

PATENT SPECIFICATION (11) 1 582 599

1 582 599

- (21) Application No. 51798/77 (22) Filed 13 Dec 1977 (19)
(31) Convention Application No. 7637581 (32) Filed 15 Dec 1976 in
(33) France (FR)
(44) Complete Specification Published 14 Jan 1981
(51) INT.CL.³ B01D 9/00
(52) Index at Acceptance B1G 10A 13 14A 9D1



(54) A METHOD AND INSTALLATION FOR THE TREATMENT OF CRYSTAL SUSPENSIONS

- (71) We, FIVES-CAIL BABCOCK, a French Body Corporate, of 7, Rue Montalivet, 75383 Paris Cedex 08, France, do hereby declare the invention for which we pray that a Patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—
- This invention relates to a method and installation for treating a suspension of crystals in a solution of the crystallizable product.
- This invention also relates to a method and installation for the production of crystallization nuclei, which can be used in non-continuous or continuous crystallization apparatus.
- A crystallization apparatus intended to employ the mother liquor efficiently and to produce crystals of a specific size, should initially be supplied with crystallization nuclei in suitable numbers for ensuring the carrying out of these conditions. When the apparatus operates with continuous production, the number of nuclei introduced should be proportional to the output of the apparatus.
- This first crystallization phase with introduction of nuclei and the beginning of their enlargement is difficult to carry out in view of the very small size of the nuclei introduced and the risk of considerable variations in the initial population of crystals by local re-melting of fines (crystals of very small size) or by agglomerations.
- This risk is particularly felt in a last sugar-making crystallization strike in view of the low purity of the syrups which are treated, their high viscosity, and the corresponding low speed of crystallization.
- Frequently, in non-continuous crystallization, this difficulty is reduced by carrying out the operation in two stages:
- In a first stage, starting from crystallization nuclei, crystals are obtained of a sufficient size to ensure that they retain their identity in the following operations (in the order of 100 to 180 μ). For this stage there is used a syrup of higher purity (for example first strike or refining syrup instead of second strike syrup for a 3-strike layout) and the operation is carried out by starting with an excess quantity of nuclei and producing deliberate partial remelting during the cycle to return in a rather empirical way to the intended population of crystals; this method of operation does not obviate the existence, sometimes, of insufficient crystal populations, resulting in the formation of fines in the second stage of crystallization.
- In a second stage, the massecuite obtained from the preceding stage known as the footings, is used as nuclei for the remainder of the crystallization operation, the crystallizer being supplied by the normal syrup which is to be employed. (in the preceding case, the second strike syrup).
- This procedure has the disadvantage of requiring the introduction at the level of the last crystallization strike of a relatively pure syrup (to constitute the footings) and hence there is less good utilisation of the suspension issuing from the crystallization room, other things being equal. Furthermore, such a method is difficult to reproduce in a reliable manner in an entirely continuous process, bearing in mind the difficulties referred to hereinbefore of controlling the first stage of the crystallization (enlarging the nuclei to 100 to 180 μ).
- It is well known that in order to obtain optimum exhausting of the suspension it is necessary to have a high population of crystals, to increase the crystallization surface and reduce the average distance between crystals; on the other hand the smaller crystals have to be sufficiently large so as not to run the risk of remelting in the treatment cycle, and so as to limit the grain size and dispersion of the crystals obtained. These two requirements control the lower and upper limits of the grain size range of the crystals produced according to the invention.
- According to one aspect of the present invention there is provided a method of treating a suspension of crystals in a solution of crystallizable product which method comprises subjecting a suspension in a portion of a solution of the crystallizable product of crystals produced in a crystallization apparatus to a centrifugal separation during the course of which crystals whose size is larger than a predetermined maximum dimension are separated from the suspension and from smaller crystals, the crystals separated from the suspension being broken up and then recycled into the said suspension

before it is subjected to the separation.

