

[54] METHOD AND DEVICE FOR FEEDING CRYSTALS AND MELTS

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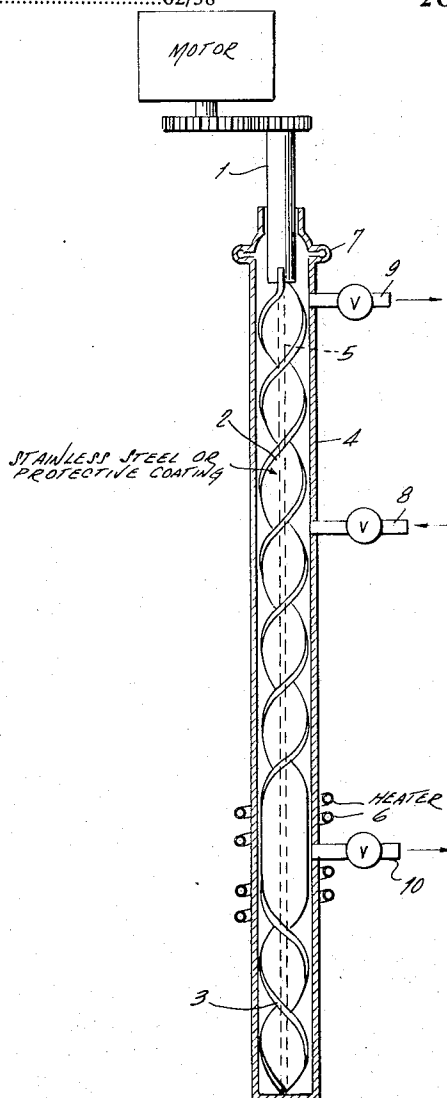
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[57] ABSTRACT

Apparatus for feeding crystals and melts in a crystallization column comprising a rigid strip freely rotatably mounted in the column, the edges of said strip bearing against the inside of the column and the strip being twisted spirally in one direction about its longitudinal axis, at least one end of the strip being connected to a driving means adapted to rotate the strip about its longitudinal axis.

