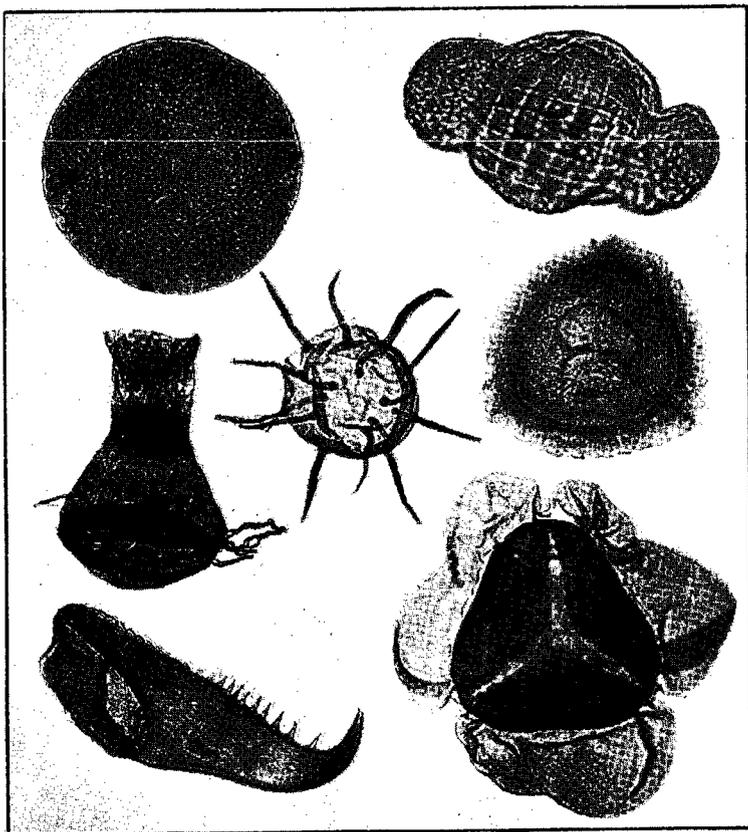


# OKLAHOMA GEOLOGY NOTES



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## GASTRIOCERAS IN RED EAGLE LIMESTONE

An item of Oklahoma geology is tucked away in a recent report on the Sacramento Mountains of New Mexico. The cephalopod *Gastrioceras drakei* Miller is stated to occur in the Red Eagle limestone in the quarry south of U. S. Highway 60 about a mile east of Burbank, Osage County, Oklahoma. The fact is stated by Otte as a personal communication from A. L. Bowsher and in a discussion of the age of the Laborcita formation.

Laborcita is a new name for the Early Permian unit in the La Luz Canyon area that had been referred to the Bursum formation. It is the formation from which the cephalopods described by Boese and by Miller were collected.

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--C. C. B.

## PERMIAN OPHIUROID OCCURRENCES

In 1958 H. W. Miller, Jr., described an ophiuran (snake star-fish) from the Americus limestone of central Kansas. His report is the first to mention an occurrence in Permian rocks. Hattin has now reported and figured the same species from the Speiser shale of Butler County, Kansas. He also mentioned a third specimen, this one from the Cottonwood limestone of Osage County, Oklahoma ( $N\frac{1}{2}$   $NE\frac{1}{4}$  sec. 2, T. 28 N., R. 5 E.).

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Hattin, D. E., An occurrence of *Ophiuraster burrisi* Miller; Jour. Paleontology, vol. 33, p. 1125-1126.

--C. C. B.

## PERMIAN SALT BEDS IN LAVERNE GAS AREA HARPER COUNTY, OKLAHOMA

Louise Jordan

Salt in the Permian of western Oklahoma presents the seismologist with one of the more troublesome problems he may encounter in seismic studies of the area. Salt is also a factor in drilling of wells and in the cementation of casing. From a different point of view, salt beds of sufficient thickness have an economic value in that they are suitable for construction of washed-out caverns in which liquid petroleum hydrocarbons or atomic wastes might be stored. The location of potash salts in an evaporite sequence also could be of economic importance.

Gasoline-plant operators in Texas have developed 110 storage caverns in salt layers; 17 of these are in Carson, Gray and Hutchinson Counties in the panhandle of Texas (Oil and Gas Journal, 1959). Phillips Petroleum Company has developed storage capacity for 3,390,000 barrels of butane and propane in 12 caverns in Hutchinson County, Texas. In Kansas, prepared caverns in salt layers number 40 and have a total capacity of 2,740,000 barrels. Thirty of these washed-out cavities are along the eastern boundary of the Hutchinson salt member of the Wellington formation in McPherson, Reno and Kingman Counties of south-central Kansas; and seven at one locality in Grant County in southwestern Kansas are constructed in the Blaine evaporite sequence. Oklahoma has two underground storage caverns in salt layers of the Blaine evaporite sequence: one with a capacity of 16,000 barrels in Elk City Field, Beckham County, (Jordan, 1959, and Saye, 1956), and one with a capacity of 33,000 barrels in the Camrick District of Beaver County (Oil and Gas Journal, 1959).

In the Laverne Gas Area of Harper and Beaver Counties, salt beds occur at four stratigraphic positions: 1) below the Blaine formation in beds equivalent to the Flowerpot shale, 2) below the Cimarron anhydrite (Stone Corral of surface nomenclature), and 3) in two stratigraphic positions in the Wellington formation. The salt strata are quite extensive in distribution as salt at these positions is recorded in Kansas to the north (Jewett, 1956, p. 26, and Kulstad, 1950, p. 241), and occur in all directions away from the field. Logs used to illustrate positions of the salt strata include gamma ray, neutron-gamma ray, caliper and guard logs of Cities Service Oil Company No. 1 Dunnaway "B" in NW $\frac{1}{4}$  SE $\frac{1}{4}$  sec. 9, T. 26 N., R. 25 W. (Figures 1-3).

To those unfamiliar with such types of investigations made in a drilled hole, a brief summary of some of the physical properties of salt, anhydrite and shale as measured by each type of log is given below. Salt and anhydrite have relatively low natural radioactivity and can not be differentiated by gamma ray alone. Neutron-gamma ray logs measure induced radioactivity of rocks, but, where a hole is enlarged owing to salt solution, the hydrogen-ion of water in the drilling mud filling the hole acts as a neutron absorber. The position of salt is indicated then by diminished recorded radioactivity; hence under this condition, salt and shale may not be differentiated on a neutron-gamma ray log.