The method according to the present invention generally uses the breaking up and screening of crystals by a wet process after they have
5 been put in suspension in a saturated solution to form a magma.

Very preferably, the suspension is subjected to a second centrifugal separation during which crystals, whose size is smaller than a predetermined minimum dimension, are removed together with a liquid phase of the suspension and the crystals which are separated from the suspension during the second separation are used as crystallization nuclei for a crystallization apparatus. The first separation is carried out in a first separator generally by passing the magma over a screen with large openings. The liquid phase which carries along the crystals of smaller size is directed to a second separator which
10 effects the second separation with a screen with smaller openings. The oversize of the first separator is subjected to breaking up and is then recycled. The oversize of the second separator constituted by selected crystals is put into suspension again in a saturated solution to be used as crystallization nuclei. The liquid residue of this separator containing fines is put in a state of sub-saturation by addition of water and re-heating and is then recycled.

The breaking up of the larger crystals is effected selectively. The crystals are in fact slowed down during their travel by friction in the air. This slowing-down is more considerable in proportion as the specific surface area of the crystals is larger, and therefore in proportion as its size is smaller. From a given initial speed and a selected travel distance it is possible to gather the small crystals without breaking them, whilst the large crystals retain a speed of impact capable of causing them to break up.

The preparation of the magma which is to be subjected to two separating stages and of the seeding magma is carried out in mixers which are advantageously provided with systems for regulating the crystals content of the magma and level regulating devices.

The following description refers to the accompanying drawings which illustrate exemplary embodiments of the invention and wherein:

50 Figure 1 shows a diagram of an installation for carrying out the method proposed by the invention used with continuously operating crystallizer;

55 Figure 2 shows a diagrammatic view of another installation for carrying out the method according to the invention when applied to a crystallizer with non-continuous operation;

60 Figure 3 shows a section through a separator used in the installation shown in Figures 1 and 2; and

Figure 4 shows a section through another separator used in the installation shown in Figures 1 and 2.

65 The diagram shown in Figure 1 shows the principle of continuous crystallization of a

third strike in sugar making with continuous addition of selection crystallization nuclei. It will be understood that the details of the diagram used for the understanding of the method, are not intended to have any limiting character, 70 likewise the nature of the product treated or the type of apparatus described in the present specification. To mark the general character of the method, the sugar making terms are indicated in brackets in the following description. 75

The diagram of Figure 1 shows a continuous crystallization apparatus 31 (third strike continuous boiling) supplied at 32 by the solution which is to be treated (second strike low purity molasses) and at 33 by a suspension of crystallization nuclei (magma 3). In the apparatus 31, the solution is kept in supersaturation by evaporation under a vacuum, which results in the deposit of the crystallizable material, the latter assembling on the crystallization nuclei which become larger and larger. The suspension of crystals (third strike massecuite) is extracted at 34. The crystallization is continued by cooling in a mixer 35. Separation is carried out in a continuous centrifugal separator 36 which gives on the one hand a liquid phase 37 (molasses) and also crystals 38 (third strike sugar). These crystals fall into a paste-forming mixer 39 in to which there is poured at 40 a solution which is or the same purity as that introduced at 33 into the crystallizer (second strike low purity molasses) to form a suspension (magma 2). This suspension is either removed at 41 to obtain refined crystals or used to obtain crystallization nuclei at a higher stage of purity (second strike continuous boiling). A portion of the suspension is taken off by a volumetric pump 42 to be directed towards a paste-forming mixer 43, and to be mixed therein with a solution introduced at 44, of the same kind as that used in the previously mentioned mixer 39, and the crushed products 45 issuing from a continuous centrifugal separator 46, which will be described hereinafter with reference to Figure 3. This separator is designed in order to carry out the shock breaking up of the crystals which are too large to pass through the openings of the separator screen.

