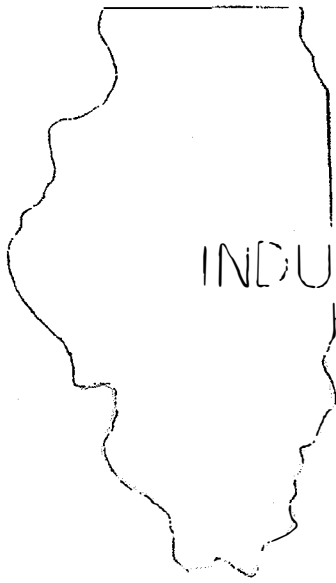


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BENEFICIATION OF KAOLINITE CLAY FROM
SILICA SAND WASHINGS

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ABSTRACT

In the processing of St. Peter Sandstone for the production of silica sand, substantial amounts of clay material and fine-grained sand are washed out and discarded. Although it has been known for many years that this clay material was relatively pure kaolinite, no commercially useful techniques for separating the clay from the sand in these washings have as yet been reported.

After the purity and high quality of the kaolinite existing in these wastes was verified in the laboratories of the Illinois State Geological Survey, a process was developed to separate the clay from the fine-grained sand by using hydrocyclones. Additional tests were conducted on this beneficiated clay to measure some of its commercial properties. The results of these tests suggest areas of possible industrial application.

BENEFICIATION OF KAOLINITE CLAY FROM SILICA SAND WASHINGS

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INTRODUCTION

Silica sand has been mined in Illinois from the St. Peter Sandstone since the turn of the century, especially in the area around Ottawa, on the Illinois River, in the north-central part of the state. Production of this sand, consumed mainly by the glass and foundry industries but also sought after for many other uses, has grown with the economy, and last year's output in Illinois exceeded 4,000,000 tons.

Such an important commodity was given the early attention of the Illinois State Geological Survey, and a complete report by J. E. Lamar was issued in 1928. The mining and processing of the sand has changed very little during the intervening 40 years, so that much of Lamar's data and descriptions are applicable today. More recently, the stratigraphy of the St. Peter Sandstone in north-central Illinois has become better known from the work of Willman and Payne (1942). We have borrowed generously from Lamar's publication and we wish to acknowledge the contribution of both of these studies to our work.

Lamar's report showed that the St. Peter Sandstone contained a small amount of kaolinite clay. Most uses of silica sand require extreme purity, and thus most of the sand shipped today is washed. The washing process removes both fine-grained sand and clay, which always has been discarded in waste pits or ponds. Even though chemical analyses in Lamar's report showed the clay portion of this slime waste to be a remarkably pure kaolinite, no commercially useful techniques for separating the clay from the fine-grained sand have been reported. This paper, therefore, describes our successful attempt to beneficiate these silica plant washings and, by effecting a clean separation, recover a pure kaolinite clay.

GEOLOGY AND MINERALOGY

The St. Peter Sandstone is early Middle Ordovician in age (over 450 million years old) and crops out at four areas in Illinois (fig. 1). However, silica sand is currently being mined and washed from the St. Peter only in LaSalle and Ogle Counties. In these areas, the formation varies from 140 to over 200 feet thick. It is mined to a maximum depth of only about 90 feet, due to water problems and increasing iron content at depth. The overburden in the outcrop areas is glacial drift of variable thickness.

The St. Peter Sandstone is very poorly cemented and consolidated. Its principal lithologic features are its purity, homogeneity, and roundness of the sand grains. It is an extremely pure orthoquartzite, containing only about 0.1 percent heavy minerals and 1 to 5 percent clay minerals. The heavy minerals are mostly zircon, with some tourmaline and anatase, and these occur mainly in the finer grained sand fractions.

The clay minerals are principally kaolinite at the outcrops, with minor amounts of illite. Away from the outcrop areas and deeper in the Illinois Basin, where the formation water changes from fresh to saline, the clay mineral present in the St. Peter is pure illite. This distribution of clay minerals suggests an alteration of pre-existing unknown material to kaolinite due to flushing by fresh ground water at the outcrop and in the aquifer. The clay is rather easily removed from the sand by water-washing, leaving a very pure sand (99.8 percent SiO₂).

