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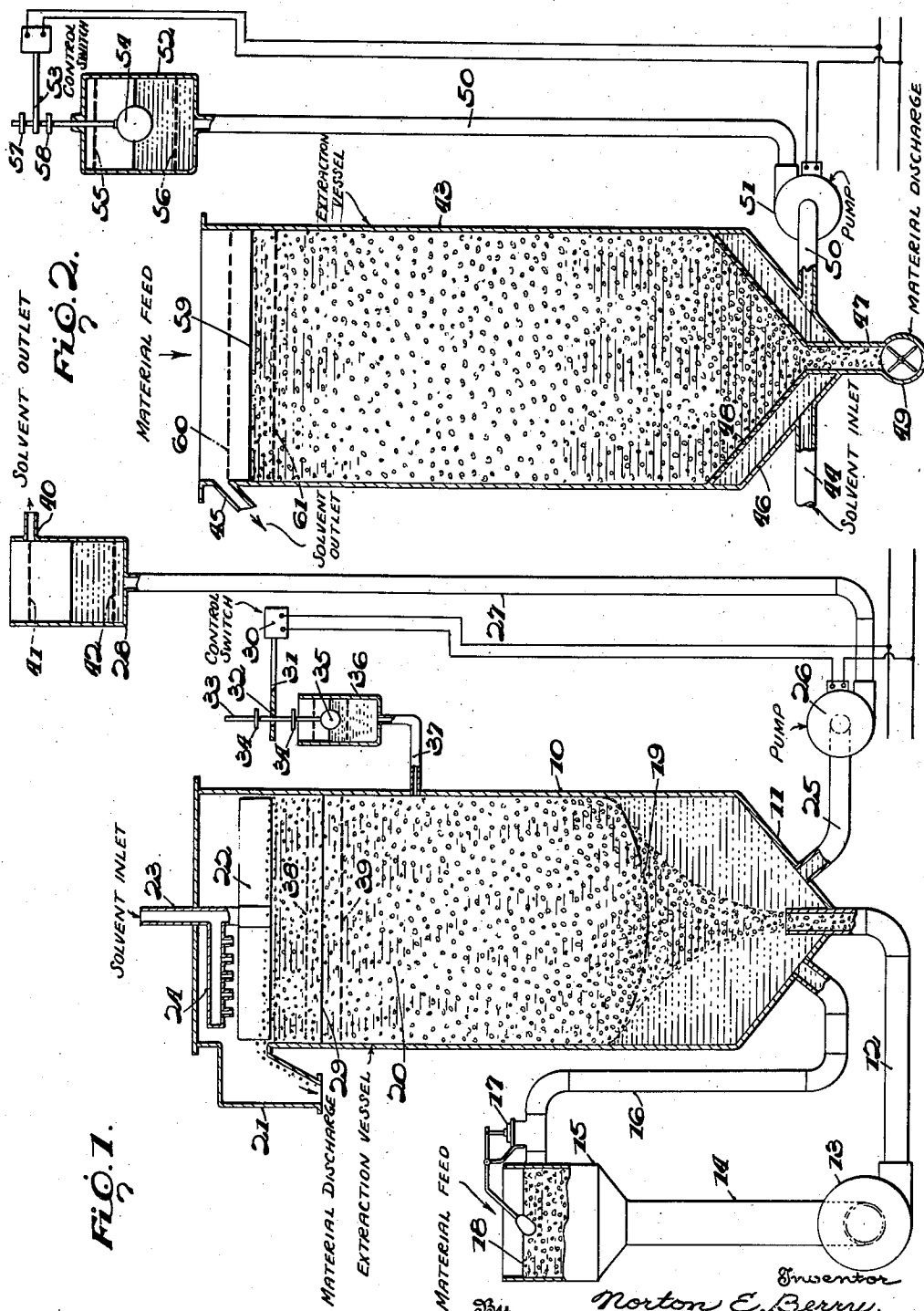
N. E. BERRY

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SOLVENT EXTRACTION OF SOLID MATERIALS

