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PATENT SPECIFICATION

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(54) THE SEPARATION OF ISOTOPES

(71) We, KRAFTWERK UNION AKTIENGESELLSCHAFT, of Wiesenstrasse 35, 4330 Mülheim (Ruhr), Germany (Fed. Rep.), a German body corporate, 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 the separation of isotopes.

The separation of originally gaseous mixtures of isotope components, more particularly isotopic compounds, can be effected by utilising the selective excitation of one isotope component, which is rendered possible at very low temperatures, by electromagnetic radiation of suitable wavelength and energy density, wherein the excited compound is thus enabled to undergo chemical reaction with a reactant supplied thereto. Such a process is proposed, for example, in German Offenlegungsschrift 2 447 762. In the process described in this Offenlegungsschrift a gasesous isotopic substance is mixed together with a gaseous reactant, adiabatically expanded in common therewith and then one isotope component is subjected to selective excitation, preferentially as compared with other isotope components, employing laser radiation of an appropriate wavelength, before condensation is allowed to take place. For the process to be economic, it is necessary for as much as possible of the incident radiation to be efficiently used. Hence, it is desirable for the isotope component which is to be excited to have a good absorption coefficient in relation to the laser radiation used. This means that the effectiveness of isotope separation by this process depends both upon the availability of a laser wavelength at which the absorption of the isotope components to be separated is highly selective, and upon the presence of a sufficiently high absorption coefficient in the isotope component to be excited at this wavelength.

According to one aspect of the present invention there is provided an isotope separation process for the preferential removal of one isotope component as compared with a second isotope component from a gaseous isotopic substance, wherein a quantity of the said gaseous isotopic substance and at least a stoichiometric quantity of a gaseous reactant are expanded separately from one another adiabatically, or substantially so, to attain temperatures below 100 K, the expanded isotopic substance mixes with the expanded reactant in a condensation chamber cooled to below 100 K, condensation of the reactant takes place in the chamber to form solid particles in which molecules of the said isotopic substance are incorporated, and the said solid particles are collected and subjected to selective excitation employing electromagnetic irradiation at an excitation frequency of the said one isotope component whereby that component is enabled (preferentially as compared with the second isotope component) to undergo chemical reaction with the reactant, whereafter the resulting solid material is subjected to a treatment for separating out a product of the chemical reaction which product is relatively rich in the said one isotope component as compared with the second isotope component.

In an embodiment of the invention the gaseous isotopic substance and reactant are mixed together at such a low temperature that substantially no reaction occurs before irradiation. Since the isotopic substance is in the solid state during irradiation it is possible to inject the radiation at frequencies having low extinction coefficients in the isotopic compound to be excited, because, in contrast to the gaseous state, a high density of material is present, so that as a whole high absorption of the light quanta, and hence a relatively high selective excitation propability can still be

expected. In the case of the isotopic susbtance UF_6, the visible spectrum lies in the wavelength range 310 nm$\leq\lambda\leq$410 nm, but wavelengths in this range which have sufficient selectivity also have low extinction. Alternatively, of course, combined vibrational states in the infra-red range which also often have low extinction can be used. An example of such a vibrational state is $3\nu_3$, where ν_3 is a fundamental vibration of UF_6.

The basic course of the process is as follows, with reference to uranium hexafluoride as the isotopic substance to be separated. The UF_6 and reactant gas are cooled separately from one another by substantially adiabatic expansion. The molar ratio of UF_6 to the said reactant gas, which may be hydrogen bromide or hydrogen iodide, is preferably in the range from 1:1 to 1:1000. The expansion conditions are chosen such that, after expansion, a temperature is attained at which substantially no reaction occurs between the gases upon mixing, and the reactant becomes supersaturated and condenses on cooled surfaces in the chamber. An auxiliary gas may be mixed with the UF_6 prior to expansion to increase the effective adiabatic coefficient of the UF_6. The ratio of the admixed auxiliary gas to the UF_6 may be in the range from 1:1 to 1:10. The reactant is expanded separately through a nozzle which may be a ring or slit nozzle surrounding a central nozzle through which the UF_6 is expanded. The nozzles are designed such that the relative velocity of the two gas streams is as low as possible. Sufficient space is provided in the chamber for the gases to intermix completely. During or after this intermixing, the reactant gas condenses to form solid (e.g. crystalline) particles on cooled surfaces within the chamber. The UF_6 is incorporated in or added to the matrix of the condensing reactant. If an auxiliary gas is being used, it is desirable for the quantity of auxiliary gas incorporated with the UF_6 in the condensing reactant to be low in order that the selectivity of the excitation process may not be disturbed to an admissible extent and the desired photoreaction between UF_6 and the reactant may not be hindered. For example, helium is suitable as an auxiliary gas because it is only sparingly incorporated.