SILICA PLANT OPERATIONS

Silica sand is mined from the St. Peter Sandstone at seven separate sites in Illinois. Six plants are located in LaSalle County, and one is in Ogle County.

All of the mining is done by open-pit methods. The overburden, which varies from 6 to 30 feet in thickness, is removed by bulldozer and scrapers. The sand body is then drilled from the top and the face blasted down with light charges of dynamite. The sand is mined in a single face in all but one case, where it is taken in two benches because of its greater minable thickness and the significant difference in grain size that exists between the benches. The loose sandstone is either mechanically loaded or hydraulically sluiced down into sumps. It is transported to the plant by trucks, conveyors, or pipelines.

Although the plants vary in the type of equipment they possess, the general method of processing the sand is similar in all cases. The mined rock is reduced to sand sizes by crushing and screening. This sand is scrubbed and washed with excess water and then drained. As stated previously, almost all of Illinois' silica sand production is washed, although some unwashed sand is still shipped to foundries.

The washed and drained sand is dried and is either sold without sizing or screened for special uses. Two Illinois plants also grind washed sand to make a silica flour product. Another plant coats a portion of its sand production with a thermo-setting resin for use in foundry sand cores.



Fig. 1 - Location of the four St. Peter Sandstone outcrop areas in Illinois. Size of circles reflects relative extent of the outcrops.

Because the washing operation generates a clay-rich waste product, the beneficiation of which will be discussed in this paper, a closer look at this technique is in order. The simplest washing operation involves passing jets of water upward through a fluidized bed of sand in a concrete tank. The overflow water carries fine-grained sand and clay up and out of the bed; this overflow is then pumped to the waste pond. More complex washing schemes utilize scrubbers, followed by either mechanical classification or hydrocyclones, or a combination of both. The different ratios of sand to clay among the wastes of the seven plants reflect the variable nature and degree of washing of the raw sand.

The washing plant wastes are pumped to settling ponds where the clarified water is reclaimed for plant reuse. The waste undergoes a natural size separation along the length of the pond, with the sand and larger silt sizes settling first and forming a delta at the point of ingress. The clay eventually settles farther out in the pond; completely sand- and silt-free clay can be obtained by sampling the bottom surfaces of these settling ponds at the maximum distance from the influent points.

The seven operating plants (whose names appear in the Appendix of this paper) were each visited on at least two occasions to determine if there were any significant difference in the washing plant outflows on separate days. The sand-clay ratio within each plant remained substantially the same. Between plants, the sand-clay ratio varied considerably, owing to variations in the clay content of the sandstone at the particular locality and the washing method applied to the crude sand. The clay from each plant was found to be mainly kaolinite of rather high purity, with little variation in its character from plant to plant.

LABORATORY EXAMINATION OF RAW WASTE

Sieve Analysis

The methods of washing the clay out of the sand utilized in each of the plants vary considerably in their efficiency. Table 1 shows three typical examples of the size fractions occurring in plant outwashes sampled at the discharges to the tailings pond.

Plant "B," the most modern of the three, uses a spiral classifier in the circuit, whereas the other two operations use only the washing and settling technique. In every case, however, the sand from the washer is almost completely free (less than 0.1 percent) of the clay material that occurs in the natural sandstone.

Sizing by Hydrometer Method

The less than 270-mesh fractions obtained by wet-sieving of plant outflows were analyzed for size distribution by the hydrometer method. Table 2

TABLE 1 - SIEVE ANALYSES OF THREE SILICA PLANT WASTE OUTFLOWS

Size (mesh)	Plant A (%)	Plant B (%)	Plant C (%)
+ 48	0.23	0.00	0.37
- 48—+ 65	1.29	0.14	0.85
- 65—+100	6.09	0.92	1.65
-100—+150	22.95	3.34	4.53
-150—+200	24.00	9.34	12.00
-200—+325	15.81	12.77	18.90
-325—+400	2.81	3.57	5.01
-400	26.82	69.92	56.69

