

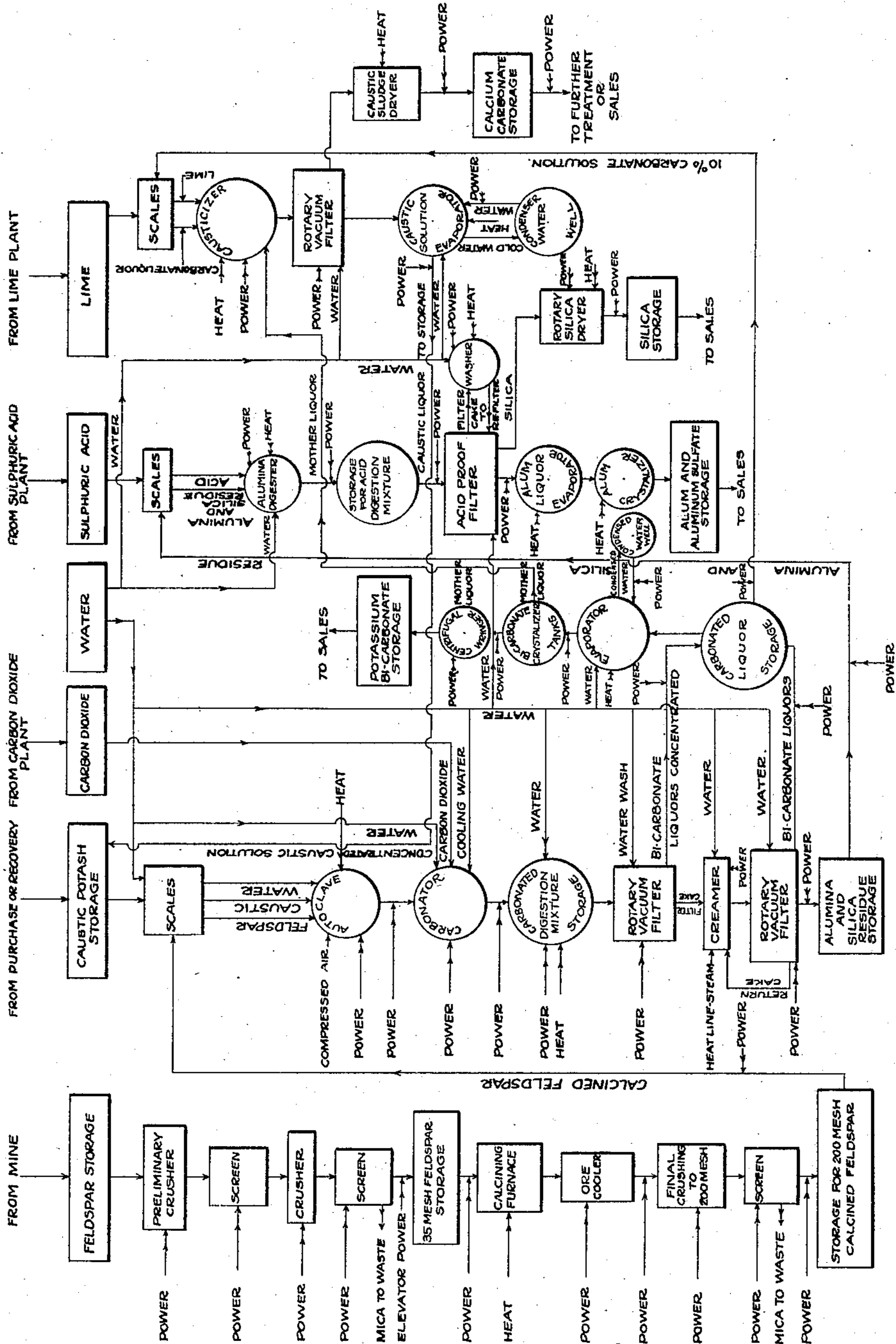
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PROCESS OF SEPARATING THE CONSTITUENTS OF MINERAL SILICATES

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PROCESS OF SEPARATING THE CONSTITUENTS OF MINERAL SILICATES

Original application filed May 17, 1922, Serial No. 561,704. Divided and this application filed February 7, 1924. Serial No. 691,217.