2 Claims, 2 Drawing Figures



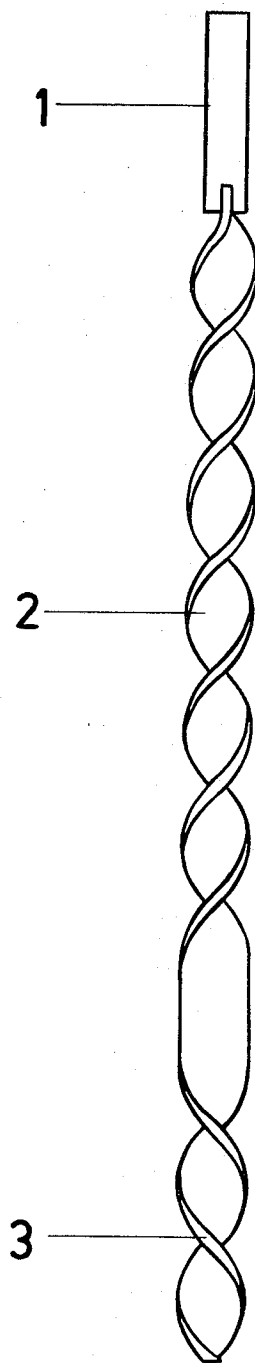


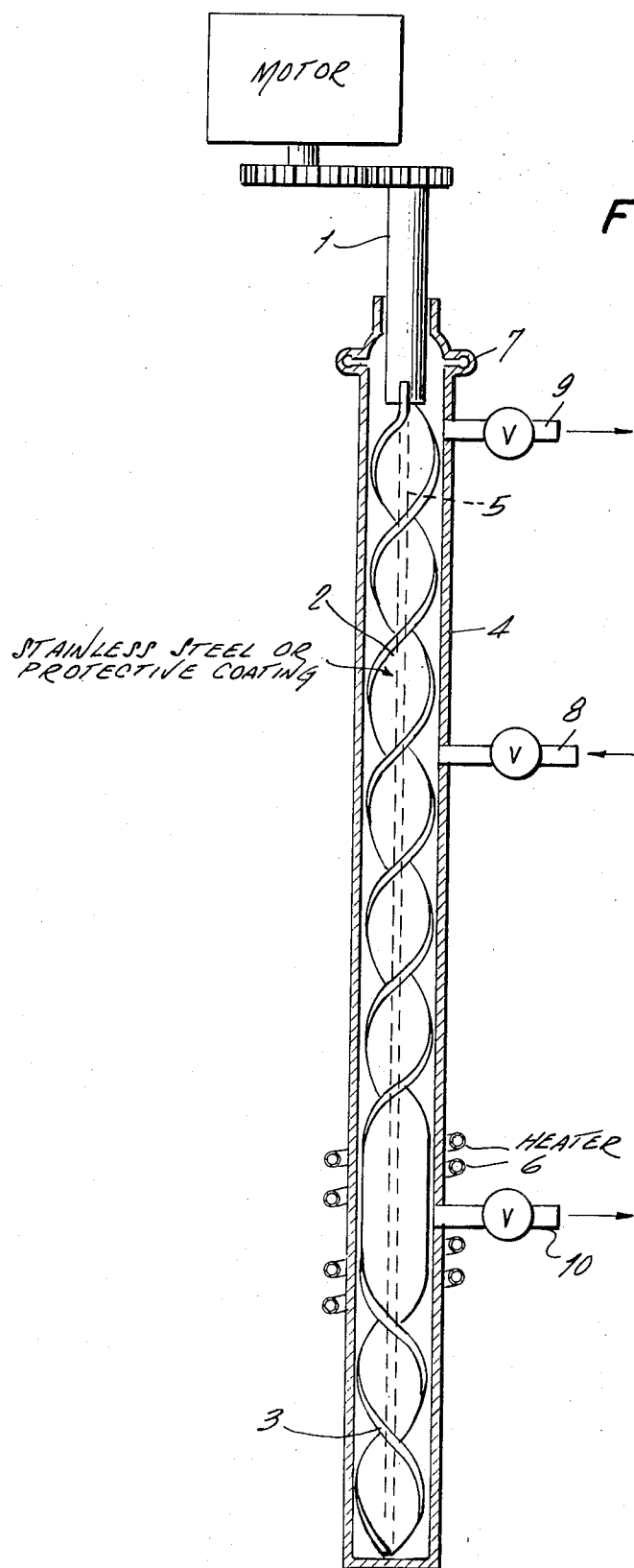
Fig.1

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METHOD AND DEVICE FOR FEEDING CRYSTALS AND MELTS

The present invention is directed to apparatus for feeding crystals and melts in a crystallization column and is more specifically directed to an improvement in the impeller used for this purpose.

It is known in the prior art to carry out crystallization in a vertical column. In such a device a mixture of melt and crystals are supplied to the center of the column and moved between a cooler at the top of the column and a heater at its base. The liquid and solid move in counter-current flow to one another. As a result, the lower melting fraction (which comprises the lower melting mixed crystal component or the eutectic as the case may be) is removed at the top of the column and the higher melting fraction (pure substance or higher melting mixed crystal component as the case may be) is removed from the lower end of the column.

The counter-current of the crystals and melt in this apparatus is carried out by means of a spiral. This comprises a coil spring (preferably of steel wire) with square or lenticular cross section which rotates in the annular space between concentric stationary tubes. These tubes are commonly made of glass but, of course, any other suitable material inert to their contents may be used. An effort was made to revise this design for use on a micro scale but great difficulties arose. Even when amounts as large as 100 mgs were used the feed coil was too weak (due to its necessarily small size) to function adequately. The comparative high stresses produced by accumulating of crystals coupled with the reduction in strength of the coil itself because of its size prevented proper operation of such a device. Moreover, on such a small scale minor variations in heat control tended to cause substantial crystal accumulations. This contributed to the already difficult problem.

It has been found that quantities of crystals and melts of the foregoing order of magnitude (and amounts even much smaller) can be fed safely and successfully in a crystallization column if the column is provided with a feeding means in accordance with the present invention. This comprises a rigid strip twisted spirally about its longitudinal axis. This strip is placed within a cylindrical column and its edges bear against the sides of the column. It is so proportioned that it can turn freely within the column and has one end connected with a driving means causing rotation about the longitudinal axis. It has been found that this apparatus is suitable for amounts as small as 10 mgs. Of course, it can also be used for much larger quantities.