In the case of sugar making installations where remelting of the third strike is carried out directly (in the particular case of beet sugar making) there will be introduced directly into the paste-forming mixer 43 a controlled flow of sugar crystals issuing from the separator 36, before remelting. 120

The suspension obtained in the mixer 43 is taken up by a volumetric pump 47, to be directed towards the separator 46. The crystals passing through the openings of the screens of this separator are removed at 48 with the liquid phase and brought to a second continuous centrifugal drier 49 shown in Figure 4. The crystals whose sizes are larger than those of the openings of the screen of the separator 49, are removed at 50. These are crystals whose sizes are width 130

the fixed range. They fall into a paste-forming mixer 51 which receives at 52 a solution of the same purity (second strike syrup) as that which is to be exhausted by crystallization. The magma thus constituted (magma 3) is taken up by a volumetric pump 53 to be injected into the continuous crystallization apparatus 31 (third strike boiling). The crystals of smaller sizes than the openings of the screen of the separator 49 contained in the separated suspension 54 are remelted by injecting a small proportion of water at 55 and reheating at 56, bringing the suspension to subsaturation; the solution obtained is recycled into the mixer 39.

In the industrial realisation of this layout, the operation of the installation can be facilitated by automatic regulation. By way of non-limitive example we shall describe hereinafter the best method of regulation which would be suitable for this use.

The content of crystallization nuclei of the magma in the mixer 51 is measured by a conductivity meter 61. The measurement is transmitted to a regulating device 62 which actuates a valve 63 situated on the conduit 52 so as to keep this content equal to a fixed reference value.

By manual control 64 acting on the speed change gear 65 the flow of suspension of nuclei (magma 3) taken from the mixer 51 is regulated so as to obtain in the crystallizer the population of crystals corresponding to the rate of operation imposed. A level measurement 66 acting on the regulating device 67 controls a change speed gear 68 which adjusts the delivery of the pump 42 taking the suspension of crystals (magma 2) from the mixer 39. A measurement of conductivity 69 acting on the regulating device 70 regulates the throughflow of a valve 71 placed in the conduit 44 so as to keep the content of crystals of the magma in the mixer 43 constant.

Figure 2 shows an installation similar to the preceding installation for a crystallization apparatus with non-continuous operation 31'. In this case the flow taken off by the pump 42' is adjusted by the speed change gear 68' controlled from a distance by a switch 64' so as to constitute a reserve of nuclei suspension (magma 3). The level of the mixer 51' is indicated at a reading device 66. The pump 53' is started up each time it is necessary to take off magma.

The separator 46 shown in Figure 3 comprises a conical basket 1 rotating at a high speed, and in this basket the suspension of product of be separated arrives by way of the supply tube 2. The liquid phase passes through the screen 3 the size of whose openings is selected to allow passage the upper cut of the selected crystals which are thus carried along with the liquid. This product circulates on the wall of the basket and is then ejected through the orifices 4 arranged at the periphery of the basket. It is collected in a circular container 5 and is discharged through an outlet pipe 6. The solid phase is ejected substantially at the tangential

speed of the basket above the strap 7, to be projected on to a metal sleeve 8. A number of sleeves of different diameters are provided in order to make it possible to select a travel length in accordance with the products which are to be dealt with, the sleeve 9 shown in broken lines being the sleeve of larger diameter. The peripheral speed of the basket and the travel length are selected so that the largest crystals which are projected arrive at the sleeve with such a speed that they break up by shock effect and the small crystals which are slowed down to a greater extent by friction in the air do not break up or are subjected only to partial erosion. The products, even energetically centrifuged, are sticky in the pulverised state; in order to prevent accumulation of products, which would modify the crystal break-up conditions, the sleeve is cleaned by rotating scrapers 10.

The number and the rotational speed of these scrapers are such that the shock conditions are somewhat variable. It would be quite possible and in accordance with the present invention to receive the product on a rotating sleeve acted upon by fixed scrapers. The crushed products fall into the annular chamber 11 to be discharged by way of the orifice 12. The scrapers are mounted on supports 13 fixed in an operating position on the plate 14 which drives them in rotational movement. The driving shaft 15 of this plate is hollow in order to allow the passage of the supply tube; it is guided in rotational movement by a bearing 16 integral with the cover of the separator, and driven in rotational movement by a transmission 17 and a geared motor unit 18.

