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DIATOMITES OF THE PACIFIC NORTHWEST  
AS FILTER-AIDS

By

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# DIATOMITES OF THE PACIFIC NORTHWEST AS FILTER-AIDS <sup>1</sup>

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## INTRODUCTION

### PURPOSE OF INVESTIGATION

Diatomite, also known as diatomaceous earth, diatomaceous silica, kieselguhr, and moler, has been used extensively for many years, but only during the past two decades has it become an important industrial mineral. United States sales for 1936-38 totaled almost 280,000 tons,<sup>7</sup> but relatively little (with the exception of that sold by an Oregon company affiliated with a larger California company) was sold by Pacific Northwest producers. In attempting to develop some of the many Pacific Northwest diatomite deposits, these producers have been handicapped by lack of published technical information regarding processing methods and the suitability of different deposits for specific uses. To determine some of the physical and chemical properties of known Pacific Northwest diatomites, the present investigation was started in 1938 by the Bureau of Mines in cooperation with the College of Mines, University of Washington. Figure 1 shows the location of deposits sampled.

Because most relatively pure diatomites can be used as concrete admixtures or as inert fillers in rubber, paints, and plastics, this investigation concentrated upon determining some filter-aid characteristics and their relationship to other physical properties. A filter-aid is a powdered material that, when added to solutions to be filtered, improves flow rate, filtrate clarity, or both. Some chemical analyses also were made.

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<sup>1</sup> Work on manuscript completed May 1944.

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<sup>5</sup> Research fellow, University of Washington, 1940-41.

<sup>6</sup> Research fellow, University of Washington, 1941-42.

<sup>7</sup> U. S. Bureau of Mines, Minerals Yearbook—Review of 1940: 1941, pp. 1241-42.

for the first part of the investigation, permission to construct a similar bomb, and for commercial filter-aids; the Kittitas Diatomite Co., Kittitas, Wash., the Dia-Causti-Lite Co., Quincy, Wash., the Deer Park Natural Pigments Co., Spokane, Wash., the Oromite Co., Terrebonne, Oreg., and the Owyhee Processing Co., Grand View, Idaho, for samples of their processed products; to all other diatomite property owners or leasers who helped to obtain samples or information for this investiga-

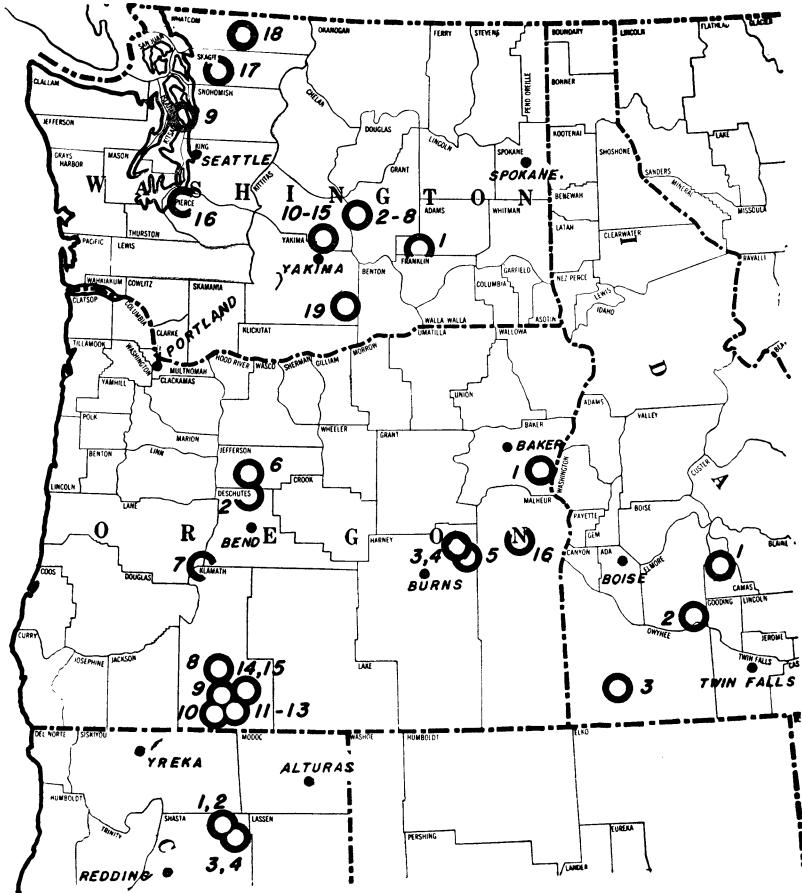


FIGURE 1.—Map showing location of sampled Pacific Northwest diatomite deposits.

tion; and to the Works Progress Administration for laboratory assistance under Project 2923 in grinding samples during the first phases of the investigation.

### PROPERTIES AND USES OF DIATOMITE

When diatoms—microscopic, flowerless water plants related to algae—die and then decay, oil and a silica shell remain. Beds of these skeletal, amorphous silica<sup>8</sup> remains are called diatomite, and 1 cubic

<sup>8</sup> Some evidence of high cristobalite has been reported. Mulyran, Henry, *Geology, mining, and processing of diatomite at Lompoc, Santa Barbara County, Calif.*: Trans., Am. Inst. Min. and Met. Eng., vol. 129, 1938, pp. 469-500.



inch may contain as many as 50,000,000 skeletons of one or more of the known 10,000 species. Because diatoms live in all types of water all over the world, but in greatest number in Arctic and Antarctic regions, only a microscopic examination shows whether the diatomite consists of fresh- or marine-water diatom skeletons or whether it consists of warm- or cold-water diatom skeletons.

**PHYSICAL PROPERTIES <sup>9</sup>**

*Color.*—When pure, the color is almost white, but impurities such as carbonaceous matter or iron oxide may darken it.

*Optical.*—Diatomite is isotropic, and the refractive index ranges from 1.41 to 1.46, which is almost the same as that for opaline silica.

*Density.*—The apparent density for powdered diatomite varies from 7 to 20 pounds per cubic foot and to 60 pounds per cubic foot for lump material containing impurities.

*Bed moisture.*—Bed moisture may be greater than 65 percent, but in arid regions it usually varies from 15 to 25 percent.

*True specific gravity.*—The true specific gravity of diatomite is 2.1–2.2, or the same as opaline silica, such as opal. Table 1 gives the true specific gravities of four diatomites, as determined in this investigation with picnometers.

TABLE 1.—*True specific gravity of four diatomites*

<i>Sample</i>	<i>Sp. gr.</i>
Commercial filter-aid from California.....	2.063
Commercial powder from central Washington.....	1.927
Crude, impure, containing carbonaceous matter and clay, from Western Washington.....	2.355
Crude from central Washington.....	1.936

*Fusion point.*—When pure, diatomite should have the same fusion point as pure silica, 1,710° C. (3,110° F.).

*Thermal conductivity.*—The low thermal conductivity of diatomite increases with an increase in temperature and generally increases with an increase in density or weight per unit volume.

*Thermal expansion.*—Unless diatomite has been subjected to high temperatures, it has little thermal expansion. If, however, the material has been subjected to high temperatures, thermal expansion depends upon the silica form present.

*Absorption.*—Diatomite, either natural or calcined, has only weak adsorptive powers but has excellent absorptive powers. Acids, liquid fertilizers, nitroglycerine, alcohol, and water are appreciably absorbed by diatomite, which in some instances may absorb as much as three times its own weight of alcohol to form a plastic mass.

*Adsorption.*—Salts are adsorbed by diatomite—sodium thiocyanate less than sodium nitrate, and organic liquids (generally) less than water. Diatomite fragments cause an increase in adsorption, because more working or effective surface is exposed per unit weight. An alkaline, alcoholic phenolphthalein solution, when added to some diatomites, changes from pink to colorless.<sup>10 11</sup>

<sup>9</sup> Essentially represents an abstract from: Hatmaker, Paul, Diatomite: Bureau of Mines Inf. Circ. 6391, 1931, 20 pp.  
<sup>10</sup> Calvert, Robert, Diatomaceous Earth: The Chemical Catalogue Co., New York City, 1930, 252 pp.  
<sup>11</sup> Hoeglund, E. J., and Autzler, J. E., Adsorption by Diatomaceous Filters: Jour. Phys. Chem., vol. 40, 1936, pp. 415–422.

*Colloidal.*—It is doubted whether many diatomite uses are based upon its colloidal properties; instead, most applications are based upon rather well-understood chemical and physical characteristics that do not involve colloids.

#### CHEMICAL PROPERTIES

Goodwin<sup>12</sup> states that diatomite acts the same as ordinary opal upon ignition, giving up combined water between 500° (932° F.) and 800° C. (1,472° F.), and is usually chemically inert, although impurities such as clay and carbon may affect its chemical properties. He further states that diatomite is insoluble in neutral solutions, insoluble in acids other than HF, and insoluble in cold, caustic alkaline solutions such as NaCO<sub>3</sub>.

#### USES

Because this report deals primarily with Pacific Northwest diatomites' filter-aid characteristics, little more than a mere listing will be given regarding other uses. More complete information may be obtained from the references cited and the bibliography. Hatmaker<sup>13</sup> states:

Diatomite has many important uses because of the wide adaptability of its extraordinary physical properties. It has rapidly assumed greater and greater importance in modern industry as markets are developed and industry becomes familiar with the various benefits to be derived from its use. One of the interesting features in respect to its marketing is that new uses continually are being found for diatomite which more than make up for the decrease in some of the older ones. An example of this is the employment of diatomite as an absorbent in dynamite. It is no longer used in this way, and what was once a major use now is of negligible importance. On the other hand it is comparatively recently that diatomite has been employed as a concrete admixture. Its use as a filler for battery boxes has grown rapidly in recent years.

The more important uses are: First, as a filtration medium; second, as a heat insulation material; and third, as an admixture in concrete.

#### MINOR USES

In addition to the three major uses, Davis,<sup>14</sup> Calvert,<sup>15</sup> and Hatmaker<sup>16</sup> list a number of minor uses, some of which are as follows:

1. Filler for battery boxes, hard rubber, phonograph records, paints and varnishes, oilcloths, and insecticides.
2. Abrasive in polishing and cleansing powders for enamel, glass, furniture, sinks, teeth (dentists' powders), and metal.
3. Absorbent for fertilizers, catalysts, and disinfectants.
4. Reagent in the manufacture of casein glue, sodium silicate, dyes, and a source of pure silica.
5. Packing material used in shipment of chemical that would cause combustion with organic substances if the two materials came in contact.
6. Constituent of cements, crayons, porous chemical ware, tile glazes, and dusting powders.
7. Filler for absorption towers.

*Light-weight refractories.*—Although diatomite has refractory properties similar to fire clays, low mechanical strength has limited its use for this purpose. It has been reported<sup>17</sup> that some Russian

<sup>12</sup> Goodwin, Norris, *Diatomaceous Earth: Chem. and Met. Eng.*, 1920, p. 1158.

<sup>13</sup> Hatmaker, Paul, Page 8 of work cited in footnote 9.

<sup>14</sup> Davis, C. N., *Diatomaceous Earth: Bureau of Mines Rept. of Investigation 2718*, 1925, 14 pp.

<sup>15</sup> Calvert, Robert, Work cited in footnote 10.

<sup>16</sup> Hatmaker, Paul, Work cited in footnote 9.

<sup>17</sup> Basilevich, A. S., *Refractories: Ogneuporui*, vol. 2, No. 2, 1934, pp. 32-38. *Chem. Abs.*, vol. 29, 1934, p. 2323.

diatomites, when mixed with quartzite, sawdust, and 40 to 50 percent water and then fired to 1,200° C. (2,192° F., or about cone 6), produce a lightweight refractory, which may be used up to 1,300° C. (2,372° F., or about cone 11–12).

*Catalyst.*—Diatomite has been used as a catalyst carrier, but relatively recent investigations have indicated that in some instances the diatomite acts as a catalyst. Waterman<sup>18</sup> states that diatomite is used as a catalyst, in the presence of nickel, in naphthalene hydrogenation at high pressures and temperatures above 200° C. (392° F.). Fifteen years before this, Hastings<sup>19</sup> mentioned that diatomite accelerated oil polymerization. At 250° C. (482° F.), polymerization was accompanied by partial decomposition of the oil with the formation of free acids. Although no direct relationship was found between oil composition and degree of polymerization, it was found that diatomite had little effect in a vacuum.

#### FILTER-AID

Since 1876, when sugar refiners first found diatomite filter-aids useful in their processes, the use of such aids has increased until the sugar-products industry is the largest diatomite filter-aid consumer. The powdered diatomite is usually added directly to the liquid to be filtered, and the mixture is then pumped into pressure filters with filter-aid pre-coated filter cloths. Cane-sugar refineries commonly use more filter-aid per ton of sugar than beet-sugar refineries use. A beet-sugar-refinery flow sheet<sup>20</sup> shows that 0.5 pound filter-aid per ton of beets and 10 pounds filter-aid precoat per 100 square feet filter surface were used.

European sugar companies have found that diatomite filter-aids are superior to charcoal for clarification, but that diatomite and animal charcoal mixtures are better than either used alone. One company, which used 500 grams diatomite per 1,000 liters solution or 250 grams per square centimeter of filtering surface, obtained filter cakes 2 cm. thick.<sup>21</sup>

Other important applications using diatomite filter-aids in their clarification processes include mineral, vegetable, and animal oils and fruit-juice extractions. Metallurgical solutions, cleaning fluids, varnishes, lacquers, and liquid soaps also are purified using diatomite filter-aids. A Pacific Northwest dry-cleaning company employs the following general procedure:

Filter cloths are pre-coated using a concentrated cleaning solution mixed with about 6 pounds filter-aid per 100 square feet of filtering surface. After pre-coating the filter cloths, the washer is filled with clean solvent, clothes, and about 1 pound of filter-aid per 100 pounds of clothes, and the cleaning is started.

The filter-aid type and the quantity used have to be determined carefully for any given filtration process. Two important factors

<sup>18</sup> Waterman, H. I., Hydrogenation of Naphthalene in the Presence of Nickel: *Rec. Trav. Chim.*, vol. 53, 1934, p. 821. *Chem. Abs.*, vol. 28, 1934, p. 6136.

<sup>19</sup> Hastings, Elliott, *Diatomaceous Earth: Rock Products*, vol. 32, March 16, 1929, pp. 50–54.

<sup>20</sup> Eisenbest, A., Elliot, R. D., and Sullivan, E. J., Use of Super-Cel in the Sugar Refining Industry: *Ind. and Eng. Chem.*, vol. 21, 1929, pp. 676–684.

<sup>21</sup> Maksimov, O. B., Action of Diatomite at High Temperatures: *Bull., Far Eastern Brouch Arad. Sc. U. S. S. R.*, No. 9, 1934, p. 117. *Chem. Abs.*, vol. 29, 1934, p. 3867.

Ofner, Rudolf, Speedex, a Highly Potent Diatomite: *Listy Cukrovar.*, vol. 55, 1937, p. 481. *Chem. Abs.*, vol. 31, 1937, p. 2976.

involved in the selection are (1) required filtrate characteristics, such as clarity and flow rate, and (2) whether the filter-aid cost is offset by either (or both) an improved product or an increased output. For example, wine filtrates must be free of impurities that are detrimental,<sup>22</sup> whereas some metallurgical filtrates may be relatively full of impurities.

#### PURPOSE OF A FILTER-AID

Filter-aids are used primarily to (1) increase flow rate, (2) remove visible suspended matter, and (3) eliminate bacteria. Carman's<sup>23</sup> experimental results indicated that a filter-aid's main function was to form a porous cake structure giving filter cakes a more open texture, which allows higher flow rates and gives greater rigidity to the filter cake. Higher filtering pressures also may be used. However, if too much filter-aid is used, the increased cake thickness tends to counterbalance these advantages.

#### TYPES OF FILTER-AIDS

Commercial filter-aids may be grouped into three types, each suited for different filtration conditions, as follows:

1. Natural filter-aids. Filter-aids produced by drying, milling, and air-separating diatomite.

2. Calcined filter-aids. Diatomites that have been milled, calcined, and air-separated. Air separation may precede, follow, or precede and follow calcination.

3. Calcined, chemically treated filter-aids. Air-separated diatomite that has been calcined in the presence of a chemical, usually an alkali salt.

*Effect of calcination.*—According to Calvert<sup>24</sup> and others, calcination, usually between 927° and 982° C. (1700°–1800° F.), improves diatomite filter-aids. Calcination causes clay to sinter to diatom edges and act as sand grains, with resulting improved filter rate and in some instances an improved clarity.<sup>25</sup> Thatcher<sup>26</sup> claims that clay forms anhydrous aluminum silicate, which acts as sand and is harmless in a filter-aid. Stockton<sup>27</sup> combined calcination and air separation to remove fines, either by sintering them to larger pieces or by removing them in an air stream. Calcination also removes last traces of moisture and any carbonaceous material present.

Among the first patents regarding diatomite processing was one issued to Calvert, et al.,<sup>28</sup> covering calcining diatomite in the presence of a fusible alkali salt. In the same year, a patent was issued to Ritche<sup>29</sup> covering two or more calcinations in the presence of an alkali. Later patents include those of Hoodless and Stockton. Hoodless<sup>30</sup> mixed fine carbon with diatomite and claimed the mixture increased

<sup>22</sup> Ruth, B. F., *Studies in Filtration, Derivation of General Filtration Equations*: Ind. and Eng. Chem., vol. 27, 1935, pp. 708–728.

Ribereau, Gayon, *Filtration of Wine*: Bull. Assoc. Chem., vol. 54, 1937, p. 730. Chem. Abs., vol. 31, 1937, p. 8817.

<sup>23</sup> Carman, P. C., *The Action of Filter-aids*: Ind. and Eng. Chem., vol. 30, 1938, pp. 1163–67.

<sup>24</sup> Calvert, Robert, Dern, Karl, and Alles, Gordon A., *Diatomite for Filtration*: U. S. Patent 1,502,547, 1924.

Thatcher, H. S., *Recovery of Filtration Material*: U. S. Patent 1,571,074, 1926.

<sup>25</sup> Kraner, H. H., *Effect of Heat Treatment on Diatomaceous Earth*: Jour. Amer. Ceram. Soc., vol. 11, 1928, pp. 875–83.

<sup>26</sup> Thatcher, H. S., *Filter Aid*: U. S. Patent 1,477,394, 1923; British Patent 234,735, 1923.

<sup>27</sup> Stockton, McKinley, *Apparatus for Separation (Air)*: U. S. Patent 2,018,039, 1935.

<sup>28</sup> Calvert, Robert, Dern, Karl, and Alles, Gordon A., *Comminuting and Blending Diatomaceous Earth*: U. S. Patents 1,936,300 and 1,936,301, 1935.

<sup>29</sup> Calvert, Dern, and Alles, Work cited in footnote 24.

<sup>30</sup> Ritche, A., *Treatment of Diatomite*: U. S. Patent 1,503,133, 1924.

<sup>30</sup> Hoodless, W. H., *Kieselguhr for Filtering*: U. S. Patent 1,589,531, June 23, 1926.

filtration and bleaching properties. Stockton<sup>31</sup> patented a process in which he claimed a sodium silicate application to either calcined or uncalcined diatomite filter-aids improved their quality.

#### FACTORS AFFECTING FILTRATION

Six factors affecting filtration and its control are:

1. Filter-aid percent (within limits).
2. Filtration temperature.
3. Pressure.
4. Liquid dilution (percent solids).
5. Liquid fluidity (inverse of viscosity).
6. Filtration time.

Calvert<sup>32</sup> states that many slow filtrations have been made sufficiently rapid to be practicable by adding more filter-aid, and that within limits the rate is proportional to the amount added. Temperature affects viscosity, and in sugar solutions the effect is pronounced. The viscosity of a 60-percent sucrose solution at 75° C. is one-third the viscosity at 45° C., and only one-twelfth the viscosity at 15° C. Although more filtrate is obtained from less concentrated solutions, sugar manufacturers found it cheaper to filter 60° Brix solutions than to evaporate the increased water from 20° Brix solution. A highly fluid solution also has a tendency to break the filter cake and to carry slimes through with the filtrate. Although an increase in filtration time increases the total filtrate volume, the filtration rate decreases as the filter-cake thickness increases with the longer filtration time. Other things being equal, an increase in pressure will increase filtration rate.

Some of the above factors will be discussed further under "Preparation and Test Methods," and "Special Tests."

### PREPARATION AND TEST METHODS

Before diatomite can be used as a filter-aid, it must be processed, and, although many patents have been granted covering processing, all are essentially the same with minor variations. Milling is probably the most important step. A typical method consists in air-drying the diatomite, passing it through a hammer mill and then a rotary calciner or drier, depending on the process. The material is then air-sized, the fines being used as a filter-aid and the coarse either returned to the mill or used as an admixture, or used as an insulation material, or rejected. This method is simple, but inefficient when compared with the complicated and extensive processes now used.

#### COMMERCIAL PREPARATION

##### PROSPECTING AND EXPLORATION

Diatomite in compact deposits can be sampled from outcrops and examined physically and chemically to determine the purer beds and to determine possible uses for each bed. Many deposits may be examined by core drilling; others by tunnels, trenches, etc. Auger drills are often used. Deposits may be drilled under water by driving a pipe and then drilling inside the pipe.

<sup>31</sup> Stockton, McKinley, Preparing Products from Diatomaceous Earth: U. S. Patent 1,970,204, Aug. 14, 1934.

<sup>32</sup> Calvert, Robert, p. 130 of work cited in footnote 28.

### MINING

The mining method selected depends on various factors, such as deposit's proximity to the surface, moisture, thickness, frequency and kind of impurities, and production rate required.

The usual United States practice in mining compact deposits is to use quarry methods on the surface in conjunction with shovels or draglines. Deeper deposits are mined by means of shafts, drifts, stopes, or rooms-and-pillars. Western States' deposits do not require much timbering.<sup>33</sup> Recent practice<sup>34</sup> at larger operations has been to use several methods or only underground methods. Dredging is used in several Eastern States and in eastern Canada.<sup>35</sup> Free water is usually drained off, and then the diatomite is spread for air drying.

### MILLING

Crude diatomite, either wet or field-dried, is generally conveyed to trommel screens or grizzlies. The coarse material from the screening goes to primary crushers and then to secondary mills in combination with air classifiers, either to remove fine impurities or to air-classify the diatomite according to particle sizes.<sup>36</sup>

Size classification is important, and three methods are generally used: (1) wet- or dry-screening, (2) settling in water, and (3) air-separation. Dry-screening below 200-mesh is impractical, and wet-screening is uneconomical because excess water has to be removed. Hydraulic settling requires tests for each diatomite.<sup>37</sup> Although air-separation is not entirely satisfactory, it is the method generally used. Three types of flow sheets, one for each of different sections of the United States, are common, and the many patents issued covering diatomite processing are only modifications. The patents cover such things as blending,<sup>38</sup> reduction of hammer mill speed to reduce diatom shattering during milling and final breaking by attrition during air transportation,<sup>39</sup> and removal of chert and larger diatoms by means of air separators in series.<sup>40</sup>

## LABORATORY PREPARATION

### GRINDING AND SIZING

Crude lump diatomite was broken with a hammer to smaller than 3-inch lumps and then dried on steam pipes for 48 hours. The dried material, containing 5 percent (average) moisture, was crushed in a 6-inch laboratory roll-jaw crusher set at  $\frac{3}{8}$  inch. After the crushed product was passed over a 100-mesh Sturtevant vibrating screen fitted with a brush to prevent agglomeration, the oversize (plus 100-mesh) was crushed further by 8- by 5-inch rolls set to touch lightly. Screening and crushing by the rolls were repeated several times until nothing was left on the screen except grit and cherty material.

<sup>33</sup> Cummins, A. B., and Mulryan, H., *Diatomite: Industrial Rocks and Minerals*, A. I. M. E., 1937, pp. 243-260.

<sup>34</sup> Hatmaker, Paul, Work cited in footnote 9.

<sup>35</sup> Cummins, A. D., and Mulryan, H., Work cited in footnote 33.

<sup>36</sup> Cummins, A. D., and Mulryan, H., Work cited in footnote 33.

<sup>37</sup> Calvert, Robert, pp. 84-85 of work cited in footnote 28.

<sup>38</sup> Stockton, McKinley, *Comminating and Blending Diatomaceous Earth*: U. S. Patents 1,936,300 and 1,936,301, 1935.

<sup>39</sup> Stockton, McKinley, *Milling Diatomaceous Earth*: U. S. Patent 1,991,583, 1935.

<sup>40</sup> Stockton, McKinley, *Preparing Products from Diatomaceous Earth*: U. S. Patent 1,970,204, August 14, 1934.

Filtration tests (described later) indicated that diatomite sized to minus 250- plus 325-mesh gave the highest flow rates and the highest clarities; therefore, minus 100-mesh diatomite was dried at 120° C. (248° F.) for 24 hours and then screened by a Ro-Tap machine for 5 minutes, and the minus 250- plus 325-mesh fraction obtained. Although all of the material would pass through a 325-mesh screen when screened wet, the method used was satisfactory for rough sizing. Usually, if more than one sample was obtained from the same deposit, minus 100-mesh material was used for the filtration tests, and the sample having the best flow-rate was sized as outlined above and then tested further.

*Calcined diatomite.*—Calcined diatomite dried for 24 hours at 120° C. (248° F.) was ground by lightly hand-crushing the material in a 6-inch porcelain mortar and then sizing it by screening with a Ro-Tap machine for 5 minutes to obtain the minus 250- plus 325-mesh fraction. That retained on the 250-mesh screen was recrushed and then screened, and the process was repeated until virtually all the material passed through the 250-mesh screen.

#### CALCINATION

*Without chemical additions.*—Minus 100-mesh diatomite prepared as previously described was calcined in covered fire-clay crucibles at 954°–960° C. (1750°–1760° F.) for 2 hours in a potentiometer-controlled electric muffle furnace. The calcined material, lightly crushed in a mortar, was screen-sized, and the minus 250- plus 325-mesh fraction was used as a filter-aid.

*With chemical additions.*—Minus 150-mesh chemical flux was mixed with 100 grams of minus 100-mesh diatomite by rolling them together on glazed paper. The mixture, placed in fire-clay crucibles and then covered, was calcined to 954°–960° C. for 2 hours. The calcined material, lightly crushed in a mortar, was screen-sized, and the minus 250- plus 325-mesh fraction was used as a filter-aid.

#### TEST METHODS

Each diatomite outcrop or deposit was inspected briefly, and samples ranging in size from 10 to 100 pounds were sent to the laboratory, where the following tests, in whole or in part, were made:

1. Filtration tests of sized materials to determine flow rates and clarities.
2. Calcination tests.
  - A. Effect of calcination on various physical properties.
3. Chemical analysis.
4. Pyrometric-cone-equivalent or cone-fusion test.

#### FILTRATION

Because flow rate and clarity are two important filter-aid properties, tests were made to determine them for samples prepared as previously described. For comparison purposes, all the samples received the same treatment, although different size fractions, calcination treatment, etc., probably would give results different from those obtained in this investigation.

## VACUUM FILTER APPARATUS

The first filtration apparatus tried, similar to Callingham's,<sup>41</sup> employed a double-jacketed Buchner funnel and a vacuum system (fig. 2). A damp filter cloth is placed in the funnel *A*, and receivers *D* and *E* are evacuated. The funnel *A* is brought to the same temperature as the test solution by passing either steam or water through the jacket. A sugar solution containing, say, 1 percent filter-aid, is then poured

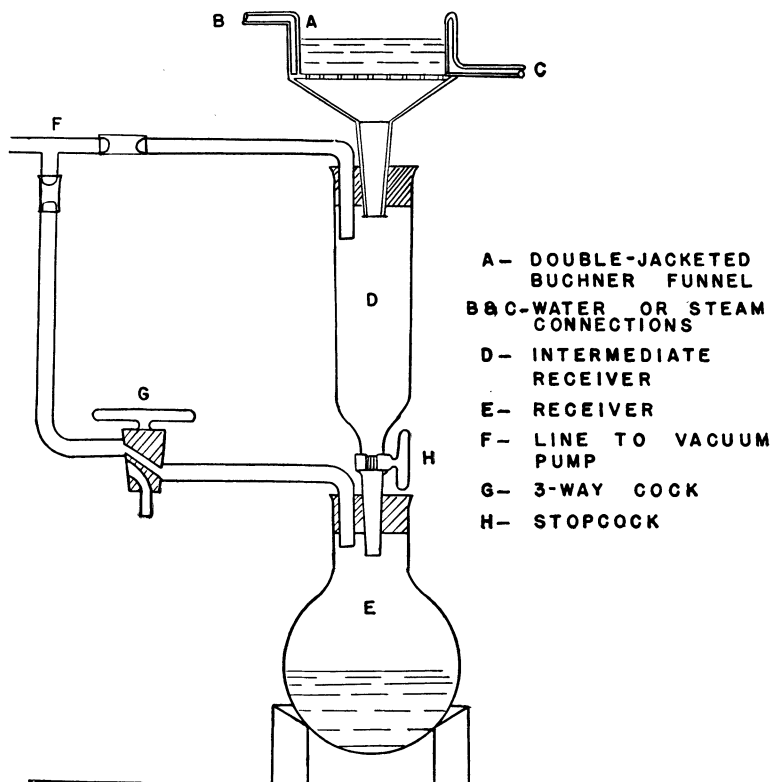


FIGURE 2.—Sketch of vacuum filter apparatus; not to scale. (After Callingham.)

into the funnel. After 2 minutes, the stopcock *H* is closed, and the 3-way cock *G* is turned, so that the vacuum is broken in the receiver *E*. The filtrate volume is then determined. If necessary, more test solution is poured into the funnel during the test.

Because duplicate results could not be obtained, as shown in tables 2 and 3, this vacuum apparatus was abandoned in favor of a pressure type.