The spiral strip described herein takes the place of the prior art feed spiral. By using this device no center portion of the column is required and thus the construction of the overall apparatus is simplified. Moreover, this potential source of heat dissipation problems is eliminated. The twisted strip as described and claimed herein is much more rigid than the comparable feed spirals and therefore permits much longer columns. On the other hand for short columns lower strength material such as glass may be used. Obviously glass is substantially inert to almost all crystallization materials and would be desirable particularly where high corrosion is an important object. It presents the clear problem due to its lack of flexural strength of high breakability particularly under the kind of stress which would be present in apparatus of this kind and, therefore, workers in the field have looked to special metal alloys in order to obtain the necessary strength and the inert characteristics which are most desirable. The present invention will permit the use of glass because of the increased strength provided by the form in which the strip is inserted.

It has been found that such a strip can be prepared using a width as small as approximately 1 mm and its minimum thickness is then about 0.2 mm. The strip can, of course, comprise any material which is inert to the substances being handled and which is stable at working temperatures. In addition to glass stainless steel is particularly suitable for the manufacture of the spiral strips of this invention. In its simplest method of construction a piece of steel plate can be cut into strips of the desired width and each strip twisted into a suitable spiral.

It should be noted that care must be taken to cut the strips in the rolling direction of the plate.

During the twisting of the strips the rotations are distributed uniformly over the entire length thereof. However, the twistability of steel strips is limited. It has been found that the length of a half turn should at best correspond to the width of the starting strip. Therefore, it is not possible to obtain the small pitches which are possible with the coil springs of the prior art. However, the practical number of turns per unit length are sufficient to achieve excellent separations. The ratio of rotating surface to the stationary column wall is better and consequently the crystal feed is more favorable than with the large feed screws of the prior art. In the case of these prior art designs the turns become clogged after a while by conglomerated crystals and the screws will turn without carrying out any feeding.

In the present device no particular problems arise with regard to maintaining the accuracy of the pitch. This is a further improvement over the prior art coil springs. All dimensions can be determined in advance because there is only a slight variation of the initial dimensions during the twisting process.

In a preferable form of the invention the strips are encased in a protective coat of an inert material such as teflon. This material is easily applied to the strips as the strips do not come in contact during manufacture either with a guide or a core of any kind. A very simple method of coating with teflon can be carried out by pushing unsintered teflon hose over the steel band which is then twisted in the usual manner. The entire completed feeding means is then sintered and the hose fuses to the metal.

In the preferred form of the invention the end opposite that connected to the driving means has one to two turns which are directed oppositely to the other turns. An extended untwisted flat portion of the strip is disposed between the turns of the spiral and the counterturns on the end opposite that connected to the driving means. These counterturns form a crystal stopper during the column crystallization at the point at which the reversal takes place. This has a favorable effect on the separating process. Moreover, this helps to prevent lifting of the feeding device by the crystals accumulated at the bottom of the column.

In the accompanying drawing, constituting a part hereof and in which like reference characters indicate like parts

FIG. 1 is a spiral strip of the present invention; and

FIG. 2 is a view partly in section showing the strip of FIG. 1 within a column.

There is provided a driving rod 1 affixed to one end of the spiral strip. The strip has twists 2 and counterturns 3. In a special embodiment of the invention the strip is hollow and its interior is provided with resistance wire 5 for generating of heat. A simple way to manufacture such strips is the installation of a resistance wire 5 in a flat tube prior to turning. The heated spiral strip often permits the feeding of material which has a tendency to agglomerate.

Moreover, the apparatus may contain in addition to internal heating devices an external heater surrounding the column. There may also be provided cooler 7 preferably adjacent the upper end of column 4. The two heating devices can supply heat of different intensities to the material being handled. It has been found that the separating effect can be improved by this means. However, it is often costly in its use of power. It is, therefore, of primary value on the laboratory scale where these matters are of minor concern only.

Column 4 is provided with feed inlet 8 for the mixture to be separated. The lower melting fraction is withdrawn at top fraction outlet 9 and the higher melting fraction exists at bottom fraction outlet 10.

The following are examples of the invention which are intended to be illustrative and not limitative.