Since the axiliary gas is used only as an aid in the improvement of the adiabatic expansion, but otherwise does not participate in the chemical reaction, such a gas need not be added in the case of substances which already have a sufficiently high adiabatic coefficient.

The solid particles thus produced are irradiated using radiation at a resonant frequency of the isotope component to be separated which has sufficient selectivity, so that a light-induced chemical reaction proceeds between the said first isotope component and the reactant. In order to ensure maximum possible effectiveness of the irradiation over as far as possible all the reaction products, the solid particles may be introduced before the irradiation into moulds, or moulded into bodies, of such shape that scattered or reflected radiation is again directed on the substance to be irradiated. Irradiation of the solid particles may take place in the condensation chamber, or in an irradiation chamber separated therefrom. Separation of that reaction product relatively rich in the said first isotope component from remaining substances may take place, for example, by fractional distillation. The fractional distillation may be carried out in the irradiation chamber (if present) or in a further chamber which should be provided with means for adjusting temperature and degree of vacuum therein. This treatment preferably takes place at such low temperatures that non-selective thermal reactions between the UF_6 and the reactant do not have any disturbing effect, in vacuum-tight chambers. To adjust the temperature of the solid particles or moulded body as is necessary for the accurate maintenance of distillation conditions, there may be applied, for example, a regulatable capacitive high-frequency treatment. As compared with other thermal control means, this renders possible a substantially uniform introduction of heat throughout the volume of the powder or moulded body.

According to another aspect of the present invention, there is provided apparatus for use to effect preferential removal of one isotope component as compared with a second isotope component from a gaseous isotopic substance, comprising a condensation chamber provided with cooling means, for maintaining it at a temperature below 100 K, and with expansion nozzles through which a quantity of the said gaseous isotopic substance and at least a stiochiometric quantity of a gaseous reactant can be expanded respectively, adiabatically or substantially so, into the chamber to attain temperatures below 100 K so that the expanded isotopic substance mixes with the expanded reactant and condensation of the reactant takes place in the chamber to form solid particles in which molecules of the said isotopic substance are incorporated, which particles are collected in the chamber, means for subjecting the collected solid particles to

selective excitation by electromagnetic irradiation at an excitation frequency of the said one isotope component so as to enable that component (preferentially as compared with the second isotope component) to undergo chemical reaction with the reactant, and means for separating out a product of the chemical reaction, which product is relatively rich in the said one isotope component as compared with the second isotope component.

For a better understanding of the present invention, and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying diagrammatic drawings, in which:—

Figure 1 shows a vertical section through one form of apparatus for putting an embodiment of the present invention into effect;

Figure 2 shows a vertical section through another form of such apparatus; and

Figure 3 shows to a larger scale a horizontal section through a part of the apparatus of Figure 2.

In the Figures, like parts are denoted by like numerals.

There is diagrammatically illustrated in Figure 1 apparatus comprising a storage chamber 1 for the gaseous isotopic substance to be separated, with which an auxiliary gas may already have been admixed. This gas, or mixture of gases, is expanded through a nozzle 31 into a cooled condensation chamber 3. Situated in a storage chamber 2 is a reactant gas, for example hydrogen iodide, which is expanded through nozzles 32 into the condensation chamber 3. The nozzles 32 may consist of a plurality of individual nozzles, or they may be in the form of a ring-shaped nozzle. The gases entering the condensation chamber 3, which have undergone considerable supercooling owing to the substantially adiabatic nature of the expansion, mix together therein. By adjustment of pressures in the storage chambers 1 and 2 and in the condensation chamber 3, and by appropriate design of the nozzles 31 and 32, it is arranged that gas streams in the condensation chamber have substantially equal velocities. This is because an appreciable relative velocity between the gaseous isotopic substance and reactant may cause undesirable, i.e. non-selective, chemical reactions to occur during the mixing operation.

A stream of the mixed materials thereafter passes through treatment chamber 35 to slowly rotating cylinders 5 maintained at low temperature, and condensed reactant is deposited on these cylinders as layers 51 having molecules of the isotopic substance incorporated therein. The temperature of the layers is made so low, for example 20 K, by cooling the cylinders that subsequent irradiation permits selective excitation of a reaction of the said one isotope component with the reactant. The cooling medium (not shown) is used to dissipate the kinetic energy of the gases which is converted into heat on impact with the cylinders, and the latent heat of condensation. By rotation of the cylinders the solid layers 51 are passed through laser beams 4, which are adjusted with regard to the direction of impingement of laser radiation so that each layer is permeated by radiation over the whole coated length of the respective cylinder. The speed of rotation of the cylinders is adjusted so that the layer thickness deposited per revolution is of such magnitude that, on the one hand, molecules of the said one isotope component situated adjacent the cylinder receive a sufficient dose of radiation, whilst on the other hand the proportion of radiation absorbed is as high as possible. In order to achieve this, it is desirable for the surfaces of the cylinders to have good reflective properties.