The separator 49 shown in Figure 4 is of conventional construction and will not be described in detail. It receives the separated liquid from the separator 46 which contained crystals whose dimensions are smaller than those of the openings of the screen of the latter apparatus. The screen 18 of the separator 49 has smaller openings. The crystals whose sizes are smaller than those of these openings are removed with the liquid phase by way of the pipe 20. The larger crystals, which are selected to constitute the crystallization, are collected in the tank 21 and discharged by way of the orifice 22.

By way of example, in the case of a third strike in sugar making it is possible to fix lower and upper dimensional limits of the crystallization nuclei at 0.050mm and 0.125mm respectively the drier 46 will have a screen with apertures of 0.130mm and the drier 49 a screen with apertures of 0.060mm. If a volumetric enlargement of 15 to 16 is allowed in the crystallization apparatus, the crystals of the massequite produced will have dimensions of between 0.125 and 0.315mm and a mean width of 0.220mm.

WHAT WE CLAIM IS:—

1. A method of treating a suspension of

70

75

80

85

90

95

100

105

110

115

120

125

130

crystals in a solution of the crystallizable product which method comprises subjecting a suspension in a portion of a solution of the crystallizable product of crystals produced in a crystallization apparatus to a centrifugal separation during the course of which crystals whose size is larger than a predetermined maximum dimension are separated from the suspension and from smaller crystals, the crystals separated from the suspension being broken up and then recycled into the said suspension before it is subjected to the separation.

2. A method according to claim 1, wherein said suspension is subjected to a second centrifugal separation during which crystals whose size is smaller than a predetermined minimum dimension are removed together with the liquid phase of the suspension and the crystals which are separated from the suspension during the second separation are used as crystallization nuclei for the seeding of a solution of a crystallizable product in a crystallization apparatus.

3. A method according to claim 2, wherein the crystals contained in the said suspension after the second separation are dissolved therein by heating and/or dilution and the solution obtained is recycled and used to form the said suspension.

4. A method according to claim 2 or 3, wherein the crystals which are separated from the suspension during the second separation are mixed with another portion of the said solution to form a seeding magma for introduction into a crystallization apparatus in a measured quantity.

5. A method according to claim 2 or 3, wherein the percentage of crystals in the said suspension is kept constant, the production of crystallization nuclei being effected continuously for supply to a continuously running crystallization apparatus.

6. A method according to claim 2 or 3, wherein the production of crystallization nuclei in the form of a seeding magma is effected continuously and the seeding magma is temporarily stored in a mixer for subsequent supply to a discontinuously running crystallization apparatus.

7. A method of treating a suspension of crystals in a mother liquor, as claimed in claim 1 and substantially as hereinbefore described.

8. A method of producing crystallization nuclei for the seeding of a solution of a solution of a crystallizable product in a crystallization apparatus, substantially as hereinbefore described with reference to Figure 1 or Figure 2 of the accompanying drawings.

9. An installation for treating a suspension of crystallizable product, which comprises suspension forming means for forming in a portion of a solution of crystallizable product a suspension of crystals produced in a crystallization apparatus, and a centrifugal separator adapted to separate from said suspension formed by said suspension-forming means crystals larger than a

predetermined maximum size, said centrifugal separator comprising a rotary basket arranged to receive therein said suspension and equipped with a screen through the openings of which the liquid phase of the suspension can pass together with crystals whose diameters are smaller than said predetermined maximum size, a metal sleeve concentric with the basket and surrounding an outlet end thereof so that crystals which are larger than said predetermined maximum size are ejected at the outlet end of the basket and are projected onto this sleeve by the centrifugal force and broken up by the shock effect, and scrapers for cleaning the surface of the sleeve exposed to the impact of the crystals, recycling means being provided for recycling the broken up crystals to said suspension forming means.