TABLE 2 - DISTRIBUTION OF FINE-GRAINED SIZES IN PLANT WASTE OUTFLOWS
BY THE HYDROMETER METHOD

Size (E.S.D.)*	Plant A (%)	Plant C (%)	Plant D (%)	Plant E (%)	Plant F (%)	Plant G (%)
greater than 53 μ †	70.8	41.0	69.0	12.0	42.5	42.0
less than 53 μ	29.2	59.0	31.0	88.0	57.5	58.0
less than 40 μ	21.5	47.3	27.5	80.4	51.2	50.4
less than 20 μ	17.5	45.3	21.2	71.9	46.3	42.3
less than 10 μ	16.0	42.2	18.2	66.3	43.2	35.5
less than 5 μ	14.4	39.5	16.3	63.1	39.9	31.0
less than 2 μ	12.1	33.4	14.7	51.5	33.6	24.8

* Equivalent spherical diameters; † μ = micron = 1/1000 millimeter; 53 μ = 270 mesh

lists the percentage of this total fraction occurring below certain size values (of equivalent spherical diameters). These data show the wide variation in efficiency of the different washing processes employed by the industry.

BENEFICIATION OF RAW WASTE BY HYDROCYCLONING

In preparation for the hydrocyclone process, the plant outflows were thickened in the laboratory to 35 percent solids. No settling aids were needed owing to the rather highly flocculated condition of the pulp. This slurry was then put through a 30 mm laboratory cyclone with a vortex-to-apex ratio of 4½:1 and a water pressure of 15 to 20 pounds per square inch. These values had been determined by preliminary experimentation to give the optimum sand-clay separation. The vortex overflow was considered a final clay product and

was removed from the system; the apex underflow was repulped and recycled. By adding a second cyclone in series to reclassify the apex underflow of the primary cyclone, and by directing the middling (vortex) product of this secondary cyclone back to the original plant outflow, a clean sand and clay separation can be effected in continuous operation. A suggested schematic flow sheet of a plant to accomplish this beneficiation is shown in figure 2.

The results of test runs on actual plant waste outflows (undried) by the method described above are given in table 3.

LABORATORY INVESTIGATION OF BENEFICIATED CLAY

The kaolinite material from the vortex was found to be uniformly fine grained. For example, the clay portion from Plant A contained 72.11 percent minus 5-micron, 83.20 percent minus 10-micron, and 99+ percent minus 20-micron material. Most of the material occurring in the size range between 10 and 20 microns was composed of aggregates rather than discrete particles. The clay material from Plant C contained more than 95 percent minus 5-micron material after passing through a hammer mill to break up aggregates.

Kaolinite, by definition, is "a hydrous aluminum silicate, $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. $\text{Al}_2\text{O}_3 = 39.5$ percent, $\text{SiO}_2 = 46.5$ percent, and $\text{H}_2\text{O} = 14.0$ percent," (Hurlbut, 1944). The close correspondence of the values in table 4 with these theoretical values attests to the purity of the kaolinitic material produced by the hydrocycloning.

TABLE 3 - SIZE DISTRIBUTIONS OF FINAL LABORATORY CYCLONE PRODUCTS

Size (mesh)	Plant A (%)	Plant B (%)	Plant C (%)
Primary Cyclone Overflow (Clay)			
+400	0.40	0.00	0.00
-400	99.60	100.00	100.00
Secondary Cyclone Underflow (Sand)			
+ 48	0.24	0.00	0.82
- 48—+ 65	1.11	0.42	1.88
- 65—+100	10.64	2.79	3.65
-100—+150	32.77	10.14	10.01
-150—+200	24.12	28.39	26.50
-200—+325	16.42	38.87	41.70
-325—+400	2.77	10.86	11.07
-400	11.93	8.53	4.37