The caliper log shows the variations in hole diameter. The position of salt is indicated by enlargement of hole size if the section in the area is known to be rock other than shale. Thus a focused-type log, such as a guard, laterolog or induction, which records electrical resistivity or con-

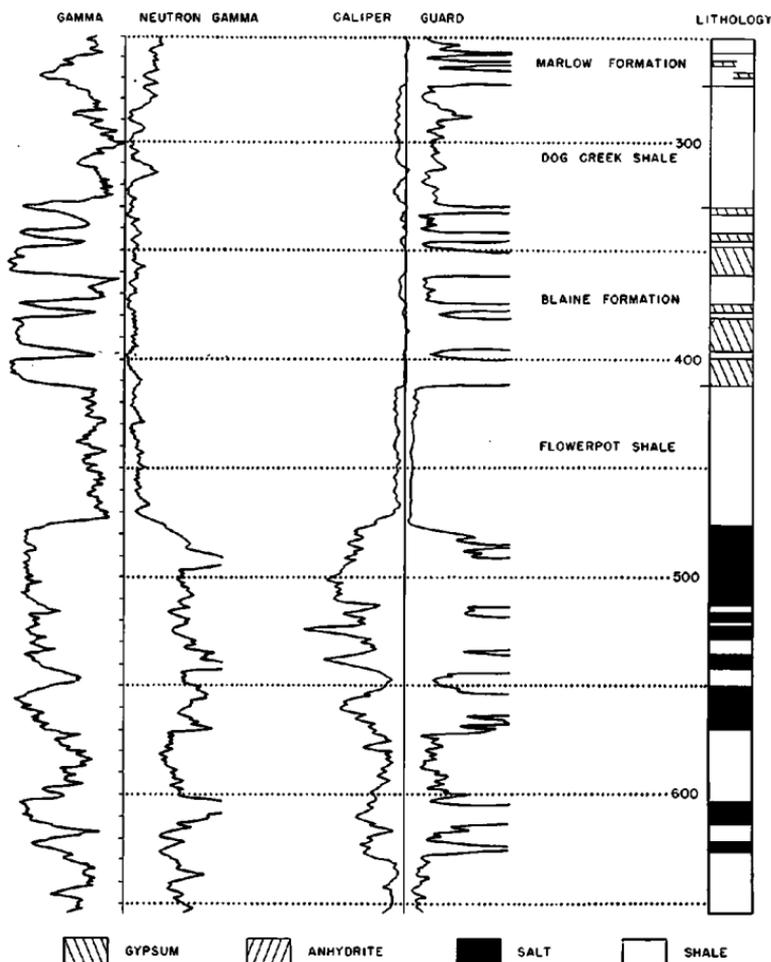


Fig. 1. Logs of Cities Service No. 1 Dunnaway "B", 250-650 feet, showing gypsum strata in Blaine formation and salt in Flowerpot shale. Leftward excursions of caliper log curve show increasing hole diameter. Rightward excursions of gamma and neutron gamma curves indicate increasing counts per second; and of guard log, increasing resistivity.

ductivity of the rock in the hole, is needed to differentiate salt and anhydrite from shale. Both anhydrite and salt have high electrical resistivity, whereas shale has low electrical resistivity. Focused-type logs have deep penetration and are much less affected by hole diameter variation than is the neutron-gamma ray log. Hence, with a combination of caliper and focused logs, salt can be distinguished from both shale and anhydrite. The neutron-gamma ray log may be used to indicate presence of a washed-out hole inasmuch as where the hole is enlarged, induced radioactivity is diminished. Unfortunately not all neutron-gamma ray logs of wells in the area have the good boundary definition between different types of rock as shown in the illustrated log (Figures 1-3) owing to various factors such as choice of time constant and of logging speed for the formations being logged, as well as presence or absence of casing, and holes not filled with fluid.

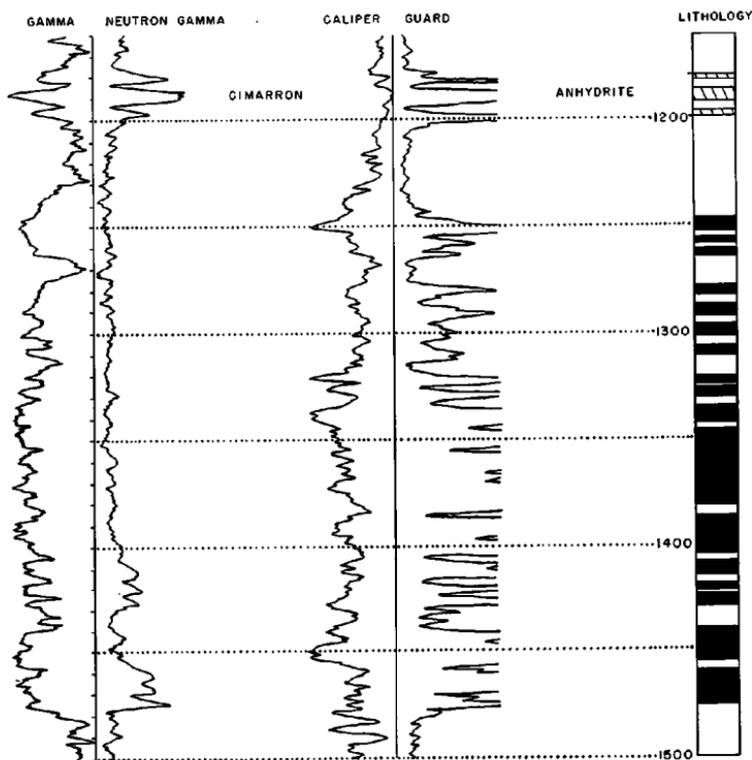


Fig. 2. Logs of Cities Service No. 1 Dunnaway "B", 1,150-1,500 feet, showing Cimarron anhydrite and salt in shale section below. Explanation of lithologic symbols given in Figure 1.

It has been assumed above that all salt in the Permian section is halite. However, potash salts exhibit high radioactivity and are soluble. They should show high radioactivity on gamma ray logs and low induced radioactivity on neutron-gamma ray logs, and might not be differentiated from shale without the use of a focused-type log.

1. *Salt below the Blaine formation (Figure 1).* Salt beds nearest the surface occur below the base of the Blaine formation in the Flowerpot shale and underlying formation. Flowerpot shale, 30 feet thick, rests upon a section essentially salt, possibly 250 feet in thickness in sec. 3 T. 25 N., R. 25 W. The salt in sec. 9, T. 26 N., R. 25 W. (Figure 1), underlying about 60 feet of Flowerpot shale, is found between 475 and 625 feet below the derrick floor, and is about 70 feet in thickness or nearly 50 percent of the total thickness. The interval containing salt in the field area ranges from 150 to 250 feet. Unfortunately few logs record top of the salt because at many places surface casing is cemented in the salt-bearing strata. Purity of the salt is not known but presumably the salt contains shale stringers normally less than one foot in thickness. (If any person has a core of the salt, a gift to the Survey would be appreciated.)

2. *Salt below the Cimarron anhydrite (Figure 2).* The Cimarron anhydrite (Stone Corral of surface nomenclature) occurs at 1,178 to 1,198 feet (Figure 2) and consists of three beds of anhydrite interbedded with shale. Top of the uppermost salt stratum below the Cimarron is at 1,246 feet and the section containing salt interbedded with shale is 230 feet thick. Although some lithologic logs of wells in the vicinity have shown thin beds (two feet or less) of anhydrite in this salt sequence, any such stringers can not be recognized by means of the illustrated logs. Other lithologic logs show as much as 200 feet of salt with minor amounts of shale. Assuming that the high resistivity section in conjunction with large-hole diameter in the Dunnaway "B" logs represents salt, approximately 125 feet or more than 50 percent of the section is primarily salt. Beds of the lower portion of the 230-foot interval contain a higher percentage of salt.

3. *Upper Wellington salt section (Figure 3).* The Wellington formation, slightly over 1,000 feet in thickness, consists of interbedded salt, anhydrite, and gray shale. Top of the Wellington formation is normally placed by means of subsurface rock samples at the top of the uppermost occurrence of gray shale above anhydrite strata. Because this horizon can not be determined by electric or radiation logs, most geologists place the top of the formation at the uppermost anhydrite stratum, which in the No. 1 Dunnaway "B" occurs at 1,836 feet. The upper 200 feet of the Wellington contains anhydrite and shale in the upper portion and shale in the lower portion. Ten salt strata ranging in thickness from 5 to 20 feet are interbedded with shale and anhydrite from 2,040 to 2,265 feet (Figure 3). From depths of 2,265 to 2,400 feet, the section is essentially anhydrite and shale.

