PART 1. INSOLUBLE RESIDUES AS AN AID IN THE STUDY OF SEDIMENTARY ROCKS

R. R. SHROCK

University of Wisconsin

Introduction.—It is a well known fact that almost every sedimentary rock leaves some insoluble residue when digested in hydrochloric acid. The amount of this material may range from less than 1% (as in very pure limestones and dolomites) to more than 99% (as in very pure sandstones). While percentages of insoluble material have been recorded from chemical analyses for many years, little attention has been paid to that material until very recently. It is the purpose of this brief discussion to call attention to the uses that may be made of insoluble residues in the study of sedimentary rocks.

POSSIBLE USES OF INSOLUBLE RESIDUES

1. All students of sedimentary rocks are aware of the uses which have been made of diagnostic fossils (both macroscopic and microscopic), heavy detrital minerals, and insoluble residues for purposes of correlation. Work now in progress on Wisconsin Silurian dolomites has shown that certain residues appear to mark definite horizons in wells and exposures. Many problems of a similar nature, involving the determination of the amount, character and possible stratigraphic significance of insoluble residues in both well cuttings and exposures are awaiting investigation in Wisconsin as well as elsewhere.

2. Paleontologists are following with interest the rapid increase in number and variety of micro-fossils (foraminifera, conodonts, scolecodonts, etc.), which are being recovered from calcareous rocks by acid digestion (See Plate V, Figs. 4, 6-8,
13). Not only are these of value for correlation but they also help to picture life conditions and relations at the time they lived. Investigation, by insoluble residue analysis, of Pre-Cambrian calcareous rocks might produce significant results concerning the amount and character of the life of that time. Much may be learned about the very early growth stages of certain organisms if their youthful shells can be found. Such shells have been recovered from certain Wisconsin dolomites, and further investigations along these lines may produce interesting results.

3. Many investigators have found that insoluble residues aid in reconstructing conditions of sedimentation. The basal beds of the Black River limestone in Indiana, Illinois and Wisconsin frequently contain a high percentage of detrital quartz. When released from the calcareous matrix, the grains are found to be well rounded and frosted (See Plate V, Figs. 5, 9). These conditions obviously suggest affinities with the underlying St. Peter sandstone. The basal beds of the St. Lawrence dolomite, the Oneota dolomite, the Black River limestone and the Devonian of Wisconsin need to be studied for their detrital content. Original chert nodules, siliceous oölites, and other insoluble substances often furnish information of value (See Plate V, Figs. 1-3). Dolocastic chert has been reported frequently and chert masses containing impressions of crystals other than those of dolomite should be looked for. Small chert nodules in the Mayville dolomite of Wisconsin contain calcitic crinoid fragments and brachiopod shells, both of which are suggestive of the origin of the chert. The detailed study of these nodules, both as to faunal content and time of origin, promises some interesting results. Much has been written concerning the significance of glauconite in sedimentary rocks. Most of the Wisconsin dolomites contain some glauconite, and investigations of this material will be aided by insoluble residue analysis.

4. It is widely recognized that almost all sedimentary rocks have undergone certain changes since deposition as sediments. Most of these are so well known that they need not be mentioned. A few, however, are cleared up considerably by light thrown on them from studies of insoluble residues. European geologists, and very recently Americans also, have reported a number of authigenic minerals from sedimentary rocks. Quartz
and feldspar are the chief ones, and both have been found in abundance in Wisconsin dolomites. Microcline from the Silurian dolomites occurs as prismatic crystals with well developed brachy- and macropinacoidal faces. The crystals are composed of a shell of authigenic origin built around, and in crystallographic continuity with, a nuclear detrital grain of microcline (See Plate V, Figs. 11-12). Other kinds of authigenic feldspar have been found in insoluble residues from Wisconsin rocks (e.g. the Mendota dolomite), but they have not yet been definitely identified. Their thorough study constitutes an important problem. The recovery and accurate identification of all authigenic minerals in sedimentary rocks are needed to understand adequately one of the important changes that takes place in a sedimentary rock after deposition (See Plate 5, Fig. 10).