EXAMPLE 1

Twenty-five mg of mixed crystals of 50 percent by weight of azobenzene and 50 percent by weight stilbene are introduced into a crystallization column having an inside diameter of 1 mm, a strip of stainless steel with a usable length of 50 mm rotates within the column. The strip has a cross section of 1 mm $\times$ 0.25 mm with 25 half turns or twists and one counterturn. Rotation is at a speed of 300 rpm. The mixed compounds are crystallized and after a separating period of 5 minutes there appears in the column 15 mm of virtually colorless stilbene, a 5 mm yellow-orange transition zone, 25 mm of red-orange azobenzene and 5 mm of eutectic forming impurities. These impurities are mostly air oxidation products of azobenzene. This separation was deemed satisfactory in all respects.

EXAMPLE 2

The same procedure as set forth in Example 1 was followed except for the following variations: the inside diameter of the column was 2 mm and the steel strip had a usable length of 100 mm. Its cross section was 2 mm $\times$ 0.6 mm, and there were 25 half turns (twists) and 2 counterturns. The strip was rotated at 200 rpm and the separating time was 10 minutes. 200 mg of the mixed crystal substance were introduced and substantially the same results were obtained as with Example 1.

It has been found that in general longer columns require more time for the establishment of the equilibrium. As the column diameter increases (and turn intervals increase also) the optimum rotational speeds diminish.

EXAMPLE 3

In this Example a crystallization column with an inside diameter of 7 mm was used. A strip of stainless steel having a usable length of 1,000 mm, a cross section of 7 mm $\times$ 1 mm, 80 twists and 2 counterturns was rotated in the column at a speed of 100 rpm. The charge was 30 g of technical grade caprolactam (also known as "extract lactam") as a eutectic forming substance and the material is crystallized. After a separating time of 60 minutes 15 cm dark brown impurity concentrate is found at one end of the column. Next to it is 10 cm of brown to light brown transition material and there are 75 cm of pure white caprolactam. This separation is satisfactory for practical use.

EXAMPLE 4

This separation was carried out in a manner similar to that of Example 3 but with the following differences: The inside diameter of the column was 25 mm and it contained a strip

having a useful length of 1,500 mm, a cross section of 25 $\times$ 8 mm and there was 35 twists and 1 counterturn. The strip was rotated at 50 rpm for 60 minutes. The charge was 400 g of the same material as used in the Example 3.

After the indicated separating time a 35 cm dark brown impurity concentrate zone was noted. Next to this zone was a 25 cm brown to light brown transition zone followed by 90 cm of pure white caprolactam. This separation was deemed satisfactory in all respects.

EXAMPLE 5

In this Example a hollow strip containing an internal heating device was used. The current was supplied by sliding contacts and the width of the flat tubes which were compressed from thin tubes was 14 mm, the thickness was 3.5 mm and the usable length was 170 mm. There were 6 twists in this strip. The rotational speeds were between 50 and 100 rpm.

The column was charged with the crystal substance of Example 1 and the operation took place without the necessity of tempering the outer column wall. The response to the power supply was extremely rapid and this system is particularly suitable for column crystallization of substances which crystallize only with great difficulty. It appears that the temperature gradient obtainable between the feeding device and the column wall aids in the crystallization process.

While only a limited number of embodiments of the foregoing invention have been described it is nonetheless to be broadly construed and not limited except by the character of the claims appended hereto.

What is claimed is:

1. A vertical crystallization column apparatus for separating crystals and melt into higher and lower melting fractions comprising: a rigid strip having edges, said strip being freely rotatably mounted in said column, said strip having an upper and bottom section having twists thereon, the twists of one section being directed oppositely to that of the other portion of said rigid strip and an extended untwisted flat portion forming the junction between the twists of the upper section and the twists of the bottom section, such that said higher melting fraction is urged by said upper section toward the untwisted flat portion whereby said untwisted flat portion facilitates separation.

2. An apparatus for feeding crystals and melts in a crystallization column, comprising a rigid strip having edges freely rotatably mounted in said column, said strip twisted spirally in one direction about its longitudinal axis, said strip being connected to a driving means whereby said strip is adapted for rotation about said axis, said strip being a flattened tube, and said heating device comprising a resistance wire in said tube connected to a source of electricity.

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