4. *Lower Wellington salt section (Figure 3).* The lower salt section from 2,400 to 2,490 feet contains the thickest salt bed, 31 feet, in the Wellington. In this 90-foot interval, there is approximately 49 feet of salt, 17 feet of anhydrite, and 24 feet of shale. From 2,490 feet to 2,765 feet, the Wellington is essentially anhydrite with thin (less than four feet) stringers of shale.

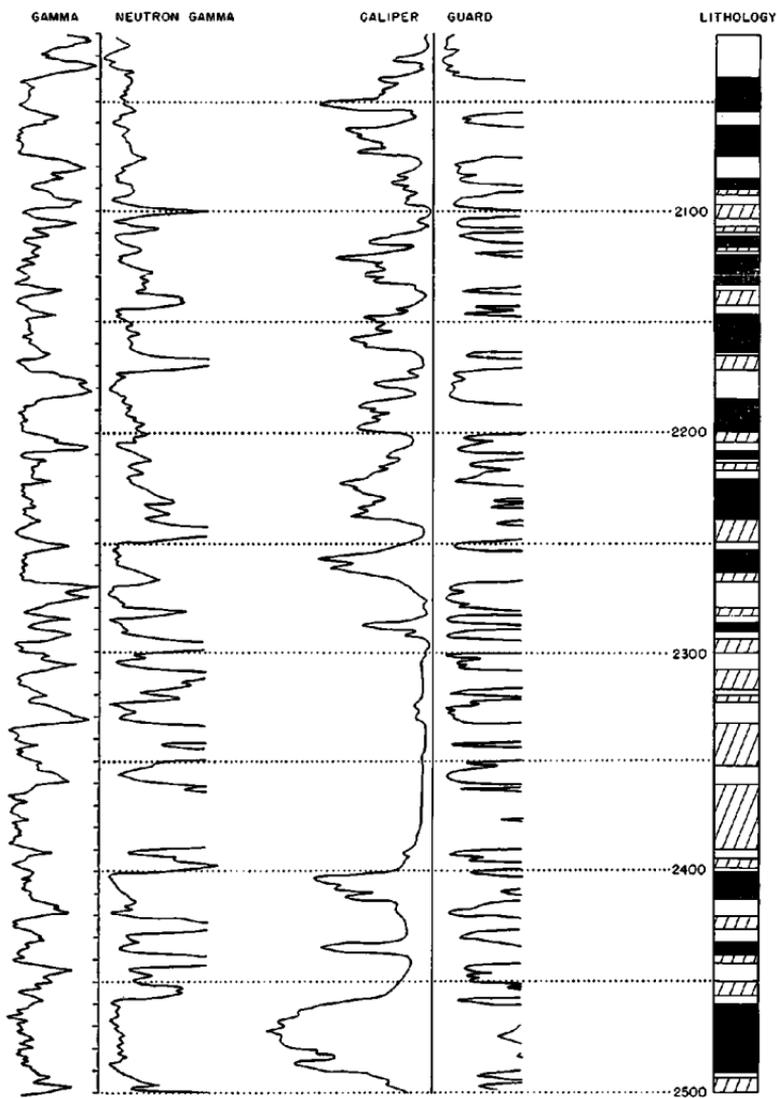


Fig. 3. Logs of Cities Service No. 1 Dunnaway "B", 2,030-2,500 feet, showing upper and lower salt beds in Wellington formation. Explanation of lithologic symbols given in Figure 1.

About two miles south in sec. 21, T. 26 N., R. 25 W. the caliper log of the Cities Service No. 1 Shuman "A" is closely similar to that of the Dunnaway "B" well in the sections below the Blaine and Cimarron, and practically identical in the Wellington formation. Unfortunately, the radiation log (gamma and neutron) is of poor quality for definition of an individual stratum. A present project of the Oklahoma Geological Survey is a study of the evaporite deposits in the Permian of western Oklahoma. Because caliper logs are not normally distributed through commercial services, the Survey would appreciate any such logs sent to it.

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#### MORE OKLAHOMA PERMIAN INSECTS

Permian fossil insects have been known since 1864, the first ones from Germany. Later they were reported from Czechoslovakia, United States, Anstralia, Russia, and other countries. Sellards first published on American Permian insects (the insects earlier found in Pennsylvanian are probably Pennsylvanian in age) from Kansas in 1906. Raasch found fossil insects in a dolomite bed of the Midco member of the Wellington formation in 1939. Carpenter described some of the forms in 1947. On December 11, 1959, Tasch and Zimmerman reported additional finds. They discovered an additional fossil layer eight to 10 feet above the bed found by Raasch and traced both beds across Kay County into Kansas. The preliminary note is "New Permian insects discovered in Kansas and Oklahoma", by Paul Tasch and T. R. Zimmerman, *Science*, new series, vol. 130, no. 3389, p. 1656.

—C. C. B.

## FLORINITES PELUCIDUS AND ENDOSPORITES ORNATUS WITH OBSERVATIONS ON THEIR MORPHOLOGY\*

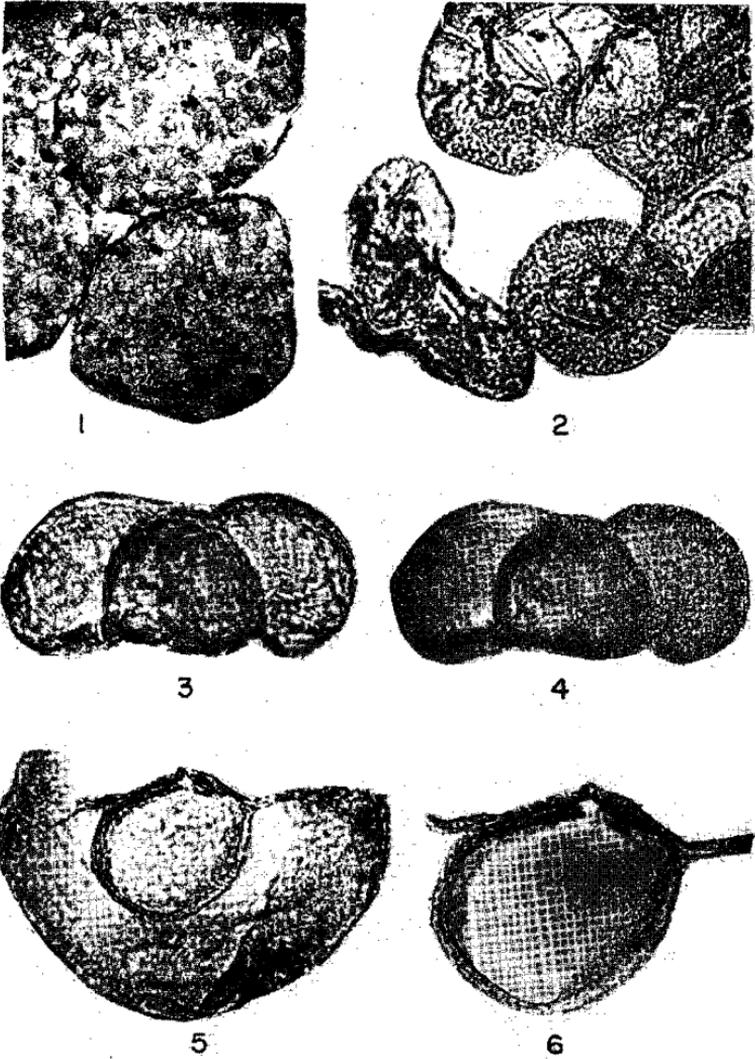
L. R. Wilson

A restudy of the types of *Endosporites pelucidus* Wilson and Coe, 1940 (Wilson, 1958) showed that they are not *Endosporites* as the genus is presently understood but rather a species of *Florinites* closely related to the type species, *F. antiquus* Schopf, 1944. Recognizing this fact the species was transferred to *Florinites* as *F. pelucidus*. Recently acquired coal ball peels containing pollen sacs and pollen of *Cordaianthus schuleri* give additional information about the morphology and Cordaitalean affinity of *Florinites*. Also outside of these pollen sacs are various other spores including several sectioned spores of *Endosporites ornatus* which provide morphological data not previously observed.