10. An installation according to claim 9, wherein the said separator is designed to receive sleeves of different diameters.

11. An installation according to claim 9 or 10, wherein the scrapers of the said separator are mounted on rotary arms the axis of rotation of which is identical with that of the basket.

12. An installation according to claim 9, 10 or 11, which also comprises a mixer for forming therein the suspension of crystals and for supplying the suspension to the said centrifugal separator, the outlet for the crystals of the said separator being connected to an inlet of the mixer.

13. An installation according to claim 12, further comprising a second centrifugal separator for receiving the suspension of crystals which has been filtered through the screen of the first separator, the openings of the screen of the second separator being smaller than those of the screen of the first separator, and a second mixer for receiving the crystals separated from the suspension in this second drier and for forming therein a seeding magma, the outlet of this second mixer being connected to a variable-delivery pump for supplying the seeding magma to a crystallization apparatus.

14. An installation according to claim 12 or 13, characterised in that each mixer comprises an element for measuring the crystals content of the product contained in the mixer and a regulating device for controlling a valve situated in a pipe for supplying the mixer with solution in accordance with the indications received from the measuring element in order to keep the content of crystals in the product equal to a reference value.

15. An installation according to claim 12, wherein the second mixer comprises a level regulating device for controlling the supplying of the first mixer.

16. An installation for treating a suspension of crystals in a solution of the crystallizable product, as claimed in claim 9, and substantially as hereinbefore described.

17. An installation for producing crystallization nuclei substantially as hereinbefore de-

5

10

15

20

25

30

35

40

45

50

55

60

65

70

75

80

85

90

95

100

105

110

115

120

125

130

scribed with reference to Figure 1 or Figure 2 or as modified by reference to Figure 3 and/or Figure 4 of the accompanying drawings.

5 18. A crystallization apparatus incorporating an installation as claimed in any of claims 9 to 17.

10 19. A crystallization apparatus substantially as hereinbefore described with reference to Figure 1 or Figure 2 of the accompanying drawings.

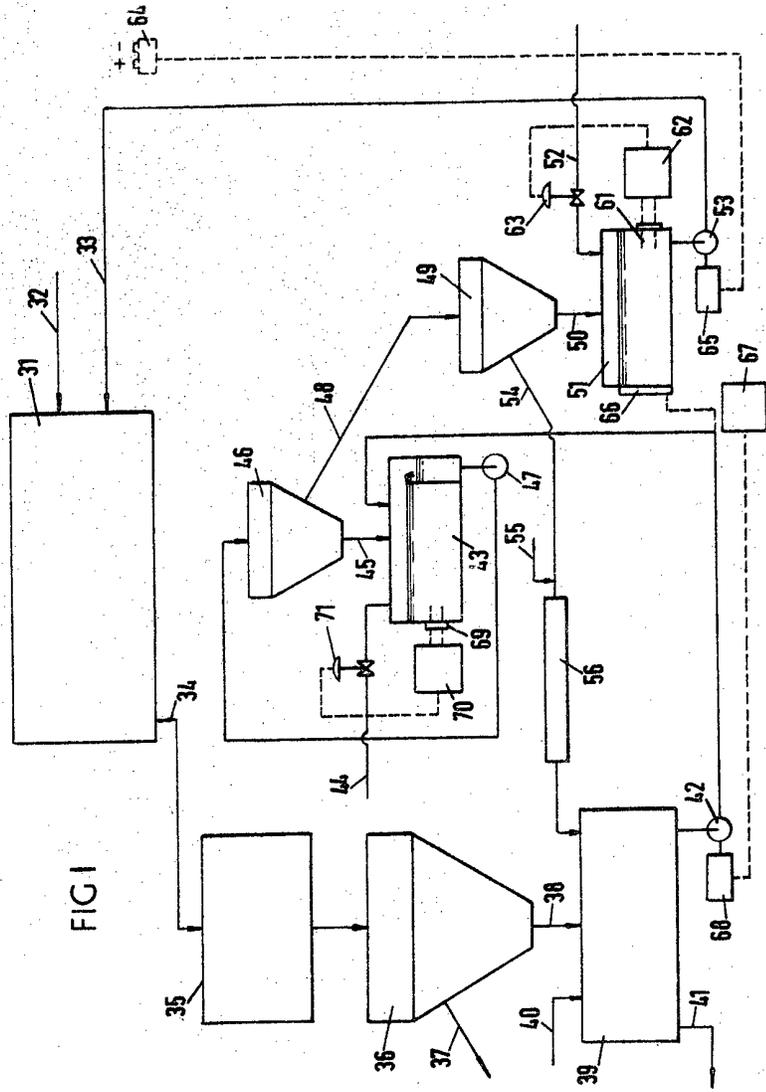
20. Crystals when obtained from a crystallization apparatus as claimed in claim 17 or 18.