Recent work by the writer on certain Wisconsin dolomites shows that, when these rocks are treated for a short time with a weak solution of hydrochloric acid, the crystals are not only separated from each other but are also broken up into cleavage fragments. This fragmentation is due to the fact that solution proceeds most rapidly along the cleavage planes of the mineral. Small rhombs of carbonate carried by certain spring waters may have originated by the solution action just described. Investigations of the way various minerals behave during solution might produce significant results.

5. Students of sedimentary rocks are familiar with the way English, Scotch and French petrologists have utilized heavy detrital minerals in determining provenances of sediments. Similar work has been initiated in America recently, and many problems have been outlined and suggested. The presence of detrital minerals in certain of the limestones on Anticosti island suggests that it might be very much worth while to recover the insoluble residues of all calcareous rocks lying on the periphery, or upon the main mass, of the Canadian Shield, and study them along with the heavy detrital minerals from the clastic rocks to determine the relations between the sediments and the crystalline terrane from which they presumably were derived. By utilizing exposures and well cuttings studies of the calcareous formations, as well as of the clastic ones, which lie around buried or partly exhumed Wisconsin Pre-Cambrian hills might be pursued with significant results.
6. Geologists have been able, by using chemical and stratigraphic data, to estimate roughly the relative percentages of sandstone, shale and limestone in the world’s sedimentary column. Insoluble residue analysis, extended to include all types of sedimentary rocks, might well alter those estimates somewhat. In order to carry out such a program, however, it would be necessary to sample all rocks in some uniform manner.

7. Many field geologists occasionally find themselves at a loss to describe satisfactorily and accurately some sedimentary rock. A chemical analysis showing soluble material, sand, silt and clay would make this much easier and more definite. The entire Wisconsin sedimentary column needs to be studied with this in mind.

PART 2. STUDIES OF WISCONSIN SEDIMENTARY ROCKS

1. INSOLUBLE RESIDUES FROM WISCONSIN SILURIAN DOLOMITES

George B. Burpee*

Purpose.—This investigation was initiated to determine whether the lithologic units of the Wisconsin Silurian possessed characteristic insoluble residues which might be of assistance in correlating exposed formations with those encountered in wells down the dip from the outcrop.

Procedure.—The samples used in this study were collected by Dr. R. R. Shrock in eastern Wisconsin during the summers of 1930 and 1931. They were analyzed in the Sedimentation Laboratory at the University of Wisconsin with laboratory materials furnished by the Milwaukee Public Museum.

A 20-40 gram sample was crushed into fragments about one-half inch in greatest dimension, and then dissolved in a 50% solution of hydrochloric acid at a temperature slightly below boiling. It was necessary in some cases to wash the sample and treat it with fresh acid several times before all of the soluble matter was dissolved. When all possible reaction had ceased the acid was decanted and the residue washed clean of acid and

* Submitted as a thesis for the degree of Master of Arts (Geology) at the University of Wisconsin, 1932. Abstracted by R. R. Shrock.
Plate V. 1. Thin section showing siliceous oölites in a calcareous matrix (x10). 2. Same as 1, enlarged (x25), showing several siliceous oölites under crossed nicols. Note the central core of quartz. 3. Residue of siliceous oölites from same rock as 1 and 2 (x15). 4. Siliceous foraminifera (x8). 5. Detrital quartz grains in a calcareous matrix, shown in a thin section (x15). 6. Siliceous sponge spicules (x4). 7. Siliceous foraminifera (x8). 8. Several enlarged (x20) specimens of 4. 9. Residue of detrital quartz grains from same rock as 5 (x10). Note rounding and frosting. 10. Authigenic pyrite crystals (x10). 11. Authigenic microcline crystals (x25). 12. An authigenic microcline crystal, greatly magnified (x200) and with crossed nicols, showing the nuclear detrital grain around which the later authigenic shell grew (Photograph by W. L. Wilgus). 13. Chitinous jaws (x10).
clay material. From this procedure only a granular residue remained. This was then carefully washed onto a tared watch glass, dried and weighed. It was then filed for future microscopic study. No attempt was made to determine the amount of clayey matter in the residues.