The Paleozoic pollen genus *Florinites* was established by Schopf, Wilson, and Bentall in 1944 and named in honor of Dr. Rudolf Florin who has contributed much to our knowledge of Paleozoic gymnosperms. Although less than a dozen unquestioned species have been described, *Florinites* is an important genus in Pennsylvanian coals and shales. A few species are of Mississippian and Permian age and some of the pollen observed in the Lower Triassic rocks may be assignable to *Florinites*. Geographically it is one of the more widely spread Paleozoic pollen grain genera and probably has been observed wherever Carboniferous rocks have been studied for plant microfossils. *Endosporites*, like *Florinites*, has essentially the same long geologic and wide geographic range, also the number of described species is not large. Both genera appear frequently together and presumably had the same ecology.

A review of the original descriptions of the genera *Endosporites* and *Florinites* reveals an interesting refinement in palynological interpretation. In 1940, when Wilson and Coe established the genus *Endosporites*, three species were included. The basis for the genus was essentially the presence of a bladder-like structure surrounding an internal spore body. The central body possesses a trilete germinal aperture although in one species (*E. pelucidus*) this character did not appear to be definite. The eruptions in the central body of that species were interpreted as an undeveloped trilete aperture. This later proved to be an erroneous inference. In addition the central bodies of the two species *E. ornatus* and *E. angularis* are more nearly spherical than they are in *E. pelucidus*. The bladder of the first species appears round and the second angular when seen in equatorial view. In polar views the bladders of both species may be round to crescent-shape depending upon their preservation. The bladder and central body of the last species are elliptical in equatorial view. Soon after the description of the genus *Endosporites*, fossils similar to *E. pelucidus* (*Florinites pelucidus*) were found in cone-like structures resembling *Cordaianthus*. These occurred in coal balls collected at What Cheer, Iowa, above the coal from which *F. pelucidus* was described. In the same year Darrah (1940) described similar fossil strobili from the Shuler Mine, near Wauke in Dallas County,

\* Preliminary report of one study being conducted by National Science Foundation Grant No. G6589.



DESCRIPTION OF PLATE I.

Fig. 1. *Cordaianthus schuleri* (Slide No. 97) with one pollen sac and portions of three others containing pollen grains. Greatest diameter of complete pollen sac 260 microns, pollen sac wall 6 microns thick. Pollen grains 49-80 microns long, 30-57 microns wide; central body 21-39 microns long, 19-29 microns wide. All photomicrographs with Carl Zeiss Photomicroscope and Plus X film.

- Fig. 2. Detail of nine pollen grains in pollen sac of *Cordaianthus schuleri* (Slide No. 98). Most of the grains appear in equatorial view and show the central body and surrounding bladder. Dimensions of bottom central grain 51.2 microns long, 41.7 microns wide; central body, 29.5 microns long, 21.6 microns wide.
- Fig. 3. Partially compressed pollen grain of *Cordaianthus schuleri* (Slide No. 96) in longitudinal-polar section showing attachment of central body to bladder below equator in the distal hemisphere. Photomicrograph oriented with proximal side of pollen grain at top. Length of bladder 68.9 microns, greatest thickness 31.5 microns; length of central body 31.5 microns, width (polar axis) 25.6 microns; bladder wall, smooth outside, finely reticulate inside, 1.6 microns thick; central body wall, smooth externally and internally, 2 microns thick. Photographed with Neofluar 40/0.75 objective Optivar setting 2, mag. 6.3, and medium green filter.
- Fig. 4. Same as Fig. 3 but photographed with Phase 2, Neofluar 25/0.00 objective, Optivar setting 2, mag. 6.3, and medium green filter. This photomicrograph shows with better definition, by phase contrast microscopy, the association of the central body and pollen grain bladder.
- Fig. 5. *Endosporites ornatus* (Slide No. 96). Spore in transverse-polar section showing proximal hemispheric attachment of central body to the bladder wall. Greatest bladder length (diameter) 108 microns, height (polar axis) 63 microns; central body diameter 39 microns, height 35.6 microns. Bladder wall 1.2 microns thick, finely reticulate on both surfaces; central body wall 2 microns thick, finely reticulate on outer surface. Photography same as for Fig. 3.
- Fig. 6. Central body of spore shown in Fig. 5 photographed with Neofluar, 100/1.30 Oel objective, Optivar setting 2, mag. 6.3 and medium green filter. Details of bladder and central walls, the germinal structure, and the ornamentation on the central body are shown.

Iowa. The name *Cordaianthus shuleri* was given the fossils. Because of the similarity of *E. pelucidus* (*Florinites*) to the pollen in *Cordaianthus shuleri* the writer stated in Schopf, Wilson and Bentall (1944, p. 45) that *Endosporites* had been found in the male strobilus of a Cordaitalean. Understanding the present status of *E. pelucidus*, now *F. pelucidus*, it should be emphasized that *Endosporites* (*s. str.*) was not found in the strobilus of a Cordaitalean.

When the genus *Florinites* was erected by Schopf *et al.* in 1944, the type was not seen by the writer, consequently the relationship of *F. antiquus* and *E. pelucidus* was not realized.

Florin, in a note within the paper by Crookall and Morris (1952), stated that "contrary to what is said by Schopf and others, I do not think that there are any *Cordaianthus* species with the *Endosporites* type of pollen". The quotation is reprinted in Chalouet's paper (1953) announcing the discovery of a new species of *Lepidostrobus* containing megaspores of the *Triletes auritus* type, and microspores assignable to *Endosporites*. This important discovery in a heterosporous lycopod cone seems to resolve the affinity problem of *Endosporites*.

Several *Cordaianthus schuleri* sections which contain numerous pollen sacs and an abundance of pollen grains were acquired from W. C. Darrah. These were collected near Waukeet, Iowa, from the Des Moines series, Middle Pennsylvanian age. Some of the pollen grains are cut showing the relationship of the bladders and central bodies. Three pollen-sacs containing many grains of *Florinites* type pollen are illustrated in fig. 1 on Plate I. Figure 2 is a detailed equatorial view of one pollen grain and several other

grains associated with it. The pollen sacs contain pollen in various stages of development but no grains were found that could be considered still in the tetrad state.