<sup>41</sup> Callingham, W. E., Estimation of the Comparative Value of Kieselguhr as a Filter Aid in Sugar-House Practice: *Inter. Sugar Jour.*, vol. 34, 1932, pp. 274-75. *Chem. Abs.*, vol. 27, 1933, p. 622.



TABLE 2.—*Sugar-solution flow rates with vacuum filter apparatus*

Time required to filter 250 cc. 50° Brix raw-sugar solution at 60° C. with 2.5 percent diatomite filter-aid

Filter-aid	Time, seconds	Filter-aid	Time, seconds
Commercial 1.....	165	Commercial 5.....	120
Do.....	1,190	Do.....	253
Do.....	160	Do.....	852
Do.....	190	Do.....	1,280
Do.....	135	Do.....	115
Commercial 3.....	105	Do.....	21
Do.....	27		

TABLE 3.—*Glycerine flow rates with vacuum filter apparatus*

Time required to filter 250 cc. glycerine at 60° C. with 1 percent diatomite filter-aid

Filter-aid	Time, seconds	Filter-aid	Time, seconds
Commercial 1.....	196	Commercial 9.....	315
Do.....	260	Do.....	339
Commercial 2.....	226	Commercial 15.....	468
Do.....	136	Do.....	345
Commercial 3.....	69	Commercial 16.....	400
Do.....	85	Do.....	640

PRESSURE FILTER APPARATUS

A filter bomb (figs. 3 and 4) similar to one borrowed from the Dicalite Co., Los Angeles, Calif., consisting principally of a 4¼-inch-

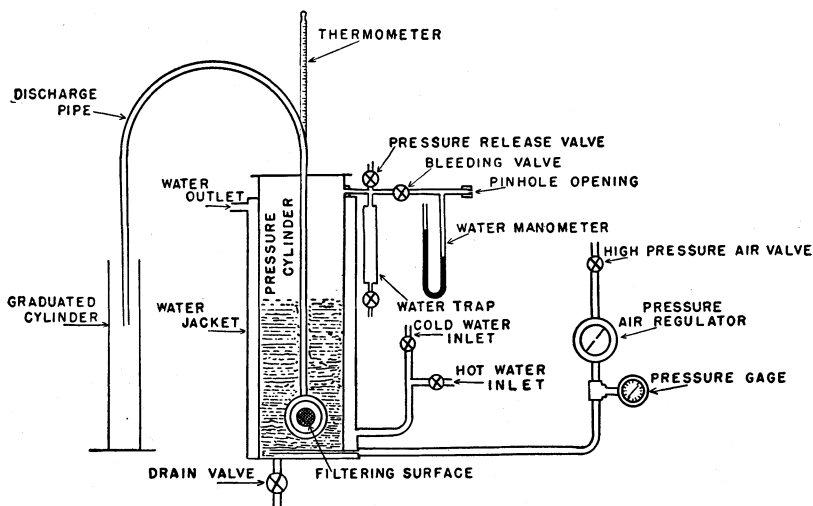


FIGURE 3.—Sketch of filter bomb.

diameter stainless-steel shell in which a suspended leaf acts as a filtering surface, was used in this investigation. The filter area could be changed by using any one of three filter-leaf caps having 0.75-, 1.0-, and 1.5-inch-diameter openings (0.442, 0.785, and 1.767 square inches). A delivery tube carried the filtrate into a graduated cylinder (outside the bomb).

Air entering at the bottom of the bomb through a coiled brass pipe with  $1/32$ -inch holes left at the top through a bleed-off line. The air, bled-off through a pinhole in the end of the bleed-off line, kept the diatomite in suspension during the filtration test. A manometer indicated

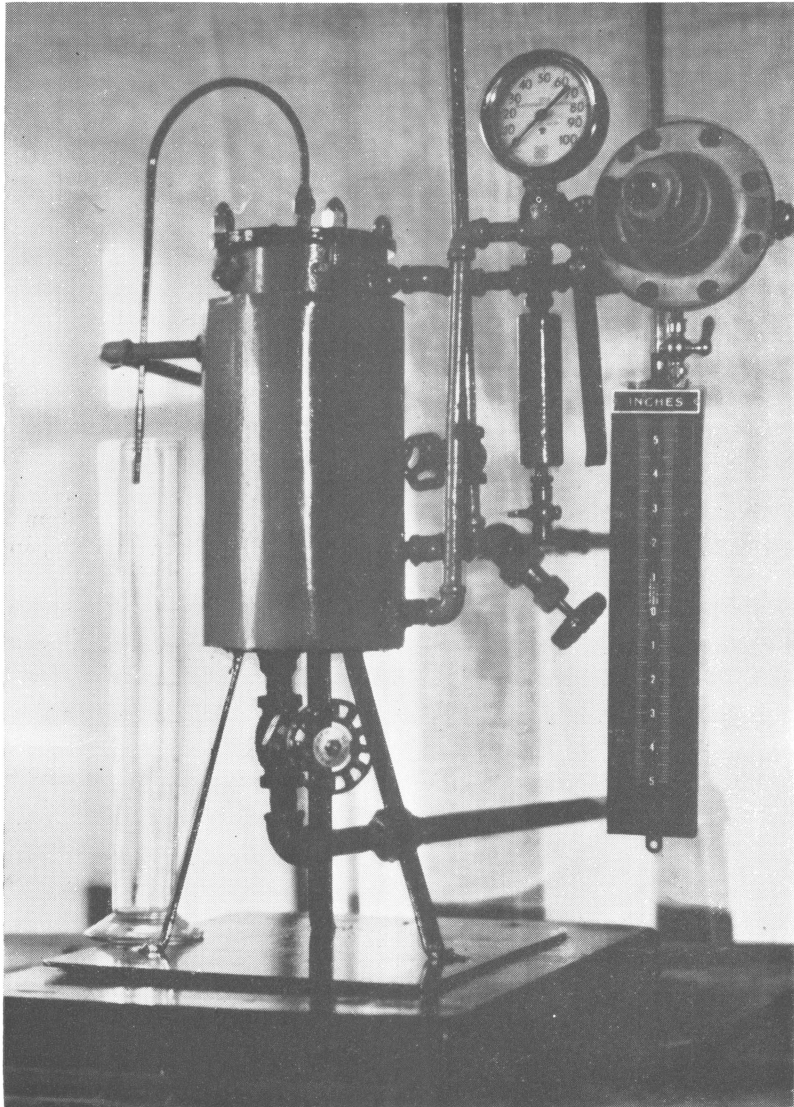


FIGURE 4.—Photograph of bomb-type filtering apparatus.

the air bleed-off, and a water trap placed before the manometer caught any sugar carried by the air. A drain valve permitted cleaning the bomb after each test. Bomb temperature was controlled by a water jacket connected to both hot- and cold-water lines.

FILTER BOMB OPERATION

The following standard conditions were employed for the filtration tests:

Filter area.....	0.785 sq. in. (1 inch diameter).
Bomb pressure.....	40 pounds per square inch.
Filter-aid.....	1.0 percent by weight.
Raw-sugar solution.....	50.5° Brix.
Temperature.....	20° C. ± 1°.
Bleeding pressure.....	1.0 inch water on manometer.
Filter cloth <sup>1</sup> .....	New cloth with ribbed side out. Cloth soaked in both hot and cold water before use.

<sup>1</sup>m-451 40-inch drill 70 x 84 weave filter cloth obtained from the California Cotton Mills, Oakland, Calif.

One percent, or 10 grams, of diatomite dried for 24 hours at 110° C. was added to 1,000 grams of standard sugar solution in the filter bomb, and the mixture was stirred for 2 minutes by air at 15 pounds pressure. The top of the bomb was tightened after 2 minutes and 50 seconds, and the pressure was increased to 40 pounds. At 3 minutes, the exhaust valve above the water trap (see fig. 3) was closed, and the bleeding pressure was adjusted to 1 inch of water on the manometer. Filtration time started as soon as the filtrate began to flow from the discharge tube. Filtrate volumes were noted at 2.5, 5.0, 10.0, and 20.0 minutes. After 20 minutes, the exhaust valve was opened, and then the bomb was cleaned for the next test.

*Standard sugar solution.*—Because raw sugar is hygroscopic and because mixing 500 grams sugar and 500 cc. water in the filter bomb gave erroneous results, a standard sugar solution was made by using 16,000 grams raw sugar, 15,850 cc. water at 20° C., and 150 cc. 40-percent formaldehyde. After this had stood 48 hours, the specific gravity was corrected to 50.5° Brix, as determined with a hydrometer. Formaldehyde prevented fermentation, which affected both flow rate and clarity. Figure 5 shows the effect of raw sugar solution concentration upon flow rate.

TESTS USED IN DETERMINING STANDARD FILTRATION CONDITIONS

Before the standard test conditions were established, tests were made to determine the effects of changing filtration conditions. The results of some of these tests are given in the following paragraphs.

*Percent filter-aid.*—Flow rates and clarities were determined for two raw-sugar solutions containing 0.25 to 3.00 percent (by weight) commercial filter-aid. (See table 4.) Even though more filtrate, with the same clarity, was obtained with the 0.5 percent addition than the 1.00 percent addition, the 1 percent addition was used in subsequent tests because it handled more conveniently.

TABLE 4.—Filter-aid percentage versus filtrate volume and clarity

Filter-aid, percent	Filtrate		Filter-aid, percent	Filtrate	
	Volume <sup>1</sup>	Clarity		Volume <sup>1</sup>	Clarity
0.25.....	92	Fair.	1.00.....	83	Good.
0.50.....	95	Good.	2.00.....	73	Excellent.
0.75.....	88	Do.	3.00.....	51	Do.

<sup>1</sup> Cc. per 20 minutes.

In the latter stages of the investigation, another series of tests was made to determine the effect of filter-aid quantity upon flow rate and clarity. Commercial 2 diatomite was added in quantities of 0.10, 0.25, 0.50, and 1.00 percent, and the flow rates and clarities were determined. The total volume of filtrate in 20 minutes decreased with an increase in filter-aid according to the following equation :

$$\text{Volume of filtrate per 20 minutes} = 280 - 110x$$

where  $x$  = percent filter-aid.

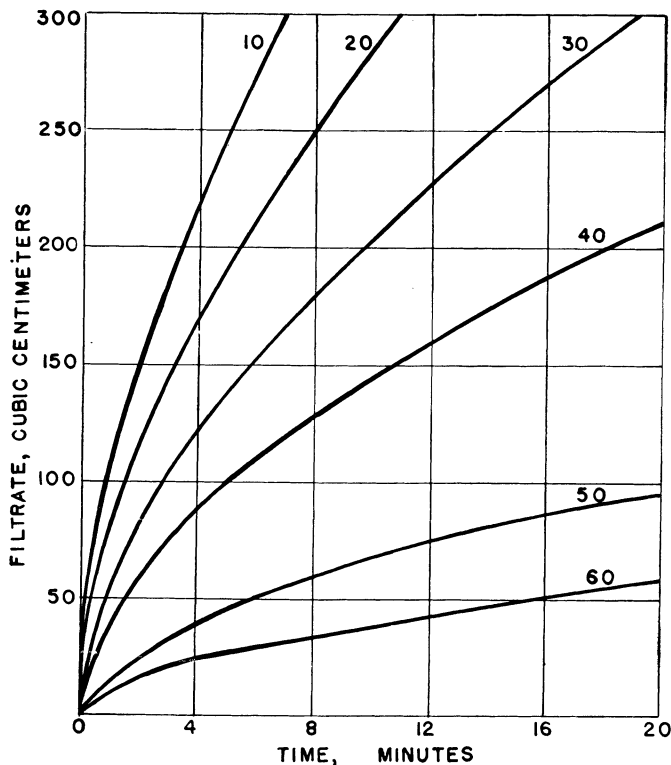


FIGURE 5.—Effect of raw-sugar solution concentration upon flow rate at 21° C. Numbers on curves represent percent raw sugar (degrees Brix).

As shown by galvanometer readings, the clarity did not increase when the quantity of filter-aid was increased from 0.5 to 1.0 percent, and less than 0.25 percent gave cloudy filtrates.

*Filtration temperature.*—The standard filtration temperature of 20° C.,  $\pm 1^\circ$ , was established after experiments were made showing the effect of temperature upon flow rate and upon viscosity. An increase in temperature increased the flow rate and decreased the viscosity, but the increased flow was not proportional to the decreased viscosity. This was probably due to a change in filter-cake formation. Figure 6 shows the effect of temperature upon flow rate (*A*) and upon viscosity (*B*) of a raw-sugar solution.

*Filtration pressure.*—An increase in filtration pressure increased the filtrate volume, but not in direct proportion to the increased pressure. (See table 5.)

*Filtration area.*—Special filtration tests made with a 50° Brix raw-sugar solution increased, but the flow rate per unit time decreased because the filter cake increased in thickness. (See table 6.)

*Filtration area.*—Special filtration tests made with a 50° Brix raw-sugar solution containing 1 percent filter-aid showed that although an increase in the filter area increased the filtrate volume, the increase was not directly proportional to the increased area. The data of table 7 indicate that to convert values obtained with the 0.75-inch (diameter)

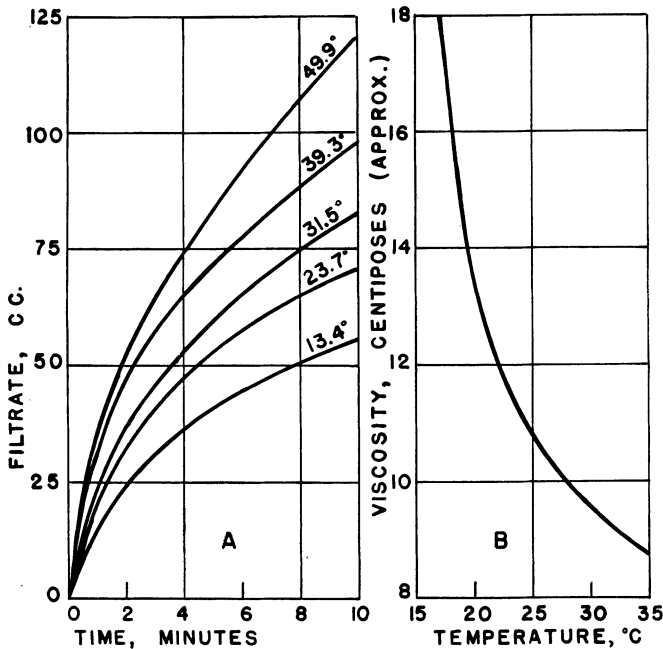


FIGURE 6.—Effect of temperature upon flow rate (A) and upon viscosity (B) of a raw-sugar solution.

opening to those obtained with the 1.00-inch opening, the former values had to be multiplied by 1.52. To convert from the 1.50-inch to the 1.00-inch opening values, a factor of 0.513 had to be used.

Other filtration tests made by using a different commercial filter-aid showed that using the empirical factors from the first tests gave calculated values for the 1.00-inch opening that deviated from the actual values more than the usual experimental error. (See table 8.) Therefore, it was concluded that the factors could only be used to estimate filtrate volume under standard conditions if different filtration areas were used, such as the 0.75-inch opening if the flow rate was too fast or the filtrate volume too large for the 1.00-inch opening.

TABLE 5.—*Effect of pressure upon filtrate volume*

Filtration pressure, lbs. in.	Filtrate volume, cc. per 10 min.	Ratio		Filtration pressure, lbs. in.	Filtrate volume, cc. per 10 min.	Ratio	
		Pressure	Volume			Pressure	Volume
10.....	53	1.00	1.00	40 <sup>1</sup> .....	131	4.00	2.47
20.....	90	2.00	1.70	50.....	141	5.00	2.68
30.....	112	3.00	2.09	55.....	151	5.50	2.85

<sup>1</sup> Standard filtration pressure.

TABLE 6.—*Effect of time upon filtrate volume and flow rate*

Time, minutes	Filtrate		Time, minutes	Filtrate	
	Volume, cc.	Flow rate, cc. per min.		Volume, cc.	Flow rate, cc. per min.
2.5.....	60	24.0	15.0.....	152	10.1
5.0.....	86	17.2	20.0.....	178	8.9
10.0.....	123	12.3			

TABLE 7.—*Effect of filter area upon flow rate*

Time, minutes	Filtrate, cc.			Time, minutes	Filtrate, cc.		
	Opening diameter, inches				Opening diameter, inches		
	0.75 <sup>1</sup>	1.00 <sup>2</sup>	1.50 <sup>3</sup>		0.75 <sup>1</sup>	1.00 <sup>2</sup>	1.50 <sup>3</sup>
2.5.....	24	37	72	15.0.....	59	90	175
5.0.....	35	53	102	20.0.....	68	101	200
10.0.....	48	73	144				

<sup>1</sup> Area=0.442 sq. in.

<sup>2</sup> Area=0.785 sq. in.

<sup>3</sup> Area=1.767 sq. in.

TABLE 8.—*Comparison of calculated and actual flow rates*

Time, minutes	Filtrate, cc.				
	Actual		Calculated		
	0.75 in.	1.00 in.	1.50 in.	0.75 to 1.00 in.	1.50 to 1.00 in.
2.5.....	44	60	133	67	68
5.0.....	61	86	186	93	95
10.0.....	82	123	260	124	133
15.0.....	100	152	316	152	162
20.0.....	118	178	362	179	186

*Effect of screen sizing.*—To arrive at an optimum screen size for a filter-aid, three Washington diatomites were dried for 24 hours at 120° C. and then screen-sized on a Ro-Tap machine for 5 minutes. Filtration tests were made with the 65/100-, 100/150-, 200/250-, 250/325- and minus 325-mesh products. Similar tests were made with three

calcined samples. As shown in figure 7, the minus 250- plus 325-mesh material gave the best flow rate with the highest clarity, whereas the minus 200- plus 250-mesh material gave the next-best results. As previously stated, all the material would pass through a 325-mesh screen if screened wet, but the method employed gave rough sizing.

*Effect of clay additions.*—In an attempt to duplicate slime conditions, 1.0 percent clay was added (in addition to the filter-aid) to a raw-sugar solution. Duplicate tests were not obtained; therefore, no clay was used in subsequent tests. In one series, a commercial filter-aid gave 170 cc. filtrate per 20 minutes, whereas the same filter-aid with a 1.00 percent clay addition gave results varying from 95 to 180 cc.

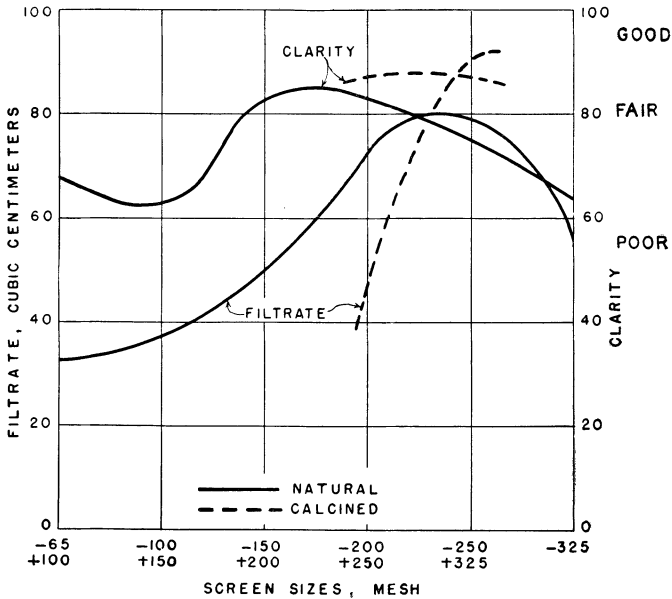


FIGURE 7.—Screen-sizing versus flow rate and clarity.

CLARITY

A simple tyndellometer, consisting of a box with two small holes in the top and containing an electric light, was tried in the first part of the investigation to test clarity. Filtrate samples were collected in test tubes, allowed to stand for 3 hours, and then placed over the holes in the box. Because colloidal matter disperses light (tyndel effect) according to the amount in suspension, a comparison of height and shape of the cone formed with that of a standard sample gave a comparison of the amount of colloidal matter present. Because it was difficult for different operators to obtain check results, this method was abandoned in favor of a colorimetric method.

*Colorimeter.*—A simple colorimeter consisting of two graduated glass tubes (one for the test solution and the other connected to a reservoir with a plunger for the standard solution) and a split-viewing tube was used to compare relative clarities. (See fig. 8.) Light from a 75-watt light in a bright metal-lined box and passing through an open-

ing covered with light-blue tracing cloth was used as a fixed light source. The unknown filtrate, diluted 1:10, was placed in the colorimetric tube to the 50 mark, and then the depth of the standard solu-

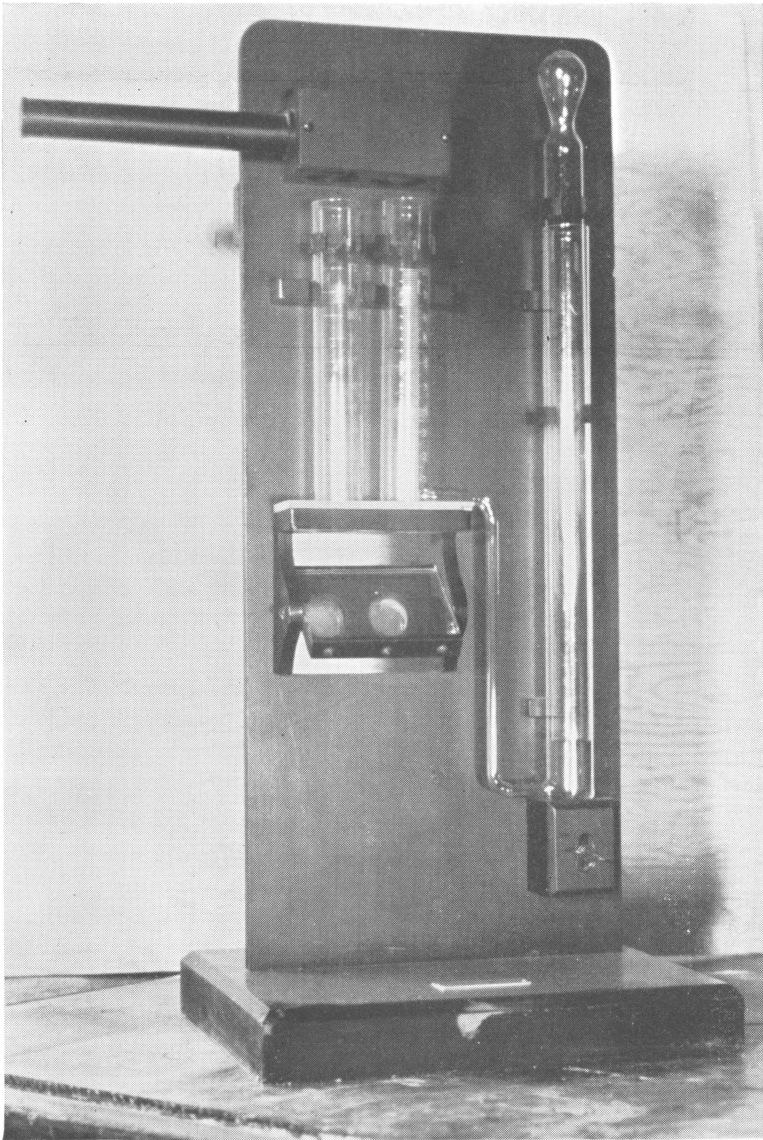


FIGURE 8.—Clarity colorimeter. In operation, the mirror under the two tubes is tilted so that light from a fixed source back of the colorimeter reflects upward through the tubes. Height of the standard liquid is varied by raising and lowering plunger at right.

tion<sup>42</sup> was varied until both halves of the split-view were the same color. The standard solution was light yellow-brown. Depth of the standard solution at point of matching color was called the standard

<sup>42</sup> Raw-sugar filtrate obtained from a commercial filter-aid and diluted 1:6.66. Two percent of 40-percent formaldehyde solution was added to prevent fermentation.



reading, but clarity varied inversely as the standard reading. A filtrate obtained with a commercial filter-aid was taken as 100 clarity, and all readings were referred to this relative standard.

If the filtrate contained a noticeable amount of suspended matter, it was found necessary to place a small piece of tracing cloth under the standard-color tube to gray the solution's color. As the solution depth varied, the effect of the cloth varied; therefore, light-meter readings were taken to show the effect with and without the cloth at various depths. The ratios obtained between these readings at various depths were used to convert tube-depth readings with the cloth to those without the cloth. Experience showed that when the clarity was below 80, filter-aid fines had passed through with the filtrate.

*Clarity classification.*—Using relative clarity, as determined by the colorimeter, as a basis, the following clarity classification was established:

<i>Relative</i>	<i>Classification</i>
Plus 105-----	Excellent.
95-105-----	Good.
80-95-----	Fair.
-80-----	Poor.

#### PYROMETRIC CONE EQUIVALENT

A relatively quick method for determining diatomite purity is to find its pyrometric cone equivalent (P. C. E.), which is similar to fusion, and compare this with that of pure quartz, cone 32½ (about 1,725° C.). Diatomite was ground to pass 65-mesh, molded into cones of proper shape by using a gum-arabic solution to give dry strength, and the P. C. E. determined by comparison with standard cones in an oxy-acetylene furnace.<sup>43</sup>

#### EFFECT OF CALCINATION UPON COLOR

Because calcination changes diatomite color, tests were made to determine color changes when diatomite was calcined with and without chemical additions. Minus 100-mesh diatomite was mixed with enough XXX-refined linseed oil to make a thick paste, which was then spread upon glass slides (plaques) and dried for 4 days. The plaque colors not only were compared with standard colors<sup>44</sup> but were also analyzed with a Hess-Ives tintometer. Glass color standards, whose principal wave lengths (in millimicrons) were red, 638; amber, 585; green, 535; blue-green, 490; and blue-violet, 450, were used in the tintometer.

Luminosities were calculated by the following method, described by Gardner<sup>45</sup>:

From the results obtained on the instrument the luminosity value and also the admixture of black may be calculated. A luminosity factor has been determined for each of the three colors. These are, Red, 0.19; Green, 0.71; and Blue-violet, 0.10. To calculate the luminosity, the red reading of the sample is multiplied by the red factor, the green by the green factor, and the blue-violet

<sup>43</sup> Wilson, Hewitt, An Oxygen-Acetylene High-Temperature Furnace: Jour. Amer. Ceram. Soc., vol. 4, 1921, pp. 835-41.

Staff, The Properties and Uses of Pyrometric Cones: The Edward Orton Jr. Ceramic Foundation, Columbus, Ohio, 44 pp.

<sup>44</sup> Maerz, A., and Paul, E. R., A Dictionary of Color: McGraw-Hill Book Co., New York, 1930, 207 pp.

<sup>45</sup> Gardner, Henry A., Physical and Chemical Examination of Paints, Varnishes, Lacquers, and Colors: Institute of Paint and Varnish Research, Washington, D. C., 1930, p. 199.

by the blue-violet factor. These three derived values are then added and the results are the total luminosity. Perfect luminosity is 100 percent, consequently the admixture of black is the difference between the total luminosity and 100 percent.

### DESCRIPTION OF DEPOSITS

Although there are a large number of diatomite deposits in the Pacific Northwest, usually only those that had been worked or described in geological reports were sampled for this investigation. In the case of a rather large area with numerous diatomite exposures, deposits that had been worked were sampled, and if the deposits were more than 10 feet thick, horizon samples were taken. Most of the deposits sampled in Oregon and in northern California had not been worked at the time of sampling (1940). No tonnage estimates were made, because no drilling or channeling was done to determine either the deposit's depth and extent or the amount of overburden.

Geologically, diatomite deposits have been or are now being formed by one of the following methods:

1. Ancient marine beds, more or less massive, with or without clay or other impurities.
2. Deposits adjacent to lakes and rivers of more recent age and probably formed by fresh-water diatoms, although both marine and fresh-water species may occur together.
3. Deposits now in the process of formation, such as swamps, lake bottoms, and bogs. These beds usually contain more carbonaceous matter than the other two types.

Lake or swamp deposits may be covered with a peaty overburden, whereas the older deposits may be covered with sedimentary beds or with lava flows. No attempt was made in this investigation to classify the beds by type, that is, fresh-water or marine, or to identify the diatom species.

Clay contents, which are given for some diatomites, were calculated from chemical analyses on the assumption that all the  $\text{Al}_2\text{O}_3$  was in the clay. One percent  $\text{Al}_2\text{O}_3$  is equivalent to 2.535 percent clay. These calculations are not exact, because some of the  $\text{Al}_2\text{O}_3$  may be in complex aluminum-silicates, such as volcanic glass, or the clay present may not have the theoretical kaolinite formula ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ).

### WASHINGTON

Diatomite deposits investigated in western Washington are now forming in bogs but, with the exception of the Parkland deposits (Washington 16), contain no more carbonaceous matter than the massive eastern Washington deposits. On an average, the western Washington deposits contain more silt and clay than the eastern Washington deposits but do not contain cherty, or opalized, material found in most of the eastern Washington deposits, particularly those in Grant County. Figure 9 shows the location of Washington diatomite deposits sampled for this investigation.

