic loops appear on the pattern (unless the slight hump at  $825^{\circ}C$ , immediately following the  $800^{\circ}C$  endotherm, can be so interpreted). Endothermic reactions may be observed at  $125-150^{\circ}C$ ,  $350^{\circ}C$ ,  $425^{\circ}C$ ,  $800^{\circ}C$ ,  $925^{\circ}C$ , and  $1010^{\circ}C$ . The low-temperature region ( $100-200^{\circ}C$ ) is due to the evolution of the crystalline water. The ragged appearance of the low-temperature endotherm is due to the solution of the sample in its own water of crystallization. This effect is also observed when the sample is heated rapidly to  $150^{\circ}C$ . The residue is usually left in the form of a delicate, papery ball, and all trace of the fibrous nature of the original crystals is destroyed. The higher temperature endotherms are due to a further reorientation and destruction of the crystallinity of the substance with the evolution of  $SO_4$ , predominantly at  $800^{\circ}C$ .

#### INTERPRETATIONS

Of the several minerals of the halotrichite group, two appear pertinent to this discussion: halotrichite and pickeringite. A complete

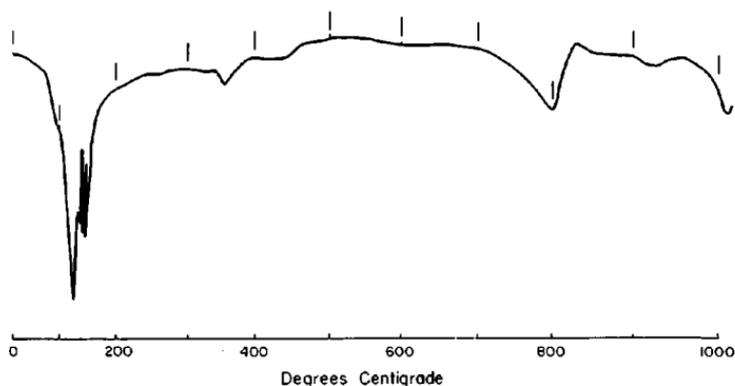


Figure 3. Differential thermal analysis pattern (room temperature to 1,020° C) of mineral identified as pickeringite, Cimarron County, Oklahoma.

Heating rate:	10° C/min	Purging gas:	nitrogen
Inert standard:	alpha alumina	Manometer setting:	2.0
Calibration:	1.0 @ 500 $\mu$ V		

series extends between these two members, with a partial series extending between them and apjohnite. The name pickeringite is applied in cases where Mg exceeds Fe, and halotrichite where Fe exceeds Mg. The adjective manganean is used when applicable (Palache, Berman, and Frondel, 1951, p. 525). The term "apjohnite" applies only when Mn exceeds (Mg + Fe).

The mineral investigated in this study is identified as a member of the halotrichite group, upon the basis of X-ray diffractometry. The chemical analysis (table II) indicates that it is closest to the magnesium-rich, or pickeringite, end member. The small amount of manganese present in the sample might justify the appellation manganean pickeringite.

The aluminum content is slightly higher than that of the ideal composition of pickeringite, suggesting the presence of some other aluminum-rich phase. Readjustment of the percentage of  $Al_2O_3$  in the sample to the ideal composition suggests that as much as 15 percent of the sample may be alunogenite. Some of the X-ray-diffraction lines (table I) match  $d$  values for alunogenite. However, conclusive data concerning the presence of this mineral are not available.

#### OCCURRENCE OF PICKERINGITE

Pickeringite and halotrichite occur commonly as weathering products of aluminum and pyrite-rich sedimentary rocks. They are often observed as recent efflorescences in coal-mine workings. Common associates found with these minerals are melanterite, alunogenite, gypsum, and epsomite. Alunogenite may be of a fibrous nature and difficult to distinguish as a separate phase from the halotrichite minerals (Palache, Berman, and Frondel, 1951, p. 526). Pickeringite is known

to occur on the western coast of South America in the Cerro Pintados near Iquique, Tarapacá, and at Quetena and Chuquicamata in Chile. In the United States, pickeringite has been found near Tucumcari and Las Vegas, New Mexico. Other occurrences of pickeringite are in Sonora County, California, and near Fallon, Nevada (Palache, Berman, and Frondel, 1951, p. 526).

The deposit from which this sample was collected has no economic potential. Only about 30 pounds of the material was observed on the floor of the abandoned coal mine in Cimarron County (Branson, personal communication).

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