In the original illustration of *Florinites* (Schopf, et al. 1944, Plate II, figs. 13, 13a) the distal surface was drawn as a single line, suggesting a central body about which a bladder was attached some distance below its distal pole. A similar interpretation is also given by Potonié and Kremp (1954, p. 149, fig. 84). The section of *Cordaitanthus schuleri* pollen illustrated on Plate I, figs. 3 and 4 is in agreement with the drawings of Schopf and of Potonié and Kremp. The two photomicrographs, fig. 3, with bright light, and fig. 4, with phase contrast, are of a slightly compressed specimen. The longitudinal and polar axes are shown in the photomicrographs. No evidence of a double wall is apparent on the distal side, where the central body is attached to the bladder, and no attachment of the central body occurs on the proximal side. Other specimens in the same pollen sac support these conclusions. The single wall at the distal side appears to be at variance with Florin's (1936) observations. The silicified fossils studied by Florin may be better preserved than the calcified Iowa material and reveal greater detail. An additional point of variance is the construction of the central body. Many of the so-called walls inside the central body appear as folds or creases when viewed with phase contrast optics. These may have formed during fossilization. Further study of well preserved specimens should be made with the available new optics to resolve these problems.

The germinal apparatus in *Florinites* appears to be an indefinite rupture on the distal side of the central body. In the early stages this rupture may roughly simulate a trilete mark but later it becomes more irregular and seen in distal surface view may appear as an angular aperture surrounded by several flap-like folds.

Two of the several *Endosporites ornatus* spores found in the coal ball peels with the *Cordaitanthus strobili* are cut in polar axial section. One of these is illustrated by figs. 5 and 6 on Plate I. The attachment of the central body has been variously interpreted by Wilson and Coe, Schopf, et al., and Potonié. Although not definitely stated in the original description by Wilson and Coe (1940) the authors interpreted the central body as being completely surrounded by a thick intine which kept it centrally supported. It was reasoned that upon fossilization the intine disappeared and the central body became displaced. In the Annotated Synopsis of Paleozoic Spores (Schopf, et al., 1944, Plate 2, figs. 14, 14a) the central body is illustrated as being attached both proximally and distally to the bladder. Potonié (1952, p. 152, fig. 3, C and D), and Potonié and Kremp (1954, p. 149, fig. 81) have illustrated the attachment as only on the proximal side. The *Endosporites ornatus* section illustrated here shows the central body attached to the bladder only on its proximal surface. In the figures approximately one-third of the central body's circumference is shown in contact with the bladder. Both walls are distinct though apparently fused. This and other sections support the interpreted central body and bladder relationship described by Potonié (1952).

The sections also indicate that the trilete germinal structure is well developed in the central body wall and that in the bladder wall it appears to be initially not as marked.

Figure 6 shows a finely reticulate external surface on the central body wall of *Endosporites ornatus*. In the *Cordaianthus* sections the central body walls are smooth.

The establishment of affinities for *Endosporites* and *Florinites* has considerably advanced Paleozoic spore and pollen paleoecology. It is now possible to recognize these two important spore types as natural floristic units rather than as two unknown spore genera. In the Croweburg coal seam Wilson and Hoffmeister (1956, p. 38, fig. 4) showed that *Calamospora* (Calamarian spores), *Endosporites*, and *Florinites* reached the maximum of their relative abundance in the top third of the seam. This same abundance and position has been observed in numerous other Oklahoma coal seams and appears to represent a successional stage in the coal swamp development. If this biofacies in coal deposits proves to be constant it will be useful in stratigraphic studies.

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## SODIUM HYPOCHLORITE, A NEW OXIDIZING AGENT FOR THE PREPARATION OF MICROFOSSILS

William S. Hoffmeister\*

In trying to find a good bleaching agent for acid-insoluble microfossils, Miss Janie M. Heard, a technician at the Jersey Production Research Company, Tulsa, Oklahoma, found that sodium hypochlorite was not only an excellent bleaching agent, but also a safe, efficient, and cheap oxidizing agent for coals and other carbonaceous material. The new method is referred to as the Heard Method.

To oxidize bituminous coals and carbonaceous shales, workers in palynology have long depended on Schulze's solution (one part of a saturated aqueous solution of  $KClO_3$  with two or three parts of cold concentrated  $HNO_3$ ), or the dry method, in which concentrated  $HNO_3$  is added to a mixture of equal parts of coal and dry  $KClO_3$ . As far as the writer knows, prior to the Heard method, no suitable oxidizing technique had been devised for breaking down anthracite coals. The microfossils in anthracite coals had to be studied in usually unsatisfactory thin sections.

It is not necessary to go into the steps comprising the Heard Method of oxidation; the method differs from those of many published accounts of oxidation, such as the Schulze's solution or the dry method, only by the substitution of a 5.25 percent solution of sodium hypochlorite, which is the ordinary household Clorox, for other oxidizing agents. We have achieved very good results in using the Heard Method to break down both bituminous and anthracite coals as well as carbonaceous shales. It is often desirable to follow the sodium hypochlorite treatment with a base such as  $NH_4OH$ .

It is assumed that the sodium hypochlorite breaks down into nascent oxygen and free chlorine upon contact with an oxidizable material. This action is shown by the following chemical reaction:



The advantages of sodium hypochlorite over Schulze's solution and the dry method are listed below:

- (1) *It is cheap.* Common household Clorox, which is a 5.25 percent solution of sodium hypochlorite, is adequate.
- (2) *It is fast.* Most bituminous coals and carbonaceous shales break down immediately upon being treated with sodium hypochlorite.
- (3) *It is safe.* No violent reaction occurs when bituminous coals are treated with sodium hypochlorite, nor is there any danger of acid burns. This is in particular contrast to the dry method in which there is always the possibility of an explosion.
- (4) *It is gentle.* Tests so far have shown that the acid-insoluble microfossils extracted by sodium hypochlorite are better preserved than those extracted with most oxidizing agents.
- (5) *It breaks down anthracite coals.* Sodium hypochlorite has been used successfully to break down these coals, where Schulze's solution and other chemicals have failed. The process for treat-

\* Jersey Production Research Company, Box 801, Tulsa, Oklahoma. The writer wishes to thank the company for permission to publish this article.

ing anthracite coals with sodium hypochlorite is somewhat longer than that involved in oxidizing bituminous coals, although we have broken down some of these coals in a period of two days. When the coals are resistant to the treatment, more sodium hypochlorite is added as needed. Sometimes it is desirable to remove the used sodium hypochlorite and to add a fresh solution. By repeating this treatment, the coals are gently broken down. A small amount of diluted HCl added to the mixture will increase the oxidation action.

- (6) *It is a good bleaching agent.* Acid-insoluble microfossils such as chitinozoans and fragments of graptolites found in the residues after treatment with sodium hypochlorite are bleached so that minor details can be recognized for more accurate determinations.

## STARFISH IMPRESSIONS FROM THE HILLTOP SHALE

Philip A. Chenoweth

Fossil starfish are among the rarer of Paleozoic fossil animals and are considered as the choice specimens of many museums. This is probably due, however, not so much to the relative scarcity of these creatures in the seas of Paleozoic time as to the accidents of preservation and the destruction of the skeletons by weathering. None has been described from Oklahoma and only a single specimen has heretofore been known, this being one in the collection of the Oklahoma Geological Survey simply listed as having been found "near Barnsdall" (Osage County). Attempts to find the exact locality from which this fossil was collected have been unsuccessful.