### ADAMS COUNTY

#### WASHINGTON 1

From the old workings of a deposit in the NE  $\frac{1}{4}$  sec. 25, T. 16 N., R. 30 E. To reach the deposit by road, turn north 10 miles east of Othello, on the Othello-Cunningham road, for 1.8 miles to G. W. Klep-

hardt farmhouse (owner in 1940) and then east 1.1 miles. The deposit, on the west side of an arroyo and beneath a minimum of 5 feet of weathered basalt and soil overburden, appears to be 10 to 15 feet thick and 300 feet long. These estimates are based upon exposures from old

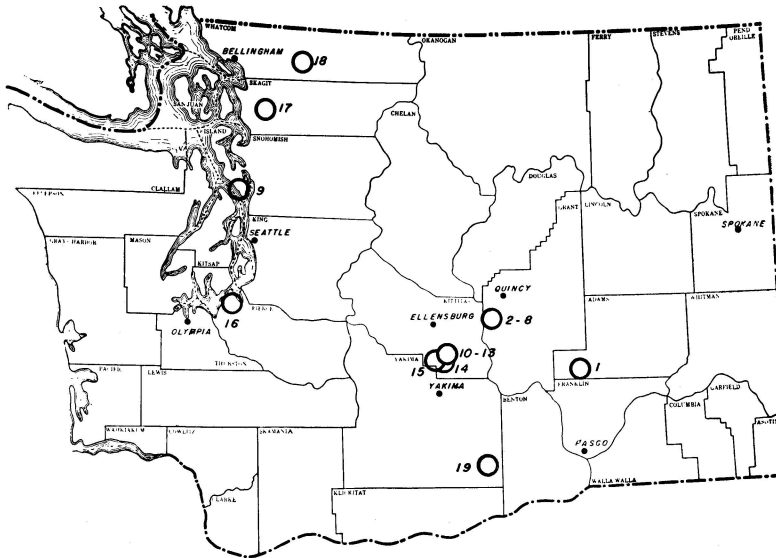


FIGURE 9.—Map showing location of sampled Washington diatomite deposits.



FIGURE 10.—Entrance to abandoned workings at Washington 1 deposit.

mining operations. About 20 years ago, the material was mined by room-and-pillar methods. Figure 10 shows an entrance to some of the old workings. A channel sample, after rejecting large chert nodules, gave an analysis of 89.5 percent  $\text{SiO}_2$ , 1.9 percent  $\text{Al}_2\text{O}_3$ , 1.3

percent  $\text{Fe}_2\text{O}_3$ , and 4.9 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 4.8 percent. The P. C. E. was cone 29.

Filtration tests using minus 100-mesh material gave a filtrate of 22 cc. per 20 minutes with poor clarity.

#### GRANT COUNTY

There are a number of diatomite exposures along the west-central border of Grant County (T. 18 N., R. 23 E.), 13 to 16 miles south and west of Quincy, some of which have been mined intermittently during the past 20 years. Most of the exposures, 10 to 15 feet thick, overlain with basalt, are in the S $\frac{1}{2}$  secs. 15, 16, and 17, N $\frac{1}{2}$  secs. 20 and 21, all sec. 22, and N $\frac{1}{2}$  sec. 27. Other exploited deposits occur in the center of sec. 9 and the SE $\frac{1}{4}$  sec. 29, N $\frac{1}{2}$  and SE $\frac{1}{4}$  sec 32, and W $\frac{1}{2}$  sec. 33. All these deposits may be reached by county roads branching from the Ellensburg-Ritzville State highway. Several deposits are visible from the highway. Samples were taken from 7 exposures in this area. Several roadcut exposures were found in sec. 29, T. 17 N., R. 24 E. Because these materials contained a relatively large amount of volcanic ash, no laboratory tests were made.

Filtration tests with minus 100-mesh material indicated that, with the exception of Washington 6, all the samples gave about 45 cc. filtrate in 20 minutes with fair to good clarity. Washington 6 gave an average of 65 cc. The bottom one-third of Washington 6, 7, and 8 gave the same results with about 80 cc. with good clarity. Calcination, especially with chemical additions, improved all the flow rates, to some as high as 250 cc., but the clarities were decreased.

#### WASHINGTON 2

From an exposure in the S $\frac{1}{2}$  sec. 17, T. 18 N., R. 23 E. The deposit, on the south side of a gully and beneath 2 to 5 feet of soil overburden, appears to be 10 feet thick and 150 feet long. These estimates are based upon small-scale mining operations in 1938. A channel sample gave an analysis of 89.1 percent  $\text{SiO}_2$ , 2.0 percent  $\text{Al}_2\text{O}_3$ , 2.1 percent  $\text{Fe}_2\text{O}_3$ , and 4.5 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 5.1 percent. The P. C. E. was cone 29.

Filtration tests with minus 100-mesh material gave a filtrate of 49 cc. in 20 minutes. Minus 250- plus 325-mesh material gave 39 cc. with fair clarity. Calcining minus 100-mesh diatomite to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 48 cc. and the clarity to excellent. As shown in table 9, material calcined to 1,600° F. and with 5 percent NaCl gave the best flow rate—260 cc. per 20 minutes.

TABLE 9.—Calcination and sizing effects upon Washington 2 filter-aid properties

	Filtrate	
	cc./20 min.	Clarity
Uncalcined:		
Minus 100-mesh.....	49	
-250+325-mesh.....	39	Fair.
Calcined:		
Minus 100-mesh calcined to 1,750° F. and sized to -250+325-mesh.....	48	Excellent.

## WASHINGTON 3

From the central part of sec. 9, T. 18 N., R. 23 E. At the time the deposit was visited (1940), some mining was being done by room-and-pillar method, but it was impossible to determine the size of the operations because the adit was boarded up. The exposed diatomite was 10 feet thick and covered with 2 to 10 feet of overburden. A channel sample had a P. C. E. of cone 26-27.

Filtration tests with minus 100-mesh material gave a filtrate of 33 cc. in 20 minutes with poor clarity. Minus 250- plus 325-mesh material gave 52 cc. with fair clarity.

## WASHINGTON 4

Three horizon samples from the south-central part of sec. 9, T. 18 N., R. 23 E. The diatomite bed, 15 feet thick and apparently dipping

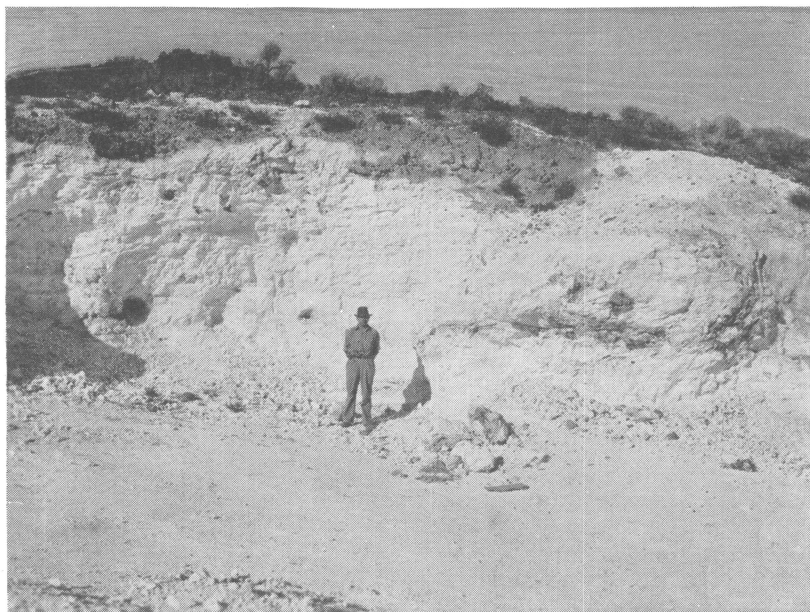


FIGURE 11.—Washington 4 deposit, Grant County. Note dark, cherty material at right center.

6° N. 50° E., as exposed by mining operations, forms an elliptical knoll about 200 by 400 feet and is covered by 1 to 8 feet of soil and basalt. In some places basalt forms the entire capping.

Channel samples were taken from 0 to 3 feet (Washington 4a), 3 to 6 feet (Washington 4b), and 9 to 12 feet (Washington 4c), as measured from the top of a face exposed by mining operations. (See fig. 11.) None of the chert shown in figure 11 was included in the samples. Washington 4a gave a chemical analysis of 87.6 percent  $\text{SiO}_2$ , 1.2 percent  $\text{Al}_2\text{O}_3$ , 1.4 percent  $\text{Fe}_2\text{O}_3$ , and 5.3 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 3.0 percent. The P. C. E. value for Washington 4a was cone 30, and for Washington 4b cone 31.

Filtration tests with minus 100-mesh material gave an average filtrate for all three samples of 44 cc. in 20 minutes with good to excellent clarity. Sizing the material to minus 250- plus 325-mesh increased the

average flow rate to 58 cc. and decreased the clarity to fair. As shown in table 10, minus 100-mesh material calcined to 1,750° F. with 4 percent NaCl and the calcined product sized to minus 250- plus 325-mesh gave the best flow rate—175 cc. in 20 minutes.

TABLE 10.—*Calcination and sizing effects upon Washington 4 filter-aid properties*

UNCALCINED				
Sample	Interval, feet	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	0-3	-100	43	Excellent.
b.....	3-6	-100	46	Good.
c.....	9-12	-100	44	Good.
a.....	0-3	-250+325	59	-----
b.....	3-6	-250+325	59	-----
c.....	9-12	-250+325	56	Fair.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	9-12	-250+325	57	Fair.
c+4% NaCl.....	9-12	-250+325	175	Fair.

#### WASHINGTON 5

From the center of sec. 26, T. 18 N., R. 23 E. The deposit, about 10 feet thick, appeared to be similar to Washington 4.

Filtration tests with minus 100-mesh material gave a filtrate of 45 cc. in 20 minutes with fair clarity. Sizing to minus 250- plus 325-mesh only slightly improved the flow rate (48 cc.), but the clarity was improved to good. Calcining minus 100-mesh material and then sizing to minus 250- plus 325-mesh only increased the flow rate to 51 cc.; and calcining with 4 percent NaCl and sizing to the same screen fraction only increased the flow rate to 74 cc. The clarity for the calcined material without the chemical addition was poor and with the chemical addition was fair.

#### WASHINGTON 6

Three horizon samples from the SE $\frac{1}{4}$  sec. 29, T. 18 N., R. 23 E. The deposit, which is exposed for 400 feet, varies in thickness from 10 to 40 feet with from 1 to 8 feet of overburden and apparently dips 10° NE. Little chert was seen. Figure 12 shows the exposed north face.

Channel samples were taken to represent the upper one-third (Washington 6a), the middle one-third (Washington 6b), and the lower one-third (Washington 6c). Washington 6b gave a chemical analysis of 82.2 percent SiO<sub>2</sub>, 6.7 percent Al<sub>2</sub>O<sub>3</sub>, 5.2 percent Fe<sub>2</sub>O<sub>3</sub>, and 4.2 percent ignition loss; Washington 6c gave 84.8 percent SiO<sub>2</sub>, 4.1 percent Al<sub>2</sub>O<sub>3</sub>, 6.9 percent Fe<sub>2</sub>O<sub>3</sub>, and 4.1 percent ignition loss. The clay equivalents for the Al<sub>2</sub>O<sub>3</sub> are 17.0 percent for Washington 6b and 10.4 percent for Washington 6c. The P. C. E. for the latter sample was cone 27, as compared to cone 23 for the former.

Filtration tests with minus 100-mesh material achieved the best results with Washington 6c, 77 cc. in 20 minutes with good clarity, and the poorest with Washington 6a, 44 cc. with good clarity. Sizing Washington 6b and 6c diatomite to minus 250- plus 325-mesh increased

the flow rates to 83 cc. and 86 cc., respectively. As shown in table 11, minus 100-mesh Washington 6c calcined to 1,750° F. with 4 percent NaCl and the calcined product sized to minus 250- plus 325-mesh gave the best flow rate, 250 cc in 20 minutes, but the clarity was poor.



FIGURE 12.—Washington 6 deposit, Grant County.

TABLE 11.—Calcination and sizing effects upon Washington 6 filter-aid properties

Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Upper third.....	-100	44	Good.
b.....	Middle third.....	-100	64	Fair.
c.....	Lower third.....	-100	77	Good.
b.....	Middle third.....	-250+325	83	
c.....	Lower third.....	-250+325	86	Good.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	Lower third.....	-250+325	144	Poor.
c+4% NaCl.....	Lower third.....	-250+325	250	Poor.

#### WASHINGTON 7

From the SE $\frac{1}{4}$  sec. 32, T. 18 N., R. 23 E., about 500 feet north of the old Quincy-Vantage Ferry highway. Channel samples were taken

from the upper one-third (Washington 7a), the middle one-third (Washington 7b), and the lower one-third (Washington 7c). No chert nodules were included in the samples.

Filtration tests with minus 100-mesh material obtained the best flow rates with Washington 7c, 80 cc. in 20 minutes with good clarity. The other two samples averaged 38 cc. with good clarity. Sizing Washington 7c material to minus 250- plus 325-mesh lowered the flow rate to 73 cc. with fair clarity. As shown in table 12, minus 100-mesh Washington 7c diatomite calcined to 1,750° F. with 4 percent NaCl, and the calcined product sized to minus 250- plus 325-mesh gave the best flow rate, 240 cc. in 20 minutes with poor clarity.

TABLE 12.—*Calcination and sizing effects upon Washington 7 filter-aid properties*

UNCALCINED				
Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Upper third.....	-100	35	Good.
b.....	Middle third.....	-100	40	Good.
c.....	Lower third.....	-100	80	Good.
c.....	Lower third.....	-250+325	73	Fair.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	Lower third.....	-250+325	218	Fair.
c+4% NaCl.....	Lower third.....	-250+325	240	Fair.

#### WASHINGTON 8

From the SE $\frac{1}{4}$  sec. 32, T. 18 N., R. 23 E., about 500 feet east of Washington 7. Channel samples were taken from the upper one-third (Washington 8a), the middle one-third (Washington 8b), and the lower one-third (Washington 8c). No chert nodules were included in the samples.

Washington 8b gave a chemical analysis of 77.7 percent SiO<sub>2</sub>, 9.4 percent Al<sub>2</sub>O<sub>3</sub>, 6.2 percent Fe<sub>2</sub>O<sub>3</sub>, and 5.5 percent ignition loss, and Washington 8c gave 69.2 percent SiO<sub>2</sub>, 2.1 percent Al<sub>2</sub>O<sub>3</sub>, 1.8 percent Fe<sub>2</sub>O<sub>3</sub>, and 5.3 percent ignition loss. The clay equivalents for the Al<sub>2</sub>O<sub>3</sub> are 23.8 percent for Washington 8b and 5.3 percent for Washington 8c. The P. C. E. for the latter sample was cone 28, as compared to cone 15 for the former.

Filtration tests with minus 100-mesh material achieved the best results with Washington 8c, 84 cc. in 20 minutes with good clarity, and the poorest with Washington 8a, 31 cc. with fair clarity. Sizing the material from Washington 8b and 8c to minus 250- plus 325-mesh increased the flow rates to 82 cc. and 80 cc., respectively. As shown in table 13, minus 100-mesh Washington 8c diatomite calcined to 1,750° F. with 4 percent NaCl and then sized to minus 250- plus 325-mesh gave the best flow rate—251 cc. in 20 minutes, but with poor clarity.



TABLE 13.—*Calcination and sizing effects upon Washington 8 filter-aid properties*

UNCALCINED				
Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Upper third.....	-100	31	Fair.
b.....	Middle third.....	-100	48	Good.
c.....	Lower third.....	-100	84	Do.
b.....	Middle third.....	-250+325	82	
c.....	Lower third.....	-250+325	80	Good.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	Lower third.....	-250+325	193	Fair.
c+4% NaCl.....	Lower third.....	-250+325	251	Poor.

## ISLAND COUNTY

## WASHINGTON 9

From the SE $\frac{1}{4}$  sec. 29, T. 29 N., R. 3 E., along Useless Bay, about 2 $\frac{1}{2}$  miles north of Maxwellton.

Filtration tests with minus 250- plus 325-mesh material gave 78 cc. in 20 minutes with poor clarity. Minus 100-mesh material calcined to 1,750° F. and then sized to minus 250- plus 325-mesh increased the flow rate to 93 cc. and the clarity to excellent.

## KITITAS COUNTY

There are a number of diatomite deposit exposures in Kittitas County in the Squaw Creek (an intermittent stream) area, T. 15 N., R. 19-23 E., about 17 miles southeast of Ellensburg, but only two are mined at present. Several others have been mined intermittently during the last 20 years. According to Mackin,<sup>46</sup> diatomite exposures are found over an area about 10 miles square, and all have the same general stratigraphic relationships. Most of the exposures are 10 to 20 feet thick and are overlain with several feet of clay and sand, which in turn is overlain with 3 to 6 feet of weathered basalt. Channel samples were taken for this investigation from 6 exposures in this area—4 from operating and test pits of the largest diatomite producer in the State.

## WASHINGTON 10

From sec. 15, T. 15 N., R. 20 E., 17.2 miles south of Kittitas. A diatomite bed, apparently flat-lying, 10 to 20 feet thick and exposed by mining operations for 400 feet was channel-sampled at the west operating pit (mining company's designation). (Fig. 13.) Few chert nodules were seen. Chemical analysis gave 91.1 percent SiO<sub>2</sub>, 1.3 percent Al<sub>2</sub>O<sub>3</sub>, 2.0 percent Fe<sub>2</sub>O<sub>3</sub>, and 5.1 percent ignition loss. The clay equivalent for the Al<sub>2</sub>O<sub>3</sub> is 3.3 percent. The P. C. E. value was cone 30.

<sup>46</sup> Mackin, J. Hoover, assoc. prof. geol., Univ. of Wash., personal communications and conversations in 1942-43.

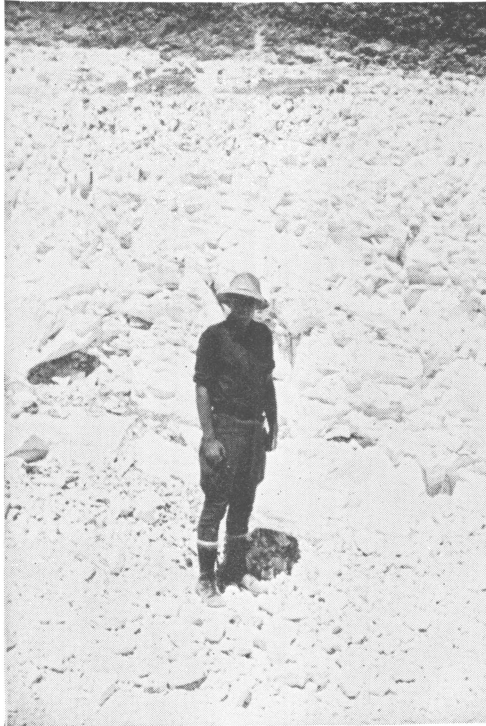


FIGURE 13.—Washington 10 deposit, Kittitas County. According to the company mining the deposit, about 2 feet of diatomite is left as the floor of the quarry.

Filtration tests with minus 100-mesh material gave a filtrate of 59 cc. in 20 minutes. (See table 14.)

TABLE 14.—*Calcination and sizing effects upon Washington 10, 11, and 12 filter-aid properties*

UNCALCINED			
Sample	Screen size, mesh	Filtrate	
		cc./20 min.	Clarity
Washington 10.....	-100	59	Poor. Do.
11.....	-100	63	
12.....	-100	52	
Washington 11.....	-250+325	125	
12.....	-250+325	115	
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN RESIZED			
Washington 11.....	-250+325	70	Excellent. Do.
12.....	-250+325	82	

#### WASHINGTON 11

From sec. 15, T. 15 N., R. 20 E. and the same deposit as Washington 10, but from the east operating pit (mining company's designation). The east pit appeared to contain more iron, but chemical analysis

showed the iron contents were almost identical. As seen in figure 14, the upper part of Washington 11 is iron-stained. Chemical analysis gave 89.6 percent  $\text{SiO}_2$ , 2.9 percent  $\text{Al}_2\text{O}_3$ , 2.1 percent  $\text{Fe}_2\text{O}_3$ , and 4.2 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 7.4 percent—4.1 percent more than for Washington 10. The P. C. E. value was cone 28.

Filtration tests with minus 100-mesh material gave a filtrate of 63 cc. in 20 minutes. Sizing the material to minus 250- plus 325-mesh increased the flow rate to 125 cc., but with poor clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 82 cc. and the clarity to excellent. (See table 14.)

#### WASHINGTON 12

From sec. 15, T. 15 N., R. 20 E., from a test pit about 500 feet west of Washington 10 in what appeared to be an extension of the deposit being mined. Chemical analysis gave 90.5 percent  $\text{SiO}_2$ , 2.0 percent  $\text{Al}_2\text{O}_3$ , 2.0 percent  $\text{Fe}_2\text{O}_3$ , and 3.6 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 5.1 percent. The P. C. E. value was cone 29–30.



FIGURE 14.—Washington 11 deposit, Kittitas County.

Filtration tests with minus 100-mesh material gave a filtrate of 52 cc. in 20 minutes. Sizing the material to minus 250- plus 325-mesh increased the flow rate to 115 cc., but with poor clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 70 cc. and the clarity to excellent. (See table 14.)

#### WASHINGTON 13

From sec. 10, T. 15 N., R. 20 E., about 1 mile north of Washington 10, 11, and 12. This deposit, designated by the mining company as "the cherty pit" because it contains chert nodules or concretions, has an exposed mining face about 10 feet thick and 150 feet long. At present, the company is not using diatomite from this deposit. A channel sample, not including nodules, gave an analysis of 88.3 percent  $\text{SiO}_2$ , 3.9 percent  $\text{Al}_2\text{O}_3$ , 2.9 percent  $\text{Fe}_2\text{O}_3$ , and 4.8 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 9.9 percent—higher than any other tested in this immediate area. The P. C. E. value was cone 29. No filtration tests were made.

#### WASHINGTON 14

From the  $W\frac{1}{2}$  sec. 14, T. 15 N., R. 19 E., which is 13.8 miles north of Yakima on U. S. Highway 97. The deposit is 1.2 miles east of a foot suspension bridge across the Yakima River at Roza station (Northern Pacific Railroad). The deposit was worked in 1926, and the old working face, 10 feet thick and 200 feet long along a hillside, is covered with 8 inches of gray clay and 5 feet of rocky soil. A few green, cherty nodules were noticed in the face. A channel sample, not including chert nodules, gave a chemical analysis of 89.0 percent  $\text{SiO}_2$ , 3.0 percent  $\text{Al}_2\text{O}_3$ , 3.9 percent  $\text{Fe}_2\text{O}_3$ , and 4.4 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 7.6 percent. The P. C. E. value was cone 28-minus.

Filtration tests with minus 250- plus 325-mesh material gave a filtrate of 39 cc. with fair clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 49 cc. and increased the clarity to good.

#### WASHINGTON 15

From about 1.5 miles west of Roza station in the  $NE\frac{1}{4}$  sec. 17, T. 15 N., R. 19 E. Considerable tonnage was mined in 1919, but the deposit has not been worked since then. An old working face about 200 feet long along a hillside was covered with slides until only a 4-foot thickness of diatomite is exposed. The diatomite is covered with 2 feet of clay, 3 feet of micaceous sand, and 2 feet of basalt. Chemical analysis of a channel sample taken of the diatomite gave 88.7 percent  $\text{SiO}_2$ , 2.3 percent  $\text{Al}_2\text{O}_3$ , 3.1 percent  $\text{Fe}_2\text{O}_3$ , and 5.4 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 5.8 percent. The P. C. E. value was cone 29 plus. No filtration tests were made.

#### PIERCE COUNTY

#### WASHINGTON 16

From a bog deposit in the central  $\frac{1}{2}$  east  $\frac{1}{2}$  sec. 8, T. 19 N., R. 3 E., 2 miles west of Parkland. The deposit covers an area of 30 acres to

a reported depth of 15 to 30 feet and during the winter months is covered with water to a depth of 5 feet in some places. Auger samples were black when wet and brown when dry. Chemical analysis gave 67.3 percent  $\text{SiO}_2$ , 13.8 percent  $\text{Al}_2\text{O}_3$ , 2.4 percent  $\text{Fe}_2\text{O}_3$ , and 10.9 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 35.0 percent. The P. C. E. value was cone 14-15, one of the lowest of any sample tested.

Filtration tests with minus 250- plus 325-mesh material gave a filtrate of 40 cc. in 20 minutes with poor clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 118 cc. and the clarity to fair.

#### SKAGIT COUNTY

##### WASHINGTON 17

From the SE $\frac{1}{4}$  sec. 25, T. 34 N., R. 4 E.,  $\frac{1}{2}$  mile south of the Big Lake post office and  $\frac{1}{4}$  mile east, where a 6-foot thick white-diatomite deposit is exposed by the Nookachcamp Creek. No attempt was made to determine the area of the deposit, which appeared to cover 15 to 20 acres. Samples were taken about every 25 feet for a distance of 200 feet along the creek bank where diatomite was exposed. Chemical analysis gave 73.5 percent  $\text{SiO}_2$ , 15.1 percent  $\text{Al}_2\text{O}_3$ , 3.9 percent  $\text{Fe}_2\text{O}_3$ , and 4.3 percent ignition loss. The clay equivalent of the  $\text{Al}_2\text{O}_3$  is 38.3 percent. The P. C. E. value was cone 11.

Filtration tests with minus 250- plus 325-mesh material gave a filtrate of 62 cc. in 20 minutes with poor clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh lowered the flow rate to 48 cc. but increased the clarity to fair.

#### WHATCOM COUNTY

##### WASHINGTON 18

From the SE $\frac{1}{4}$  sec. 30, T. 38 N., R. 8 E., from a deposit along the Concrete-Baker Lake Highway about 2 miles west of Baker Lake. There is a known area of 75 acres but no information regarding the depth. Auger samples gave a chemical analysis of 85.9 percent  $\text{SiO}_2$ , 5.6 percent  $\text{Al}_2\text{O}_3$ , 0.8 percent  $\text{Fe}_2\text{O}_3$ , and 14.2 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 14.2 percent. The P. C. E. value was cone 28.

Filtration tests with minus 250- plus 325-mesh material gave a filtrate of 112 cc. in 20 minutes, but with poor clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate slightly, to 122 cc., but increased the clarity to good.

#### YAKIMA COUNTY

##### WASHINGTON 19

From NE $\frac{1}{4}$  sec. 2, T. 10 N., R. 22 E. To reach the deposit, 2.4 miles west of Sunnyside on U. S. 410 turn north to the Maple Grove road for 1.9 miles to the last main irrigation canal, follow it 0.6 mile east to a dry wash, and then north for 0.7 mile. The diatomite outcrops at only one place in the wash; therefore, no estimate was made of

either the extent or thickness of the deposit. The deposit, as exposed by the wash, was 4 feet thick. Since the deposit was sampled, recent development work has indicated that the thickness may be more than 15 feet. Chemical analysis of a channel sample taken of the 4 feet exposed by the wash gave 59.5 percent  $\text{SiO}_2$ , 15.1 percent  $\text{Al}_2\text{O}_3$ , 4.6 percent  $\text{Fe}_2\text{O}_3$ , and 9.9 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 38.3 percent. The P. C. E. value was cone 4-minus, the lowest of all the samples tested for P. C. E.

Filtration tests with minus 250- plus 325-mesh material gave a filtrate of 43 cc. in 20 minutes with poor clarity. Calcining minus 100-mesh material to  $1,750^\circ\text{F}$ . and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 202 cc., but the clarity did not change.

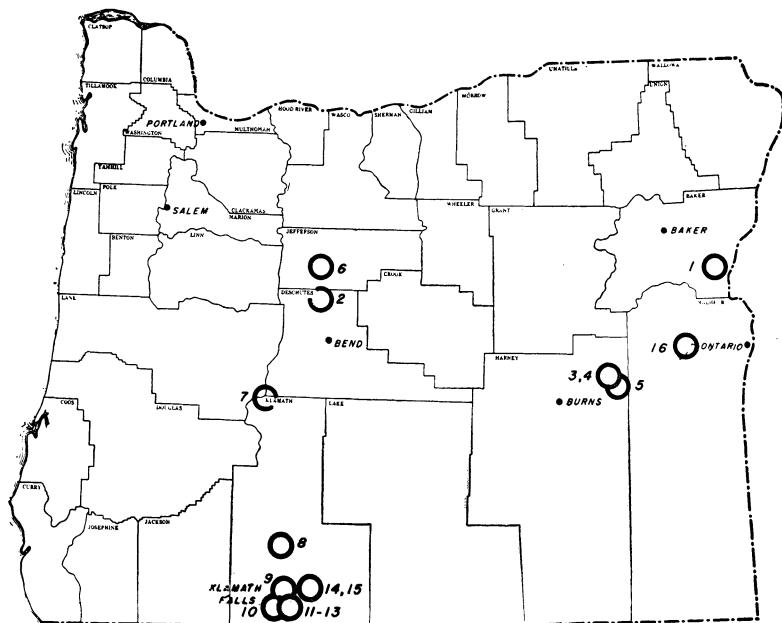


FIGURE 15.—Map showing location of sampled Oregon diatomite deposits.

### OREGON

Moore<sup>47</sup> describes the known diatomite deposits in eastern Oregon. Fifteen areas described by him were sampled for this investigation. A deposit in Jefferson County, not described, also was sampled.

Although most of the beds were thicker than those found in Washington, more were more highly contaminated with either other sediments or volcanic ash. This was especially true of the material in the Klamath Falls area. None of the sampled Oregon deposits contained chert nodules found in the east-central Washington deposits. Figure 15 shows the location of Oregon diatomite deposits sampled for this investigation.

<sup>47</sup> Moore, B. M., *Nonmetallic Mineral Resources of Eastern Oregon*: U. S. Geol. Survey Bull. 875, 1937, pp. 17-118, plates 1, 5, 8, and 11.

## BAKER COUNTY

## OREGON 1

From the center of sec. 28, T. 11 S., R. 43 E. To reach the deposit, turn north 2.5 miles south of Durkee on Baker-Ontario highway for 0.2 mile. The sample was taken from a small cut in the east side of a hill. The P. C. E. value was cone 15.