21. Crystals according to claim 20 and composed of sugar.

15

20

STANLEY, POPPLEWELL,
FRANCIS & ROSS
Chartered Patent Agents
1 Dyers' Buildings
Holborn
London EC1



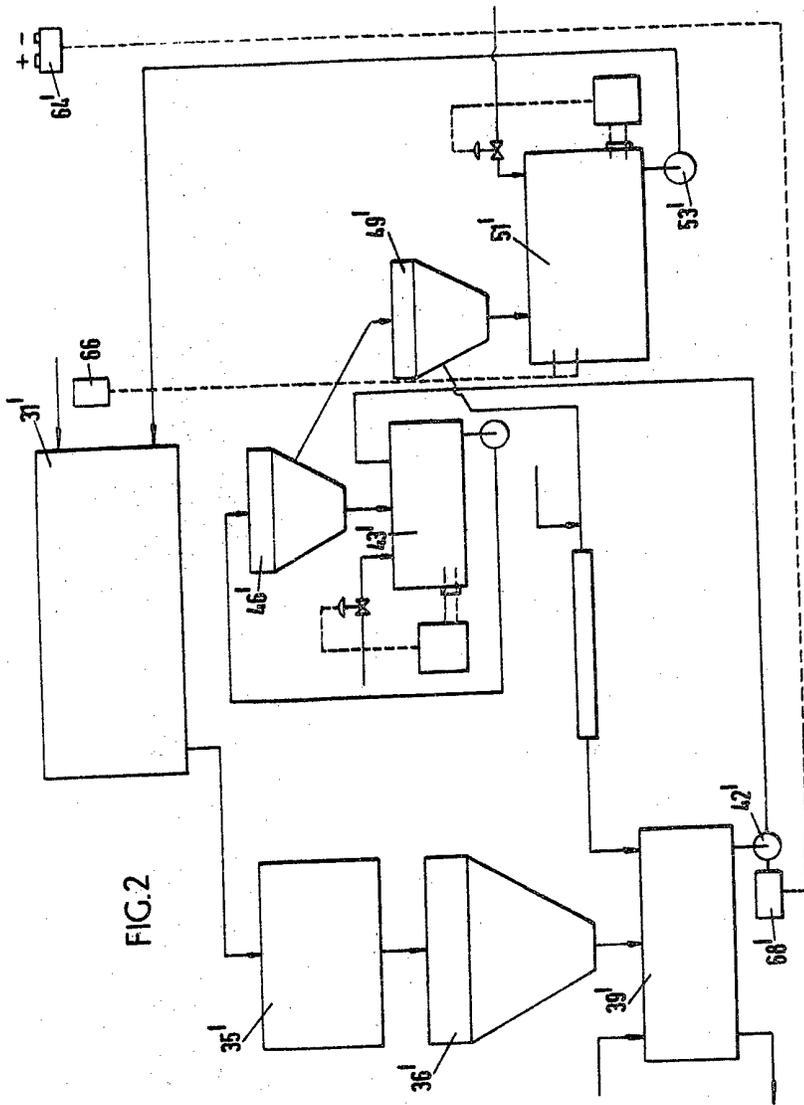


FIG. 2

1582599

COMPLETE SPECIFICATION

4 SHEETS

This drawing is a reproduction of