By means of this irradiation, the said one isotope component is selectively excited, whereby it is enabled to undergo chemical reaction with the reactant, for example hydrogen iodide. Hence, the original first and second isotope components should not, after the reaction, be present in like compounds but at least to some extent as different compounds.

The chemical reaction resulting from laser radiation in this example proceeds in accordance with the following equation:

$$UF_6+HI \rightarrow UF_5+HF+I$$

The excited first isotope component (in this example 235-U) is therefore then present preferentially as UF_5. Under the conditions described hereinabove UF_5 is a solid which like UF_6 evaporates only at substantially higher temperatures.

The layers 51 are thereafter mechanically scraped off the cylinders by means of scrapers 52, and fall in powder form to the base of the container which is closed by a vacuum valve 61.

Situated below the vacuum valve 61 is a vacuum chamber 6 into which the powder 51 can be discharged by opening the valve 61. Fractional distillation of the powder is then performed in this vacuum chamber 6. For this operation, there are provided two capacitor electrodes 62 which are connected to a high-frequency generator (not shown). When the generator is switched on, a substantially uniform heating of the powder 51 situated between the electrodes takes place. First the

reactant hydrogen iodide and HF produced in the reaction are evaporated off at a temperature of about 170 K, and pass through extraction ducts 74 and 75 and through a pump 66 to a collecting vessel 67. A separator 63 for removing any entrained UF_6 and UF_5 is provided between the ducts 74 and 75. UF_5 and UF_6 evaporate only at substantially higher temperatures, so that during evaporation of HI and HF no reactions which might disturb the selectivity of the chemical conversion should take place.

The actual separation of UF_6 from the UF_5 which is relatively rich in the said first isotope component, and iodine can be achieved simply by pumping off or recondensing the UF_6 and I_2 from the chambers 73 and 63 at relatively high temperatures because of large differences in the vapour pressures of these substances. UF_6 can be recondensed into a vessel 64 from the vessels 73 and 63 at a temperature of 250 K, and iodine can be recondensed into a vessel 65 from the vessels 73 and 63 at a temperature of 310 K. The vessels 64 and 65 are maintained at a temperature about 190 K.

The removal of U-235 enriched uranium in the form of UF_5 which has remained in the vessels 63 and 73 may be achieved by passing fluorine gas from a storage container 76 into the vessels 63 and 73 whereby the UF_5 is converted into UF_6 and condensing the UF_6 thus formed in a vessel 77. Alternatively, the UF_5 may be pumped out of the vessels under a suitable pressure.

In this apparatus, the streams from the nozzles 31 and 32 impinge upon the cylinders 5 at a relatively high velocity and their kinetic energy is reconverted into thermal energy. This energy must be dissipated by appropriate cooling of the cylinders 5, which ultimately involves additional expenditure of energy and money. The apparatus shown in Figure 2 has been designed with a view to reducing this expenditure.

The fundamental difference between this apparatus and that illustrated in Figure 1 is that the nozzles 31 and 32 are not disposed in a stationary vessel wall, but at the periphery of a rotatable disc-like drum 33 of uniform strength (giving a uniform distribution of forces during rotation). The nozzles 31 and 32 are so mounted that the gas streams issue tangentially and tend to accelerate the drum 33, or at least to maintain it at a very high speed of revolution, due to recoil forces thus produced. An electric motor 50 is provided and can assist this movement or effect a regenerative breaking and thus be used to regulate the rotation and to monitor it by measuring current or voltage absorbed or supplied. The speed of rotation in a run-in condition is so high that the velocity of the gas streams relative to the wall of the chamber 35 is low and, on impingement on a stationary cooled collecting surface 54 of substantially U-shaped cross-section, disposed in outwardly surrounding relationship to the path of the nozzles during rotation thereof, and concave towards that path, secured in the chamber 35 by stays 39, substantially no significant amount of kinetic energy is converted into heat. Hence cooling means provided for the wall 54 do not need to be as efficient as those provided for the cylinders 51 in Figure 1.