Filtration tests with minus 100-mesh material gave a filtrate of 38 cc. in 20 minutes with good clarity. Sizing the material to minus 250- plus 325-mesh increased the flow rate to 73 cc. but decreased the clarity to fair. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 253 cc., but the clarity was decreased further to poor.

## DESCHUTES COUNTY

## OREGON 2

From the S<sup>1</sup>/<sub>2</sub>, NE<sup>1</sup>/<sub>4</sub>, sec. 16, T. 14 S., R. 12 E. The deposit (about 17 miles northwest of Bend) at the quarry is about 30 feet thick and is covered with 6 to 8 feet of soil overburden. Only about the top 20 feet are being used for filter-aid manufacture, and several distinct beds in this upper section are mined as separate units. Channel samples were taken of the middle bed (Oregon 2a) and the lower bed (Oregon 2b) in the quarry face. The P. C. E. value for Oregon 2b was cone 27.

Filtration tests with minus 100-mesh Oregon 2a diatomite gave a filtrate of 42 cc. in 20 minutes with poor clarity. Similarly sized Oregon 2b gave a flow rate of 73 cc. with good clarity, and the flow rate was only increased to 76 cc. by sizing to minus 250- plus 325-mesh. Calcining minus 100-mesh Oregon 2b to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 112 cc., but the clarity was reduced to fair. Oregon 2b material with 4 percent NaCl calcined to 1,750° F. and then sized to minus 250- plus 325-mesh gave the best flow-rate—232 cc. in 20 minutes—but the clarity was poor. (See table 15.)

TABLE 15.—*Calcination and sizing effects upon Oregon 2 filter-aid properties*

UNCALCINED				
Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Middle bed <sup>1</sup> .....	—100	42	Poor.
b.....	Lower bed <sup>1</sup> .....	—100	73	Good.
b.....	Lower bed <sup>1</sup> .....	—250+325	76	Do.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
b.....	Lower bed <sup>1</sup> .....	—250+325	116	Fair.
b+4% NaCl.....	do.....	—250+325	232	Poor.

<sup>1</sup> Beds as exposed by quarry face.

## HARNEY COUNTY

## OREGON 3

From the NE $\frac{1}{4}$  sec. 17, T. 20 S., R. 36 E., and from one of a series of deposits that have been mined in the past and the diatomite used locally to insulate root cellars. The deposit, about 6 miles northeast of Drewsey, on the Drewsey-Beulah road, forms a knoll 150 feet in diameter and 35 feet high with little soil overburden. The total thickness may not be represented by the height of the knoll. At the time the material was sampled, it felt like volcanic ash, but microscopic examination revealed little ash but rather large diatoms.

Filtration tests with minus 100-mesh material gave a filtrate of 51 cc. in 20 minutes with fair clarity. Sizing the diatomite to minus 250-plus 325-mesh increased the flow rate to 67 cc., but the clarity was poor. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 241 cc. and the clarity to fair. Calcining minus 100-mesh diatomite with 4 percent NaCl and then sizing the calcined product to minus 250-plus 325-mesh increased the flow rate to 347 cc. but decreased the clarity to poor.

## OREGON 4

From another knoll 300 feet east of Oregon 3, but at a higher elevation. This material was not as coarse as the previous sample.

Filtration tests with minus 100-mesh material gave a filtrate of 71 cc. in 20 minutes with fair clarity. Sizing the diatomite to minus 250-plus 325-mesh increased the flow rate to 101 cc., but the clarity was poor. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 150 cc. and the clarity to fair. Calcining minus 100-mesh diatomite with 4 percent NaCl and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 385 cc. but decreased the clarity to poor.

## OREGON 5

From a road cut on the Burns-Ontario Highway (Oregon 54) 12.4 miles west of Juntura and either in the SE $\frac{1}{4}$  sec. 35 or the SW $\frac{1}{4}$  sec. 36, T. 20 S., R. 36 E., and about 5 miles southeast of Oregon 3. The deposit, as exposed by the road cut, has a triangular cross section 100 feet long and 20 feet high (fig. 16). Clayey soil forms the overburden on the sides of the triangle, and both diatomite and soil are covered with about 2 feet of gravel.

Filtration tests with minus 100-mesh material from a channel sample taken diagonally across the deposit gave a filtrate of 43 cc. in 20 minutes with excellent clarity. Sizing the diatomite to minus 250- plus 325-mesh increased the flow rate to 59 cc. with good clarity. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 80 cc., but the clarity was reduced to poor. Calcining minus 100-mesh diatomite with 4 percent NaCl and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 152 cc., but the clarity remained poor.



## JEFFERSON COUNTY

## OREGON 6

From the NW $\frac{1}{4}$  sec. 25, T. 11 S., R. 11 E., on the Warm Springs Indian Reservation and submitted by J. W. Elliott, superintendent, Warm Springs Agency, Warm Springs, Oreg. The deposit, about 45 feet thick, is between lava flows and exposed in a river gorge. The basalt covering is reported to vary from 3 to 15 feet.

Channel samples were taken of the entire face (Oregon 6a), from 0 to 5 feet (Oregon 6b), 10 to 15 feet (Oregon 6c), 20 to 25 feet (Oregon 6d), 30 to 35 feet (Oregon 6e), and from 40 to 45 feet (Oregon 6f), as measured from the top of a face exposed in the gorge. Oregon



FIGURE 16.—Oregon 5 deposit, Harney County. Dark areas are clayey soil. Note gravel at top left.

6a gave a chemical analysis of 83.5 percent  $\text{SiO}_2$ , 8.3 percent  $\text{Al}_2\text{O}_3$ , 2.9 percent  $\text{Fe}_2\text{O}_3$ , and 3.6 percent ignition loss. The clay equivalent for the  $\text{Al}_2\text{O}_3$  is 21.0 percent. The P. C. E. value for the same sample was cone 16.

Filtration tests with either minus 100-mesh or minus 250- plus 325-mesh material indicated that, with the exception of Oregon 6f, the flow rates were about the same for all samples. As shown in table 15, calcining minus 100-mesh material with or without the addition of 4 percent NaCl increased the flow rates but lowered the clarities. Calcined Oregon 6f gave about the same results after calcination as the other samples, although the uncalcined sized material's flow rates were only about one-third those of the other similarly treated samples. The 20- to 25-foot (Oregon 6d) and the 30- to 35-foot (Oregon 6e) samples gave the fastest flow rates. (See table 16.)

TABLE 16.—*Calcination and sizing effect upon Oregon 6 filter-aid properties*

UNCALCINED				
Sample	Interval, feet	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
b.....	0-5	-100	35	Good.
c.....	10-15	-100	42	Do.
d.....	20-25	-100	50	Excellent.
e.....	30-35	-100	43	Good.
f.....	40-45	-100	15	Do.
a.....	0-45	-250+325	32	Poor.
c.....	10-15	-250+325	39	Fair.
d.....	20-25	-250+325	46	Do.
e.....	30-35	-250+325	53	Do.
f.....	40-45	-250+325	17	Do.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
a.....	0-45	-250+325	82	Poor.
b.....	0-5	-250+325	76	Fair.
c.....	10-15	-250+325	76	Poor.
d.....	20-25	-250+325	96	Fair.
e.....	30-35	-250+325	98	Do.
f.....	40-45	-250+325	85	Do.
MINUS 100-MESH PLUS 4 PERCENT NaCl CALCINED TO 1,750° F. AND THEN SIZED				
b.....	0-5	-250+325	167	Fair.
c.....	10-15	-250+325	156	Poor.
d.....	20-25	-250+325	194	Do.
e.....	30-35	-250+325	193	Fair.
f.....	40-45	-250+325	117	Poor.

## KLAMATH COUNTY

## OREGON 7

From a bank of Odell Creek, about 0.3 mile east of the Davis Lake Ranger Station, in the south-center of sec. 11, T. 23 S., R. 7 E. According to Moore,<sup>48</sup> the diatomite represents the last stage of filling Davis Lake, which was formed by a lava flow that dammed the old channel of Odell Creek for a length of 3 miles and a width of 1.5 miles. The deposits at Davis Lake are interesting because they show how other deposits, notably the one at Terrebonne, have been formed.<sup>49</sup> Channel samples were taken from 0 to 1 feet (Oregon 7a), 1 to 2 feet (Oregon 7b), and 2 to 3 feet (Oregon 7c), as measured from the top of the creek bank. All three samples contained organic material.

Filtration tests with minus 100-mesh material showed that the top 1 foot (Oregon 7a) gave the best flow rate, 66 cc. in 20 minutes, and Oregon 7c the poorest, 42 cc. All had good clarity. Sizing Oregon 7a to minus 250- plus 325-mesh did not change either the flow rate or the clarity. Calcining minus 100-mesh Oregon 7a to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 95 cc. but reduced the clarity to fair. Calcining minus 100-mesh Oregon 7a with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 190 cc., but the clarity was poor. (See table 17.)

<sup>48</sup> Moore, Bernard N., p. 23 of work cited in footnote 47.<sup>49</sup> Moore, Bernard N., p. 23 of work cited in footnote 47.

TABLE 17.—*Calcination and sizing effects upon Oregon 7 filter-aid properties*

UNCALCINED					
Sample	Interval, feet	Screen size, mesh	Filtrate		
			cc./20 min.	Clarity	
a.....	0-1	-100	66	Good.	
b.....	1-2	-100	59	Do.	
c.....	2-3	-100	42	Do.	
a.....	0-1	-250+325	66	Do.	
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED					
a.....	0-1	-250+325	95	Fair.	
a+4% NaCl.....	0-1	-250+325	190	Poor.	

## OREGON 8

From a road cut on the Chiloquin-Sprague River highway (0.6 miles north of the Chiloquin railroad station) in the NW $\frac{1}{4}$  sec. 35, T. 34 S., R. 7 E. The diatomite exposure, the largest of a number of cuts north of the railroad station, was 400 feet long, 8 feet deep at the thickest part, and covered with 6 feet of overburden. The P. C. E. value for Oregon 8b was cone 7-8. Channel samples were taken from the upper one-third (Oregon 8a), the middle one-third (Oregon 8b), and the lower one-third (Oregon 8c).

Filtration tests with minus 100-mesh material showed little difference in flow rate from the top to the bottom sample. Oregon 8a gave the best results, 35 cc. in 20 minutes with good clarity, and Oregon 8b the poorest, 26 cc. with poor clarity. Sizing Oregon 8b material to minus 250- plus 325-mesh increased the flow rate to 44 cc.; but when Oregon 8c was similarly sized, the same results were obtained as with the minus 100-mesh material. Calcining minus 100-mesh Oregon 8c diatomite to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 104 cc. but reduced the clarity to fair. Calcining minus 100-mesh Oregon 8c with 4 percent NaCl to 1,750° F. and sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 189 cc., but the clarity was reduced to poor. (See table 18.)

TABLE 18.—*Calcining and sizing effects upon Oregon 8 filter-aid properties*

UNCALCINED				
Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Upper third.....	-100	35	Good.
b.....	Middle third.....	-100	26	Poor.
c.....	Lower third.....	-100	31	Good.
b.....	Middle third.....	-250+325	44	
c.....	Lower third.....	-250+325	31	Good.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	Lower third.....	-250+325	104	Fair.
c+4% NaCl.....	do.....	-250+325	189	Poor.

## OREGON 9

From the larger of two road cuts on the Bend-Klamath Falls Highway (U. S. 97) in T. 38 S., R. 9 E. The cut, 400 feet long, is 2.8 miles north of Ninth Avenue and Main Street, Klamath Falls. A diatomite thickness of 40 feet is exposed by the road and an additional 15 feet by a railroad cut, making a total exposure of 55 feet. Figure 17 shows the nature of the overburden, which is 6 to 15 feet thick, and the surface staining on the diatomite. Both a fault plane and a faulted 6-inch ash stratum (displaced 9 inches) may be seen at the south end of the cut. The P. C. E. value for Oregon 7b was cone 12; for Oregon 7c, cone 13-14, and for Oregon 7d, cone 14. Channel samples were taken from 5 to 10 feet (Oregon 9a), 15 to 20 feet



FIGURE 17.—Oregon 9 deposit, Klamath County. Note darkening by surface stains and angular blocks in the overburden.

(Oregon 9b), 25 to 30 feet (Oregon 9c), and 35 to 40 feet (Oregon 9d), as measured from the top of the bed. No samples were taken below the road level.

Filtration tests with material sized to minus 100-mesh gave an average filtrate for all 4 samples of 18 cc. in 20 minutes, with fair to good clarity. Sizing Oregon 9b, c, and d to minus 250- plus 325-mesh increased the average flow rate to 60 cc. Calcining minus 100-mesh Oregon 9d diatomite to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 110 cc. with good clarity. Calcining minus 100-mesh Oregon 9d with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 163 cc., but the clarity was reduced to poor. (See table 19.)

TABLE 19.—*Calcination and sizing effects upon Oregon 9 filter-aid properties*

UNCALCINED				
Sample	Interval, feet	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	5-10	-100	15	Fair.
b.....	15-20	-100	18	Do.
c.....	25-30	-100	18	Do.
d.....	35-40	-100	20	Good.
b.....	15-20	-250+325	65	
c.....	25-30	-250+325	60	
d.....	35-40	-250+325	55	
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
d.....	35-40	-250+325	110	Good.
d+4% NaCl.....	35-40	-250+325	163	Fair.

## OREGON 10

From a road cut 2.5 miles south of the Link River Bridge, Klamath Falls, on the Klamath Falls-Redding Highway (U. S. 97) in the NE $\frac{1}{4}$  sec. 18, T. 39 S., R. 9 E. This cut is the largest of a series both along the Redding Highway and the Klamath Falls-Ashland Highway (Oregon 66). The sampled bed, exposed for 400 feet and having a thickness of 10 feet, is flat-lying and appears to be the base of an oval-shaped hill, which rises to an estimated maximum height of 100 feet above the top of the diatomite. In contrast with most talus material from other deposits in the Klamath Falls area, the talus was powdery and not blocky. Diagonal channel samples were taken from the north 130 feet (Oregon 10a), the middle 140 feet (Oregon 10b), and the south 130 feet (Oregon 10c).

Filtration tests with minus 100-mesh material gave an average filtrate for all three samples of 25 cc. in 20 minutes, with poor to fair clarity. Sizing Oregon 10c to minus 250- plus 325-mesh only increased the flow rate to 31 cc. Calcining minus 100-mesh Oregon 10c to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 70 cc. Calcining minus 100-mesh Oregon 10c with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 119 cc. (See table 20.)

TABLE 20.—*Calcination and sizing effects upon Oregon 10 filter-aid properties*

UNCALCINED				
Sample	Interval, feet	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	North 130.....	-100	23	Fair.
b.....	Middle 140.....	-100	24	Do.
c.....	South 130.....	-100	27	Poor.
c.....	South 130.....	-250+325	31	
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	South 130.....	-250+325	70	
c+4% NaCl.....	do.....	-250+325	119	

## OREGON 11

From a railroad cut 0.2 mile east of the Lost River Bridge, Olene, Oreg., in the NE $\frac{1}{4}$  sec. 14, T. 39 S., R. 10 E. The deposit, which contains volcanic-ash streaks, is exposed for a distance of 400 feet and a thickness of 20 feet. The bed appears to be flat-lying. Several small outcrops below the railroad indicate the bed might reach a thickness of 50 feet. Diagonal channel samples were taken of the west 200 feet (Oregon 11a) and the east 200 feet (Oregon 11b). The P. C. E. value for both samples was cone 8-9.

Filtration tests with minus 100-mesh material gave filtrates of 25 cc. in 20 minutes, with poor clarity for both samples. Sizing the diatomite to minus 250- plus 325-mesh increased both flow rates to 45 cc. and increased the clarity to good. Calcining minus 100-mesh Oregon 11a to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 95 cc. but reduced the clarity to fair. Calcining minus 100-mesh Oregon 11a with 4 percent NaCl to 1,750° F. and then sizing the calcined product increased the flow rate to 172 cc. in 20 minutes, but the clarity was reduced to poor.

## OREGON 12

From the NE $\frac{1}{4}$  sec. 14, T. 39 S., R. 10 E., about 0.1 mile east of Oregon 11. The deposit looked similar to the previous deposit but did not have apparent ash streaks.

Filtration tests with minus 100-mesh material gave a filtrate of 41 cc. in 20 minutes with fair clarity. Sizing to minus 250- plus 325-mesh decreased the flow rate to 27 cc. but increased the clarity to good. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 67 cc. with fair clarity. Calcining minus 100-mesh material with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 92 cc. in 20 minutes, but with poor clarity.

## OREGON 13

From a railroad cut 0.1 mile west of Oregon 11 in the NE $\frac{1}{4}$  sec. 14, T. 39 S., R. 10 E. The exposure is about 10 feet thick and 150 feet long. Considerable volcanic ash is intercalated with the diatomite.

Filtration tests with minus 100-mesh material gave a filtrate of 44 cc. in 20 minutes with good clarity. Sizing to minus 250- plus 325-mesh lowered the flow rate to 37 cc. and the clarity to fair. After calcining the material, either with or without NaCl, and then sizing the calcined product, no filter cake was formed during the filtration tests. The calcination temperature might have been too high and sintered the individual diatom pieces together.

## OREGON 14

From a road cut 1.0 mile east of Dairy on the Klamath Falls-Lakeview Highway (Oregon 66) in the SW $\frac{1}{4}$  sec. 27, T. 38 S., R. 11 $\frac{1}{2}$  E. The deposit is exposed for a distance of 200 feet and an average thickness of 6 feet. The overburden, 1 to 2 feet thick, appears to be a series of 3- or 4-inch-thick basalt flows that have weathered in a platy struc-

ture. In some instances, the overburden overhangs the diatomite by 2 feet. (See fig. 18.) A diagonal channel sample was taken.

Filtration tests with minus 100-mesh material gave a filtrate of 29 cc. in 20 minutes with excellent clarity. Sizing the diatomite to minus 250- plus 325-mesh had no effect upon the flow rate, although it reduced the clarity to good. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 165 cc. in 20 minutes, but the clarity was reduced to fair. Calcining minus 100-mesh material with 4 percent NaCl to 1,750° F. increased the flow rate to 322 cc., but the clarity was reduced further to poor.

#### OREGON 15

From a road cut 1.3 miles west of Oregon 14 and in the NE $\frac{1}{4}$  sec. 33, T. 38 S., R. 11 $\frac{1}{2}$  E. Diatomite is exposed for a distance of 250 feet and an average thickness of 10 feet. A mixture of diatomite and clayey soil comprises the overburden, which has a maximum thickness of 4 feet. The P. C. E. value was cone 7-8.



FIGURE 18.—Oregon 14 deposit, Klamath County. Note overhanging platy overburden.

Filtration tests with minus 100-mesh material gave a filtrate of 21 cc. in 20 minutes with good clarity. Sizing to minus 250- plus 325-mesh increased the flow rate to 35 cc. and the clarity was good. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product increased the flow rate to 97 cc., but the clarity was fair. Calcining minus 100-mesh material with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 158 cc., but the clarity was poor.

#### MALHEUR COUNTY

Extensive diatomite beds in north-central Malheur County and 2 to 12 miles north and northeast of Harper are intermingled with other

sediments and tuff and are covered with a basalt capping (in most places). Moore<sup>50</sup> describes the area as follows:

The Harper diatomite district extends in a general way from the Malheur River northwestward to Westfall, a distance of about 12 miles. The district is an irregular basin cut out of the old plateau surface of central Oregon by the Malheur River and its tributary Bully Creek. It is roughly mushroom shaped, its base lying parallel to the Malheur River and a little south. It is constricted between Harper and Westfall but spreads out in an irregular fashion along Bully Creek. The basin occupies more than 50 square miles, much of which is underlain by high-grade diatomite.

#### OREGON 16

From an old working face in the north-center sec. 34, T. 18 S., R. 41 E., 8.6 miles north of Harper, on the Harper-Westfall road, and 0.5 mile northeast of the road. As shown in figure 19, the face is streaked with iron-stained strata, and there is little overburden—about 6 inches.

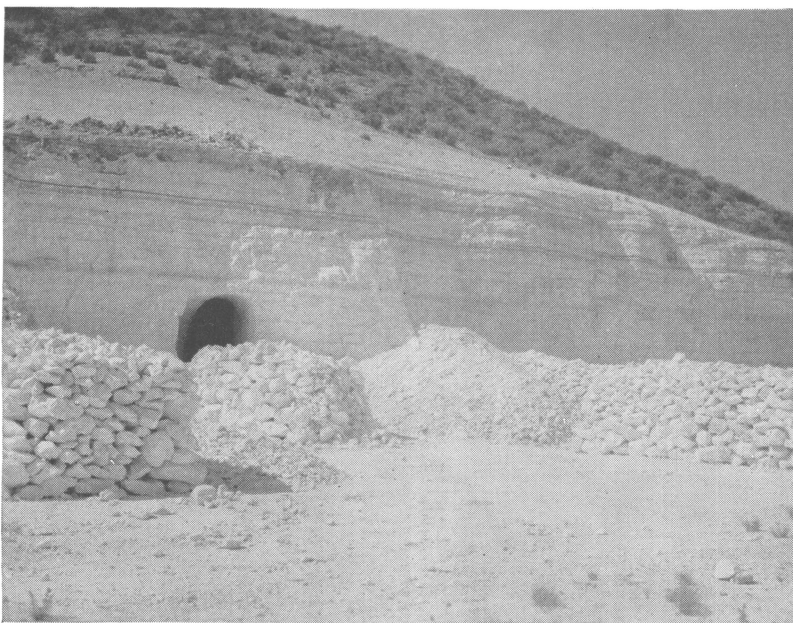


FIGURE 19.—Oregon 16 deposit, Malheur County. Note iron-stained strata and thin overburden. Piles are diatomite pieces stacked for air-drying. The deposit was not being mined when visited in 1940.

Some volcanic-ash strata also were present, but they do not show in the photograph. The working face is about 200 feet along and 8 to 20 feet high. Channel samples were taken of the upper one-third (Oregon 16a), middle one-third (Oregon 16b), and lower one-third (Oregon 16c) near the center of the face.

Filtration tests with minus 100-mesh material, Oregon 16b (middle third), gave the best filtration results—73 cc. in 20 minutes with fair clarity. The top and bottom thirds gave 54 cc. and 49 cc., respectively. Sizing Oregon 16b to minus 250- plus 325-mesh increased the clarity to good but did not affect the flow rate. Calcining minus 100-mesh Oregon 16b to 1,750° F. and then sizing the calcined product to minus

<sup>50</sup> Moore, Bernard N., p. 65 of work cited in footnote 47.



250- plus 325-mesh increased the flow rate to 191 cc. in 20 minutes. Calcining minus 100-mesh Oregon 16b with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh only increased the flow rate to 210 cc. and lowered the clarity to poor. (See table 21.)

TABLE 21.—*Calcination and sizing effects upon Oregon 16 filter-aid properties*

Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Upper third.....	-100	54	Fair.
b.....	Middle third.....	-100	73	Do.
c.....	Lower third.....	-100	49	Do.
b.....	Middle third.....	-250+325	75	Good.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
b.....	Middle third.....	-250+325	191	Fair.
b+4% NaCl.....	Middle third.....	-250+325	210	Poor.

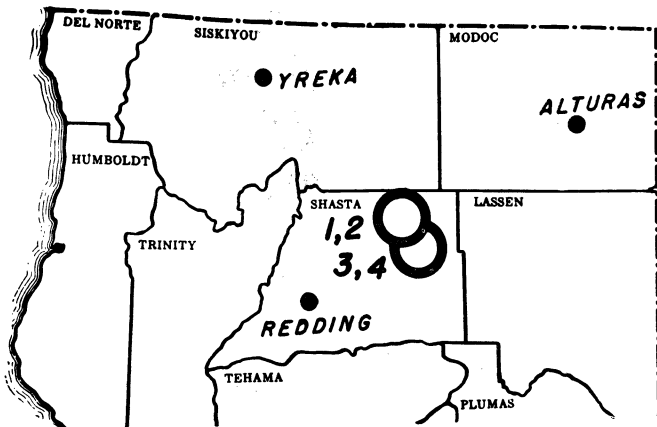


FIGURE 20.—Map showing location of sampled California diatomite deposits.

## CALIFORNIA

### SHASTA COUNTY

The only California deposits sampled were those in the vicinity of Britton Lake and Fall River Mills in northeastern Shasta County. (See figure 20.) This area is described by Logan<sup>51</sup> as follows:

Among the largest deposits of diatomaceous earth in the state are those along the Pit River from the region a few miles east of the mouth of Hat Creek on both streams, downstream to Pit 3 powerhouse. The material varies in quality as regards compactness and tint, in different localities, but there is an immense tonnage of good grade in sight. The region mentioned \* \* \* stretches for over 12 miles east and west, and four miles north and south. \* \* \* The full extent of diatomaceous earth deposits in Shasta County has never been definitely determined and there is probably a great deal more of it than has been realized. \* \* \*. The deposits were formed where the relatively recent volcanic flows

<sup>51</sup> Logan, C. A., Shasta County: Diatomaceous Earth: California State Mining Bureau, Mining in California, April 1926, pp. 163-66.

dammed the rivers and creeks, forming lakes in which the siliceous deposits accumulated. \* \* \* Later volcanic flows then covered and preserved the deposits. The modern streams have cut through the lava in places, exposing the diatomite.

#### CALIFORNIA 1

From a 30-foot road cut in the SW $\frac{1}{4}$  sec. 18, T. 37 N., R. 3 E., about 0.5 mile southwest of the Pit River Lodge, or 3.4 miles southwest of the junction between the Cayton Post Office Road and the Bartle-Cayton-Burney Highway (California 89).<sup>1</sup> The cut was 15 feet deep, but overhanging and dense brush made it impossible to estimate thickness of the overburden.

Filtration tests with minus 100-mesh material gave a filtrate of 94 cc. in 20 minutes with fair clarity. Sizing to minus 250- plus 325-mesh increased the flow rate to 109 cc. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus



FIGURE 21.—California 2 deposit, Shasta County.

325-mesh made little change in the flow rate (106 cc.), but the clarity was increased to good. Calcining minus 100-mesh diatomite with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 275 cc. in 20 minutes, but the clarity was reduced to poor.

#### CALIFORNIA 2

From a road cut 0.4 mile north of the Britton Lake Bridge on the Bartle-Cayton-Burney Highway (California 89) in the SW $\frac{1}{4}$ SE $\frac{1}{4}$  sec. 21, T. 37 N., R. 3 E. (Fig. 21.) This was one of a number of exposures (about 2 miles east and south of California 1) from the level of Britton Lake to the top of the surrounding hills—a vertical distance of 350 to 450 feet. The diatomite bed exposed by the road cut was about 60 feet thick at its thickest point, and overburden varied from 2 to 10 feet. Channel samples were taken at 25 to 30 feet (California 2a) and at 35 to 40 feet (California 2b), as measured from the top of the exposure.

Filtration tests with minus 100-mesh California 2a diatomite gave a filtrate of 23 cc. in 20 minutes, and California 2b material gave 31 cc., with good clarity. Sizing California 2b to minus 250- plus 325-mesh only increased the flow rate to 37 cc. Calcining minus 100-mesh California 2b to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 52 cc. in 20 minutes, and the clarity was reduced to fair. (See table 22.).

TABLE 22.—*Calcination and sizing effects upon California 2 filter-aid properties*

UNCALCINED				
Sample	Interval, feet	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	25-30	-100	23	Good.
b.....	35-40	-100	31	Do.
b.....	35-40	-250+325	37	Do.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
b.....	35-40	-250+325	52	Fair.

### CALIFORNIA 3

From a road cut on the Redding-Alturas Highway (U. S. 299), 7.1 miles west of Falls River Mills, at the east end of the Hat Creek Bridge in the NE $\frac{1}{4}$ NE $\frac{1}{4}$  sec. 20, T. 36 N., R. 4 E. The deposit, as exposed by the road cut, is 350 feet long, 40 feet thick (maximum), and is covered with 2 to 10 feet of soil overburden. The dip is 3° to 5° W. A small prospecting tunnel had been driven into the west side of the deposit. Channel samples were taken at 0 to 5 feet (California 3a) and 10 to 15 feet (California 3b), as measured from the top of the deposit near the center of the cut.

Filtration tests with minus 100-mesh material gave a filtrate of 78 cc. in 20 minutes with California 3a and 83 cc. with California 3b. Both had excellent clarity. Sizing the latter material to minus 250- plus 325-mesh did not change the flow rate but lowered the clarity to good. Calcining minus 100-mesh California 3b to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh lowered the flow rate to 65 cc. and the clarity to fair. Calcining the same material with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 147 cc. in 20 minutes but lowered the clarity to poor. (See table 23.)

TABLE 23.—*Calcination and sizing effects upon California 3 filter-aid properties*

UNCALCINED				
Sample	Interval, feet	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	0-5	-100	78	Excellent.
b.....	10-15	-100	83	Do.
b.....	10-15	-250+325	84	Good.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
b.....	10-15	-250+325	65	Fair.
b+4% NaCl.....	10-15	-250+325	147	Poor.

## CALIFORNIA 4

From a road cut 0.2 mile east of California 4 on the Redding-Alturas Highway (U. S. 299) in the NE $\frac{1}{4}$ NE $\frac{1}{4}$  sec. 20, T. 36 N., R 4 E. The deposit, as exposed by the road cut, is 200 feet long, 30 feet thick, and dips 15° S. 25° W. It rests conformably (apparently) upon 2 to 4 inches of alluvial material, which, in turn, rests upon volcanic ash. (See fig. 22.)