Results.—Three general types of insoluble residues were found. First, almost all of the samples analyzed contained a small percentage (0.5%—3%) of very fine to fine authigenic quartz, and a second mineral then unidentified and thought also to be quartz with a peculiar crystal habit, but now known to be authigenic feldspar (microcline). The quartz consists of clear, anhedral or euhedral prismatic crystals with rhombohedral terminations. The feldspar usually occurs as euhedral prismatic crystals with well developed brachy- and macrobinacoidal faces. They sometimes have a nucleus consisting of a detrital grain of microcline. In these cases the authigenic shell of the crystal is developed in crystallographic continuity with the nuclear grain. Second, silicified fossils although far less abundant than the authigenic minerals just described comprise the diagnostic fraction of the residues. In order of abundance these fossils are sponge spicules, silicified foraminifera, fragments of brachiopod shells, internal casts of ostracods and bryozoans, fragments of crinoids and corals, and minute silicified gastropods or glauconitic casts of such shells. The foraminifera, and possibly the ostracods, have stratigraphic significance. It might be added that a residue composed chiefly of rounded detrital grains of quartz was found in the basal beds of the Devonian, which immediately overlie the Silurian at Cedarburg.

The Mayville dolomite is characterized by residues of chert and hexactinellid sponge spicules. The amount of the residue ranges from less than 1% to as much as 15%, but these percentages do not include the chert nodules which are abundant at certain horizons throughout the formation. The Byron dolomite rarely contains more than 2% of insoluble material, but has a distinctive foraminiferal-ostracod fauna which has been found at Burlington, Waukesha (in what may be the so-called Wauke-sha beds), and in well cuttings from a deep well near Racine. The Coral beds as exposed in the vicinity of Valders contain from 3%—26% of insoluble material, consisting largely of small crystals of quartz and feldspar (Burpee, without optical study,
called all of the crystals quartz). The Racine beds, as developed in the vicinity of Racine, contain only a very small residue (seldom over 1%), consisting of a little chert, some very fine anhe- dral crystals of either quartz or feldspar and a few masses of marcasite (?). A reef facies of the Racine ("Guelph") at Cedarburg contains no appreciable amount of insoluble material, but is overlaid unconformably by Devonian strata which carry a distinctive residue of rounded detrital quartz grains. The Waubakee dolomite, youngest of the Silurian formations of Wisconsin and known only from a few scattered exposures in the vicinity of Milwaukee, contains no appreciable residue.

In summary it may be stated that the Silurian dolomites of Wisconsin carry a diversity of insoluble material, usually in small amount; but that with the exception of certain silicified fossils, the residues so far obtained and studied do not have stratigraphic significance. The foraminifera, and possibly the ostracods, seem to be limited to a narrow stratigraphic range in the Byron formation, and may with further investigations become a useful horizon marker.

2. THE INSOLUBLE RESIDUES OF THE ONEOTA DOLOMITE OF WESTERN WISCONSIN.

Joseph J. Drindak*

Purpose.—This study was undertaken to ascertain the amount and character of the insoluble material in the Oneota dolomite, and in the immediately underlying arenaceous strata, in western Wisconsin; and to determine whether that insoluble material might be of stratigraphic or economic importance.

Procedure.—Samples were collected in the summer of 1932 with the aid of E. H. Powell and R. R. Shrock. They were taken in vertical sections about five feet apart unless there was a distinct change in lithology, in which case a sample was taken where the change occurred, and then the same procedure as before was followed. In one section samples were taken every six inches in the lower part and every foot in the upper part. The analytical work was carried on in the Sedimentation Labora-

* Submitted as a thesis for the degree of Bachelor of Philosophy (Geology) at the University of Wisconsin, 1933. Abstracted by R. R. Shrock.
tory at the University of Wisconsin, with laboratory materials furnished by the Milwaukee Public Museum.

About a 20-gram sample was crushed into small fragments averaging about one-half inch in greatest dimension, wetted with distilled water and then dissolved in a 50% solution of hydrochloric acid. In some instances the sample had to be washed and treated with new acid several times before all of the soluble rock was dissolved. After solution had ceased completely the acid was decanted, and the remaining insoluble residue was washed until all of the finely divided silt and clay had been removed and only a granular residue remained. This was then washed onto a tared watch glass, dried, weighed, and studied.