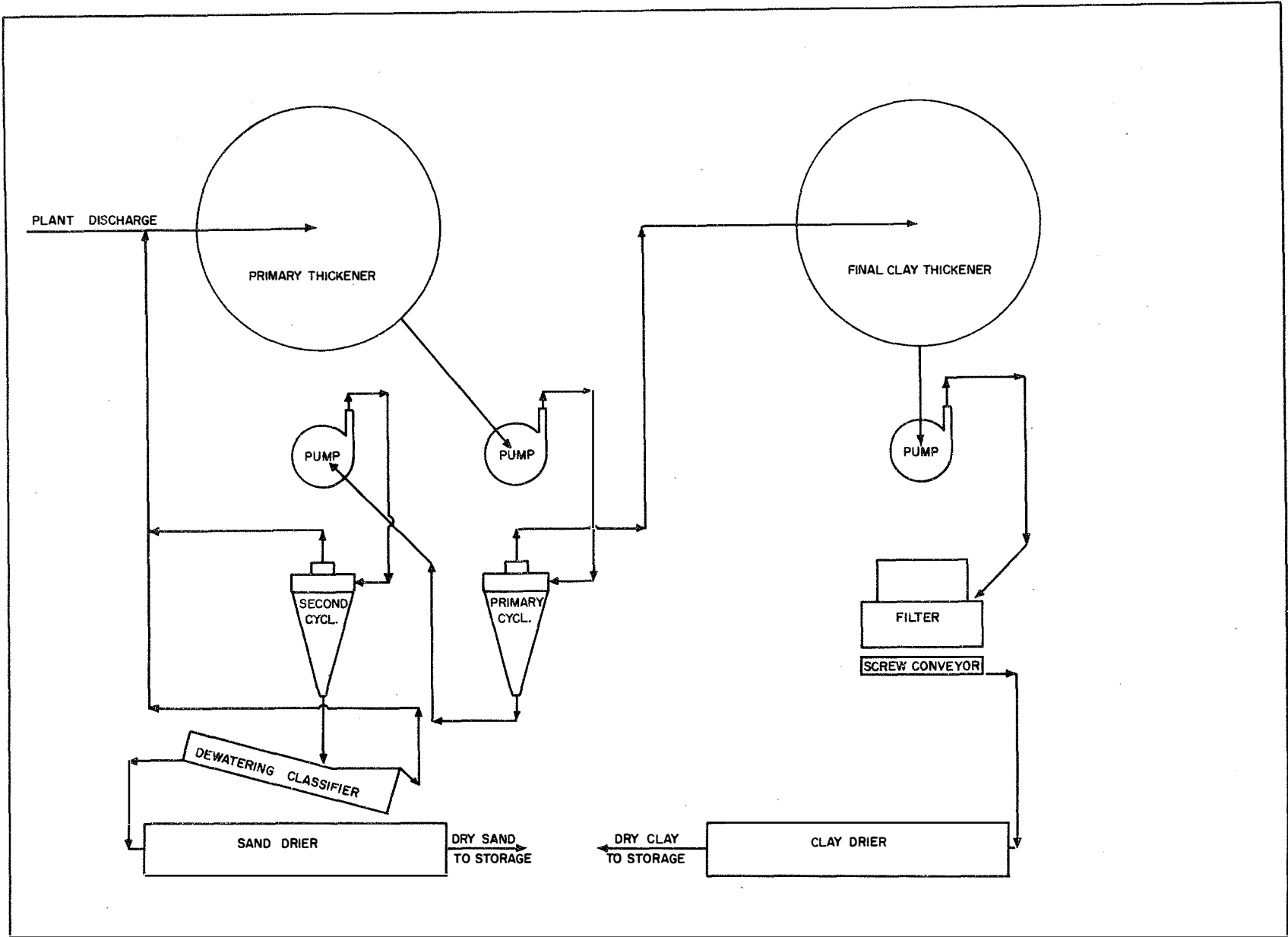


Fig. 2 - Suggested schematic flow-sheet for separating clay from fine sand in silica plant waste discharge.

TABLE 4 - PARTIAL CHEMICAL ANALYSES OF FINAL LABORATORY CYCLONE PRODUCTS*

	Plant A underflow (%)	Plant A clay (%)	Plant B clay (%)	Plant C clay (%)
SiO ₂	96.17	44.41	49.23	45.32
Al ₂ O ₃	2.11	38.08	34.11	36.10
Loss on ignition	0.84	13.38	11.85	13.13

* Analyses by L. R. Camp, Illinois State Geological Survey

Additional tests conducted on the clays obtained by the hydrocyclone procedure included tests for brightness, iron content, pyrometric cone equivalent, and surface area. The results are shown in table 5.

The X-ray diffraction patterns of clays beneficiated from all of the plant wastes show the kaolinite to be extremely well crystallized. Study of the clay's surface morphology with the scanning electron microscope reveals excellent development of crystal shape and size in the natural sandstone. The crystallinity, shape, and size of the clay crystals in the sandstone indicate an authigenic origin. However, clay that had passed through the mining, washing, and beneficiation processes was observed to have deteriorated in crystal shape and size.