Filtration tests with minus 100-mesh material from a channel sample taken diagonally across the deposit gave a filtrate of 36 cc. in 20 minutes with excellent clarity. Sizing the diatomite to minus 250- plus 325-mesh increased the flow rate to 47 cc. but decreased the



FIGURE 22.—California 4 deposit, Shasta County. Note ash and thin alluvial-material stratum.

clarity to good. Calcining minus 100-mesh material to 1,750° F. and then sizing the product to minus 250- plus 325-mesh increased the flow rate to 63 cc., but the clarity was further decreased to fair. Calcining minus 100-mesh diatomite with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 141 cc. in 20 minutes. The clarity remained fair.

## CALIFORNIA 5

From a road cut 5.0 miles west of Fall River Mills on the Redding-Alturas Highway (U. S. 299) in either the SE $\frac{1}{4}$ SE $\frac{1}{4}$  sec. 9 or the NE $\frac{1}{4}$ NE $\frac{1}{4}$  sec. 16, T. 36 N., R. 4 E. This was the largest of a series of road cuts extending for a distance of 0.3 mile along the

highway. All were covered with what appeared to be glacial material. The P. C. E. value for the diatomite was cone 32.

Filtration tests with minus 100-mesh material from a channel sample taken diagonally across the deposit gave a filtrate of 43 cc. with good clarity. Sizing the diatomite to minus 250- plus 325-mesh increased the flow rate to 67 cc., but the clarity was reduced to fair. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 89 cc. Calcining minus 100-mesh diatomite with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 270 cc., but the clarity was reduced to poor.

#### IDAHO

Three diatomite samples were taken from deposits in southwestern Idaho—one each from Camas, Elmore, and Owyhee Counties. (See fig. 23.) Idaho 1 contained more visible volcanic ash strata and was faulted more than any other deposit sampled.

#### CAMAS COUNTY

#### IDAHO 1

From a badly faulted deposit containing numerous volcanic-ash strata, in either the SW $\frac{1}{4}$  sec. 34 or the SE $\frac{1}{4}$  sec. 33, T. 2 N., R. 14 E. (Boise meridian), 8.2 miles north of Fairfield and 0.9 mile inside the Sawtooth National Forest. Old mining operations have exposed a "J"-shaped face about 300 feet long and 20 feet high. Three to six feet of soil forms the overburden. Numerous volcanic-ash strata 1 to 18 inches thick were present. Although most of the 18 fault displacements observed were only 1 to 3 inches, there was a 30-inch displacement at the east end of the pit. As shown in figure 24, a 12- to

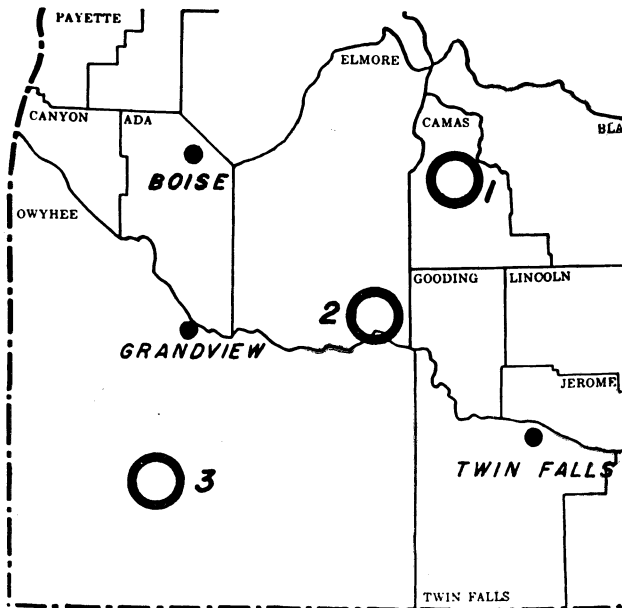


FIGURE 23.—Map showing location of sampled Idaho diatomite deposits.

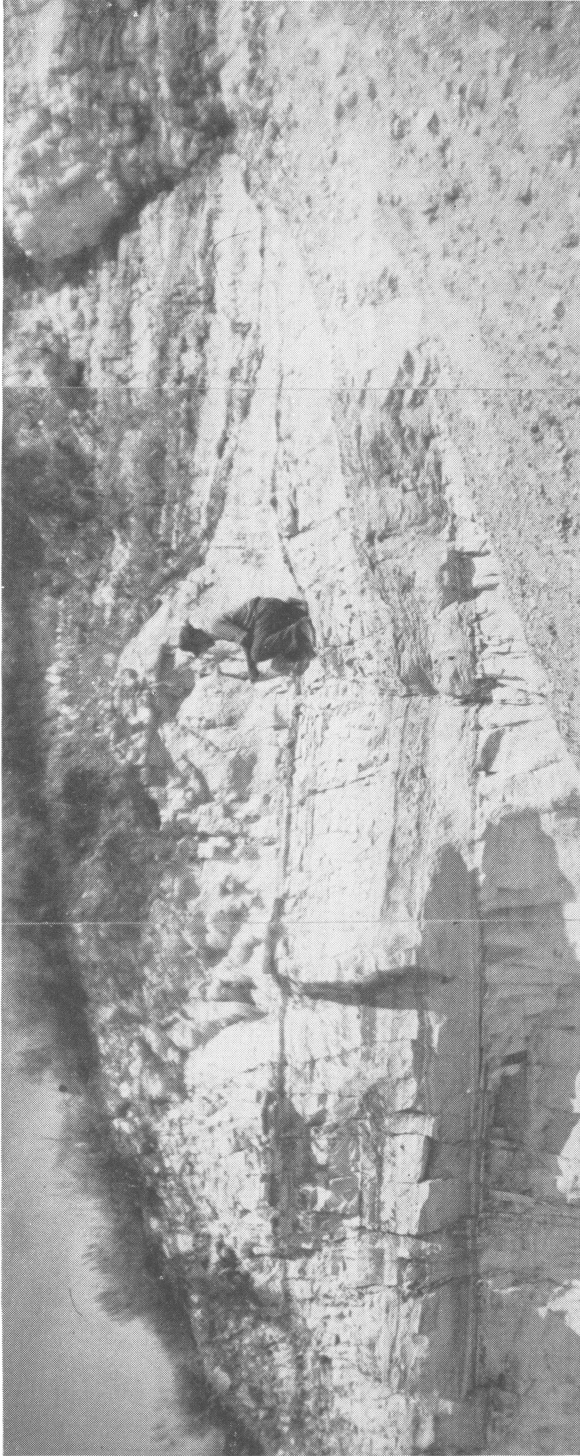


FIGURE 24.—Idaho 1 deposit, Camas County, north face. Dark stratum just below center represents 12 to 18 inches of volcanic ash, and dark stratum just above center represents 2 inches of volcanic ash. Note small fault displacements at left.

18-inch and a 2-inch volcanic-ash strata formed three rather distinct diatomite strata. Channel samples (from the west center) were taken of the diatomite above the 2-inch ash stratum (Idaho 1a), between the 2-inch and the 12- to 18-inch stratum (Idaho 1b), and below the 12- to 18-inch stratum (Idaho 1c).

Filtration tests with minus 100-mesh material gave the best results with the lower diatomite—Idaho 1c—121 cc. in 20 minutes with poor clarity. The other two samples, similarly sized, gave an average of 48 cc. with fair to poor clarity. Sizing Idaho 1a to minus 250- plus 325-mesh lowered the flow rate to 51 cc. but increased the clarity to fair. Calcining minus 100-mesh Idaho 1a to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh gave a filtrate of 78 cc. in 20 minutes with fair clarity. Calcining minus 100-mesh Idaho 1a with 4 percent NaCl to 1,750° F. and then sizing the product to minus 250- plus 325-mesh increased the flow rate to 197 cc., but with poor clarity. (See table 24.)

TABLE 24.—*Calcination and sizing effects upon Idaho 1 filter-aid properties*

UNCALCINED				
Sample	Interval	Screen size, mesh	Filtrate	
			cc./20 min.	Clarity
a.....	Upper.....	-100	47	Fair.
b.....	Middle.....	-100	49	Poor.
c.....	Lower.....	-100	121	Do.
c.....	do.....	-250+325	51	Fair.
MINUS 100-MESH CALCINED TO 1,750° F. AND THEN SIZED				
c.....	Lower.....	-250+325	78	Fair.
c+4% NaCl.....	do.....	-250+325	197	Poor.

#### ELMORE COUNTY

##### IDAHO 2

From road cut 2 miles up Bellmare Creek, or 4 miles northeast of King Hill, and probably in the SW $\frac{1}{4}$  sec. 27, T. 4 S., R 11 E. (Boise meridian). The deposit, as exposed by the road cut, was as 50 feet long and 8 to 15 feet thick.

Filtration tests with minus 100-mesh material gave a filtrate of 54 cc. in 20 minutes with poor clarity. Sizing the diatomite to minus 250- plus 325-mesh lowered the flow rate to 39 cc. but raised the clarity to fair. Calcining minus 100-mesh material to 1,750° F. and then sizing the product to minus 250- plus 325-mesh increased the flow rate to 83 cc. in 20 minutes with poor clarity. Calcining minus 100-mesh material with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 155 cc. in 20 minutes.

#### OWYHEE COUNTY

##### IDAHO 3

From a storage bin of an operating company in Owyhee County. The deposit, about 70 miles south of the plant, was not visited because of road conditions (1940). The company has 18 160-acre claims, which

comprise all of secs. 34 and 35, S $\frac{1}{2}$  sec. 36, T. 11 S., R. 2 W., and all of secs. 2 and 3, T. 12 S., R. 2 W. (Boise meridian).

Filtration tests with minus 100-mesh material gave a filtrate of 45 cc. in 20 minutes with good clarity. Sizing the diatomite to minus 250- plus 325-mesh increased the flow rate to 52 cc. but reduced the clarity to fair. Calcining minus 100-mesh material to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate to 144 cc. in 20 minutes with good clarity.

### COMMERCIAL FILTER-AIDS

Thirteen commercial filter-aids and 5 commercial concrete admixtures were examined. The samples were tested as received. As shown in table 25, samples Commercial 1 and Commercial 16 also were tested after sizing to minus 250- plus 325-mesh and after calcining to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh.

The data of table 25 indicate that the calcined materials have the highest flow rates. Sizing and calcination increased the flow rates of both sample 1, a commercial filter-aid, and sample 16, a commercial concrete admixture.

TABLE 25.—Flow rates, clarities, P. C. E. values, chemical compositions, and theoretical clay contents of commercial filter-aids

Sample No.	Commercial Preparation	Flow rate		Relative clarity	P. C. E., cone	Percent				
		Cc.	Min.			SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Ign. loss	Theoretical clay
1	Dried	84	20			82.1	5.3	2.8	6.0	13.4
1B	(1) Dried	83	20	Good						
1C	(2) Dried	106	20	do						
2	Calcined	174	20	do	18	89.4	4.3	2.6	.9	10.9
3	do	526	20			89.6	3.8	2.5	.0	6.3
4	do	285	5	Fair						
5	do	477	10	do		86.0	5.2	1.8	.1	13.2
6	do	264	4	Poor						
7	do	243	3	do						
8 <sup>3</sup>	Dried					83.9	5.5	3.0	4.5	13.9
9	do	109	20			87.0	3.5	2.5	4.1	8.9
10	do	248	20							
11	Calcined	226	10							
12	do	294	3	Fair						
13 <sup>3</sup>	Dried					90.9	2.7	1.4	4.4	6.8
14	Calcined					90.5	3.1	1.7	.6	7.8
15 <sup>3</sup>	Dried					82.3	5.0	2.5	4.8	12.8
16 <sup>3</sup>	do	18	20		31+	90.5	2.0	3.5	5.2	5.1
16B	(1) do	69	20	Fair						
16C	(2) do	131	20	Poor						
17 <sup>3</sup>	Dried	12	20			89.5	1.9	3.7	5.0	4.8
18	Calcined	352	10	Poor						

<sup>1</sup> Sized in laboratory, by screening, to -250+325 mesh.

<sup>2</sup> Calcined in laboratory to 1,750° F. and the calcined product sized to -250+325 mesh.

<sup>3</sup> Sold as either an admixture or an insulation material.

Chemical analyses of 11 of the samples showed that the calcined samples had an average ignition loss of 0.4 percent, as compared with 4.9 percent for the dried samples. Although alkalis were not determined, they probably would be greater in the calcined samples because NaCl or other alkalis are usually added before calcination.

*Comparison between advertised and laboratory flow rates.*—Table 26 gives the comparison between the flow-rate ratios advertised by the producing company and the ratios as determined in the laboratory. There was good agreement between the two sets of values, except for



the fastest, in which case the laboratory value was only 75 percent of the advertised. This might be accounted for by the fact that the flow rate was so fast that it was difficult to maintain a filter cake during the test period.

TABLE 26.—*Flow-rate ratios of six commercial filter-aids. Both advertised and laboratory ratios are given*

Commercial sample No.	Flow-rate ratio		Flow-rate group <sup>1</sup>
	Advertised	Laboratory	
1.....	1	1	Medium.
3.....	2	2	Very fast.
4.....	6	6	Do.
5.....	8	7	Do.
6.....	9	8	Do.
7.....	18	12	Do.

<sup>1</sup> See table 38.

## SPECIAL TESTS

### EFFECT OF CALCINATION WITH VARIOUS CHEMICALS

Three series of calcination tests were made to determine the effect of chemical additions on filter-aid properties. In the first series, salt (NaCl) was used; in the second, borax ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ); and in the third, soda ash (65 percent  $\text{Na}_2\text{CO}_3$ ). Four minus 100-mesh Washington diatomites were tested, and the additions of chemicals were 0.0, 0.5, 1.0, 2.0, and 4.0 percent. The mixtures were calcined to 1,750° F. and sized to minus 250- plus 325-mesh. Table 27 gives the changes in total flow, and figure 25 shows the general improvement in flow rate.

TABLE 27.—*Effect of chemical additions upon filter rates of calcined diatomites, cc. per 20 minutes*

Sample	Chemical addition <sup>1</sup>				Increase in flow rate, percent <sup>2</sup>		
	Percent	Salt	Borax	Soda ash	Salt	Borax	Soda ash
Washington 2.....	0.0	48	48	48	0.0	0.0	0.0
	.5	63	56	57	38.4	16.7	18.7
	1.0	68	72	58	41.7	50.0	20.8
	2.0	97	95	63	102.1	97.9	28.6
	4.0	149	175	129	210.4	264.6	168.8
Washington 11.....	.0	82	82	82	.0	.0	.0
	.5	100	118	154	21.9	30.5	87.8
	1.0	115	131	148	40.3	59.8	80.5
	2.0	125	151	176	51.5	84.1	114.6
	4.0	218	192	162	165.9	134.1	97.6
Washington 16.....	.0	118	118	118	.0	.0	.0
	.5	178	156	113	50.9	32.2	-4.2
	1.0	256	165	200	116.9	49.8	69.5
	2.0	255	197	178	116.1	66.9	50.8
	4.0	243	242	194	105.9	105.1	64.4
Washington 17.....	.0	48	48	48	.0	.0	.0
	.5	89	101	83	85.5	110.4	72.9
	1.0	94	105	93	95.8	118.8	93.8
	2.0	109	124	92	127.1	158.3	91.7
	4.0	141	162	139	193.8	237.5	189.6

<sup>1</sup> Minus 100-mesh diatomite calcined with indicated chemical to 1,750° F. and then sized to -250+325 mesh.

<sup>2</sup> Improvement in flow rate over calcined diatomite with no chemical addition.

Upon the basis of average improvement in flow rate over that of calcined diatomite with no chemical additions for these four samples, there is little difference between the three chemicals up to 1 percent. Borax increased the flow the greatest amount, salt next, and soda ash the least when more than 1 percent was used. An increase in chemical addition increased the flow rate but not in proportion to the amount added. There was no general change in clarities.

Other tests of Washington 2 calcined with either 4 percent  $\text{CaCO}_3$  or with 4 percent  $\text{BaCO}_3$  only increased the flow rate about 45 percent, corresponding to values for 1 percent salt or 1 percent borax. Wash-

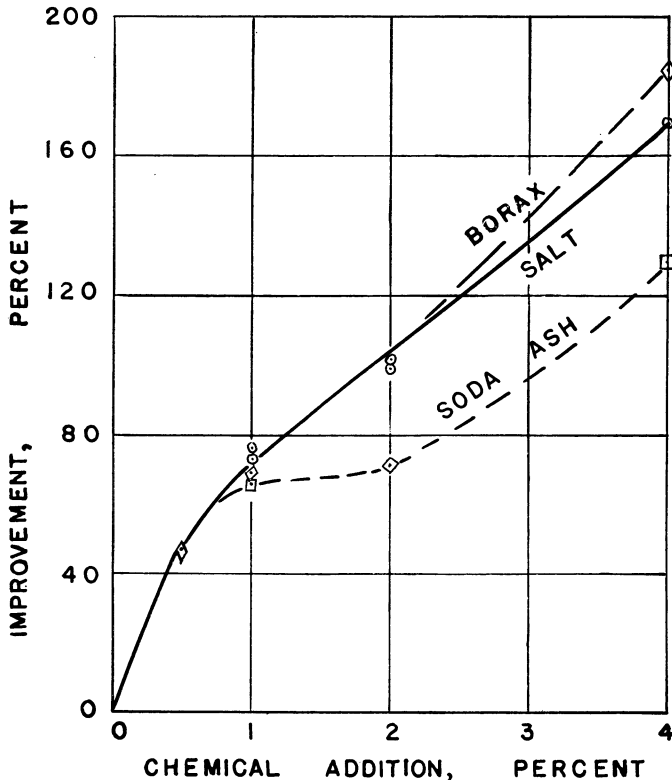


FIGURE 25.—Relationship between flow-rate increase and chemical additions.

ington 17, calcined with either 2 percent fluorite or 2 percent zinc hydrosulfite, only increased the flow rate about 60 percent, which was less than the increases obtained with 0.5 percent salt, borax, or soda ash.

#### ESTIMATED COST OF CALCINATION AND CHEMICAL ADDITIONS

Filtration tests showed that only the calcined filter-aids had fast or very fast flow rates and that most of these had been calcined with a chemical addition ( $\text{NaCl}$ ). Accordingly, a quadrant chart (fig. 26) was drawn to estimate the cost of calcination with and without a chemical added, over and above the cost of drying. It was assumed that the present rotary driers used in the Pacific Northwest diatomite plants could be converted to calciners with little cost. No estimate for amortization of the drier conversion was included.

EXAMPLE SHOWING USE OF ESTIMATING CHART

For an example problem, the following assumptions were made:

Calcination temperature.....	1,750° F.
Drying temperature.....	300° F.
Mean specific heat.....	0.26.
Kiln efficiency.....	20 percent for coal.
Do.....	30 percent for oil.
Heating value of coal.....	12,000 B. t. u. per pound.
Heating value of oil.....	144,000 B. t. u. per gallon.
Cost of coal.....	\$5 per ton.
Cost of oil.....	6 cents per gallon.
Chemical addition.....	4 percent.
Cost of chemical.....	\$26 per ton.

*Method.*—Follow the heavy dashed line vertically downward from the middle of the horizontal line (calcination temperature) in the upper-left quadrant to the diagonal line representing 300° F. drying

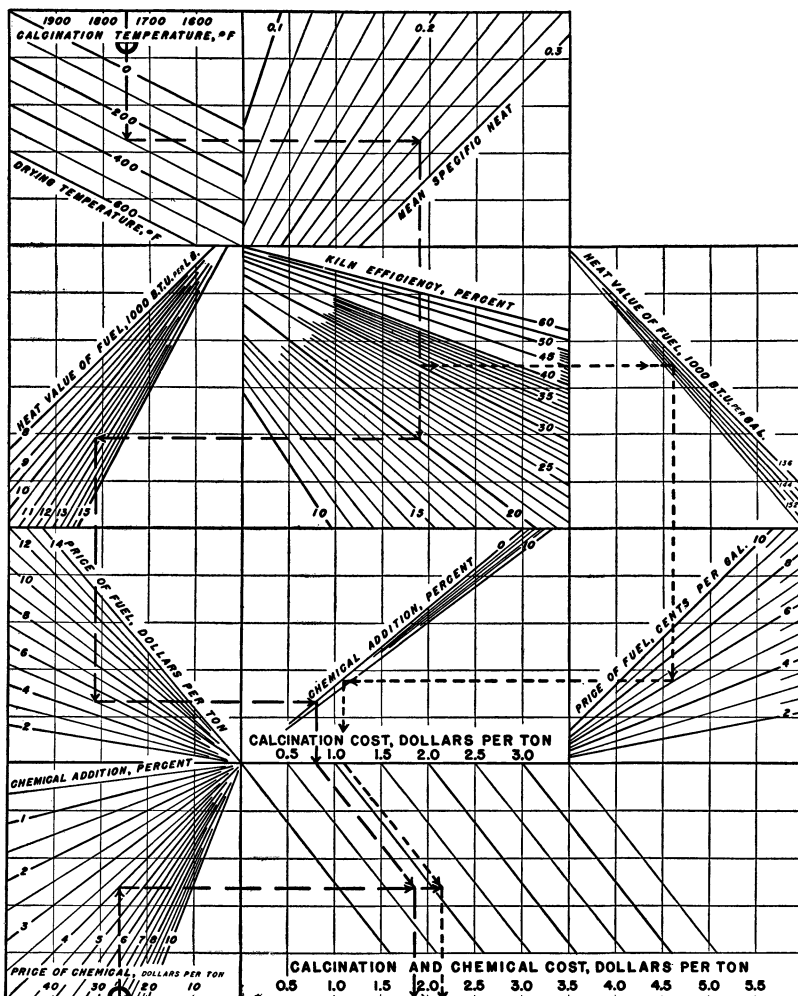


FIGURE 26.—Quadrant chart for estimating additional cost of calcination and chemical additions over and above drying.

temperature; then horizontally to the right, to the radial line representing a 0.26 specific heat; then vertically downward to the radial line for 20 percent efficiency; horizontally to the left to radial line for 12,000 B. t. u.; vertically downward to the radial line for \$5 per ton; horizontally to the right to radial line for 4 percent chemical addition; and then vertically downward to the base line of the quadrant, which shows the calcination cost, in dollars per ton, above drying cost. This last quadrant is a correction factor for the added weight of the chemical addition, because all additions were made on the basis of 1 ton of dried diatomite as 100; that is, a 4-percent addition would equal 80 pounds of chemical per ton of diatomite.

To find the additional cost for the chemical, start at the horizontal line in the lower-left quadrant; go vertically upward to the radial line for 4 percent chemical addition; then horizontally to the right to intersect the heavy dashed line paralleling the light guide lines; and then vertically downward to the base line of the lower-right quadrant, which shows the estimated additional cost for calcination and the addition of a chemical. The last diagonal line was drawn from the line representing additional cost for calcining. The cost for oil is found similarly, except that in the third quadrant (kiln efficiency, percent) the direction is right instead of left.

In the above example, the additional cost, over and above drying, for calcining 1 ton of diatomite with 4 percent chemical addition and using coal as a fuel would be about \$1.80; using oil as a fuel, the cost would be about \$2.15.

#### EFFECT OF CALCINATION RATE AND TEMPERATURE

To find the effect of calcination rate and temperature, two series of tests were made with Washington diatomite (from the same deposit as Washington 10) that had been pulverized in a Raymond hammer mill. In the first series, the temperature was held constant for half an hour, a sample was taken, and then the temperature was increased to the next higher temperature. In the second series, the temperature was held for 2 hours before being increased. The results of these tests are given in table 28 and figure 27.

TABLE 28.—Effect of calcination temperature and time upon filtration properties

Calcination temperature, °F.	Calcination time, 0.5 hour		Calcination time, 2 hours	
	Flow rate, cc./20 min.	Clarity, galv. defl. <sup>1</sup>	Flow rate, cc./20 min.	Clarity, galv. defl. <sup>1</sup>
Uncalcined.....	46	8.5	46	8.5
392.....	48	-----	41	-----
752.....	52	-----	63	9.5
1,112.....	68	-----	76	9.5
1,472.....	76	11.0	86	10.0
1,652.....	92	11.0	105	10.5
1,832.....	116	12.5	141	13.0
2,012.....	155	14.5	166	14.5
2,192.....	141	15.5	230	15.5
2,300.....	135	17.0	-----	-----

<sup>1</sup> Large galvanometer deflection caused by more suspended matter; see later description of apparatus.

The data from these tests indicate that for a given diatomite, a longer heating period will produce a filter-aid with the same properties as one heated for a shorter period but to a higher temperature.

For example, the sample tested gave the same flow rate, 120 cc. per 20 minutes, by either heating for 2 hours at 1,750° F. or heating for 0.5 hour at 1,860° F. The data also shows that an increase in temperature increased the flow rate but decreased the clarity; therefore, the calcination temperature used should be chosen to give specific properties.

For the sample tested, better clarity was obtained with the 2-hour calcination for a given flow rate than was obtained with the 0.5-hour calcination. A filter-aid with a flow rate of 120 cc. per 20 minutes would have a clarity of 12 (galvanometer deflection) for the 2-hour calcination or a clarity of 13 for the 0.5-hour calcination.

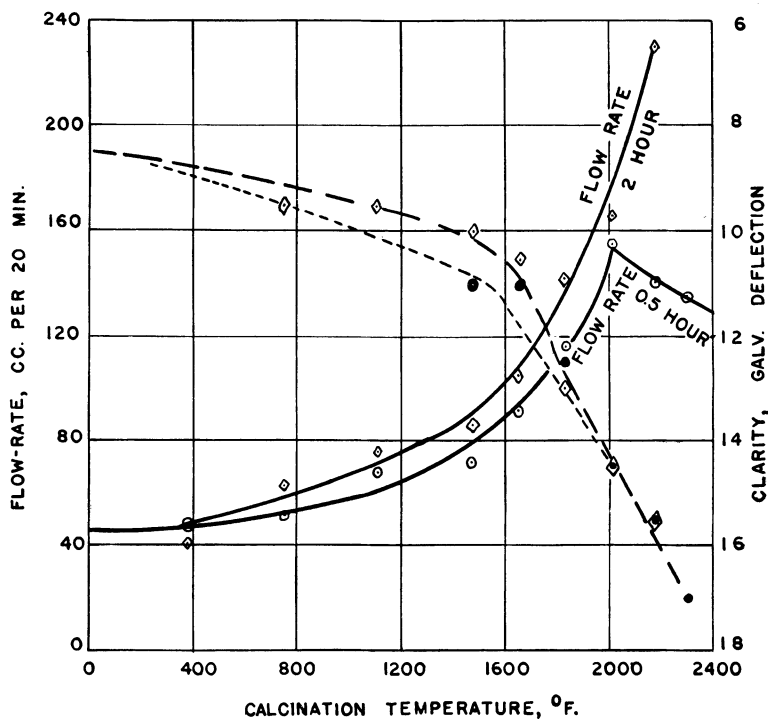


FIGURE 27.—Effect of calcination time and temperature upon flow rate and clarity.

#### EFFECT OF PREMIXING FILTER-AID AND SUGAR SOLUTION

During the investigation, it was found that duplicate flow rates were difficult to obtain with some samples; therefore, in the latter stages of the investigation, tests were made to determine the effect of premixing the filter-aid with the sugar solution before the mixture was added to the filter bomb. Mixing was done with a high-speed kitchen-type mixer.

Duplicate results were easier to obtain by premixing; therefore, it is recommended that for further work the filter-aids be premixed before being added to the filter bomb. The total volume obtained in 20 minutes was about 5 percent less than when not premixed. This loss in volume probably was caused by the formation of a better filter cake at the start of the test.

**SPECIAL CLARITY TESTS**

Although relative clarities, determined with a colorimeter, were used in this investigation, it was thought that a more precise method might be developed; therefore, in the latter part of the investigation, the following methods were tried:

1. Specific resistance (electric).
2. Inorganic chemicals added to the sugar solution, and both the filtrate and the unfiltered solutions analyzed.
3. Measurement of the Tyndall effect by photoelectric cells.

*Specific electric resistance.*—The specific resistance of 5 representative filtered and unfiltered solutions were so similar that it was impossible to use this method. Further investigation of this method might be justified if more precise apparatus were used.

*Chemical additions.*—Calcium oxalate, ferric hydroxide, and barium sulfate precipitates were added in varying concentrations to unfiltered sugar solutions, which were then filtered. The small quantities of chemicals passing through with the filtrate made accurate analysis difficult and duplicate results hard to obtain. This method was also unsatisfactory from the standpoint of the time consumed in making the chemical analyses.

*Tyndallmeter.*—The use of a tyndallmeter for determining the clarity of a sugar solution is described by Cummins and Weymouth,<sup>52</sup> as follows:

The amicroscopic turbidity of filtered liquids is due to the presence of minute particles too small to be visible by transmitted light. Such particles, however, have the capacity of scattering light, which may be observed by examining the liquid at an angle to the path of a strong beam of light passing through the liquid. This is the well-known Tyndall phenomenon. All colloidal systems possess this property of scattering light, and it has been recognized that the intensity of the scattered light depends upon the concentration of the particles in the liquid as well as upon the size of the particles and other properties of the liquid or dispersion medium. A measurement of the intensity of the Tyndall beam, therefore, provides a means of estimating the relative amount of colloidal impurities, in a liquid, or conversely its degree of clarification or "clarity." For well filtered sugar liquors the intensity of the Tyndall beam is relatively low, and a satisfactory apparatus must be capable of refined measurements with light of such low intensity.

The apparatus used in this investigation, sketched in figure 28, consisted of a projector using a 500-watt clear globe and a 3-inch lens, which projected light into a glass container with optically ground parallel sides. Two photoelectric cells parallel to the light beam measured the amount of light scattered by the suspended matter in the cell. Both cells were connected to a galvanometer, and the larger the deflection, the more material in suspension. An ammeter and a variable resistance were included in the light circuit to keep the light intensity constant.

The results obtained with this apparatus were satisfactory, and it is recommended that for further clarity work similar or more sensitive apparatus be used.