Results.—The insoluble constituents of the Oneota dolomite show considerable diversity. (1) Silicified corals and sponge spicules, mainly monaxons, are present throughout the formation in the sections studied, but do not seem to have stratigraphic significance. (2) Quartz is abundantly represented in the lower part of the formation by rounded and frosted detrital grains, and throughout the formation by minute crystals and clusters of crystals. (3) Siliceous oolites are usually present in the oolitic beds of the formation, and may show concentric structure. (4) Glauconite is abundant in certain beds (“Green speckled beds”)* and is common throughout the formation in small amount. Dolocastic chert was encountered in some beds and mica flakes are not uncommon in the arenaceous strata immediately underlying the base of the formation.

On the basis of lithology and field relations, and to a very limited extent on the basis of insoluble residue content, it is possible to subdivide the Oneota dolomite into several fairly distinct zones, as shown in Fig. 1. These have interesting, though rarely distinctive, residues.

Zone 1 belongs below the Oneota dolomite and is probably to be correlated with the Madison sandstone of the central and eastern parts of Wisconsin. It bears such a close relation to the basal part of the Oneota, however, that it was thought advisable to study its insoluble content along with that of the over-

* The basal part of the Oneota dolomite is characterized by thin beds of bluish-gray dolomite speckled or spotted with roughly spheroidal masses of greenish, or when weathered yellowish or slightly bluish, clay-like matter composed in large part of glauconite. These beds are specially designated in the sections of Fig. 1.
Fig. 1. Chart of stratigraphic sections showing the relative amounts of soluble and insoluble material at various horizons in the Oneota dolomite of western Wisconsin. The Madison-Oneota boundary is uncertain in some sections, hence it is not indicated.
lying formation. The base of zone 1 was taken at the top of the Jordan sandstone where the first strong dolomite influence appeared. The top of the zone, which has been reported as an unconformity, could not always be determined, hence no definite contact between the top of the Madison and the base of the Oneota is indicated in the sections in Fig. 1. Sand lenses and layers, dolomitically-cemented arenaceous beds, shale layers and conglomerates characterize the zone, but neither their presence nor number is consistent. The residues are composed mainly of rounded and frosted detrital grains of quartz in the sandstone layers, and both detrital and authigenic quartz in the dolomitic beds. Chert, glauconite and mica flakes were also obtained from some of the residues, but usually in small amount. The insoluble material tends to decrease upward in the zone.

Zone 2 comprises the basal part of the Oneota dolomite and is characterized by a diversity of lithology. The "green speckled beds" are always prominent, but there are also oölitic beds, shale layers, algal layers and conglomerates, all of which vary in thickness and number. All are highly dolomitic and contain small percentages of insoluble material. The detrital quartz of zone 1 gives way upward to authigenic quartz pretty largely, which occurs in the form of single crystals or crystal clusters. Chert is found in the algal layers, while the oölitic beds yield siliceous oölites. Glaucnites is found especially abundant in the "green speckled beds" and may make up as much as 30% of the residues. Sponge spicules and casts of cup corals occur sparsely throughout the zone.

Zone 3 is characterized by a well developed algal biostrome. The insoluble material is present in small amount, and consists of authigenic quartz, chert masses, siliceous oölites and rare sponge spicules. Large chert masses may be seen in this zone in the field but they are not considered in percentages of insoluble material, for in the collection of samples every effort was made to obtain dolomite free of visible chert.

Zone 4 is an interval of thin-bedded dolomites, designated as "punky beds"* in the field, alternating with thicker strata of the same general lithology. The insoluble content is quite small, and is composed mainly of fine authigenic quartz, small chert masses,

* These fine-grained, thin-bedded dolomites have been designated in the field as the "punky beds", because when struck with a hammer they give a thud.
siliceous oölites, dolocastic chert, sponge spicules and glauconite grains.