Bulk densities of random samples of the cyclone overflows were found to be in the range of 10 to 14 pounds per cubic foot after being ground in a Raymond mill.

TABLE 5 - MISCELLANEOUS PROPERTIES OF BENEFICIATED CLAY

Samples	Brightness* (%)	Fe ₂ O ₃ (%)	P.C.E.**	Surface area [†] (m ² /g)	% Illite by X-ray ^{††}
Plant A	57.5	0.54	33+	16.7	2
Plant B	55.0				1-2
Plant C	67.5	0.83	33	16.0	2
Plant D	44.0	1.36	29		4
Plant E	63.2	1.60	33		5
Plant F	55.5	1.27	30		3
Plant G					4
Georgia kaolin	76-87	0.3-0.7	33		

* Compared to MgO - 100%; ** Pyrometric Cone Equivalent; † Dynamic gas adsorption;

†† Less than 2-micron size material

USES OF KAOLINITE

There are numerous uses for kaolinite. The greatest volume is consumed in the paper industry, where it is used as both filler and coating agent. Kaolinitic clay also is used as an extender in the rubber industry; as an agent to give body to printing inks and to make adhesives for such materials as laminated fiberboard and corrugated board; as an extender in paints to reduce their cost and improve suspension of pigments; and as a filler in plastics to make them resistant to chemicals, give them opacity, and to improve surface finish, ease of molding, and flow characteristics. Kaolinite also is used in mastics, putties, caulking compounds, in the conditioning of leather and agricultural chemicals, and as a carrying agent or diluent in pesticides.

Each of these uses demands special characteristics. Brightness, purity, and iron content are of particular importance in the paper, refractories, and pottery industries, whereas the particle size and crystallinity are more important in the extender-additive uses, in printing inks, adhesives, pesticides, filler-type applications, and agricultural chemical conditioning.

SUMMARY

A commercially feasible process has been developed for the recovery of kaolinite clay as an industrial mineral product from the washings of Illinois silica sand plants.

The process involves hydrocycloning the plant outwash in two or more stages, resulting in a sand-free clay and a very low clay-content sand. Both of these products will meet commercial specifications.

The purity of the kaolinite was found to be good. This has been shown by X-ray and chemical analyses. Pyrometric cones and the brightness-meter tests show that the material meets most of the specifications for pure kaolinite. The iron content no doubt would render it less desirable for paper clay than other commercially available clays; however, for most other uses, the material would be quite satisfactory.

REFERENCES

- Hurlbut, C. S., Jr., 1944, Manual of mineralogy: John Wiley and Sons, Inc., N. Y.
- Lamar, J. E., 1928, Geology and economic resources of the St. Peter Sandstone in Illinois: Illinois Geol. Survey Bull. 53, 175 p.
- Willman, H. B., and J. N. Payne, 1942, Geology and mineral resources of the Marseilles, Ottawa, and Streator Quadrangles, with Introduction to mineral resources by W. H. Voskuil: Illinois Geol. Survey Bull. 66, 388 p.

APPENDIX

SILICA SAND PLANTS OPERATING IN ILLINOIS

Bellrose Silica Company
P. O. Box 460
Ottawa, Illinois 61350

Arrowhead Silica Company
Troy Grove, Illinois 61372

Shabbona Silica Company
P. O. Box 205
Serena, Illinois 60549

Ottawa Silica Company
P. O. Box 577
Ottawa, Illinois 61350

Wedron Silica Company
Wedron, Illinois 60557

Pure Silica Company
Troy Grove, Illinois 61372

Martin-Marietta Corporation
Manley Sand Division
110 East Main Street
Rockton, Illinois 61062