Filed May 8, 1941

2 Sheets-Sheet 1



Inventor
Norton E. Berry.
Cameron, Kerkam & Sutton
Attorneys

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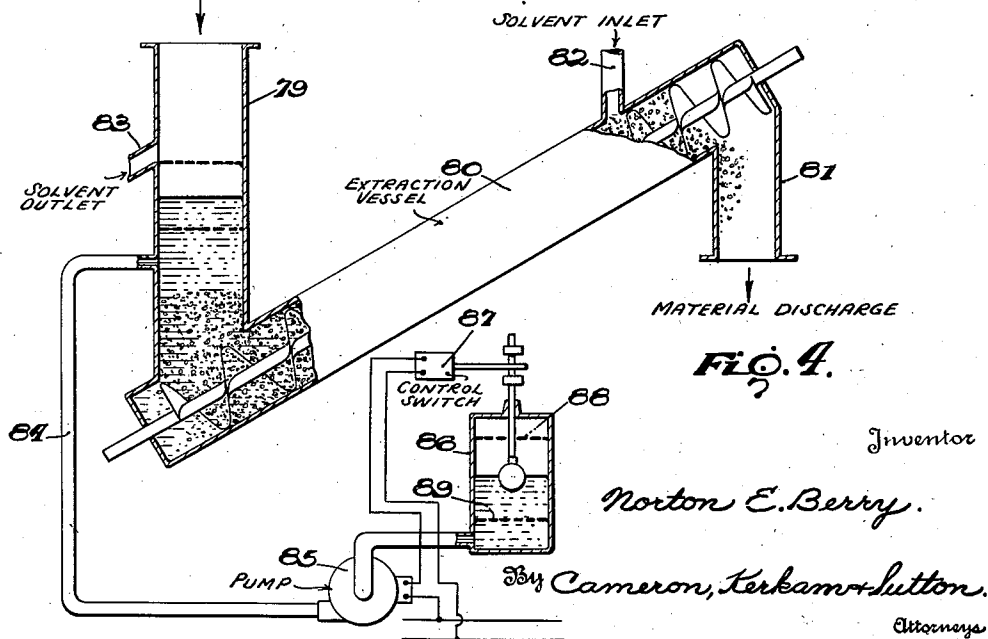
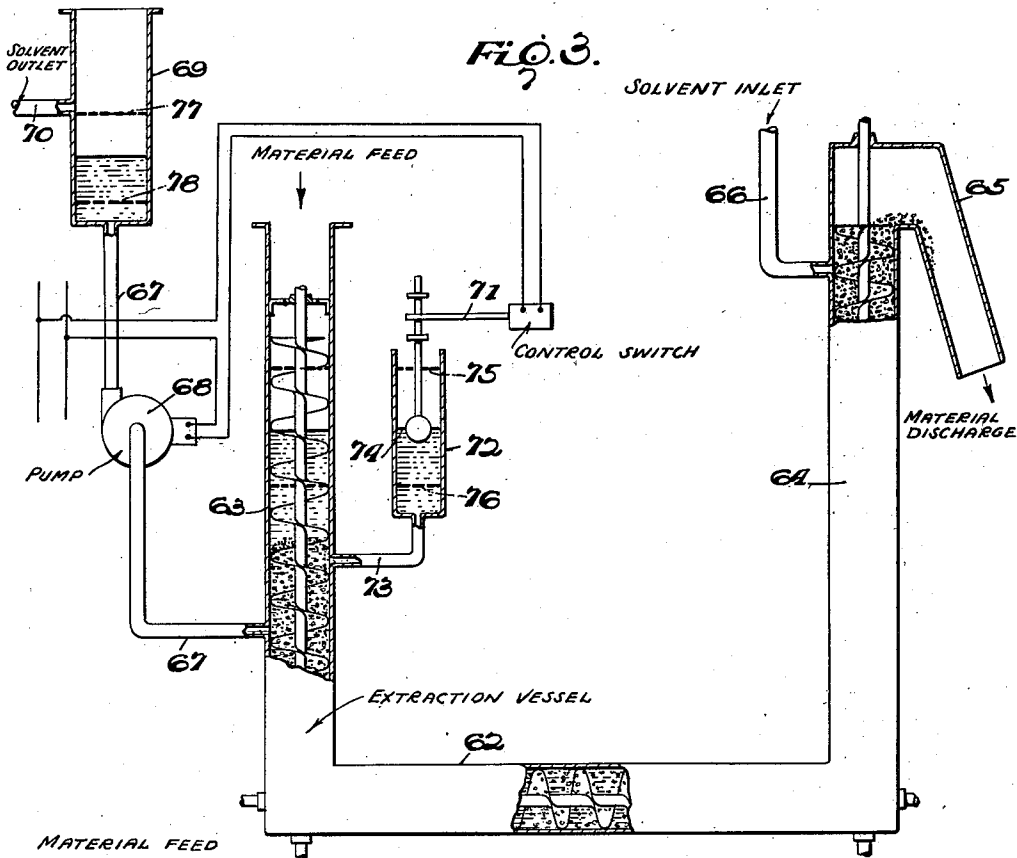
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UNITED STATES PATENT OFFICE