The principle of the invention is herein explained, and the best mode in which we have contemplated applying that principle, so as to distinguish it from other inventions. The
5 subject matter of this application is divided out of our pending application Serial No. 561,704 upon processes of separating the constituents of mineral silicates.

Our invention relates to processes for separating the constituents of mineral silicates, and particularly to processes for extracting the potassium and other values from potash feldspar. This invention relates to improvements in a process of this general character
10 disclosed in our pending application for U. S. Letters Patent, Serial No. 407,724, as well as the improvements disclosed in said application Serial No. 561,704, from which this subject matter has been divided out. The claims
15 of this application are limited to our improved method of recovering the potassium values in two fractions for sales and for reuse, as hereinafter fully described.

The annexed drawing and the following
25 description set forth in detail certain steps embodying our invention, the disclosed steps, however, constituting but one of the various methods in which the principle of the said invention may be carried out.

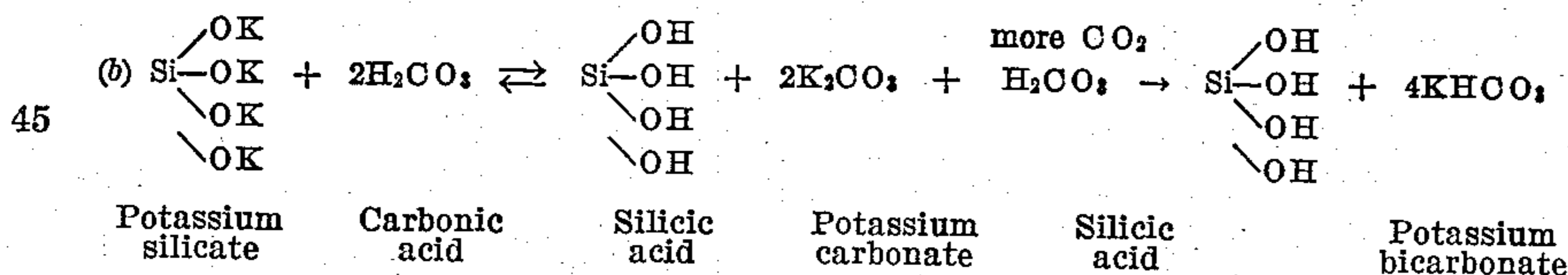
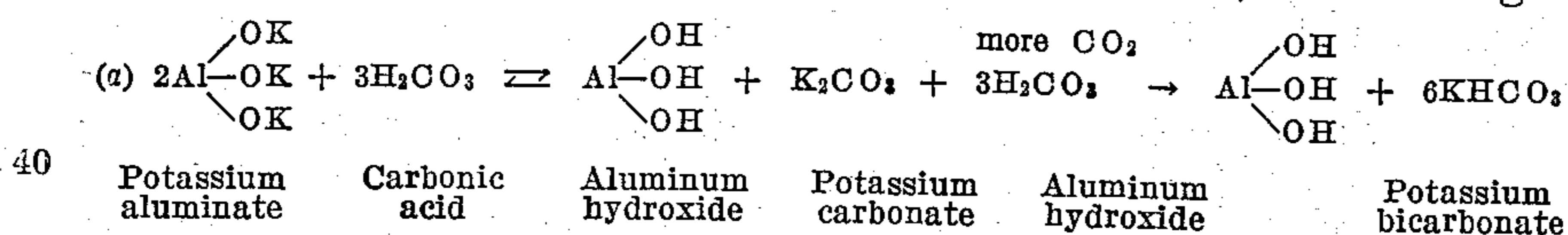
30 The drawing represents a flow diagram of our improved process.

We first crush high grade crystalline potash-feldspar to about thirty-five (35) mesh, in order that a subsequent calcining
35 can be thoroughly and economically effected. We then calcine this crushed ore until the same is converted to an amorphous condition, in order that a subsequent digestion with caustic potash may be effected to convert the
40 ore constituents into water soluble or acid soluble compounds. This calcining is effected upon the bed of a furnace or in any other suitable manner by heating from one (1) to two (2) hours at from 800° to 1050° C. After
45 the ore has thus been reduced to an amorphous condition, we then further crush the same to about two hundred (200) mesh after which the same is digested with the caustic potash. The digestion of the ore with caustic potash
50 is not carried to a point where all of the ore