Zone 5 is characterized by thick, massive beds of dolomite, somewhat cherty in places and often containing a discontinuous algal biostrome or a conglomerate horizon. Authigenic quartz crystals, both singly and in clusters, are common in this zone along with spongy chert. Chert and siliceous cement increase upward in some sections. The large masses of chert are not included in the residue percentages.

Zone 6 represents a sequence of arenaceous strata above the Oneota dolomite and probably correlates with either the New Richmond or St. Peter sandstone. The insoluble residue which comprises nearly the entire rock consists of siliceous cementing material, detrital quartz grains and a few siliceous oölites.

In summary it may be said that this study of the Oneota dolomite has shown that underlying and overlying formations (Madison sandstone below and New Richmond or St. Peter sandstone above) can be sharply differentiated from the Oneota on the basis of the amount and character of the residues; but that it is not possible to identify definite zones or horizons within the dolomite formation with any degree of success.

3. A SEDIMENTATIONAL STUDY OF A PART OF THE TREMPEALEAU FORMATION IN SOUTHERN WISCONSIN.

Bernhard O. Hougen.*

Purpose.—This study was made to determine: (1) the percentages of soluble (calcareous) and insoluble (sand, silt and clay) materials in selected exposures of the Trempealeau formation in southern Wisconsin; and (2) whether the insoluble constituents possessed characteristics which might be used either for interpreting conditions of sedimentation at the time of deposition or for correlation purposes.

Procedure.—Samples were collected, with the aid of G. O. Raasch, from four exposures near Avoca, Muscoda, Gotham and Kingston, along or near the Wisconsin river valley (See Fig. 1).

*Submitted as a thesis for the degree of Bachelor of Philosophy (Geology) at the University of Wisconsin, 1933. Abstracted by R. K. Shrock.
Hand specimens (about 2"x3"x3") were taken every three feet throughout the section unless individual strata were under that figure in thickness, in which case several small fragments were taken from each of the thin beds in the three-foot zone.

After a microscopic examination of the sample was made (both before and after crushing), about 20 grams of the crushed rock (fragments about ½ inch in greatest dimension) were digested in a 50% solution of hydrochloric acid at a temperature slightly below boiling for twenty four hours. The sample was then washed and treated with new acid. This was continued until all solution action ceased. The acid was then decanted and the residue washed free from acid with distilled water. The residue was then dried and weighed, after which it was moistened and poured into a specially devised water classifier, where the clay content was separated from the sand-silt fraction.* The latter fraction, saved in the process of separation, was then dried and weighed. Percentages of soluble material, sand-silt material and clay were then computed. It must be emphasized that while the methods used did not give highly accurate results, nevertheless, they are quite satisfactory for the purposes intended because of the variation of the formation both vertically and horizontally.

Results.—The insoluble residues from the Trempealeau formation in the four sections studied consist mainly of clay; silt; rounded and angular quartz grains; authigenic quartz, and possibly feldspar, crystals; and glauconite. The soluble material is almost entirely calcite and dolomite (relative percentages were not determined), with a very small amount of iron oxide.

The results of the analyses are shown on Fig. 1. The St. Lawrence dolomite always is impure carrying as much as 30% of insoluble material. The Lodi shale carries a surprising amount of soluble matter, in some cases well over 50%. It is also apparent that the clay and sand-silt material may or may not be fairly evenly balanced. The Jordan sandstone was found to contain a considerable amount of calcareous material in most of the samples. This situation is not always apparent in outcrops. The main importance of the insoluble residue data is to give a more accu-

*Considerable experimentation was necessary before the proper procedure and apparatus were discovered. Finally, however, it was possible to remove the clay (particles less than ½60 mm. in largest dimension) rather successfully.
rate picture of rock composition than has been available before. In themselves the residues do not possess characteristics which are distinctive, and hence are of little value for correlation purposes.

**LOCATIONS**
1. NE ¼ Sec. 18, 8 N, 1IE.
2. NE ¼ Sec. 14, 8 N, 1IE.
3. SE ¼ Sec. 28, 9 N, 2IE.
4. Sec. 24, 1 mile SW of Kingston.

Fig. 1. Chart of stratigraphic sections showing the relative amounts of soluble matter, sand and silt, and clay at various horizons of the Trempealeau formation in southern Wisconsin. Sections were measured by G. O. Rasch.