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SOLVENT EXTRACTION OF SOLID MATERIALS

Norton E. Berry, Summit, N. J., assignor to General Foods Corporation, New York, N. Y., a corporation of Delaware

Application May 8, 1941, Serial No. 392,590

4 Claims. (Cl. 23—270)

This invention relates to the solvent extraction of solid materials and is directed to the provision of an improved system of countercurrent liquid-solid extraction adapted for the treatment of such materials as cacao beans, soy beans, coffee beans, sugar beet cossettes, coarse mineral sands and the like.

It is generally recognized in the operation of any liquid-solid extraction system, whether of the single or multiple liquid phase type, that the efficiency of the extraction is increased as the velocity of movement of the solvent relative to the material extracted is increased. For this reason it is considered good practice in batch procedures to employ some form of agitation and in continuous processes to employ the highest velocity of solvent flow feasible under the conditions obtaining in each particular case. However, there are practical and economic considerations which normally limit the velocities obtainable in countercurrent procedures of the type now in use, and in a great many instances, as where a high degree of extraction is desired, these limitations entail a serious loss in efficiency of the extraction operation over what it might otherwise be.

Accordingly, one of the objects of the present invention is to provide a countercurrent liquid-solid extraction procedure wherein higher velocities of movement of solvent relative to the material extracted are obtained than have heretofore been possible with the same quantity of solvent.

Another object of the invention is to provide a continuous liquid-solid extraction system of greater efficiency than the countercurrent extraction procedures heretofore employed.

A further object of the invention is to provide a countercurrent liquid-solid extraction process wherein lesser quantities of solvent are required to obtain given velocities of movement of solvent relative to the material extracted than have been required with the procedures heretofore employed.

I have found that the above objects can be attained by imparting an alternating forward and backward movement to the body of solvent in contact with the material while maintaining a desired net forward flow of solvent through the extraction apparatus countercurrently to the material. Compared with the velocity obtainable with a unidirectional movement of solvent through the material at the same net flow, the actual velocity of movement of the solvent may thus be made considerably greater not only in the forward but in the backward direction. Hence an increased velocity of movement of solvent rela-

tive to the material being extracted is obtainable with any given quantity of solvent, and conversely, any given velocity of movement of solvent is obtainable with a lesser quantity of solvent than required when unidirectional movement of solvent through the material is employed. The frequency and extent of the alternating movement of the solvent relative to the material can be adjusted to suit conditions in any particular case, but the extent of movement in each direction should not be so great as to interfere with proper countercurrent operation.

Any suitable method and apparatus can be employed to effect the desired alternation of the direction of movement of solvent. Assuming for example a normal countercurrent flow of solvent and solid material into and out of the extraction apparatus, the body of solvent in the apparatus may be moved back and forth through the material being extracted by means of a suitable pumping device. Alternatively the relative extent of movement of the body of solvent in the forward and reverse directions may be regulated so that a predetermined quantity of solvent is discharged from the apparatus during each cycle, the quantity discharged being of course compensated by the introduction of an equivalent quantity of fresh solvent at the inlet end of the apparatus. In both cases the level of solvent in the apparatus will vary but since the magnitude of the reversals is preferably made small, such variation is not objectionable and in fact provides a convenient means of regulating the reversal of flow.