Condensation products formed in the chamber are thrown on to the wall 54 and form a coating 51 which is removed by a scraper 55, which is slowly rotated by a shaft 56. The freed particles are conveyed through an aperture 57 into a combined irradiation and separating arrangement. This arrangement comprises a housing 7 divided by a central wall 78 into two compartments, which are designed so that one may be closed off from the said chamber and inlet aperture 57 by a respective vacuum valve 72 whilst the other compartment is open. A rocking flap 71 is provided so that particles 51 trickling through the aperture 57 pass either into one compartment or the other. In Figure 2 the left-hand compartment is being filled whilst the right-hand compartment is being kept vacuum-tight by the valve 72. The particles 51 pass into the space between two electrodes 62, which are connected to a high-frequency generator (not shown) as in Figure 1. In the right-hand compartment, the space between the two electrodes 62 is completely filled with particles 51. Two laser devices 41 and 42 are provided in a direction perpendicular to the plane of the drawing, as illustrated in Figure 3 only. These laser devices irradiate the particles 51, whereby selective excitation of the said one isotope component takes place and chemical reaction with the reactant is rendered possible. The output of the laser devices and the cross-section and the shape of the layer of particles 51 through which the radiation is to pass are designed such that a substantially uniform absorption of radiation takes place over the whole cross-section of the particles 51. For this purpose, and for a better utilisation of the radiation which may be scattered by the particles, it is desirable, for example, to make the inner surfaces of the vessel 73 reflective and to provide only narrow windows for entrance of the radiation. After irradiation, the high-frequency generator (not shown) is switched on, so that fractional distillation

may take place, and distillation products are discharged through a duct 74, in the same way as illustrated in Figure 1.

Subsequently the rocking flap 71 is thrown over and the right-hand vacuum valve 72 is opened. Then, the right-hand compartment is filled with particles 51 from the aperture 57. Meanwhile, the left-hand compartment is closed by its vacuum valve 72 and the particles 51 situated between the electrodes 62 are irradiated as described hereinabove with reference to the right-hand compartment.

The gaseous isotopic substance and reactant are supplied from storage chambers 1 and 2 respectively by way of a chamber in a shaft 34 mounted, for example, in ball bearings 36, and then through channels in the drum 33. Lip seals 37 are disposed about the shaft to ensure separation of the gaseous isotopic substance and reactant before they are brought together upon leaving the rotating nozzles 31 and 32. The motor provided *inter alia* for initiating rotation of the drum may be constructed as a sleeve-gapped motor 50, as illustrated here. The thickness of the chamber wall 35*a* at the point where the motor is installed is substantially less than the normal wall thickness and surrounding housing portions are, in use, evacuated. Opposite outer field windings surrounding the chamber wall 35*a* there are mounted on the shaft 34 the associated rotor parts. It is possible for this motor to be constructed either as a synchronous motor or as an asynchronous motor. Instead of this motor, the drive means for the drum could be, for example, a turbine. However, since the design of the drive means has no effect upon the actual separating operation, no further details in this respect will be described.

With appropriate low-friction mounting of the shaft 34—for example, on gas bearings, magnetic bearings or the like—no separate drive means is required, and the recoil forces of the gas streams leaving the nozzles bring the drum 33 to such a velocity that the velocity of impact of particles in the streams on the receiver wall is so low that the requirements imposed on cooling means therefor are relatively slight. For this reason, apparatus constructed on these principles may be suitable also for other technical purposes, for example for gas liquefaction.

The apparatus and method used for the actual isotope-specific excitation of the solid particles may be selected from a variety of known possibilities. Examples of such methods are multiple-quantum absorption using short light impulses, and a two-step process in which radiation corresponding to the 3 ν_3-vibration is injected in the first step and a corresponding UV vibration is used in the second step. For UF_6, the wave number of the 3 ν_3 vibration is 1873 cm^{-1} and that of a suitable UV radiation is about 30,000 cm^{-1}.

WHAT WE CLAIM IS:—

1. An isotope separation process for the preferential removal of one isotope component as compared with a second isotope component from a gaseous isotopic substance, wherein a quantity of the said gaseous isotopic substance and at least a stoichiometric quantity of a gaseous reactant are expanded separately from one another adiabatically, or substantially so, to attain temperatures below 100 K, the expanded isotopic substance mixes with the expanded reactant in a condensation chamber cooled to below 100 K, condensation of the reactant takes place in the chamber to form solid particles in which molecules of the said isotopic substance are incorporated, and the said solid particles are collected and subjected to selective excitation employing electromagnetic irradiation at an excitation frequency of the said one isotope component whereby that component is enabled (preferentially as compared with the second isotope component) to undergo chemical reaction with the reactant, whereafter the resulting solid material is subjected to a treatment for separating out a product of the chemical reaction which product is relatively rich in the said one isotope component as compared with the second isotope component.