**FILTER-CAKE WEIGHT LOSS**

Because the thicknesses of filter cakes varied with different filter-aids, it was thought that there might be a relationship between the thickness of the filter cake and the filter-aid's filtration properties.

<sup>52</sup> Cummins, A. B., and Weymouth, L. E., Filtration of Sugar Solutions. Some Factors Determined by Laboratory Test Procedures: Ind. Eng. Chem., vol. 34, 1942, pp. 392-98.

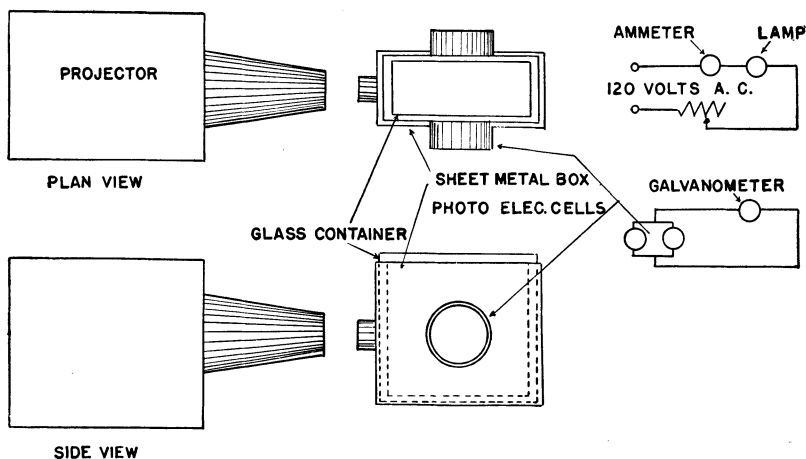


FIGURE 28.—Diagrammatic sketch of tyndallmeter.

Tests were made to determine the air-drying and ignition losses of filter cakes formed by calcined filter-aids having different flow rates. It was assumed that the weight losses were proportional to the cake thickness, because it was impossible to remove all the filter cakes without breaking them.

The data in table 29 show that there is no relationship between weight loss and filtration properties. The differences between the losses appear to be insignificant for correlation purposes.

TABLE 29.—Filtration properties vs. filter cake weight loss

Sample	Calcined with—		Filtrate, cc./20 min.	Clarity	Weight loss, percent		
	Chemical	Percent			Air-dry- ing <sup>1</sup>	Ignition, 1,000° C. <sup>2</sup>	Total <sup>1</sup>
Washington 2.....	Salt.....	2.0	97	Good.....	36.1	63.6	76.7
Do.....	Borax.....	4.0	175	Poor.....	36.4	62.2	76.0
Washington 11.....	.....	.0	82	Excellent.....	35.3	65.1	77.5
Do.....	Salt.....	1.0	115	do.....	26.1	65.7	74.6
Do.....	do.....	2.0	125	Good.....	36.0	65.4	77.8
Do.....	Borax.....	1.0	131	do.....	33.4	68.6	79.1
Do.....	Soda ash.....	4.0	161	do.....	39.0	63.4	78.7
Do.....	Borax.....	4.0	192	do.....	29.8	70.1	79.0
Do.....	Salt.....	4.0	218	Excellent.....	28.8	62.8	73.5
Washington 16.....	.....	.0	118	Fair.....	32.4	57.6	71.3
Do.....	Borax.....	1.0	165	Excellent.....	33.9	60.0	73.8
Do.....	Soda ash.....	4.0	194	Poor.....	25.5	59.7	63.3
Do.....	do.....	1.0	200	do.....	33.7	56.2	71.0
Do.....	Salt.....	4.0	243	do.....	30.7	58.1	71.0
Do.....	do.....	1.0	255	do.....	34.2	55.0	70.4
Washington 17.....	Borax.....	4.0	162	Good.....	36.4	57.2	72.8
Commercial 2.....	.....	.....	174	do.....	35.3	66.5	78.5

<sup>1</sup> Wet basis.<sup>2</sup> Air-dried basis.

## EFFECT OF CALCINATION ON pH

The pH values of 10 minus 100-mesh diatomites were determined both before and after calcining (no chemical added) to 1,750° F. Ten grams of diatomite and 100 cc. of tap water were boiled, cooled, the solids allowed to settle, and then the supernatant liquid filtered through a washed asbestos filter. A LaMotte roulette comparator was used to determine the pH values. The tap water had a pH of 7.2.

The results given in table 30 show that calcination increased the  $pH$  for all but three samples, which remained unchanged. The uncalcined diatomites had an average  $pH$  of 7.4, and the calcined an average of 7.9, or an increase of 0.5  $pH$ . This increase in  $pH$  might have an effect upon filtering some materials.

TABLE 30.—Effect of calcination upon  $pH$  values

Sample	$pH$		
	Uncalcined	Calcined	Calcined minus uncalcined
Washington 1.....	8.3	8.9	0.6
Washington 9.....	6.6	7.7	1.1
Washington 11.....	7.3	7.3	.0
Washington 12.....	7.3	7.5	.2
Washington 14.....	7.6	7.6	.0
Washington 16.....	7.4	7.4	.0
Washington 18.....	6.0	7.3	1.3
Commercial 1.....	8.2	8.8	.6
Commercial 16.....	7.3	7.7	.4
Commercial 17.....	8.1	8.9	.8

NOTE.—Water used had a  $pH$  of 7.2.

#### SIZING AND SIZE DETERMINATION

Although screening crushed diatomite was a rough sizing procedure and showed the effect of particle size and distribution, more refined methods were necessary to show more accurately the effect of size upon filtration properties; therefore, the following sizing and size-determination methods were employed:

1. Air classification.
2. Water classification and specific surface determinations.
3. Microscopic count and measurement.

#### AIR CLASSIFICATION

*Air-analyzer.*—Six size fractions, 0–5, 5–10, 10–15, 15–20, 20–25, and 25–75 microns, were obtained from four commercial filter-aids by means of a Roller air analyzer.<sup>53</sup> These results are given in table 31. This method was not satisfactory because it was too time-consuming to obtain fractions large enough for filtration tests.

The data of table 31 indicate that an increase in the amount of 0–5-micron material decreased the flow rate.

TABLE 31.—Size distribution of four commercial filter-aids

Size range, microns	Commercial sample, No.			
	17	16	1	2
0–5.....	92	83	68	53
5–10.....	6	11	17	22
10–15.....	2	4	9	15
15–20.....	0	1	3	4
20–25.....	0	1	1	2
25–75.....	0	0	2	4
Flow rate, cc./20 min.....	100 18	100 28	100 84	100 174

<sup>53</sup> Roller, R. S., Separation and Size Distribution of Microscopic Particles—An Air Analyzer for Fine Particles: Bureau Mines Tech. Paper 490, 1931, 46 pp.



*Air separator.*—The laboratory-size air separator gives a coarse product and a fine product whose sizes are controlled by fan and whizzer combinations.

To remove the finest material, a small fan is used in combination with the whizzer having the largest number of blades in the upper zone and a whizzer with half the number of blades in the lower zone. If necessary, the lower whizzer may be left out and a spacer used



FIGURE 29.—Laboratory-size air separator. The small fan and the 6- and 16-bladed whizzers are shown at the right. When the separator is used, cloth bags are clamped onto the two spigots—the larger spigot for the fine product, and the smaller for the oversize product.

instead. Different combinations of fans and whizzers give different-size products, but for any given material the combination required to give the desired products must be determined by trial and error. Figure 29, an exterior view of the separator, also shows the extra fan and two extra whizzers.

Commercial filter-aid 16 was air-sized in the separator, and the results are given in table 32, as well as the fan and whizzer combinations used. The data show that no adjustment gave less than 80 per-

cent fines, indicating that the material was too fine for the separator. The fines probably carried some coarse material. Similar results were obtained with other commercially ground diatomites sold as insulation powders or concrete admixtures.

TABLE 32.—Air-sizing of commercial 16 with different fan and whizzer combinations

Separator combination			Recovery, percent	
Fan	Whizzer, blades		Fines	Coarse
	Upper	Lower		
Small.....	16	Spacer.....	94	6
Do.....	12	do.....	94	6
Do.....	6	do.....	93	7
Do.....	24	16.....	80	20
Do.....	24	12.....	83	17
Do.....	24	6.....	83	17
Do.....	16	12.....	82	18
Do.....	16	6.....	85	15
Do.....	12	6.....	84	16
Large.....	24	Spacer.....	92	8
Do.....	16	do.....	93	7
Do.....	24	16.....	86	14

Because the commercially prepared diatomites were too finely ground for the separator, some Kittitas lump diatomite was dried, ground, screened through a 6-mesh screen, and then the minus 6-mesh material air-sized. Filtration flow rates were determined for the 6-mesh material and for five of the air-sized products. The results are given in table 33.

The beneficial effect of air-sizing is shown by the data in table 33. An increase in flow rate over the original minus 6-mesh material was obtained by air-sizing. A similarly treated Nevada diatomite improved 200 percent. This increase in flow rate was due to a change in particle size and particle-size distribution.

TABLE 33.—Air-sizing of laboratory-ground Kittitas diatomite. Six filtration flow rates also are given

Test No.	Feed	Separator combination		
		Fan	Whizzer, blades	
			Upper	Lower
1.....	Unsize material (—6 mesh).....	Small.....	24	12
2.....	Coarse from 1.....	do.....	24	12
3.....	Coarse from 2.....	do.....	24	12
4.....	Coarse from 3.....	do.....	24	12
5.....	Coarse from 4.....	do.....	24	12
6.....	Coarse from 5.....	Large.....	24	12
7.....	Coarse from 6.....	do.....	24	12
8.....	Fines from 1.....	Small.....	24	12
9.....	Coarse from 8.....	do.....	24	12
Sized product			Flow rate, cc./20 min.	Improvement, percent
Unsize (minus 6-mesh).....			95	0.0
Coarse from test 8.....			112	17.9
Coarse from test 9.....			115	21.1
Fine from test 2.....			123	29.5
Fine from test 5.....			128	34.7
Fine from test 7.....			139	45.3

## WATER CLASSIFICATION AND SPECIFIC SURFACE DETERMINATIONS

A sedimentation method described by Hinckley<sup>54</sup> was employed to determine the particle-size distribution of four commercial samples and one laboratory-prepared sample. Table 34 gives the results of these separations. Specific surface determinations and flow rates also are given. The relative specific surfaces were determined with an air-permeability apparatus designed by the National Bureau of Standards. The sample used was smaller in weight than that for which the apparatus was calibrated; therefore, the results given in tables 34 and 35 are estimates.

TABLE 34.—*Size and filtration properties of five filter-aids*

Sample	Cc.	Min.	Cc./min.	Particle-size distribution, percent less than—				Relative specific surface, cm. <sup>2</sup> per gram
				40 $\mu$	20 $\mu$	10 $\mu$	5 $\mu$	
Commercial 7 .....	420	4	105.0	88	52	4	6	5,280
Commercial 4 .....	385	10	38.5	88	67	39	6	19,070
Commercial 2 .....	174	20	8.7	94	84	68	36	26,200
Commercial 1 .....	84	20	4.2	99	98	78	31	27,150
Washington 11 .....	63	20	3.1	99	97	83	44	

TABLE 35.—*Size and filtration properties of laboratory, water-classified, Commercial 4 filter aid*

Calculated average size, microns	Relative specific surface, cm. <sup>2</sup> per gram	Flow rate			Clarity galvonometer, deflection <sup>1</sup>
		Cc.	Min.	Cc./min.	
40 .....	6,840	370	5.75	6.4	24.0
20 .....	8,760	392	8.00	4.9	23.5
10 .....	11,800	300	9.00	3.3	19.5
5 .....	17,200	117	9.00	1.3	14.0

<sup>1</sup> Large deflection caused by more suspended material.

A microscopic examination of the fractions showed that the separations were not as good as desired; therefore, 2 pounds of Commercial 4 was agitated with 50 pounds of water in a large vat and then allowed to settle. Four samples were siphoned off at arbitrarily selected heights at calculated time intervals to give size fractions with average diameters of 40, 20, 10, and 5 microns, as calculated by Stoke's Law. Each fraction was reclassified in water until large differences in particle size between the different fractions were obtained. The relative specific surface and flow rate were determined for each size fraction, and the data are given in table 35. Figure 30 shows the relationship between relative specific surface, flow rate, and clarity. For this sample, the relationship between flow rate and clarity may be shown by the following equation:

$$\text{Flow rate, cc./min.} = 0.5 (\text{clarity} - 12.5)$$

The data show that an increase in particle size increases the flow rate but decreases the clarity.

<sup>54</sup> Hinckley, W. O., Determination of Particle Size Distribution: Ind. and Eng. Chem., anal. ed., vol. 14, 1942, pp. 10-13.

## MICROSCOPIC COUNT

The average particle sizes and size distributions of 17 filter-aids were determined by microscopic count and measurement, as follows:

The filter-aids were dispersed on microslides with double-distilled turpentine, which was then evaporated by gentle heating on a hot plate. A drop of liquid Hyrax, a high-index mounting medium, was placed on the slide and then covered with a cover glass. The slide was heated to harden the Hyrax, and then cooled. Two snap clothespins held the cover glass while the slide was being heated, which helped prevent air bubbles.

The slide was placed upon the mechanical stage of a microscope in a photomicrographic apparatus, and the diatom images were measured

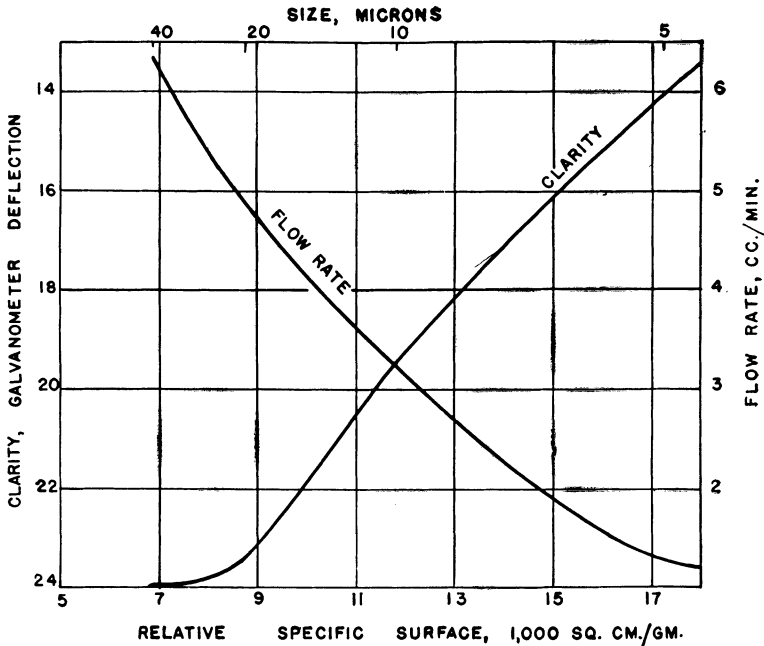


FIGURE 30.—Relationship between relative specific surface, flow rate, and clarity for Commercial 4 filter-aid.

and counted on the ground glass at magnifications of  $\times 1,000$  and  $\times 2,000$ . Seven hundred to a thousand particles were measured at  $\times 2,000$  to the closest 0.5 mm., which was equivalent to 0.25 microns. Because a few large particles would have a large effect upon the length and surface means, a correction or expectancy factor was determined by counting 3,500 to 5,000 particles at  $\times 1,000$  and measuring and counting all particles 13 microns or larger at this magnification. Using the number of particles under 13 microns counted and measured at  $\times 2,000$  and those counted at  $\times 1,000$ , an expectancy factor was calculated, that is

$$\frac{\text{Number at } \times 2000 \text{ under 13 microns}}{\text{Number at } \times 1000 \text{ under 13 microns}} = F, \text{ expectancy.}$$

The number of particles larger than 13 microns counted at  $\times 1,000$ , multiplied by the expectancy factor,  $F$ , gave the number of these particles that should have been expected at  $\times 2,000$  if more particles had been counted. Number, length, and surface means were calculated from these data, and curves were drawn to show the size distribution for number, surface, and weight. Coefficient of uniformities, which

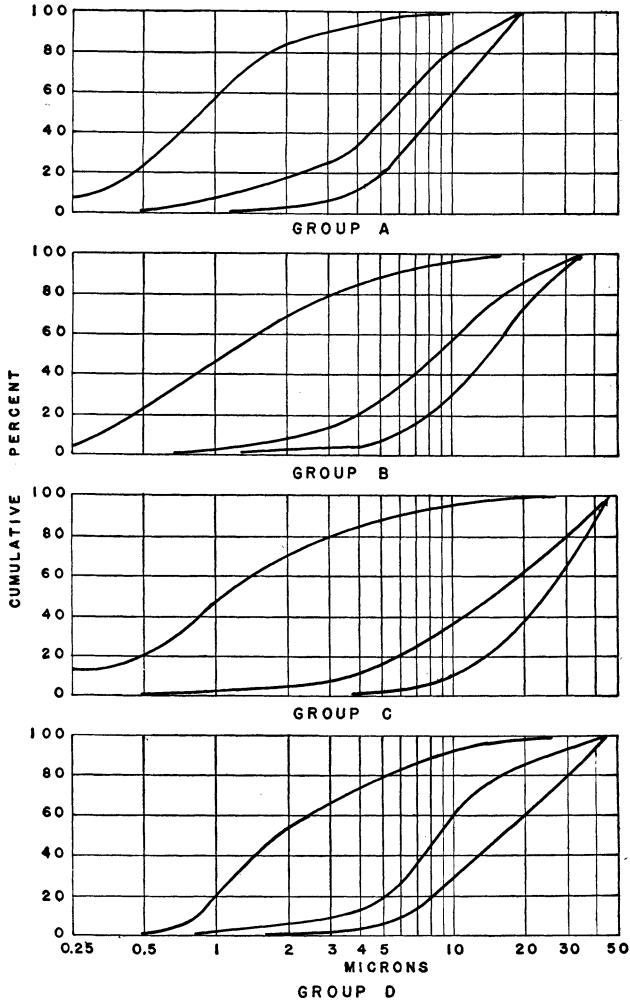


FIGURE 31.—Representative particle-size distribution curves. Upper curve shows distribution by number; the middle curve, by surface; and the bottom curve, by weight.

varies from zero to infinity, also was calculated. If all the particles were the same size,  $U$  would be infinity; if all were different sizes,  $U$  would be zero. The average probable error for all the calculations was  $\pm 3$  percent, based upon the number mean.

Figure 31 shows the four general types of size-distribution curves based upon the shapes of the percentage curves by number, surface,

and weight. The relationship between average particle size, size distribution, filtration properties, and flow tests (see later description of this test) for these 17 samples are given in table 36.

The data given for group averages in table 36 indicate that flow rate increases with an increase in average particle size as calculated for either the number or the surface mean. Clarity increases with a decrease in uniformity, as shown by the uniformity coefficient "U." No apparent relationship existed between the flow-test values and particle size, although the values for group D, with a larger size and larger flow-test values, were greater than for group A, with smaller values.

*Flow test.*—Flow tests were made with 12 filter-aids to determine whether any relationship existed between the calculated surface mean and the amount of water required to make 5 grams of material start to flow in an inclined test tube. As shown in table 36, there was no definite relationship, although an increase in the amount of water required generally indicated an increase in the specific surface.

TABLE 36.—*Relationship between average particle size, size distribution, filtration properties, and flow tests*<sup>1</sup>

GROUP A						
Sample <sup>2</sup>	Flow test, cc.	Flow rate, <sup>3</sup> cc./20 min.	Average particle size, microns			Coefficient of uniformity
			Number	Length	Surface	
Oregon 6aA.....	7.7	32P.....	1.179	3.571	7.742	0.105
Washington 16A.....	8.2	40P.....	1.391	2.930	5.887	.120
Washington 12D.....	(9)	59P.....	1.314	3.566	6.363	.099
Washington 17A.....	5.3	62P.....	1.171	2.882	6.805	.120
Washington 12A.....	11.7	115P.....	1.939	3.981	7.458	.089
Average.....	8.2	62P.....	1.399	3.386	6.851	.107
GROUP B						
Washington 2A.....	9.8	39P.....	1.735	5.097	11.348	0.074
Washington 18D.....		71E.....	1.657	4.316	9.024	.084
Washington 18E.....		79G.....	2.995	8.355	14.226	.044
Oregon 6aC.....		137G.....	3.246	7.269	11.843	.051
Washington 2C.....		149E.....	1.708	4.218	8.663	.086
Average.....		93G.....	2.268	5.851	11.021	.068
GROUP C						
Washington 17C.....	5.6	141G.....	2.215	5.832	15.661	0.062
Washington 16C.....		242F.....	2.145	6.328	14.431	.059
Commercial 7F.....	13.1	243P <sup>5</sup> .....	2.644	10.590	21.096	.113
Average.....	9.4	>209F.....	2.335	7.583	17.063	.078
GROUP D						
Washington 11B.....	11.5	82E.....	2.292	4.504	8.067	0.076
Commercial 1F.....	12.7	83G.....	2.892	6.704	14.358	.053
Commercial 2F.....	11.6	178G.....	3.402	6.772	12.403	.052
Washington 4C.....	11.2	218E.....	4.843	8.531	15.977	.038
Average.....	11.8	120G-E.....	3.357	6.628	12.701	.055

<sup>1</sup> Groups represent type particle-size distribution curves, as shown in figure 81.

<sup>2</sup> Capital letters after filter-aids refer to treatment, as follows:

A = Uncalcined minus 250- plus 325-mesh.

B = Minus 100-mesh diatomite calcined to 1,750° F. and then sized to minus 250- plus 325-mesh.

C = Minus 100-mesh diatomite calcined with 4 percent NaCl to 1,750° F. and then sized to minus 250- plus 325-mesh.

D = Minus 100-mesh diatomite sized in Roller air-analyzer—fines.

E = Minus 100-mesh diatomite sized in Roller air-analyzer—coarse.

F = As-received.

<sup>3</sup> Letters after flow rates indicate relative clarities. E=excellent, G=good, F=fair, and P=poor.

<sup>4</sup> Not determined.

<sup>5</sup> 3-minute period.

## GENERAL SUMMARY OF DATA AND CONCLUSIONS

A compilation showing the effect of sizing and calcination to 1,750° F., with and without 4 percent NaCl, on the flow rates of Pacific Northwest diatomite samples is given in table 37. Chemical analyses, percentage of clay, and pyrometric cone equivalents are also given in the same table. Table 38 classifies the samples according to their flow rates. Table 39 shows the grouping of 30 filter-aids according to their flow rates and methods of preparation. These 30 samples were the only ones from which filter-aids were prepared by all four methods, that is, (1) screening through 100-mesh; (2) sizing to minus 250- plus 325-mesh; (3) calcining minus 100-mesh diatomite to 1,750° F., and then sizing the calcined product to minus 250- plus 325-mesh; and (4) calcining minus 100-mesh diatomite with 4 percent NaCl to 1,750° F. and then sizing the calcined product to minus 250- plus 325-mesh.

## EFFECT OF SIZING AND CALCINATION UPON FLOW RATES

The data of table 38, an average increase in flow rate of 20 percent, were obtained when minus 100-mesh material was sized to minus 250- plus 325-mesh. Calcining 100-mesh material to 1,750 F. and then sizing the calcined product to minus 250- plus 325-mesh increased the flow rate 145 percent, and calcining with 4 percent NaCl increased the flow rate 340 percent. For individual samples, the increases might be more or less than the averages, but the data show that sizing and calcination, with or without a chemical addition, increase the flow rates.

TABLE 37.—Effect of sizing and calcining to 1,750° F. with and without 4 percent NaCl, upon the flow rates and clarities of Pacific Northwest diatomites

Sample	Flow rate, <sup>1</sup> cc./20 min.				P.C.E., cone	Percent				
	Uncalcined, mesh		Calcined <sup>2</sup> NaCl, percent			SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Ignition loss	Theoretical clay
	-100	-250 +325	0	4						
Washington:										
1.....	22P	(3)			29	89.5	1.9	1.3	4.9	4.8
2.....	49	39P	48E	149E	29	89.1	2.0	2.1	4.5	5.1
3.....	33P	52F			26-27					
4a.....	43E	59			30	87.6	1.2	1.4	5.3	3.0
4b.....	46G	59			31					
4c.....	44G	56	57F	175F						
5.....	45F	48G	51P	74F						
6a.....	44G									
6b.....	64F	83			23	82.2	6.7	5.2	4.2	17.0
6c.....	77G	86G	144P	250P	27	84.8	4.1	6.9	4.1	10.4
7a.....	35G									
7b.....	40G									
7c.....	80G	73F	218F	240P						
8a.....	31F	82								
8b.....	48G				15	77.7	9.4	6.2	5.5	23.8
8c.....	84G	80G	193F	251P	28	69.2	2.1	1.8	12.4	5.3
9.....		78P	93E			82.6	3.9	1.1	11.5	9.9
10.....	59				30	91.1	1.3	2.0	5.1	3.3
11.....	63	125P	82E	218E	28	89.6	2.9	2.1	4.2	7.5
12.....	52	115P	70E		29-30	90.5	2.0	2.0	3.6	5.1
13.....					29	88.3	3.9	2.9	4.8	9.9
14.....		39F	49G		28-	89.0	3.0	3.9	4.4	7.6
15.....					29+	88.7	2.3	3.1	5.4	5.8
16.....		40P	118F	242F	14-15	67.3	13.8	2.4	10.9	35.0
17.....		62P	48F	141G	11	73.5	15.1	3.9	4.3	38.3

See footnotes at end of table.

TABLE 37.—Effect of sizing and calcining to 1,750° F. with and without 4 percent NaCl, upon the flow rates and clarities of Pacific Northwest diatomites—Con.

Sample	Flow rate, 1 cc./20 min.				P.C.E., cone	Percent				
	Uncalcined, mesh		Calcined <sup>2</sup> NaCl, percent			SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Ignition loss	Theoretical clay
	-100	-250 +325	0	4						
Washington—Con.										
18.....		112P	122G		28	85.9	5.6	.8	5.9	14.2
19.....		43P	202P		4—	59.5	15.1	4.6	9.9	38.3
Oregon:										
1.....	38G	73F	253P		15					
2a.....	42P		82P	138G						
2b.....	73G	76G	116F	232P						
3.....	51F	67P	241F	347P						
4.....	71F	101P	105F	385P						
5.....										
6a.....	43E	59G	80P	152F						
6b.....		32P	82P	137G	16	83.5	8.3	2.9	3.6	21.0
6c.....	35G		76F	167F						
6d.....	42G	39F	76P	156P						
6e.....	50E	46F	96F	196P						
6f.....										
6g.....	43E	53F	98F	193F						
6h.....	15G	17F	85F	117P						
7a.....	66G	66G	95F	190P						
7b.....	59G									
7c.....	42G									
8a.....	35G									
8b.....	26P	44			7-8					
8c.....	31G	31G	104F	189P						
9a.....	15F									
9b.....	18F	65			12					
9c.....	18F	60			13-14					
9d.....	20G	55	110G	163F	14					
10a.....	23F									
10b.....	24F									
10c.....	27P	31	70	119						
11a.....	25P	45G	95F	172P	8-9					
11b.....	25P	46G			8					
12.....	41F	27G	67F	92P						
13.....	44G	37F								
14.....	29E	30G	165F	322P						
15.....	21G	35G	136F	158P	7-8					
16a.....	54F									
16b.....	73F	75G	191F	210P						
16c.....	59F									
California:										
1.....	94F	109F	106F	275P						
2a.....	23E									
2b.....	31G	37G	52F							
3a.....	78E									
3b.....	83E	84G	65F	147P						
4.....	36E	47G	63F	141F						
5.....	43G	67F	89F	270P	32					
Idaho:										
1a.....	47F									
1b.....	47P									
1c.....	121P	51F	78F	197P						
2.....	54P	39F	83P	155P						
3.....	45G	52F	144G							

<sup>1</sup> Letters after flow rates indicate relative clarities: E=excellent, G=good, F=fair, and P=poor.

<sup>2</sup> Minus 100-mesh diatomite calcined to 1,750° F. and the product then sized to minus 250- plus 325-mesh.

<sup>3</sup> Spaces marked ..... show no data obtained.