is converted into water soluble potassium silicate and potassium aluminate but there is also produced a water insoluble but an acid soluble potassium aluminum silicate. We
55 use 90% caustic potash for the alkali digestion in amount from one and one-quarter (1¼) to two (2) times the weight of the ore and also an equal amount of water, heating for from one (1) to two (2) hours at a temperature from 275° to 325° C. The operation
60 is effected in an autoclave at a pressure of from 300 to 350 lbs. per square inch. We then allow a further reaction period of about two (2) hours, resulting in a complete decomposition of the ore into the water or acid
65 soluble compounds above-mentioned. By "90% caustic potash", we denote merely a certain grade of potash. It means that caustic potash with a KOH content of ninety (90) per cent or better should be used. In the development upon which this application is
70 based, standard caustic potash was used for the digestion, such as Schieffelin or Innes-Speiden, except where the caustic was obtained from the process itself for reuse. In
75 buying the standard potash for the purpose a ninety (90) per cent grade or better was obtained. During the digestion operation considerable hydrogen is formed, after the temperature has reached substantially 200° C,
80 due to the fact that the iron walls of the autoclave are attacked by strong caustic potash, the reaction being $Fe + 2KOH = Fe(OK)_2 + H_2$. It may be that some impurity in the ore or even in the iron acts as a catalyst for this
85 reaction. The iron later appears as an impurity in the aluminum salts or possibly in the silica. In order that this evolved hydrogen may not form an explosive mixture with the oxygen of the air which is in the autoclave
90 at the start, we remove this air from the autoclave during the first part of the digestion operation by allowing the pressure to reach thirty (30) to forty (40) lbs. per square inch
95 and then allowing the oxygen to escape from the autoclave. The already formed steam displaces the air which escapes and finally the interior of the autoclave contains nothing but steam, indicated by a current of pure quickly 100

condensing steam escaping from the auto-clave valve.

We dilute the alkali digestion products with from one (1) to four (4) times their volume of water and then carbonate the mixture with carbon dioxide, maintaining a pressure of about 22 inches above atmospheric pressure, the temperature being maintained at substantially room temperature by means of a positive removal of the generated heat. We thus obtain the water soluble potash value in solution and precipitate aluminum hydroxide and ortho silicic acid. The mass action resulting from the pressure above-mentioned and the maintenance of normal temperature effects the carbonating in about forty-five (45) minutes, the same taking place in two stages, the first stage resulting in a conversion of the potassium content to potassium carbonate and taking only about five minutes and the second stage converting the potassium carbonate to bicarbonate and taking about forty (40) minutes. The reaction is based upon the action of the ionized acid (H_2CO_3) formed by the dissolving of CO_2 gas in water. There is only incomplete precipitation of the silicon and aluminum during the first stage because the potassium carbonate being strongly alkaline dissolves aluminum hydroxide and ortho silicic acid and it is not until this carbonate is very largely converted into bicarbonate, which is almost neutral in reaction, that all of the aluminum and silicon separate out. The reactions are represented, at least in principle, by the following equations:



Of course, carbonation to formation of 100 per cent bicarbonate requires just twice the amount of CO_2 that is needed for the normal carbonate. The speed of carbonation can be greatly increased by distributing the gas uniformly over the whole cross-section of the volume of liquid to obtain the most extensive contact between the gas and liquor, and also by very strong agitation. To the carbonation products we add about one-half ($1/2$) of their volume of water. We then repeatedly filter and wash, the wash water requirements being from two and one-quarter ($2\frac{1}{4}$) to four and one-half ($4\frac{1}{2}$) gallons of water per lb. of feldspar digested. These steps, of course, result in the recovery of so much of the potassium from the feldspar as was converted into

water soluble compounds during the alkali digestion period.

We convert only a part of the potassium carbonates thus obtained into sales products and the balance we causticize in order to obtain caustic potash for reuse in further digestion operations, the steps constituting these last-mentioned operations comprising the method which is particularly claimed in this application. In detail, we evaporate the carbonate solution in steps obtaining each time a saturated solution from which, as the temperature is permitted to drop, crystalline masses of potassium carbonate and potassium bicarbonate are obtained, preferably until from five (5) to ten (10) per cent of the total potassium content of the carbonate solution is removed. The potassium carbonates thus obtained also contain the sodium that is present and thus prevent the building up of the sodium content during repeated reuses of the caustic potash. The balance of the carbonate solution is concentrated to a 10 per cent solution which is suitable for a causticizing operation with lime to produce caustic potash for reuse and calcium carbonate for sale. For the recovery of the aluminum and silicon values we utilize the operations fully described, shown and claimed in the parent application Serial No. 561,704, the same constituting no part of the improvement claimed in this application.