The last-mentioned procedure has the advantage that it can be performed without material change of existing equipment, and hence it may conveniently be used to illustrate the operation of the invention, but it will be understood that the methods and apparatus specifically described in the following examples are for purposes of illustration only.

In the accompanying drawings:

Fig. 1 is a schematic showing of an apparatus suitable for practicing the invention in connection with the countercurrent extraction of solid materials lighter than the solvent; and

Figs. 2, 3, and 4 are schematic showings of three different forms of apparatus suitable for practicing the invention in connection with the countercurrent extraction of solid materials heavier than the solvent, the passage of the material through the solvent being effected by gravity in Fig. 2 and by screw propulsion in each of Figs. 3 and 4.

Fig. 1 illustrates the application of the inven-

tion to the method of countercurrent extraction of caffeine from green coffee beans disclosed in the co-pending application of T. M. Rector, Serial No. 373,039, filed January 3, 1941. In accordance with this method, the beans are passed upwardly under the action of buoyancy through a vertical column of downwardly moving caffeine solvent.

Referring to this figure, the extracting apparatus is shown as comprising a heat insulated vessel 10 through which the coffee passes upwardly and the solvent downwardly in countercurrent relation, the vessel preferably being of cylindrical form and having a coned bottom 11. Centrally fitted through the coned bottom 11 is an injector pipe 12 connected to a pump 13 having its input connected by a pipe 14 with a hopper 15, into which the coffee to be extracted is continuously fed in any suitable manner. Solvent from the vessel 10 is continuously introduced through the pipe 16 into the hopper 15, from which it flows through the pipe 14 to the pump 13, which pumps it back into the vessel 10 through the injector pipe 12. The introduction of solvent into the hopper 15 is controlled by a float valve 17, to maintain a desired level 18 therein, and the coffee and solvent in the hopper 15 form a slurry which can be readily pumped through the pipe 12 into the vessel 10. The pipe 16 preferably communicates with the vessel 10 below the outlet of injector pipe 12 to insure against any return passage of coffee there-through. The coffee introduced in the vessel 10 being lighter than the solvent rises against the bottom 19 of the column of beans 20 which substantially fills the vessel 10 and due to its buoyancy extends above the level of the solvent. Coffee from the top of the column of beans 20 is discharged through a side chute 21 by a rotary raking mechanism 22. Fresh solvent is introduced into the top of the vessel 10 through the hollow shaft 23 of the raking mechanism and an associated rotating sprayer 24, and spent solvent is discharged from the bottom of the vessel by a pipe 25.

As described in the aforesaid Rector application, the above apparatus is adapted for the continuous countercurrent extraction of caffeine from the coffee beans at a constant rate of solvent flow through the vessel 10. The present invention additionally provides for the back and forth movement of the body of solvent within the vessel to increase the velocity of movement of the solvent through the coffee beans. This result is accomplished in the form shown by alternately withdrawing from and returning to the vessel 10 a portion of the solvent therein, the frequency of alternation being conveniently regulated by the resulting rise and fall of the solvent level. In the embodiment shown in Fig. 1, this withdrawal of a part of the body of solvent from the vessel is combined with the normal discharge of solvent from the vessel, but it will be understood that this combination is not necessary.