4. **INSOLUBLE RESIDUES OF THE MENDOTA (ST. LAWRENCE) DOLOMITE.**

Ray E. Wilcox.

**Purpose.**—This investigation was undertaken to find the amount, character and possible uses of any insoluble materials that might be present in the Mendota dolomite of the Madison

---

*Submitted as a thesis for the degree of Bachelor of Philosophy (Geology) at the University of Wisconsin, 1933. Abstracted by R. R. Shrock.*
vicinity. Correlation of the Mendota with the St. Lawrence dolomite follows the practice of Wisconsin geologists.

Procedure.—Samples were collected with the aid of Mr. F. T. Thwaites. Some well cuttings were also obtained from him. The analyses were made in the Sedimentation at the University of Wisconsin, with laboratory materials furnished by the Milwaukee Public Museum.

From 25 to 50 grams of the sample were crushed to fragments averaging about 0.3 cms. in greatest dimension, placed in a beaker and wetted with distilled water, and then dissolved in a 50% solution of hydrochloric acid at a temperature slightly below boiling. It was in some cases necessary to wash the samples and then add new acid several times before all of the soluble matter was dissolved. After all action had ceased the acid was decanted and the residue washed free of acid. Care was taken not to pour off any of the residue. It was then washed onto a tared watch glass, dried, and weighed. It was then returned to a beaker, wetted and the fine clayey material removed by careful decantation. The result was a clean, granular residue whose particles were above clay dimensions. This residue was then washed onto a tared watch glass, dried and weighed. The method just outlined gave a reasonably satisfactory separation of the sand-silt and clay fractions of the original residue.

Results.—The results of the investigation are tabulated on Fig. 1. The insoluble residues consisted of the following: (1) white, green or brown clay of about the same amount in most of the samples; (2) rounded and frosted detrital grains of quartz, scattered throughout the formation, and showing incipient secondary enlargement; (3) authigenic feldspar crystals consisting of a shell of feldspar of authigenic origin around a nuclear detrital grain of microcline; (4) fairly large flakes of detrital muscovite; (5) fine to coarse, dark to light green, rounded and polished grains of glauconite; and (6) irregular masses of soft, argillaceous, cinder-like material containing ferruginous matter.

Three significant facts are apparent from the data tabulated on Fig. 1. (1) The basal part of the Mendota dolomite is marked by a thin conglomerate, which is composed of sand and clay, and
dolomite. The analyses show that the insoluble material is high (25% to over 50%). It consists of rounded and frosted grains of quartz, some of which show secondary enlargement; polished glauconite grains; mica flakes; and some silt and clay. (2) The algal layers or biostromes are conspicuous by their very low insoluble content. Obviously the water in which the algae grew must have been free of muddy matter. (3) The strata in the Pheasant Branch section, and the upper beds of the Farwells Point section, carry considerable insoluble material, but it is to be noted that algal layers are absent. Perhaps the high residues furnish the explanation for the absence of the algae.

While the amount and character of the insoluble residues of the Mendota formation are distinctly different from those of the underlying Franconia or of the overlying Lodi shale, it will be necessary to obtain much more information on all of the residues before they can be used successfully for correlation purposes. The best procedure will be to use the residues in conjunction with the lithologic data and field relations.
Fig. 1. Chart of stratigraphic sections showing the amount and character of the insoluble residues of the Mendota dolomite. The base of the dolomite is marked by the top of the conglomeric matrix.

Legend:
- Sandstone
- Dolomite
- Breccia
- Cryptocrystalline
- Conglomerate

Scale:
- % Coarse Residue
- % Fine Residue
- % Soluble

Sections:
1. Pleasant Branch
2. Farrell's Point
3. Sun Prairie Road
4. Waukesha
5. Lake Waukesha
6. U.S. Highway 15
7. Colliday's Point

Locations of Sections:
- Lake Mendota
- Lake Monona
- Lake Waukesha
- Madison

Location of Area Studied:
- Madison

Shrock—Insoluble Residues from Sedimentary Rocks.