This note describes an occurrence of starfish impressions in the Hilltop shale (Missourian) in Seminole County. The fossils have been collected in the quarry of the Wewoka Brick and Tile Company located on old U.S. route 270 about 3 miles west of Wewoka in SW $\frac{1}{4}$  sec. 11, T. 8 N., R. 7 E., Seminole County. This is the type locality of the formation, which here consists of 127 feet of bluish-gray and green shales, thin tan to buff siltstones, and in the upper part massive tan to buff siltstones. The unit rests conformably upon the Belle City limestone and is overlain unconformably by the Vamoosa formation (Virgilian). The thin siltstone beds near the middle of the formation are commonly ripple-marked and both the upper and under surfaces of the beds are covered with irregular fucoidal impressions, grooves, scratches, trails, worm castings, and other unidentifiable markings. The starfish occur both as shallow star-like impressions on the beds and as slightly raised molds. All are small: of the 8 specimens collected the average distance from tip to tip is about 14 millimeters. None is well enough preserved to permit identification, although the specimens appear to have the characteristics of forms generally assigned to the subclass Asteroidea; simple, straight, sharply tapering arms which are prolongations of the central disk (Figure 1).

According to Schuchert (1915) starfishes are most often preserved in fine-grained sandstones and muddy siltstones and least often in limestones. They usually occur as fine molds since all the calcium carbonate has been dissolved out by atmospheric waters. Conditions for their preservation were well satisfied during deposition of the Hilltop shale and it is quite possible that further search will result in the finding of more readily identified specimens.

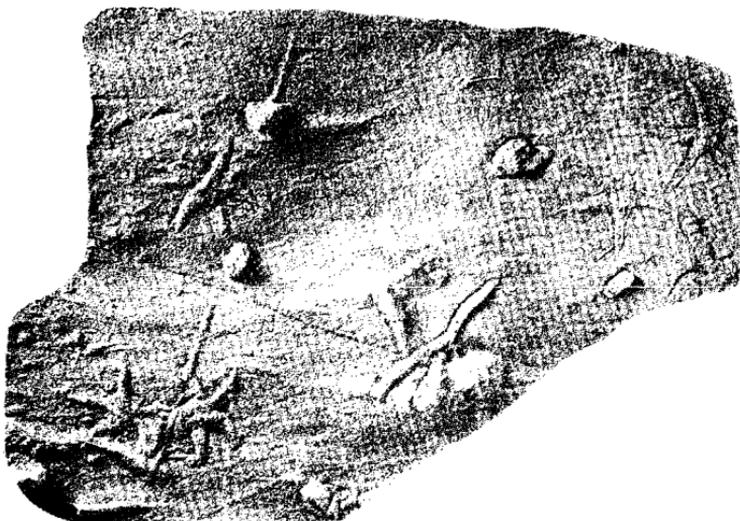


Fig. 1. Slab of siltstone from Hilltop formation showing casts of starfish. Natural size.



Fig. 2. Detail of part of same slab showing three of the starfish specimens. Twice natural size.

#### REFERENCE CITED

Schuchert, Charles, 1915, Revision of Paleozoic Stelleroidea with special reference to North American Asteroidea: U.S. Nat. Museum, Bull. 88.

## A STUDY OF THE TEMPERATURE NECESSARY TO DETERMINE THE PURITY OF GYPSUM BY DEHYDRATION

John A. Schleicher

The presence of much high-purity gypsum in the Blaine formation of western Oklahoma, which is extensively utilized in the manufacture of portland cement retarder, plasters, wall board and commercial fillers, causes a continuing interest in these deposits by the Oklahoma Geological Survey.

Recent discrepancies between chemical analyses and purity assays in the Geochemical Laboratory of the Oklahoma Geological Survey have cast considerable doubt upon the accuracy of assays for the purity of gypsum based upon dehydration at an accepted temperature. For many years the standard method for this assay (Scott, 1939, p. 214) has been as follows: a one-gram sample, ground to -60 or -100 mesh, is dried at 40° Centigrade to remove adsorbed water, and then further heated at 215° to 230° Centigrade to a constant weight. The loss in weight is then multiplied by a factor of 4.7787 and divided by the original sample weight. Multiplied by 100, the result is the percentage of pure calcium sulfate dihydrate (gypsum) in the sample. The accuracy of this method is, of course, dependent upon the presumption that all combined water associated with the gypsum is removed at or below 230° C. This presumption is borne out by the existing literature on the subject, which states that three-fourths of the combined water is given off at a temperature of 128° C., and the remaining one-fourth at or below 163° C. In actual practice, it is well known that gypsum begins to dehydrate at a temperature as low as 50° C. Some of the water of hydration is so loosely bound in the crystal structure that a portion of it will be removed, even at room-temperature, by storing the powdered sample at atmospheric pressure over an efficient desiccating agent such as concentrated sulfuric acid, phosphorus pentoxide or anhydrous magnesium perchlorate. Droste and Grim (1959) in investigating the first step in dehydration of gypsum, from the dihydrate to the hemihydrate, have shown that there is no transitional stage between the two forms. Their work, furthermore, although done in steam rather than air, indicates to this author another substantiation of the idea that there is no fixed transitional point in the initial dehydration of gypsum.

Some of the water is bound to the molecule very strongly indeed, but there has been little work done to determine the true conditions required to dehydrate completely a gypsum sample. Toward this purpose, this study was initiated.

To assist in the performance of this study under as nearly exactly known conditions as possible, a quantity of large crystals of selenite was generously donated by the United States Gypsum Company laboratory at Southard, Oklahoma, Mr. W. Dale Reynolds, Quality Superintendent. These crystals of naturally occurring  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  were presumed to be as nearly pure as possible. A subsequent average of careful assays and analyses of three samples of this material gave a purity of 99.997 percent  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . A portion of the crystals was ground by hand with porcelain mortar and pestle in the Geochemical Laboratory. Another portion was ground in the laboratory of the U. S. Gypsum Company at Southard where a laboratory-

size Raymond mill was used. The sample at the Geochemical Laboratory was ground to between 60 and 100 mesh. The sample ground in the Raymond mill, which uses a centrifugal impellor made up of small, hinged hammers to smash the particles of sample as they fall through the mill, without contacting any other surfaces, and then blows the smaller particles out through a peripheral screen, was sieved and found to contain more than 85 percent of -200 mesh particles. This differential in grain size was found to be of importance in the dehydration study, as was suspected. It was impossible to grind the selenite to such a small grain size by hand, since compaction of the powder in the mortar resulted from reduction of the particle size below 100 mesh.

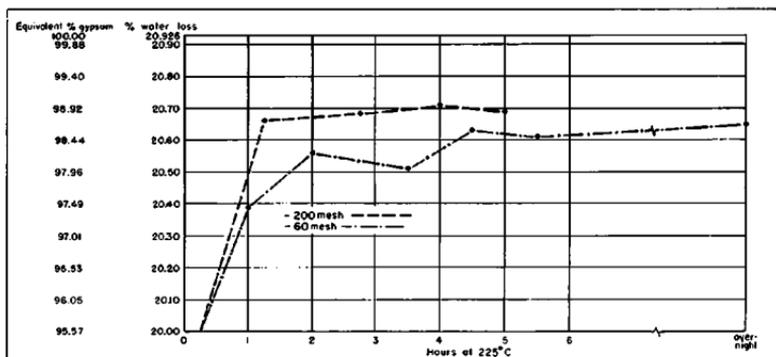


Fig. 1. Influence of particle size and duration of heating on dehydration of selenitic gypsum at 225°C. Finely ground sample is more completely dehydrated, but neither sample shows as much as 99.0 percent gypsum purity at this temperature.