In carrying out the above described operation, solvent leaving the vessel 10 through the pipe 25 is delivered by an intermittently operated pump 26 and a pipe 27 to a storage chamber or vessel 28, from which it flows back into the vessel 10 when the pump stops to cause the periodic reversal of movement of the body of solvent in the vessel. The intermittent operation of the pump 26 is conveniently controlled

by the rising and falling solvent level 29 in the vessel 10, as by means of a float operated switch 30 responsive to the solvent level and controlling the pump driving motor. As shown, the operating arm 31 of the switch has an aperture 32 through which a vertical rod 33 carrying actuating blocks 34 on either side of the operating arm 31 is freely movable. The lower end of rod 33 is attached to a float 35 in a float chamber or vessel 36 that is connected by a pipe 37 to the vessel 10. Hence the level of solvent in the vessels 10 and 36 is the same. When this level rises to a desired point as indicated by the dotted line 38, the upward movement of the float 35 closes the switch 30 and starts the operation of the pump 26. When the solvent level falls to a suitable point as indicated by the dotted line 39, the downward movement of the float 35 opens the switch 30 and stops the pump 26 so that the solvent pumped into the storage vessel 28 runs back by gravity into the vessel 10, the pump being of suitable type or suitably arranged to permit the return flow. Thus it is apparent that solvent is alternately transferred from the vessel 10 into the vessel 28 and returned from the vessel 28 into the vessel 10, which maintains alternate downward and upward movement of the body of solvent in the vessel 10.

The rates at which solvent is alternately removed from the vessel 10 by the pump 26 and returned to the vessel 10 by gravity are preferably made to exceed considerably the normal rate of solvent flow through the apparatus, so that high velocities of movement of the solvent are maintained in the vessel 10 in both directions of movement of the solvent. However, the quantities of solvent alternately removed from the vessel 10 by the pump 26 and returned to the vessel 10 by gravity are made comparatively small by suitable adjustment of the switch blocks 34 so that the alternate downward and upward movement of the solvent takes place over comparatively small distances and does not interfere with the desired countercurrent operation.

It will be seen that the withdrawal of spent solvent from the apparatus at a rate equal to the rate of introduction of fresh solvent can take place regardless of the alternate upward and downward movement of solvent in the vessel 10 as described above, so that the desired net forward flow of solvent through the apparatus is maintained. Preferably, however, this net forward flow is combined with the withdrawal of solvent from the vessel 10 by the pump 26 in order to avoid unnecessary duplication of equipment. To this end the storage vessel 28 is provided with an overflow pipe 40 through which spent solvent overflows from the vessel 28 and is discharged from the apparatus on each pumping cycle of the pump 26. The quantity of solvent returned to the vessel 10 is thus less than that withdrawn on each cycle of operation by an amount which is equal to the quantity of fresh solvent introduced into the vessel 10 through the hollow shaft 23 and sprayer 24 during the cycle.

In operation, when solvent reaches the level 38 in the vessel 10, pump 26 begins to pump solvent into the storage vessel 28. The solvent in the latter vessel rises to the level 41 of the overflow 40 and thereafter overflows until the lower level 39 is reached in the vessel 10. At this point pump 26 stops and solvent flows back into the vessel 10 while the level of solvent in the storage vessel 28 falls to the point 42. Meanwhile the

solvent level in the vessel 10 rises again to the point 38, due to the return of solvent from the storage vessel 28 and the introduction of fresh solvent, whereupon the pump 26 starts again and the cycle is repeated. The average rates of introduction of fresh solvent into the vessel 10 and discharge of spent solvent through the overflow 40 are always equal, so if desired the introduction of fresh solvent may vary from time to time or be intermittent without effect on the operation. Preferably the operation should be such that the lower limit 42 in the storage vessel 28 is kept above the upper level 38 in the vessel 10, even if the supply of fresh solvent should fail.

It will be seen that there is a substantially floating column or mass of coffee in the vessel 10, untreated coffee being continuously supplied to the bottom of the column by the injector pipe 12 while treated coffee is continuously discharged from the top of the column by the raking mechanism 22. Because of its inertia, the tendency of the column of coffee to rise and fall with variations in the solvent level is relatively slight, when the upward and downward movement of the solvent is effected at high velocity and for short periods, and consequently the velocity of movement of the solvent through and relative to the mass of coffee is relatively high and the efficiency of extraction is accordingly increased. On the other hand, the extraction is carried out without increasing the quantity of solvent necessary to maintain the desired net forward flow of solvent through the apparatus as determined by the average rate of introduction of fresh solvent into the vessel 10. Moreover, because of the high-velocity alternating movement of solvent maintained in the vessel 10, the amount of solvent used may be reduced if desired without sacrifice of efficiency.