INDUSTRIAL MINERALS NOTES SERIES

- * 1. Heavy Minerals in Illinois Glacial Sands: R. S. Shrode. 1954.
- * 2. Lightweight Brick from Clay and Peat or Shredded Corncobs: J. E. Lamar. 1955.
- * 3. (1) The Industrial Minerals Industry in Illinois in 1955: W. H. Voskuil and W. L. Busch. (2) Trace Elements and Potash in Some Illinois Gravels: J. E. Lamar and R. S. Shrode. 1956.
- 4. Subsurface Dolomite in Lake, McHenry, and Part of Northwestern Cook Counties: M. E. Ostrom. 1956.
- * 5. (1) Gypsum and Anhydrite. (2) Fluorspar for Controlling Vanadium Staining. (3) Relation of Sulfate and Chloride to Ore Deposits in the Ordovician Rocks of Jo Daviess County: J. C. Bradbury. (4) Possibilities for Calcitic Limestone Underground in Kankakee and Iroquois Counties: J. W. Baxter. 1957.
- 6. Trend in Fuel Uses in Selected Industrial Mineral Products, 1947 and 1954: W. H. Voskuil. 1957.
- 7. Outlying Occurrences of Galena, Sphalerite, and Fluorite in Illinois: J. C. Bradbury. 1957.
- 8. Origin of Illinois Sand and Gravel Deposits: J. E. Lamar and H. B. Willman. 1958.
- * 9. Shales as Source Material for Synthetic Lightweight Aggregate: W. A. White. 1959.
- *10. Recent Price and Cost Trends Relating to Stone, Sand, and Gravel Production in Illinois: H. E. Risser. 1959.
- *11. Rare Earth and Trace Element Content of an Unusual Clay on Hicks Dome in Hardin County, Illinois: J. C. Bradbury. 1960.
- *12. A Survey of Some Illinois Materials Possibly Useful as Pozzolans: W. A. White and J. S. Machin. 1961.
- 13. Summary of Illinois Mineral Industry, 1951-1959: W. L. Busch. 1961.
- *14. Illinois Stone Production in 1959: W. L. Busch. 1961.
- *15. Black and Brown Terrazzo Chips from Southern Illinois Limestones: R. D. Harvey. 1962.
- *16. Refractory Clay Resources of Illinois: W. A. White. 1962.
- 17. Pelletizing Illinois Fluorspar: H. W. Jackman, R. J. Helfinstine and Josephus Thomas, Jr. 1963.
- *18. Permanent Expansion in Bricks: W. A. White. 1964.
- 19. Binding Materials Used in Making Pellets and Briquets: G. R. Yohe. 1964.
- 20. Chemical Composition of Some Deep Limestones and Dolomites in Livingston County, Illinois: J. W. Baxter. 1964.
- 21. Illinois Natural Resources—An Industrial Development Asset: H. E. Risser. 1964.
- *22. Illinois Clays as Binders for Iron Ore Pellets: H. W. Jackman, M. B. Mirza, W. A. White, and R. J. Helfinstine. 1965.
- 23. Limestone Resources of Jefferson and Marion Counties, Illinois: J. C. Bradbury. 1965.
- 24. Thermal Expansion of Certain Illinois Limestones: R. D. Harvey. 1966.
- 25. Annotated Selected List of Industrial Minerals Publications: Compiled by J. E. Lamar. 1966.
- 26. Binders for Fluorspar Pellets: H. W. Jackman, M. B. Mirza, R. J. Helfinstine, and D. R. Dickerson. 1966.
- 27. High-Purity Limestones in Illinois: J. E. Lamar. 1966.
- 28. Illinois Clays as Binders for Iron Ore Pellets—A Further Study: H. P. Ehrlinger III, M. B. Mirza, L. R. Camp, and H. W. Jackman. 1966.
- 29. Clay and Shale Resources of Clark, Crawford, Cumberland, Edgar, Effingham, Jasper, and Vermilion Counties: W. A. White and W. E. Parham. 1967.
- 30. Lightweight Bricks Made with Clay and Expanded Plastic: H. P. Ehrlinger III, B. F. Bohor, L. R. Camp, and Suresh Khandelwal. 1967.
- 31. Clays as Binding Materials: G. R. Yohe. 1967.
- 32. Silica Sand Briquets and Pellets as a Replacement for Quartzite: H. P. Ehrlinger III, M. L. Schroder, L. R. Camp, and H. W. Jackman. 1968.
- 33. A New Use for Illinois Clay Materials in Pesticide Formulations: B. F. Bohor and Suresh Khandelwal. 1968.
- 34. Neutron Activation Analysis at the Illinois State Geological Survey: R. R. Ruch. 1968.
- 35. Computer-Calculated Lambert Conformal Conic Projection Tables for Illinois (7.5-Minute Intersections): P. B. DuMontelle, P. C. Heigold, Manoutchehr Heidari, and D. H. Swann. 1968.

* Out of print