TABLE 38.—Classification of Pacific Northwest diatomite filter-aids<sup>1</sup> according to their flow rates (cc. per 20 minutes), with 18 commercial filter-aids given for comparison

<b>VERY FAST; MORE THAN 300 CC.</b>			
Oregon 3D	Commercial 3	Commercial 6	Commercial 12
Oregon 4D	Commercial 4	Commercial 7	Commercial 18
Oregon 14D	Commercial 5	Commercial 11	
<b>FAST; 200 TO 299 CC., INCLUSIVE</b>			
Washington 6cD	Washington 11D	Oregon 1C	California 1D
Washington 7cD	Washington 16D	Oregon 2bD	California 5D
Washington 7cD	Washington 17D	Oregon 3C	Commercial 10
Washington 8cD	Washington 19C	Oregon 16bD	
<b>MEDIUM-FAST; 140 TO 199 CC., INCLUSIVE</b>			
Washington 2D	Oregon 6cD	Oregon 11aD	California 4D
Washington 4cD	Oregon 6dD	Oregon 14C	Idaho 1cD
Washington 6cC	Oregon 6eD	Oregon 15C	Idaho 2D
Washington 8cC	Oregon 7aD	Oregon 15D	Idaho 3C
Oregon 5D	Oregon 8cD	Oregon 16bC	Commercial 2
Oregon 6bD	Oregon 9dD	California 3bD	
<b>MEDIUM; 80 TO 139 CC., INCLUSIVE</b>			
Washington 6bB	Washington 18B	Oregon 6eC	California 1C
Washington 6cB	Washington 18C	Oregon 6fC	California 3bA
Washington 7cA	Oregon 2aC	Oregon 6fD	California 3bB
Washington 8aB	Oregon 2aD	Oregon 7aC	California 5C
Washington 8cA	Oregon 2bC	Oregon 8cC	Idaho 1cA
Washington 8cB	Oregon 4B	Oregon 9aC	Idaho 2C
Washington 9C	Oregon 4C	Oregon 10cD	Commercial 1
Washington 11B	Oregon 5C	Oregon 11aC	Commercial 1B
Washington 11C	Oregon 6aC	Oregon 12D	Commercial 1C
Washington 12B	Oregon 6aD	California 1A	Commercial 9
Washington 16C	Oregon 6dC	California 1B	Commercial 16C
<b>SLOW; 40 TO 79 CC., INCLUSIVE</b>			
Washington 2A	Washington 9B	Oregon 6cC	Oregon 16aA
Washington 2C	Washington 10A	Oregon 6dA	Oregon 16aB
Washington 3B	Washington 11A	Oregon 6dB	Oregon 16bB
Washington 4aA	Washington 12A	Oregon 6eA	Oregon 16cA
Washington 4aB	Washington 12C	Oregon 6eB	California 2bC
Washington 4bA	Washington 14C	Oregon 7aA	California 3aA
Washington 4bB	Washington 16B	Oregon 7aB	California 3bC
Washington 4cA	Washington 17B	Oregon 7bA	California 4B
Washington 4cB	Washington 17C	Oregon 7cA	California 4C
Washington 4cC	Washington 19B	Oregon 8aA	California 5A
Washington 5A	Oregon 1B	Oregon 8bB	California 5B
Washington 5B	Oregon 2aA	Oregon 9bB	Idaho 1aA
Washington 5C	Oregon 2bA	Oregon 9cB	Idaho 1bA
Washington 5D	Oregon 3A	Oregon 9dB	Idaho 1cB
Washington 6aA	Oregon 3B	Oregon 10cC	Idaho 1cC
Washington 6bA	Oregon 4A	Oregon 11aB	Idaho 2A
Washington 6cA	Oregon 5A	Oregon 11bB	Idaho 3A
Washington 7bA	Oregon 5B	Oregon 12A	Idaho 3B
Washington 7cB	Oregon 6bC	Oregon 12C	Commercial 16B
Washington 8bA	Oregon 6cA	Oregon 13A	
<b>VERY SLOW; LESS THAN 40 CC.</b>			
Washington 1A	Oregon 6fA	Oregon 10bA	Oregon 15B
Washington 2B	Oregon 6fB	Oregon 10cA	California 2aA
Washington 3A	Oregon 8bA	Oregon 10cB	California 2bA
Washington 7aA	Oregon 8cA	Oregon 11aA	California 2bB
Washington 8aA	Oregon 8cB	Oregon 11bA	California 4A
Washington 14B	Oregon 9aA	Oregon 12B	Idaho 2B
Oregon 1A	Oregon 9bA	Oregon 13B	Commercial 16
Oregon 6aB	Oregon 9cA	Oregon 14A	Commercial 17
Oregon 6bA	Oregon 9dA	Oregon 14B	
Oregon 6cB	Oregon 10aA	Oregon 15A	

<sup>1</sup> Capital letters after filter-aids refer to treatment, as follows:

A = Uncalcined, minus 100-mesh.

B = Uncalcined, minus 250- plus 325-mesh.

C = Minus 100-mesh diatomite calcined to 1,750° F. and then sized to minus 250- plus 325-mesh.

D = Minus 100-mesh diatomite calcined to 1,750° F. with 4 percent NaCl and then sized to minus 250- plus 325-mesh.

Commercial filter-aids tested as received, except where noted.

TABLE 39.—*Relationship between method of filter-aid preparation and flow rate*

Preparation <sup>1</sup>	NUMBER OF SAMPLES					
	Flow rate, cc. per 20 min.					
	-40	40-79	80-139	140-199	200-299	+300
A.....	9	16	5	0	0	0
B.....	9	15	6	0	0	0
C.....	0	9	15	4	2	0
D.....	0	1	3	15	8	3

Preparation <sup>1</sup>	PERCENT					
	-40	40-79	80-139	140-199	200-299	+300
A.....	7.5	13.3	4.2	0.0	0.0	0.0
B.....	7.5	12.5	5.0	.0	.0	.0
C.....	.0	7.5	12.5	3.3	1.7	.0
D.....	.0	.8	2.5	12.5	6.7	2.5

<sup>1</sup> A = Uncalcined, minus 100-mesh.

B = Uncalcined, minus 250- plus 325-mesh.

C = Minus 100-mesh diatomite calcined to 1,750° F. and then sized to minus 250- plus 325-mesh.

D = Minus 100-mesh diatomite calcined with 4 percent NaCl to 1,750° F. and then sized to minus 250- plus 325-mesh.

The data of table 39 for 30 samples show that none of the uncalcined samples had flow rates greater than 139 cc. in 20 minutes and none of the calcined samples had flow rates less than 40 cc. in 20 minutes. The data also show that calcining increased the flow rates and that calcination with 4 percent NaCl further increased the flow rates.

The data of tables 25, 37, and 38 show that none of the Pacific Northwest diatomites prepared in the laboratory and tested as filter-aids had flow rates as fast as the fastest of the commercial filter-aids tested, but some of the laboratory-prepared filter-aids would be satisfactory for some uses. As previously stated, other methods of preparation probably would give different results; therefore, each diatomite to be used as a filter-aid should be tested to find the best size and the best calcination conditions to give a product having the required filter-aid characteristics.

#### RELATIONSHIP BETWEEN CHEMICAL ANALYSIS, PYROMETRIC CONE EQUIVALENT, AND FLOW RATE

No relationship was found between chemical analysis and flow rate, although the analysis did indicate which commercial filter-aids had been calcined. The calcined materials had lower ignition losses than the uncalcined. The analysis also indicated the amount of impurities.

An increase in pyrometric cone equivalent usually indicated an increase in the SiO<sub>2</sub> content.

#### EFFECT OF CALCINATION WITH AND WITHOUT CHEMICAL ADDITIONS ON DIATOMITE COLOR

The data of table 40 show that as a general rule diatomites are grayed upon calcination and that with increasing amounts of chemical additions the colors further darken and tend toward red-buffs and dark pinks. Carbonaceous matter burns out and lightens the color (Washington 16). As shown by luminosity factors, borax reddened diatomite the least on calcination, and salt the most. A decrease in luminosity in these tests was due primarily to an increase in red. Figure 32, typical color-analysis curves, shows graying of the sample after calcination and an increase in red.

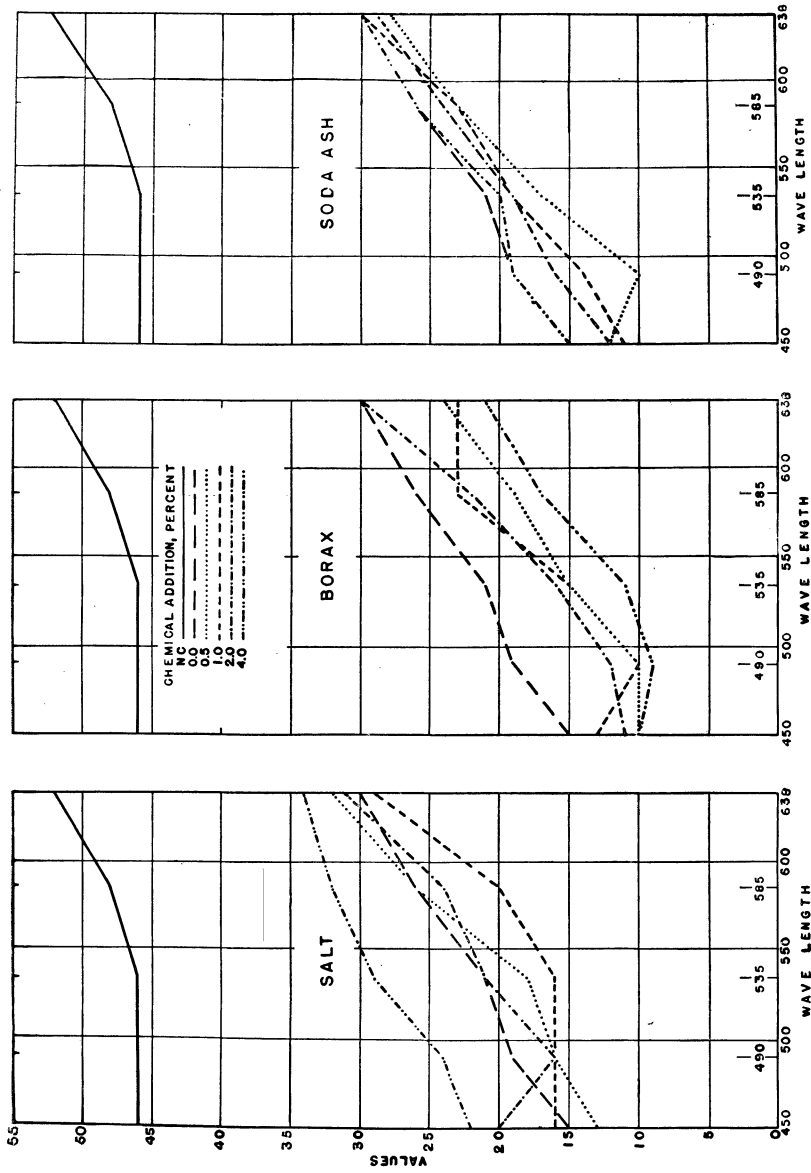


FIGURE 32.—Change in diatomite color upon calcination, both with and without chemical additions, Washington 11 sample.

*Iron-valence change upon calcination.*—Six minus 100-mesh diatomites were calcined to 1,750° F. in an oxidizing atmosphere to determine the effect of iron valence. The results given in table 41 show that, with one exception, all of the ferrous iron was changed to ferric upon calcination. Color analysis showed that the uncalcined samples containing the most ferrous iron reddened and grayed the most upon calcination.

TABLE 40.—*Effect of calcination, with and without chemical additions, upon diatomite color*

GRAY					
Washington sample No.	Treatment	Chemical addition <sup>1</sup>	Luminosity	Maerz and Paul No.	Common color name
1	Uncalcined	0	41.5	12 B 2	Flax.
2	do	0	47.1	15 E 6	Mouse-gray.
9	do	0	20.1	16 A 12	Date.
11	do	0	43.7	12 C 2	Flax.
12	do	0	43.6	12 D 2	Crash.
14	do	0	38.0	13 B 2	Elmwood.
17	do	0	22.2	13 C 3	Lido.
18	do	0	25.4	16 E 12	Bronze luster.
19	do	0	24.3	14 D 5	Sponge.
TAN					
1	Calcined	0	29.5	11 G 7	Golden wheat.
2	do	0	22.1	12 H 8	Topaz.
2	do	0.5A	18.6	12 K 9	Yucatan.
2	do	1.0A	20.3	14 H 9	Harvest.
2	do	2.0A	20.2	12 L 9	Yucatan.
2	do	4.0A	21.4	12 J 8	Topaz.
2	do	0.5B	16.2	13 J 9	Hazel.
2	do	1.0B	16.3	12 K 9	Yucatan.
2	do	2.0B	18.2	12 I 9	Topaz.
2	do	4.0B	29.3	12 E 5	India buff.
2	do	0.5S	20.2	13 H 11	Pekinese.
2	do	1.0S	18.5	13 H 12	Windsor tan.
2	do	2.0S	22.8	13 C 9	Wild honey.
2	do	4.0S	12.9	5 A 12	Fouille morte.
9	do	0	21.0	11 C 8	Muskmelon.
11	do	0	21.6	12 H 9	Harvest.
11	do	0.5A	15.9	12 H 10	Amberglow.
11	do	1.0A	17.9	12 I 9	Topaz.
11	do	2.0A	16.3	12 K 9	Yucatan.
11	do	4.0A	18.2	12 J 10	Burma.
11	do	0.5B	21.4	12 K 9	Yucatan.
11	do	1.0B	21.9	12 H 10	Amberglow.
11	do	4.0B	13.3	4 D 12	Terra cotta.
11	do	0.5S	19.1	13 C 12	Spice.
11	do	1.0S	19.4	13 C 12	Do.
11	do	2.0S	18.0	4 F 12	Mosque.
11	do	4.0S	17.1	12 D 10	Caramel.
12	do	0	21.3	12 H 9	Harvest.
16	do	0	22.8	13 F 8	Toast.
16	do	0.5A	16.5	13 F 8	Do.
16	do	1.0A	14.3	13 D 9	Tuscan tan.
16	do	2.0A	14.4	13 F 10	Pekinese.
16	do	4.0A	12.8	14 D 12	Hispano.
16	do	0.5B	22.8	13 G 8	Toast.
16	do	1.0B	18.0	13 E 8	Do.
16	do	0.5S	23.4	13 B 10	Sonora.
16	do	1.0S	14.3	13 B 12	Spice.
16	do	2.0S	16.8	6 A 11	Vassar tan.
18	do	0	24.0	12 F 6	Walnut taffy.
19	do	0	18.9	12 E 11	Caramel.

See footnote at end of table.

TABLE 40.—*Effect of calcination, with and without chemical additions, upon diatomite color—Continued*

BROWN

Washington sample No.	Treatment	Chemical addition <sup>1</sup>	Luminosity	Maerz and Paul No.	Common color name
11.....	Calcined.....	2.0B	15.6	13 L 11	Peruvian brown.
14.....	do.....	0	14.7	13 L 11	Do.
16.....	do.....	2.0B	16.6	14 D 10	Mustard brown.
16.....	do.....	4.0B	21.0	14 C 10	Coconut brown.
16.....	do.....	4.0S	19.0	6 D 12	Ginger brown.
17.....	do.....	0	15.1	14 F 12	Gold brown.
17.....	do.....	0.5A	12.4	14 D 12	Hispano.
17.....	do.....	1.0A	13.6	14 C 12	Tortoise.
17.....	do.....	2.0A	12.3	14 C 12	Do.
17.....	do.....	4.0A	12.1	6 A 12	Rust.
17.....	do.....	0.5B	14.5	6 A 12	Do.
17.....	do.....	1.0B	17.6	6 A 12	Do.
17.....	do.....	2.0B	10.8	6 B 12	Gypsy.
17.....	do.....	4.0B	12.7	6 A 12	Rust.
17.....	do.....	0.5S	14.6	6 A 12	Do.
17.....	do.....	1.0S	13.4	6 C 12	Copper brown.
17.....	do.....	2.0S	13.2	6 G 12	Terragona.
17.....	do.....	4.0S	14.8	6 F 12	Persimmon.
17.....	do.....	2.0F	16.2	14 D 12	Hispano.
17.....	do.....	0.1Z	15.4	14 F 12	Gold brown.
17.....	do.....	0.5Z	14.5	14 F 12	Do.
17.....	do.....	2.0Z	13.5	14 C 12	Tortoise.

DARK BROWN-BLACK

16.....	Uncalcined.....	0	13.4	16 A 2	Smoke brown.
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<sup>1</sup> Numbers show percent added. Letters, chemical as follows: A, soda ash; B, borax; F, fluorite; S, salt; and Z, zinc hydrosulfite.

TABLE 41.—*Effect of calcination in an oxidizing atmosphere upon iron valence*

Sample	Iron, percent						Decrease in FeO, percent
	Before calcination			After calcination			
	FeO as Fe <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Total Fe <sub>2</sub> O <sub>3</sub>	FeO as Fe <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Total Fe <sub>2</sub> O <sub>3</sub>	
Commercial 1.....	0.2	2.6	2.8	0.0	2.8	2.8	100
Washington 2.....	.1	2.0	2.1	.0	2.1	2.1	100
Washington 11.....	.0	2.1	2.1	.0	2.1	2.1	-----
Washington 16.....	1.9	.5	2.4	.2	2.2	2.4	90
Washington 17.....	1.0	2.9	3.9	.1	3.8	3.9	90
Washington 19.....	.4	4.2	4.6	.0	4.6	4.6	100

## APPENDIX

### MICROSCOPIC EXAMINATION OF FILTER-AIDS

The filter aids made in the laboratory from crude Pacific Northwest diatomites were examined microscopically after dispersing on microscopic slides with triple-distilled turpentine and mounting with "Hyrax" ( $nd=1.65\pm$ ). Twelve commercial filter-aids were similarly treated. In all, about 200 slides were examined to see whether any microscopically visible changes occurred upon calcination and whether any apparent relationship existed between diatom species and flow-rate characteristics.

As discussed under Microscopic Count (p. 62), the faster flow-rate filter-aids consisted of larger diatoms and fragments with fewer fine sizes; but, as shown in the following selected photomicrographs (figs. 33 through 43), the shape (species) of diatoms apparently had little effect on flow rate. The photomicrographs do not necessarily show the true particle-size distribution, because these illustrations were chosen to show the principal shapes of diatoms present.

Figures 33 and 34, both of filter-aids having slow flow rates (less than 40 cc. in 20 minutes), show two distinctly different types of diatoms. Again, the diatoms found in figure 34 (Oregon 15) are similar in shape to those in Washington 11 (fig. 35), but this one gave a slightly faster rate of filtration, being characterized as slow (40 to 79 cc. in 20 minutes) rather than very slow.

When diatomite Washington 11 is calcined with 4 percent NaCl and the product sized from 250- to 325-mesh, the flow rate is increased to fast (200 to 299 cc. in 20 minutes), but no visible change could be noticed in the diatom structure (fig. 39). Similarly, Washington 17 (fig. 36) before calcination had a slow flow rate, but after calcination with 4 percent NaCl (fig. 40) the rate was fast.

Figures 41, 42, and 43 represent commercial filter-aids having very fast flow rates (greater than 300 cc. in 20 minutes); nevertheless, each is composed of a different dominant species. Figures 42 and 43 are photomicrographs taken with a magnification of only  $\times 500$  instead of the  $\times 1,000$  used for previous illustrations; therefore, the diatoms that appear to be about the same size as those in the previous figures are actually about twice as large. Commercial 7 filter-aid, the fastest of all tested, although not shown by an illustration, is similar to Commercial 6 (fig. 43), which was the second fastest tested.

Only three laboratory-prepared filter-aids, Oregon 3, 4, and 14, had very fast flow rates. These were prepared by calcining minus 100-mesh diatomite with 4 percent NaCl and sizing the product from 250- to 325-mesh. Microscopically, Oregon 3 and 4 looked like Commercial 6 (fig. 43), but Oregon 14 contained none of the "banana-shaped" diatoms found in the other two. These three samples before calcination and Commercial samples 6 and 7 showed evidence of secondary silica both by rough edges and by partial disappearance of the diatom structure. This is shown by the round diatoms in figure 43.

Calcination with or without NaCl caused no apparent change in 99 percent of 200 large diatoms examined. The remaining 1 percent showed a slight change in the appearance of the knobs found on the disk-like shapes. Figure 44 shows the presence of small knobs, some less than 0.1 micron in diameter, between the large knobs. These small knobs are characteristic of all the disk-like diatoms examined.

All types of diatoms were found in all flow-rate groups, but only those calcined with a chemical were in the very fast group. The uncalcined Oregon diatoms (3, 4, and 14) were in the very slow and slow groups before calcining with 4 percent NaCl, but in the very fast group afterwards. On the basis of this microscopic examination, diatom species or shapes are not as important a factor controlling filter-aid characteristics as are particle size, particle-size distribution, and processing treatment.

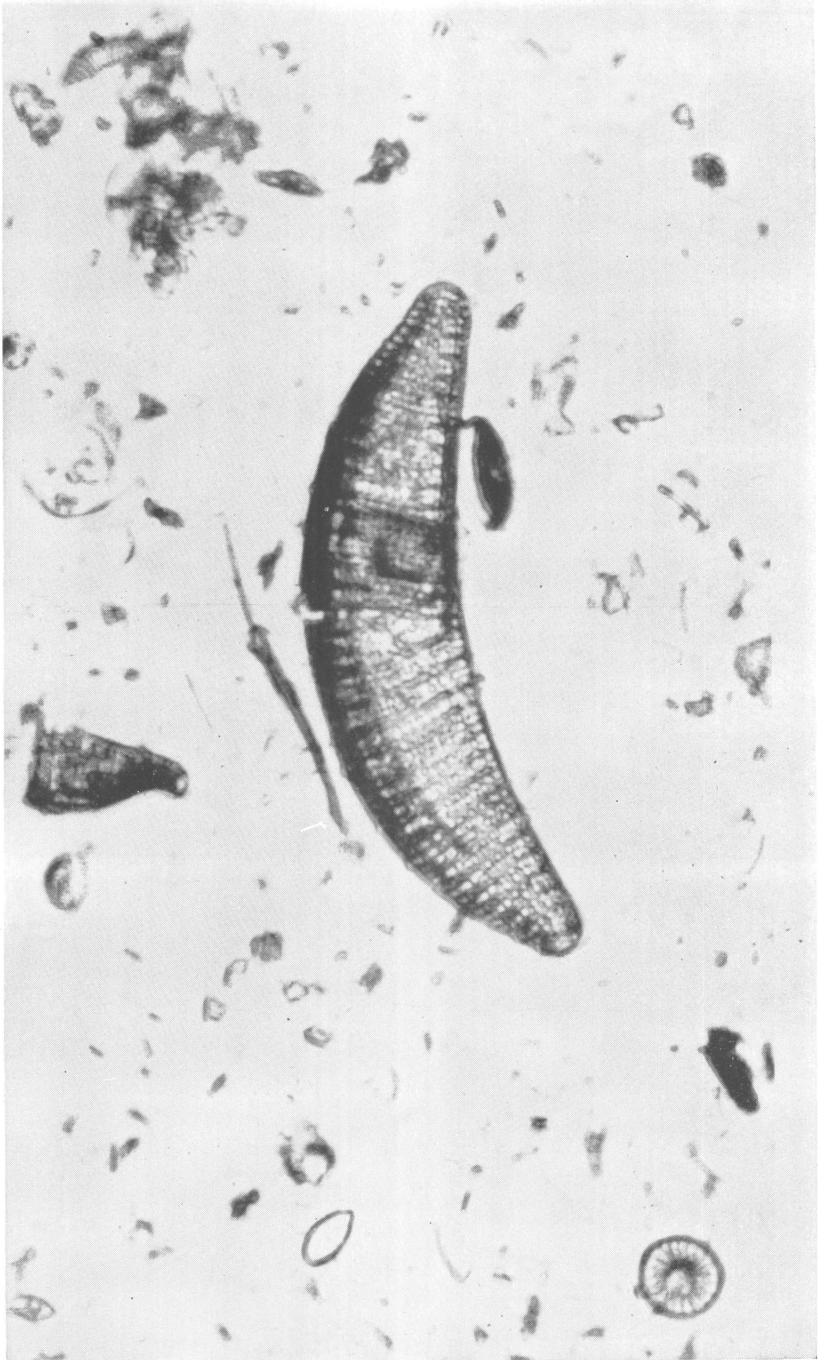


FIGURE 33.—Photomicrograph of diatoms and diatom fragments in the very slow flow-rate filter-aid made from 250- to 325-mesh diatomite from Warm Springs, Oreg. (Oregon 6a).  $\times 1,000$ . Figures 33 through 43 do not necessarily represent actual particle-size distribution.



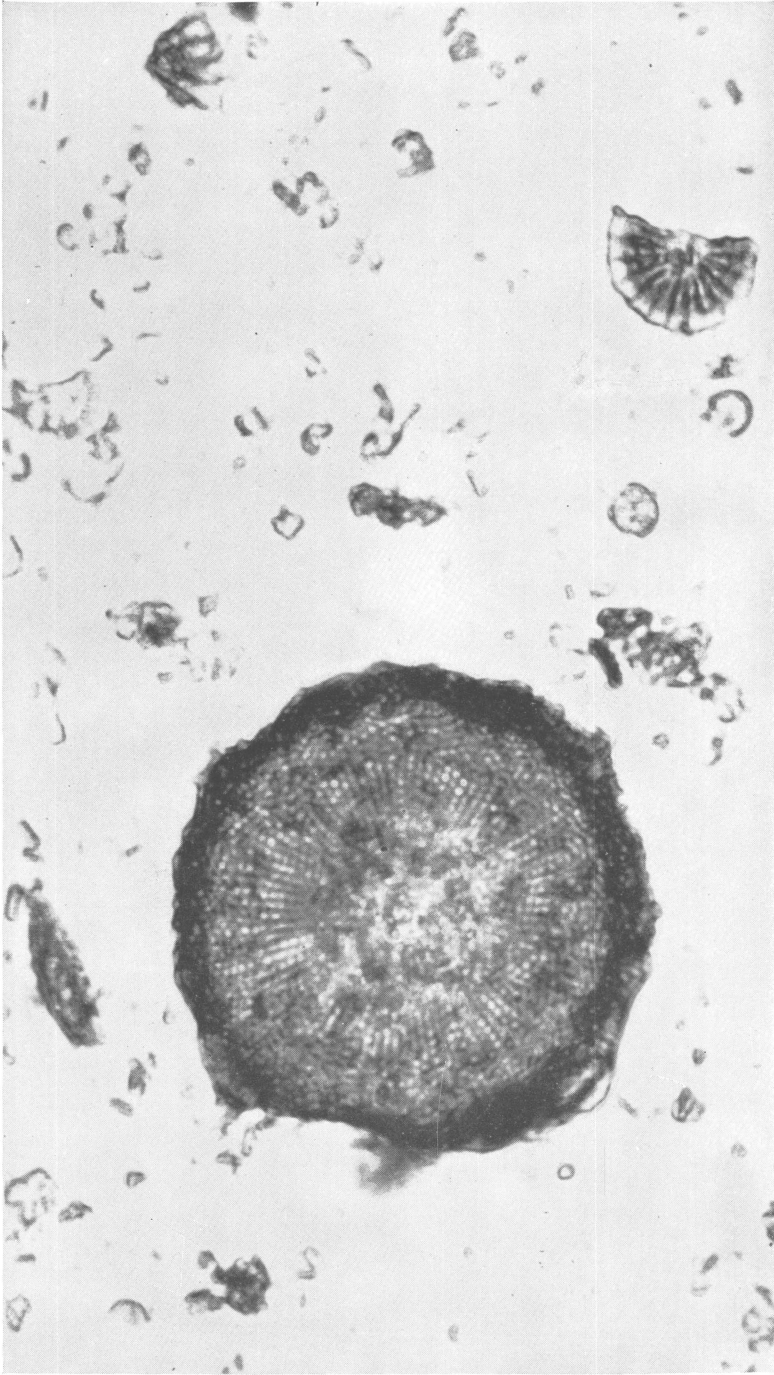


FIGURE 34.—Photomicrograph of diatoms and diatom fragments in the very slow flow-rate filter-aid made from 250- to 325-mesh diatomite from Dairy, Oreg. (Oregon 15).  $\times 1,000$ . Secondary silica on the large diatom is shown by the rough edge and especially by the clear material along the lower-right edge.

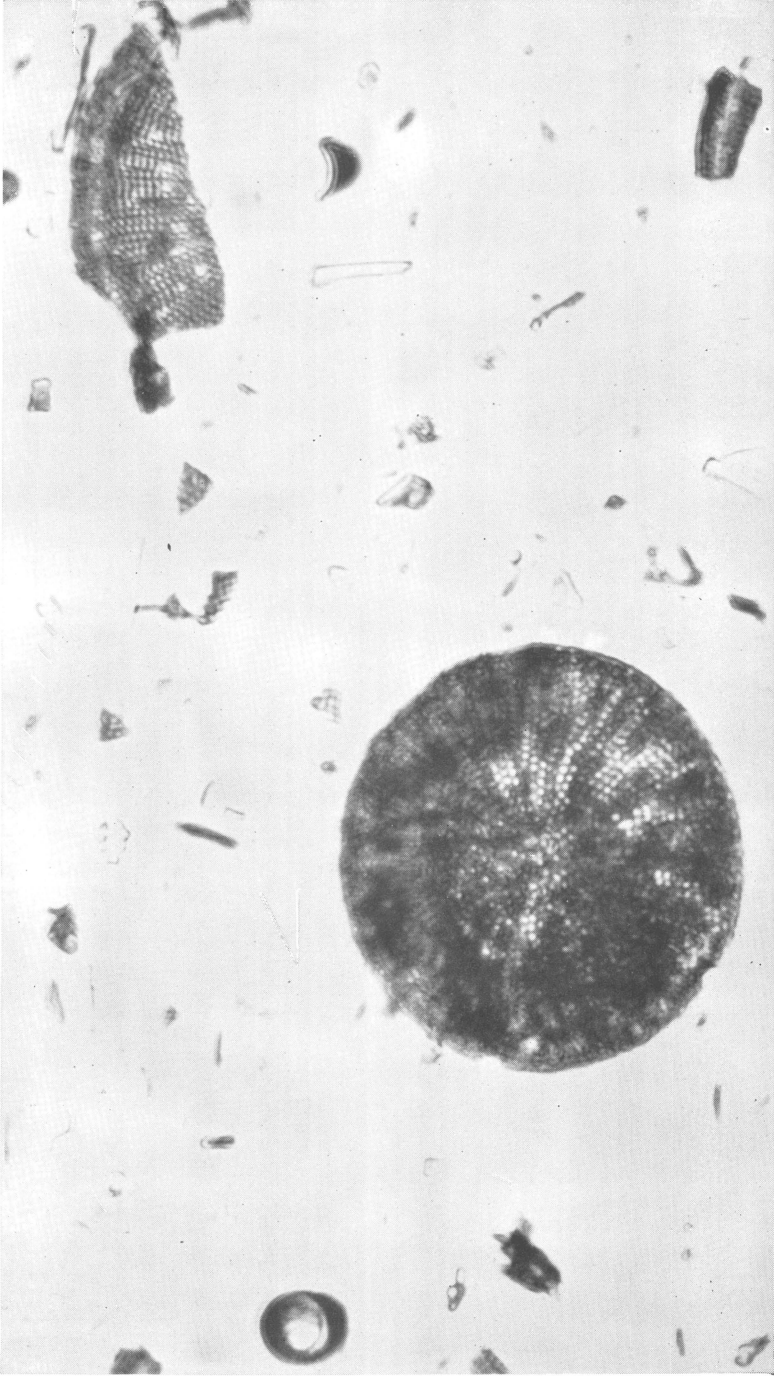


FIGURE 35.—Photomicrograph of diatoms and diatom fragments in the slow flow-rate filter-aid made from minus 100-mesh diatomite from the Squaw Creek area, Kittitas County, Washington (Washington 11).  $\times 1,000$ . The oval particle at the bottom is an end view of a small cylindrical diatom.