What we claim is:

1. In a process of separating the constituents of mineral silicates, the steps which consist, in removing the water soluble potas-

sium value by means of caustic potash digestion and carbonation; and then separating the soluble potassium carbonates into two fractions by evaporating the solution in steps, obtaining each time a saturated solution from which crystalline potassium carbonate and bicarbonate are obtained, until the sodium content is removed, and then concentrating the remainder of the water soluble potassium value to a substantially ten (10) per cent carbonate solution and causticizing.

2. In a process of separating the constituents of mineral silicates, the steps which consist, in removing the water soluble potassium value by means of caustic potash digestion and treatment with carbon dioxide; and then separating the soluble potassium carbonates

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F. E. SMITH

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VALVE SEAT

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Fig. 1

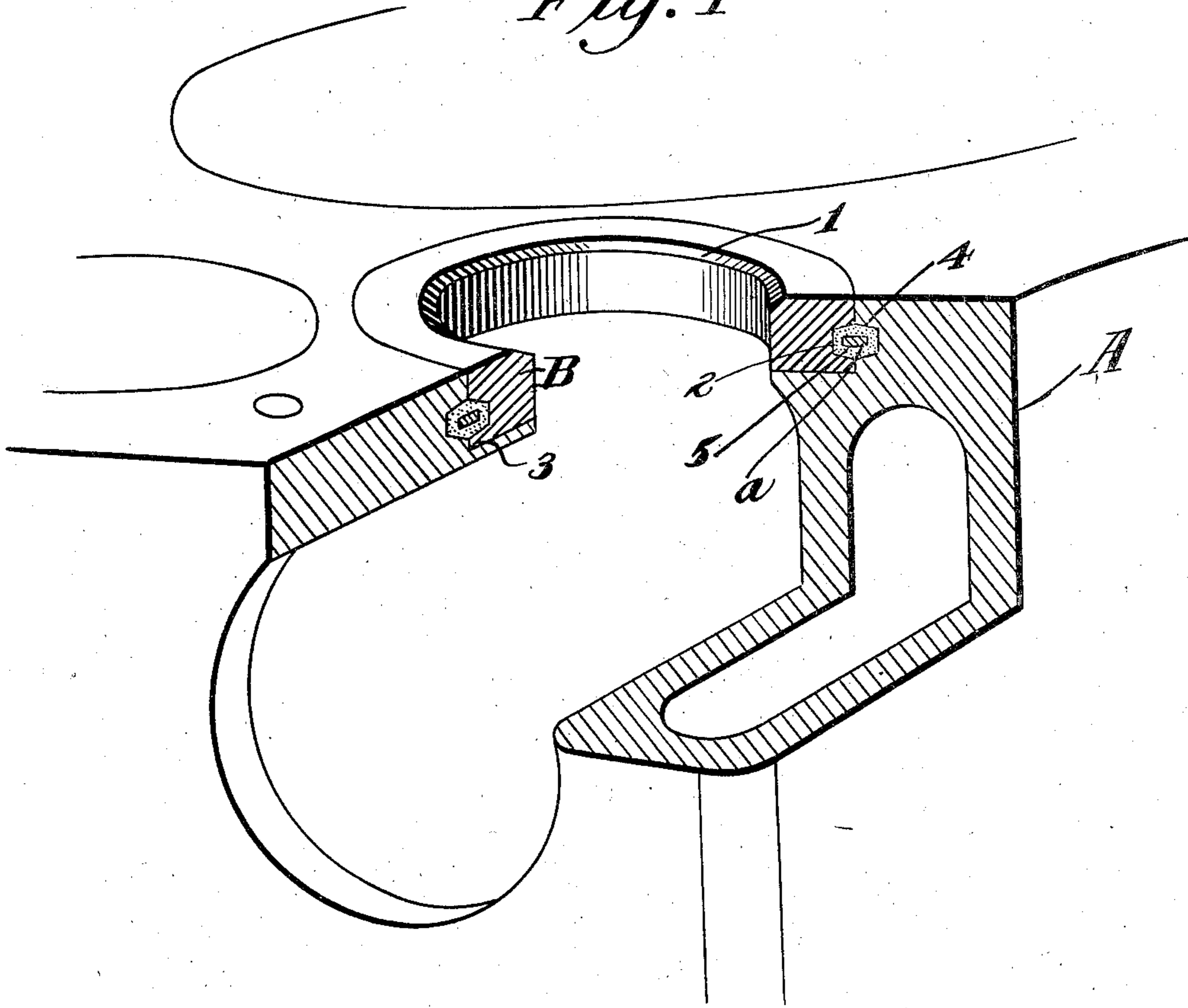
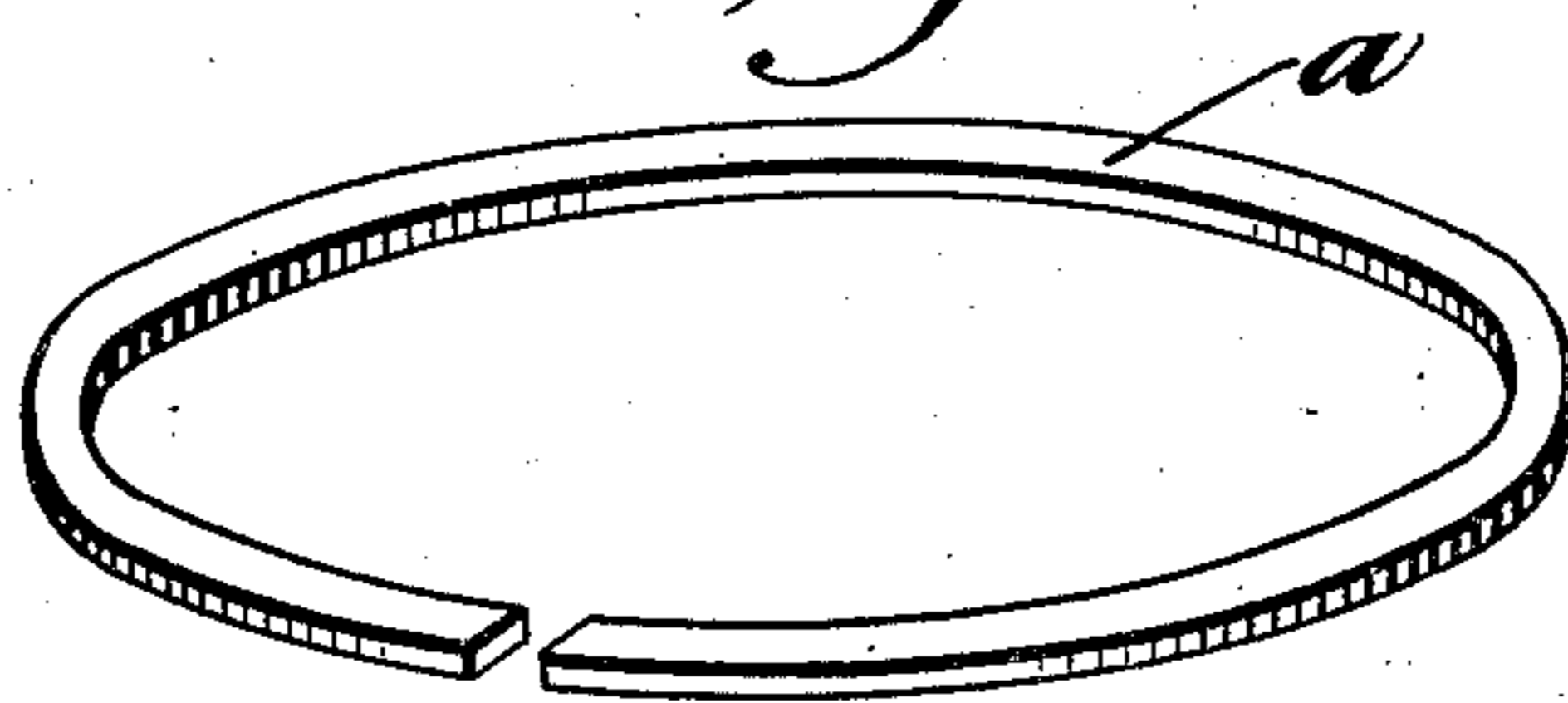


Fig. 2



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