Samples of both grindings were heated in a drying-oven at 225° C. for varying lengths of time, one sample of the -60 mesh powder being left at this temperature overnight. The result of this experiment is shown in Figure 1. It was suggested by Mr. Reynolds that a partially dehydrated gypsum was so deliquescent that it might take up water even from a strong desiccating agent. It was decided, therefore, to weigh the samples as soon as they became cool enough to be weighed with no turbulence in the analytical balance. A standard time of exactly three minutes seemed adequate. In the case of the 3½-hour sample of -60 mesh powder, the time of cooling was extended, with the result that its dehydration distorted the curve, indicating that gypsum was, in fact, a stronger desiccant than the anhydrous magnesium perchlorate used as drying agent in the cooling-desiccator. It will be noted that neither of the groups of samples was completely dehydrated at the heretofore standard temperature of 225° C., although the -200 mesh sample approached the theoretical value closer than did the -60 mesh samples. Even drying at this temperature for 16 hours (overnight) did not produce a closer assay than 98.69 percent on the -60 mesh material. It was decided, in light of this information, to determine at what temperature the selenite would be thoroughly dried. Accordingly, a number of

samples of both grindings were placed in a muffle-furnace, and the temperature raised to 300° C., held at that temperature for one hour, and a sample of each grinding removed and weighed after exactly three minutes of cooling. The temperature of the furnace was then raised to 350° C., held for one hour and a sample of the -60 mesh sample only removed and weighed. This procedure was repeated with both grindings at 400°, with only the -60 mesh at 450°, and with both grindings at 500° C. and 1000° F. (538° C.). The results are shown in figure 2. It will be seen that the

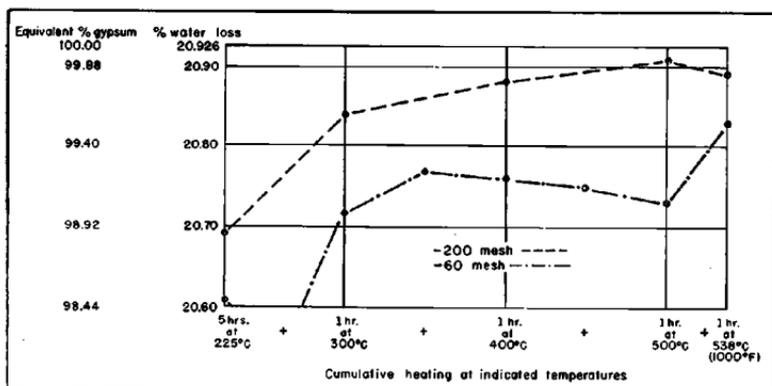


Fig. 2 Influence of particle size and duration of heating of selenite gypsum at 225-538° C. The data show that the sample ground to -200 mesh must be heated to approximately 500° C. before being nearly completely dehydrated.

-200 mesh powder approached the theoretical value, within experimental error, at both 500° and 538° C. The slight decrease seen between 500° and 538° can be said to be also experimental error. However, the -60 mesh material still was 0.46 percent away from the true value at 538°, and the continuously decreasing values between 350° and 500° are inexplicable. It was apparent that three hours at 400° was insufficient even for the -200 mesh powder, and that 500°-538° was probably the best temperature at which to effect complete dehydration.

Some gypsums, however, contain varying amounts of carbonates as impurities, and it was felt that heating to 500°-538° might cause some loss of CO<sub>2</sub> from any dolomite present. Accordingly, samples of the -200 mesh powder were mixed with a very pure powdered dolomite in the ratio of 5 grams of selenite to 0.5 grams of dolomite. Samples of this mixture were heated at 1000° F. for 1 to 4 hours. All of these samples lost some CO<sub>2</sub>, as is shown in figure 3, the weight-loss computed to the assay-percent on the basis of the 5 grams of selenite alone.

Samples of the mixture and of the unmixd selenite were then heated to 500° C. for 1 and 2 hours. (Fig. 3) Two hours at this temperature was insufficient to remove all the water of hydration from the pure selenite, but this time and temperature was ample to cause the decomposition of some of the dolomite from the mixture.

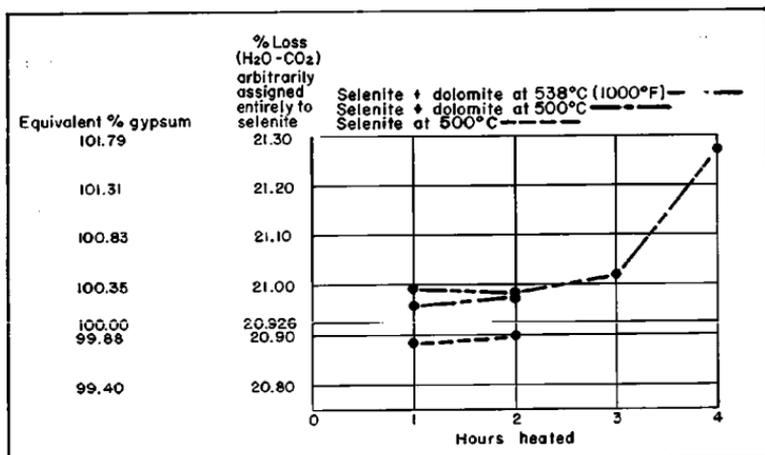


Fig. 3. Thermal decomposition of selenite gypsum compared to selenite mixed with a small amount of pure dolomite. 5.0 grams of selenite was used to minimize errors in weighing. All loss greater than 20.926 percent is loss of  $\text{CO}_2$  from the dolomite. The curves show that significant positive error can be introduced in the assay of rock gypsum containing dolomite at temperatures of  $500^\circ$  or  $538^\circ$  C.

Other samples were heated to the temperatures and for the times shown in the following table with the results obtained:

Samples	Time-Temp. Conditions				
	1 hr @ $400^\circ$	1 hr @ $450^\circ$	3 hrs @ $450^\circ$	8 hrs @ $450^\circ$	overnight @ $475^\circ$
Selenite	99.28%	99.43%	99.90%		
Selenite + Dolomite	99.37%	99.80%	99.96%	100.15%	100.02%

The samples assaying at 100.02 percent and 100.15 percent are within the limit of accuracy of the determination, considering the large conversion factor from weight-loss to percent calcium sulfate dihydrate.

To determine the time required for complete dehydration at a temperature of  $475^\circ$ , several samples of pure selenite were heated at that temperature for increasing lengths of time. The results are shown in Figure 4. The sample removed after three hours was allowed to remain in the desiccator over anhydrous magnesium perchlorate for ten minutes instead of the usual three minutes. Some water was regained by the sample, as shown.

The conclusion indicated by this study of the dehydration characteristics of calcium sulfate dihydrate seems to be that a temperature of  $450^\circ$  C. to  $538^\circ$  C. is necessary to insure the complete dehydration of the mineral at a particle size of -200 mesh. When any appreciable amount of dolomite is present as an impurity, the temperature must be not more than  $475^\circ$  C. The time required for water loss is not less than four hours, and preferably

five hours at any of these temperatures. To illustrate further this principle, a sample of rock gypsum, previously assayed by the standard method, was reported as having a purity of 94.71 percent. This was a sample of the Shimer gypsum taken from the quarry of the U. S. Gypsum quarry at Southard, Oklahoma. Upon reassaying by the new method, the true purity was found to be 97.93 percent, a difference of 3.22 percent. A difference of this magnitude would be of great importance in determining the type of use, as well as the economic value of the gypsum.