FIGURE 36.—Photomicrograph of diatoms and diatom fragments in the slow flow-rate filter-aid made from 250- to 325-mesh diatomite from Big Lake, Wash. (Washington 17).  $\times 1,000$ . Clear, semi-rounded particles in lower half are quartz grains.

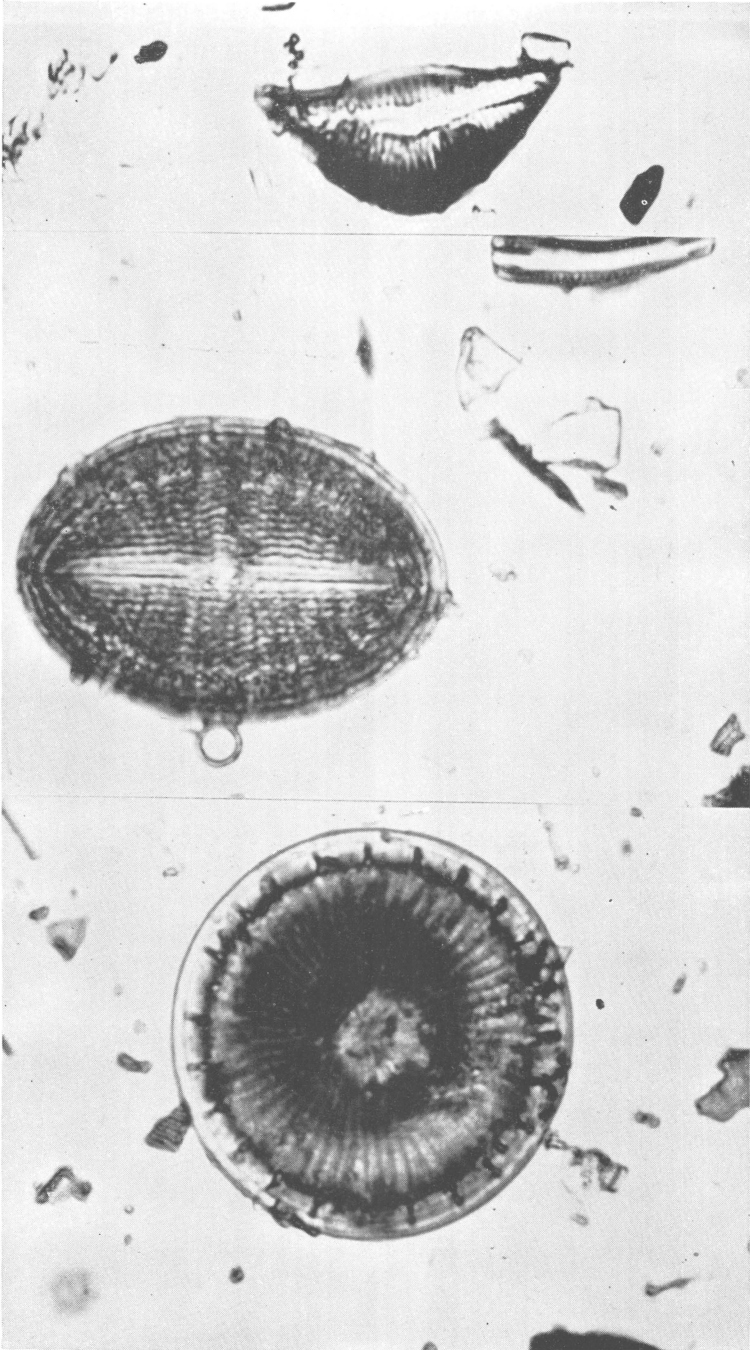


FIGURE 37.—Composite photomicrograph showing the three most abundant diatom types found in the medium flow-rate filter-aid made from calcined Terrebonne, Oreg. (Oregon 2b) diatomite sized from 250- to 325-mesh.  $\times 1,000$ .

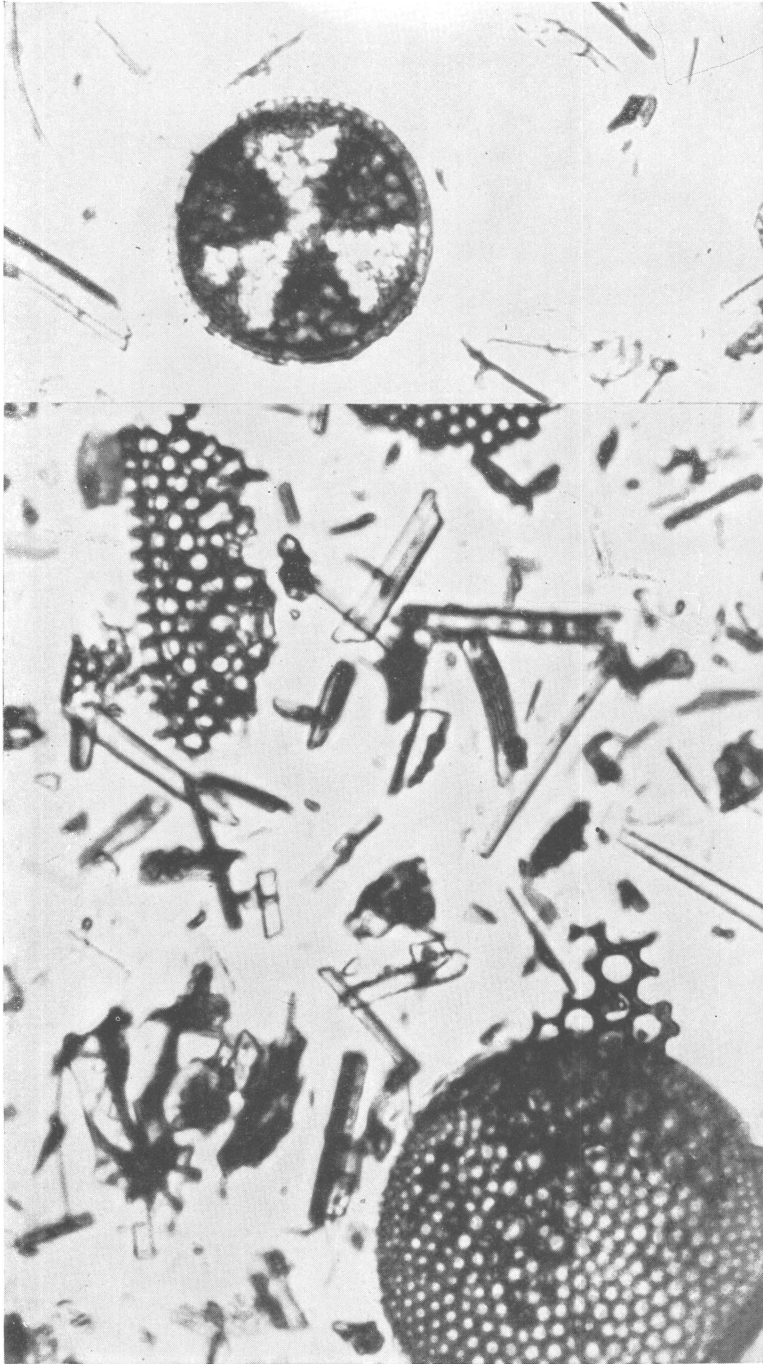


FIGURE 38.—Composite photomicrograph showing the most abundant diatom types and fragments found in the medium flow-rate Commercial 1 filter-aid.  $\times 1,000$ .



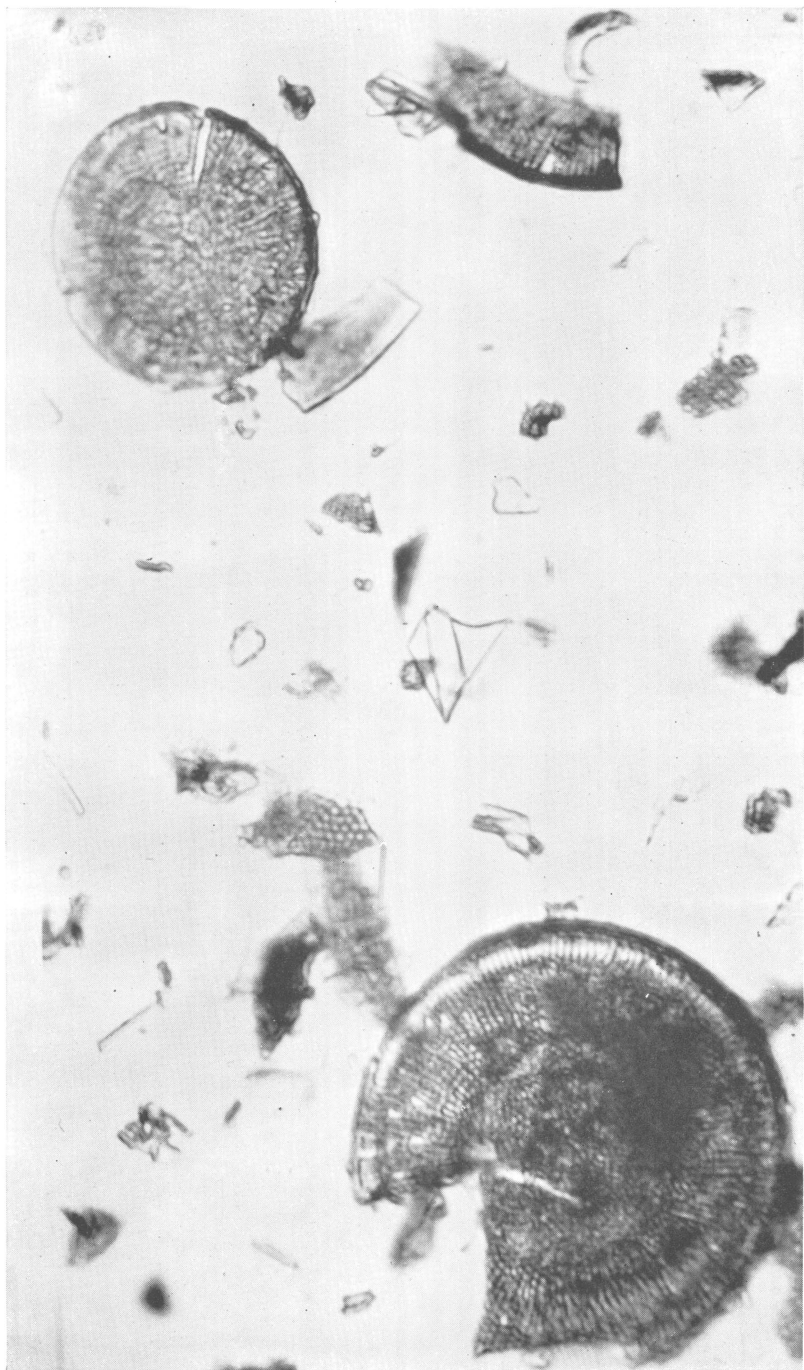


FIGURE 39.—Photomicrograph of diatoms and diatom fragments in the fast flow-rate filter-aid made from Squaw Creek, Wash. (Washington 11) diatomite calcined with 4 percent NaCl and then sized from 250- to 325-mesh.  $\times 1,000$ . Angular fragment in the center is quartz.

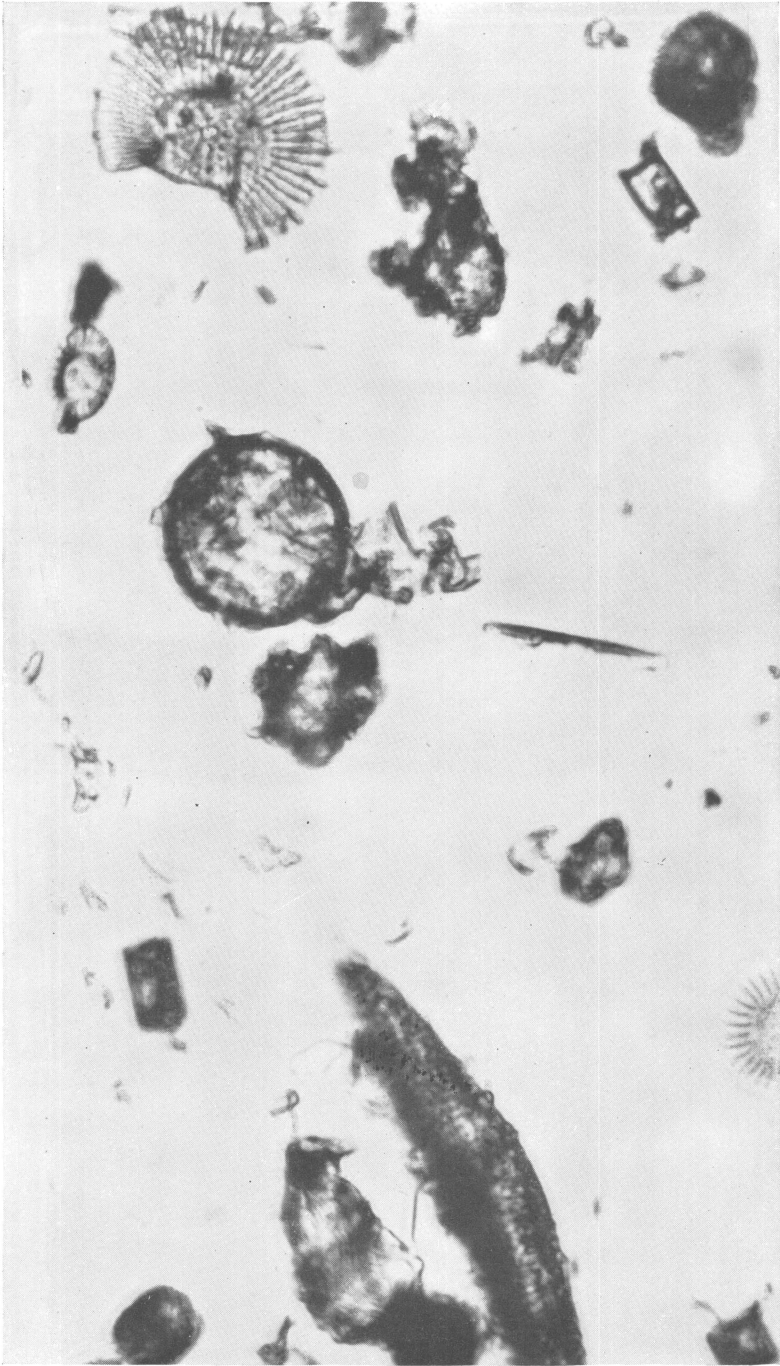


FIGURE 40.—Photomicrographs of diatoms and diatom fragments in the fast flow-rate filter-aid made from Big Lake, Wash. (Washington 17) diatomite calcined with 4 percent NaCl and then sized from 250- to 325-mesh.  $\times 1,000$ .

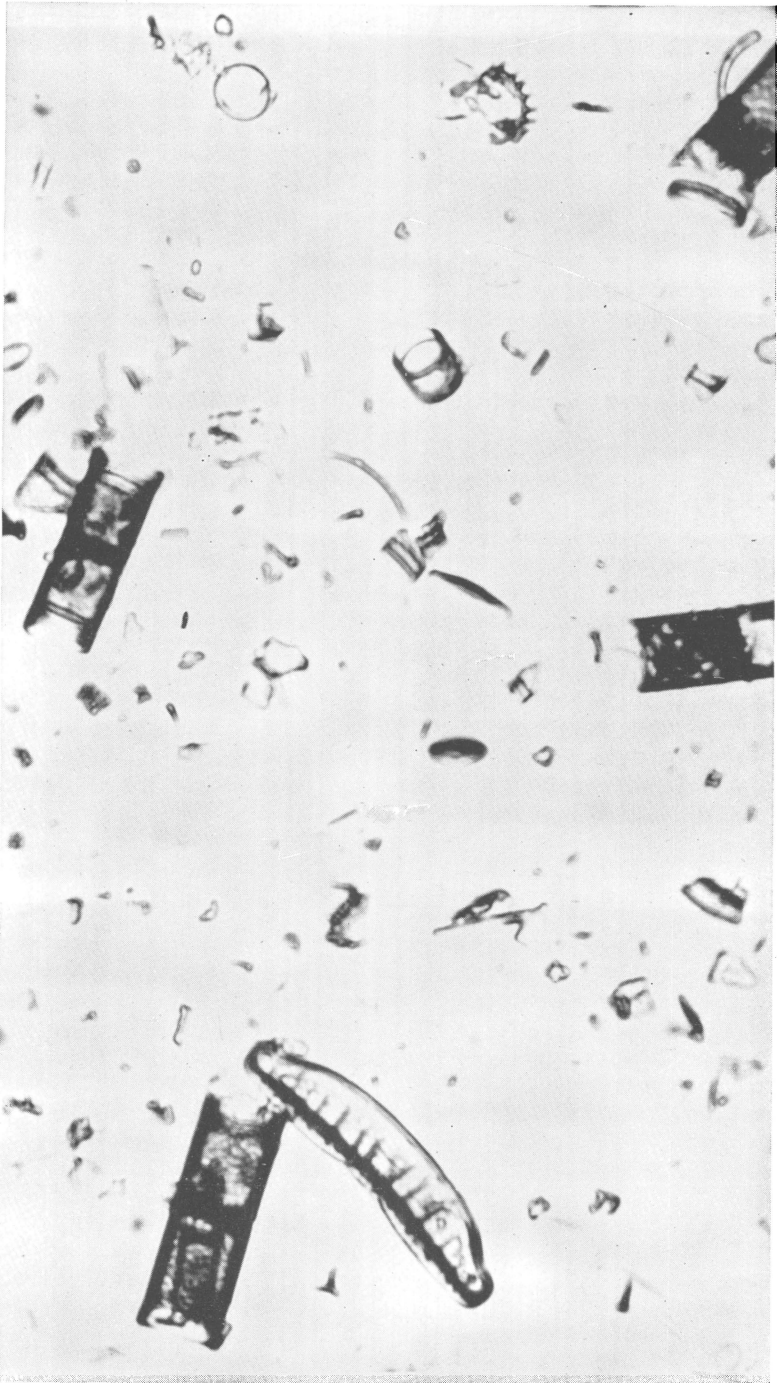


FIGURE 41.—Photomicrograph of diatoms and diatom fragments in the very fast flow-rate Commercial 4 filter-aid.  $\times 1,000$ . Note small cylindrical diatom inside larger one at lower left.



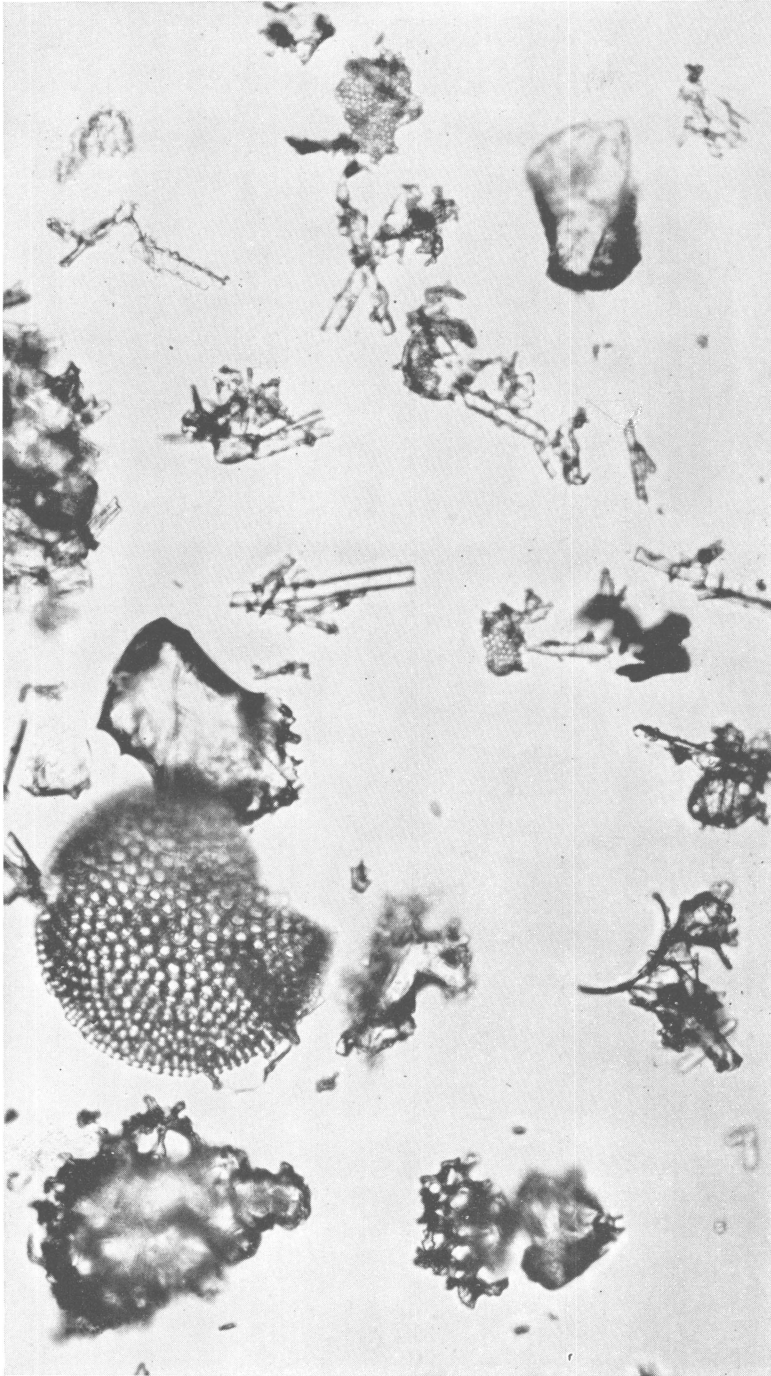


FIGURE 42.—Photomicrograph of diatoms and diatom fragments in the very fast flow-rate Commercial 5 filter-aid.  $\times 500$ . In comparison with diatoms in figures 33 through 41, the diatoms in figures 42 and 43 are about twice as large.

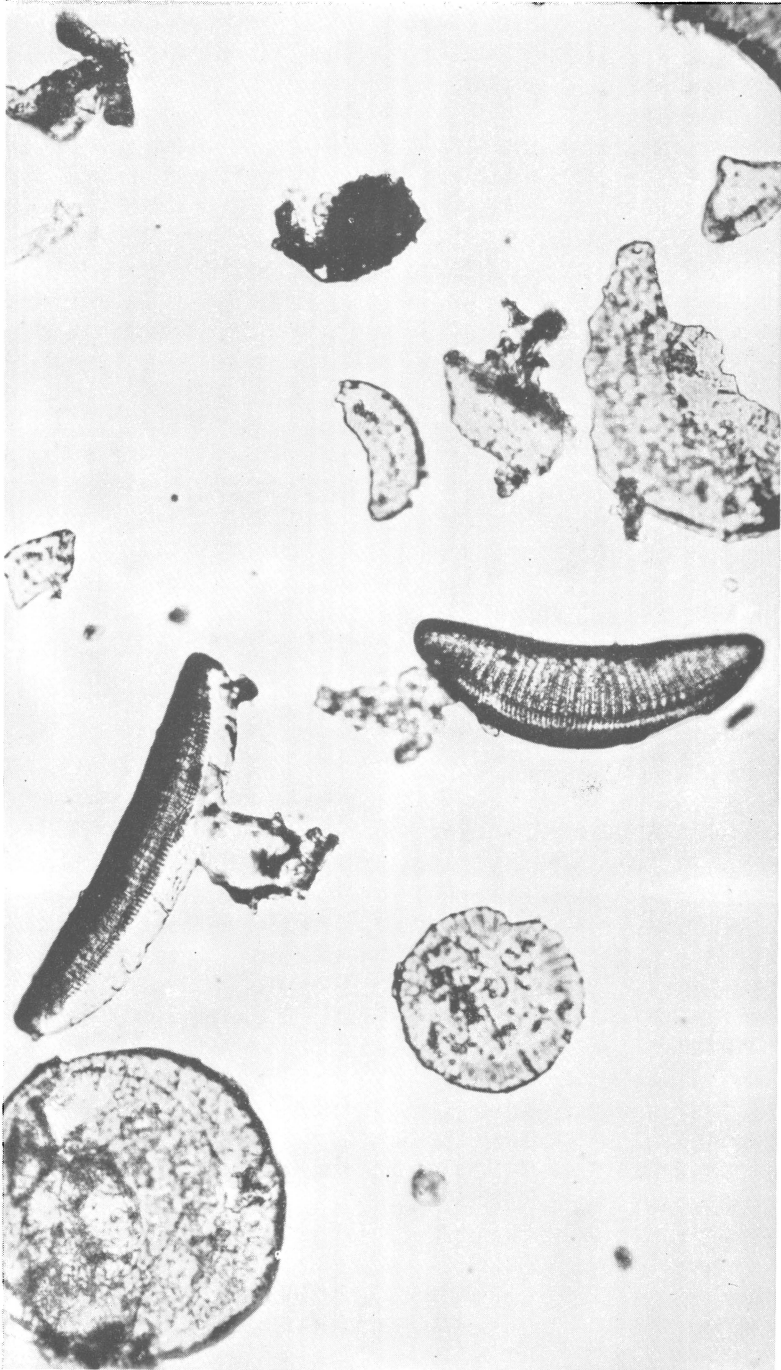


FIGURE 43.—Photomicrograph of diatoms and diatom fragments in the very fast flow-rate Commercial 6 filter-aid.  $\times 500$ .

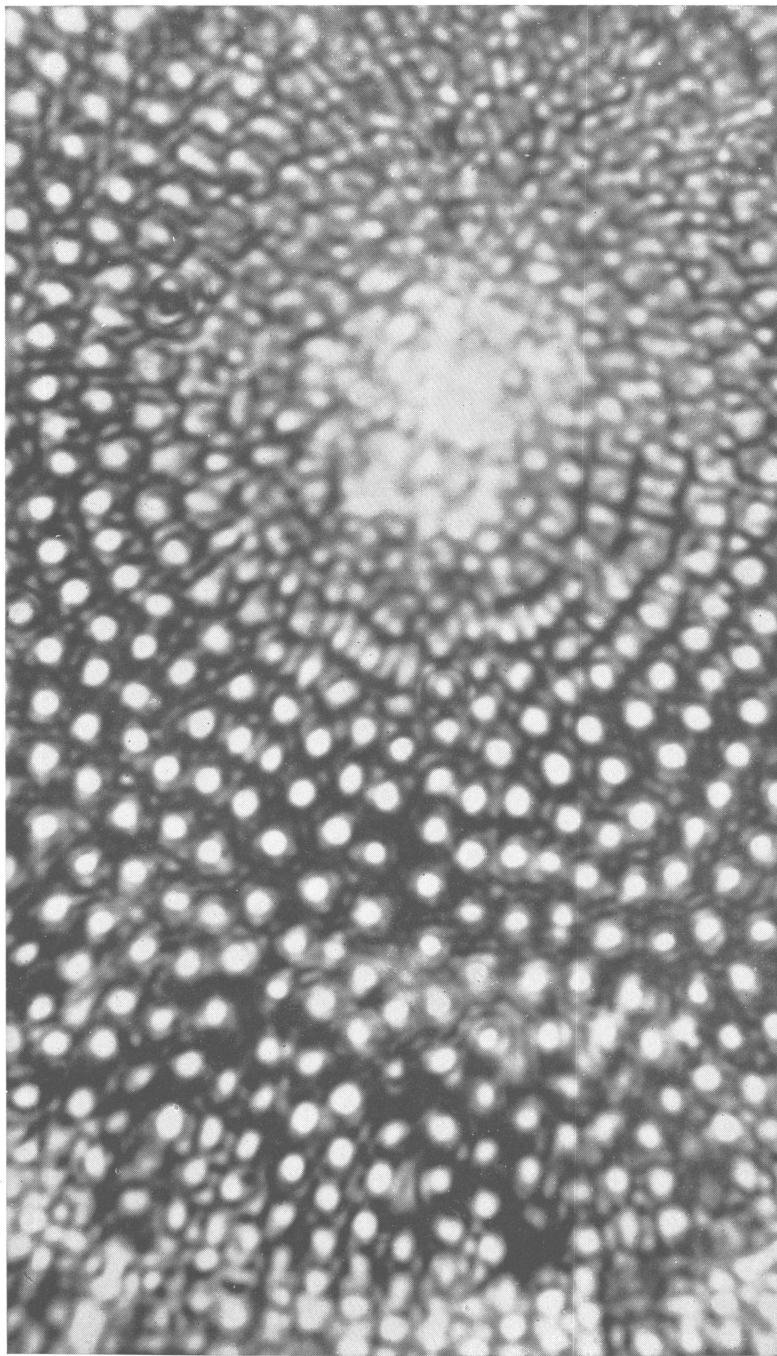


FIGURE 44.—Photomicrograph of the central part of a disk-type diatom after calcination to 1,750° F. with 4 percent NaCl, showing the small knobs between the large knobs.  $\times 6,500$ . On the illustration, 1 millimeter is equivalent to 0.154 microns.

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