the Original on a reduced scale
Sheet 3

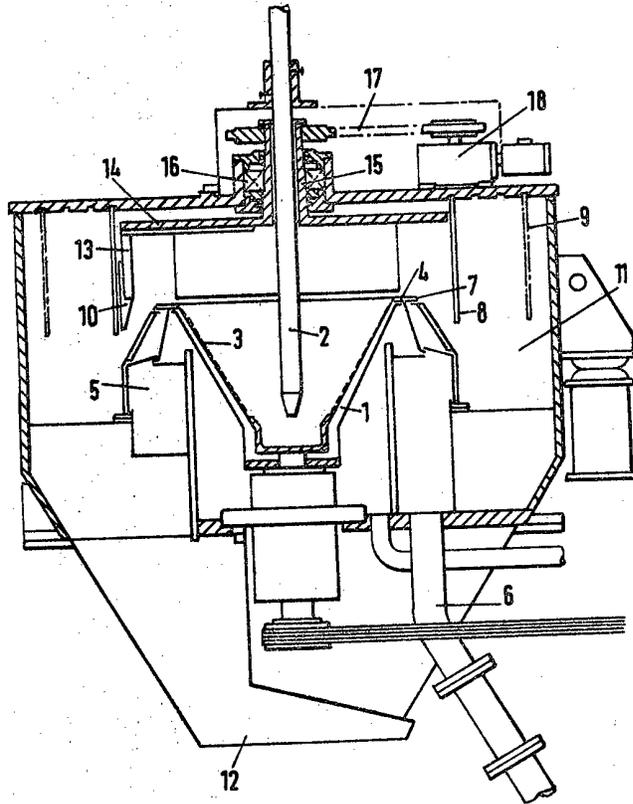


FIG. 3.

1582599

COMPLETE SPECIFICATION

4 SHEETS

*This drawing is a reproduction of
the Original on a reduced scale
Sheet 4*

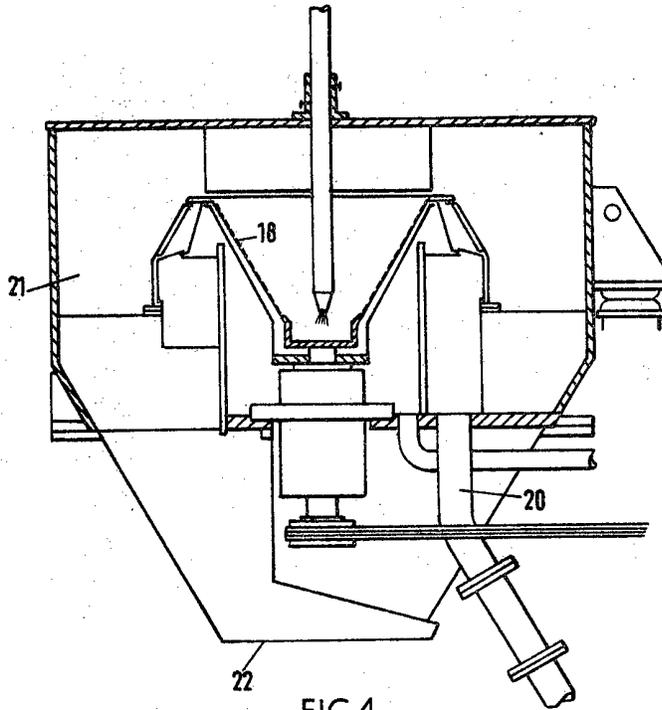


FIG. 4.