The three forms of apparatus shown diagrammatically in Figs. 2 to 4 are suitable for the extraction of solid materials heavier than the solvent. As in the case of Fig. 1, however, each of these forms embodies a low-amplitude high-velocity alternating movement of the solvent while maintaining a net forward flow through the apparatus. Referring first to Fig. 2, the solid material to be extracted is continuously introduced in any suitable manner into the top of a vertical extraction vessel 43. The solvent enters the bottom of the vessel 43 through a pipe 44 and leaves it at the top through an overflow pipe 45. The vessel 43 has a cone shaped bottom 46 centrally fitted with a pipe 47 joined to an inner cone 48 as a false bottom, the two cones together with the pipe 47 forming a space into which the solvent is introduced through the pipe 44. The solid material is supported in the vessel 43 on the inner cone 48, which is perforated for passage of the solvent through the solid material. A liquid-blocking rotary valve 49 of known construction is connected to the bottom of the pipe 47 for progressive discharge of the solid material. The solid material travels by its own weight through the solvent and finally passes through the pipe 47 into the valve 49, by which it is continuously discharged at the rate of its introduction into the vessel 43.

As in Fig. 1, the upward and downward movement of the body of solvent in the extraction vessel is accomplished by alternately withdrawing solvent from the vessel and returning it to the vessel, but in Fig. 2 this is accomplished independently of the discharge of solvent from the

vessel through pipe 45. Solvent is withdrawn from the extraction vessel by a pipe 50 and an intermittently operated pump 51 and delivered to an elevated storage vessel 52, from which the solvent returns to the extraction vessel by gravity when the pump stops. The operation of the pump can be controlled conveniently by the rise and fall of solvent in the storage vessel. In the form shown, a float operated switch 53 similar to the switch 30 of Fig. 1 controls the pump driving motor and is operated by a float 54 in response to variations of the solvent level in the vessel 52. When the solvent level reaches the desired maximum indicated by dotted line 55, the switch 53 is operated to stop the pump. The solvent then flows out of the vessel by gravity until the desired minimum level indicated by dotted line 56 is reached, whereupon the switch is operated to start the pump and the cycle is repeated. The pump 51 and conduit 50 should be proportioned to provide the desired rate of flow and velocity of movement of the solvent, and the volume of solvent withdrawn and frequency of alternation can be regulated by adjusting the position of the switch blocks 57 and 58.

The rate at which solvent is alternately removed and returned through the conduit 50 should be large in comparison with the rate of introduction of fresh solvent through the pipe 44. The level 59 of spent solvent in the vessel 43 varies between an upper limit 60 determined by overflow pipe 45 and a lower limit 61, the distance between these limits being small as compared to the height of the vessel 43 and being dependent upon the amount of fresh solvent introduced and the amount of solvent withdrawn by the pump 51. Since the volume of solvent withdrawn by the pump is equal to the volume returned by gravity, the amount of spent solvent discharged through the overflow pipe 45 is always equal to the amount of fresh solvent introduced through pipe 44.

Fig. 3 illustrates a U-shaped extraction vessel comprising a horizontal section 62 and vertical sections 63 and 64 each provided with a screw for conveying the solid material which is continuously introduced into the top of vertical section 63 and continuously discharged from vertical section 64 through a chute 65. Fresh solvent is introduced into the top of vertical section 64 through a pipe 66 and spent solvent is removed from section 63 through a conduit 67.

The outlet conduit 67 is connected through a pump 68 to a storage vessel 69 provided with an overflow pipe 70. Controlling the pump 68 is a float operated switch 71 actuated by the level of solvent in a chamber 72 connected to the vertical section 63 by a pipe 73. Rise of the solvent level 74 to a desired upper limit 75 starts the pump 68, while fall of the level 74 to a desired lower limit 76 stops the pump 68. Spent solvent is thus alternately transferred by the pump 68 from the extraction vessel to the storage vessel and returned by gravity from the latter to the extraction vessel. In this operation the level of spent solvent in the vessel 69 varies between upper and lower limits 77 and 78 and to the extent that fresh solvent is introduced through the pipe 66, spent solvent will be discharged through the overflow pipe 70.