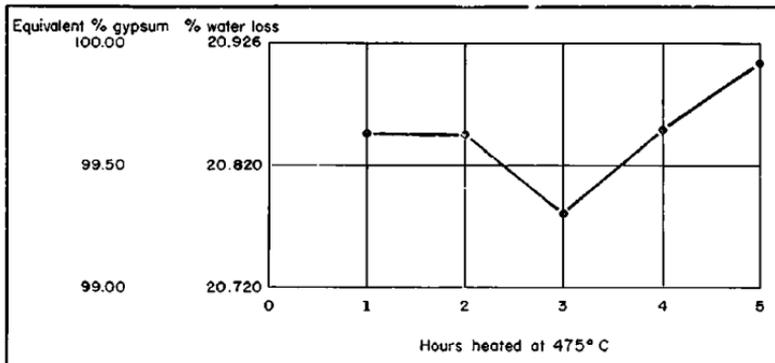


Fig. 4. Influence of duration of heating on -200 mesh selenite gypsum heated to 475° C. Sample at three hours regained water through standing in desiccator for 10 minutes before weighing; all others were weighed exactly 3 minutes after removal from furnace.

An interesting sidelight was noted during these studies. If a sample of powdered gypsum is almost, but not quite completely, dehydrated at a high temperature, and then allowed to cool and rehydrate even to a slight degree, the regained water is extremely difficult to remove, even at the elevated temperatures shown above. It might be postulated that some change in crystal structure or position of rehydration permits the formation of a stronger bond than that held by any of the original water molecules. Some further study along this line may be indicated.

#### REFERENCES CITED

- Scott's 1934, Standard methods of Chemical Analysis, N. H. Furman editor: Fifth edition, vol. 1, p. 214-216, D. van Nostrand Co.
- Droste, J. B. and Grim, R. E., 1959, A continuous investigation using an autoclave of the conversion of gypsum to hemihydrate: Amer. Mineralogist, vol. 44, nos. 7-8, July-Aug., p. 731-737.

## INDEX TO GEOLOGIC NAMES OF OKLAHOMA

The United States Geological Survey has recently released its Index to the Geologic Names of North America (Bulletin 1056-B, 215 pages). Names are indexed under each state, province, country, or territory of the type locality in groups according to age. The names are those of the Wilmarth lexicon (Bulletin 896) and of the 1936-1955 list (Bulletin 1056-A), and the index shows changes in age assignment and Federal actions on acceptance or rejection of names made before July 1, 1959.

The Oklahoma section occupies seven pages on which 559 names are listed. Of these names 171 are accepted by the Federal survey, 167 have not been considered, 59 are rejected, and 162 are subsurface names not considered as formal names. One name (Drywood) is restored to Missouri in "corrections" on page 622. Memorial shale is under Kansas, but was named for Memorial Park in Tulsa. Noxie sandstone is placed in Kansas, but the town is in Nowata County, Oklahoma. The Kinnisou shale member of the Senora is spelled "Kennison" and is placed in the "Cherokee formation." The Royer dolomite appears as Royer marble. The Paola limestone member of the Lola formation is again misspelled Paoli, as it was in Wilmarth.

It would have been wise to omit subsurface names. The book has only 162 of the 416 given by Dr. Jordan (Okla. Geol. Survey, Guide Book VI) and of those given Prue is spelled "Prune", Prosperity "Prosperity", Sikes "Sykes", and Nichlos "Nicholas". At least ten are placed in the wrong system (Patsy, Wilson, Belveal, Hotson, Vann, Mounds, Saputpa, Jefferson, Pugh, Simons).

The listing by ages is not really useful, at least for Oklahoma names, because of the numerous changes and uncertainties. The Springer and its members City Lake, Castle Rock, Lake Ardmore, Overbrook, Rod Club, and Target are placed in the Morrow. They would better have been listed under Lower Pennsylvanian (?). Listed under Lower Pennsylvanian are Big Branch, Frensley, Bostwick, Pumpkin Creek; all of them Middle Pennsylvanian of normal classification.

The Chickachoc chert is under Atoka where Taff originally put it, but it is Morrow, as all later authors have recognized.

The Ouachita units Pushmataha, Tenmile Creek, Wesley, Wildhorse Mountain, Chickasaw Creek, Markham Mill, Moyers, Jackfork, Johns Valley, Round Prairie are under Lower Pennsylvanian, but Prairie Hollow, Smithville, and Tuskahoma are under Mississippian. The Stanley is given as Mississippian and Pennsylvanian.

The Sycamore should be moved up to Middle instead of Lower Mississippian. The Tepee Creek facies of the Post Oak conglomerate is under Precambrian, but the Post Oak is under Permian.

The index would have been more useful had the units been listed alphabetically and had subsurface names been omitted.

Oklahoma names omitted are:

Lower Cretaceous

Marietta beds (in Wilmarth)

Permian

Agawan gypsum member (of Marlow formation)

Asphaltum sandstone

Gracemont shale member (of Marlow formation)

Paoli sandstone member (of Wellington formation)

Pocasset gypsum

## Pennsylvanian

Cabin Creek sandstone (member of McAlester formation)

Eaton formation (Marmaton group)

Red Oak member (of Atoka formation)

Jesse member (of Atoka formation)

Shawnee sandstone

## Ordovician

Bodine sandstone

Dixon sandstone

Oklahoma stratigraphic names proposed since 1955 (and therefore not given) are:

Ahloso member (of Caney shale). Elias 1956, emended 1957.

Bayou Manard member (of Moorefield formation). Huffman 1958.

Bowring limestone member (of Vamoosa formation). Tanner 1956.

Camp Ground member (of unnamed formation in Deese group). Hicks, published by Ramay 1957.

Clarita member (of Chimneyhill formation). Amsden 1957.

Corbin Ranch formation (of Simpson group). Harris 1957.

Cornell Ranch member (of Sycamore formation). Prestridge 1959.

Delaware Creek member (of Caney shale). Elias 1956.

Fittstown member (of Bois d'Arc formation). Amsden 1957.

Game Refuge sandstone (formation). Harlton 1959.

Gano shale (formation). Branson 1956.

Gene Autry shale member (of Golf Course formation). Elias 1956.

Golf Course formation (of Dornick Hills group). Harlton 1956.

Grindstone Creek member (of Goddard shale). Tomlinson 1959. Name is preoccupied and will be replaced by Tomlinson.

Hallett shale (formation). Branson 1956.

Ideal Quarry member (of Chimneyhill formation). Amsden 1957.

Jarvis Church chert conglomerate member (of Konawa formation). Tanner 1956.

Kiheki sandstone member (of Vamoosa formation). Tanner 1956.

Lake Ardmore formation (of Springer group). Tomlinson and McBee 1959. New unit containing sandstone member formerly called Lake Ardmore sandstone.

Lake Murray formation (of Dornick Hills group). Harlton 1953.

Lindsey Bridge member (of Moorefield formation). Huffman 1958.

Maud chert conglomerate member (of Konawa formation). Tanner 1956.

Ordinance Plant member (of Moorefield formation). Huffman 1958.

Redoak Hollow member (of Goddard formation). Elias 1956

Rhoda Creek formation (of Springer group). Elias 1956.

Sand Branch member (of Caney shale). Elias 1956.

Snomac member (of Ada formation). Tanner 1956.

Van Vacter gypsum member (of Blaine formation). Scott and Ham 1957.

Warren Ranch conglomerate facies (of Deese group). Tomlinson and McBee 1959.

West Arm formation. Harlton, published by Tomlinson and McBee 1959.

Worthey member (of Sycamore formation). Prestridge 1959.

—C. C. B.