Fig. 4 illustrates still another type of extraction apparatus to which the invention may be applied. As shown, the vessel comprises a vertical leg 79 into the top of which the solid material is introduced and a connecting inclined screw carrying section 80 through which the material is conveyed to a discharge chute 81. Fresh solvent is intro-

duced near the upper end of leg 80 through a pipe 22 and spent solvent is discharged from the leg 79 through an overflow pipe 83. As in the case of the apparatus previously described means are provided for establishing an alternating low-amplitude high-velocity movement of the body of solvent in the extraction vessel, such means comprising a pipe 84, pump 85 and storage vessel 86 provided with a float switch 87 controlling the operation of the pump. However, in this case, withdrawal of solvent from the extraction vessel takes place by gravity until the solvent level in the storage vessel reaches the upper limit 88. At this point the float switch 87 starts the pump and solvent is forced back into the extraction vessel until the level in the storage vessel drops to the lower limit 89, when the pump is stopped and the cycle is repeated.

From the foregoing description it will be apparent that the invention provides increased velocity of movement of the solvent in contact with the solid material and accordingly increased efficiency of extraction, without increasing the quantity of solvent required. In cases where it is important to extract the material to the greatest possible extent, as in decaffeinating coffee, for instance, the invention makes possible the extraction of a greater proportion of the caffeine content than has heretofore been practicable within economical limitations of commercial operation. In other cases the invention may be employed to provide more efficient and economical extraction, resulting in savings in time of extraction, quantity of solvent, etc.

It will be understood that the procedures specifically described above, as well as the forms of apparatus illustrated in the drawings, are by way of example only and that other applications of the invention, as well as variations in the details of the method and apparatus, will readily suggest themselves to those skilled in the art. Accordingly reference should be had to the appended claims for a definition of the limits of the invention.

What is claimed is:

1. Apparatus for continuous solvent extraction of solid material comprising an extraction chamber, means for flowing material to be extracted through said chamber, means for feeding solvent to and discharging it from said chamber counter-currently to the flow of said material, means for

alternately withdrawing from and returning to said chamber a portion of flowing solvent comprising a storage chamber connected to said extraction chamber by a conduit, a pump in said conduit, and means actuated by the flow of said solvent into and out of one of said chambers to control the operation of the pump.

2. Apparatus for continuous solvent extraction of solid material comprising an extraction chamber, means for flowing material to be extracted through said chamber, means for feeding solvent to and discharging it from said chamber counter-currently to the flow of said material, means for alternately withdrawing from and returning to said chamber a portion of flowing solvent comprising a storage chamber connected to said extraction chamber by a conduit, a pump in said conduit, and means actuated by the flow of said solvent into and out of said extraction chamber to control the operation of the pump.

3. Apparatus for continuous solvent extraction of solid material comprising an extraction chamber, means for flowing material to be extracted through said chamber, means for feeding solvent to and discharging it from said chamber counter-currently to the flow of said material, means for alternately withdrawing from and returning to said chamber a portion of flowing solvent comprising a storage chamber connected to said extraction chamber by a conduit, a pump in said conduit, and means actuated by the flow of said solvent into and out of said storage chamber to control the operation of the pump.

4. Apparatus for continuous solvent extraction of solid material comprising an extraction chamber, means for flowing material to be extracted through said chamber, means for feeding solvent to and discharging it from said chamber counter-currently to the flow of said material, means for alternately withdrawing from and returning to said chamber a portion of flowing solvent comprising a storage chamber connected to said extraction chamber by a conduit, said chambers being disposed so that solvent flows through said conduit by gravity in one direction, a pump in said conduit for flowing solvent therethrough in the opposite direction, and means actuated by the flow of said solvent into and out of one of said chambers to control the operation of said pump.

NORTON E. HERRY.