2. A process as claimed in claim 1, wherein the said treatment for separating products of the chemical reaction comprises fractional distillation.

3. A process as claimed in claim 1 or 2, wherein the collected solid particles are transferred from the said chamber to an irradiation chamber, separated from the condensation chamber, prior to the selective excitation and chemical reaction.

4. A process as claimed in claim 3 read as appended to claim 2, wherein the fractional distillation is carried out in the said irradiation chamber.

5. A process as claimed in claim 3 read as appended to claim 2, wherein the fractional distillation is carried out in a further chamber that is separated from the said irradiation chamber and is provided separately therefrom with means for adjusting temperature and degree of vacuum.

6. A process as claimed in claim 3, 4 or 5, wherein the said solid particles are compressed to form a compact, whilst the temperature thereof is maintained below

100 K, before transfer thereof to the said irradiation chamber.

7. A process as claimed in any preceding claim, wherein the said gaseous isotopic substance is uranium hexafluoride.

8. A process as claimed in claim 7, wherein the molar ratio of uranium hexafluoride to the said reactant is in the range from 1:1 to 1:1000.

9. A process as claimed in any preceding claim, wherein an auxiliary gas is admixed with the said gaseous isotopic substance prior to expansion thereof to increase the adiabatic coefficient thereof.

10. A process as claimed in claim 9 read as appended to claim 7, wherein the ratio of the admixed auxiliary gas to the uranium hexafluoride is in the range from 1:1 to 1:10.

11. A process as claimed in claim 9 or 10, wherein the said auxiliary gas is helium.

12 A process as claimed in any preceding claim, wherein the gaseous reactant is hydrogen bromide or hydrogen iodide.

13. An isotope separation process, for the preferential removal of one isotope component as compared with a second isotope component from a gaseous isotopic substance, substantially as hereinbefore described with reference to Figure 1, or Figures 2 and 3, of the accompanying drawings.

14. Apparatus for use to effect preferential removal of one isotope component as compared with a second isotope component from a gaseous isotopic substance, comprising a condensation chamber provided with cooling means, for maintaining it at a temperature below 100 K, and with expansion nozzles through which a quantity of the said gaseous isotopic substance and at least a stoichiometric quantity of a gaseous reactant can be expanded separately, adiabatically or substantially so, into the chamber to attain temperatures below 100 K so that the expanded isotopic substance mixes with the expanded reactant and condensation of the reactant takes place in the chamber to form solid particles in which molecules of the said isotopic substance are incorporated, which particles are collected in the chamber, means for subjecting the collected solid particles to selective excitation by electromagnetic irradiation at an excitation frequency of the said one isotope component so as to enable that component (preferentially as compared with the second isotope component) to undergo chemical reaction with the reactant, and means for separating out a product of the chemical reaction, which product is relatively rich in the said one isotope component as compared with the second isotope component.

15. Apparatus as claimed in claim 14, wherein one or more cylinders are mounted rotatably within the said condensation chamber, the or each cylinder being equipped with cooling means, and connected to drive means for rotating the cylinder steadily, the selective excitation means for irradiating the solid particles are provided adjacent to the or each cylinder, and are arranged so that, in use, radiation permeates completely through a layer of such particles collected on the or each cylinder in the course of each rotation thereof, and a scraper is arranged to remove the said layer, after irradiation, from the or each cylinder.

16. Apparatus as claimed in claim 14, wherein the said nozzles are carried by a support structure that is rotatable in the said chamber about a predetermined axis of rotation and are spaced radially from that axis and directed so that the passage of the expanding gaseous materials therethrough, to emerge into the chamber, encourages such rotation of the support structure, and wherein a stationary collecting surface within the chamber, in outwardly surrounding relationship to the path of the said nozzles during such rotation and concave towards that path, is arranged for receiving such particles, there being a scraper arranged in the chamber and rotatable to remove a collected layer of such particles from the said collecting surface, which surface is formed with an aperture positioned to deliver such removed particles to a location outside the condensation chamber, there to be subjected to the said selective excitation.

17. Apparatus as claimed in claim 16, wherein a motor is connected to the said support structure for initiating such rotation thereof.

18. Apparatus as claimed in claim 16 or 17, wherein the said support structure is formed as a disc-like drum of substantially uniform strength so as to give a uniform distribution of forces during rotation, the said nozzles being at the periphery of the drum, the drum being mounted for rotation on a central shaft through which respective channels for the isotopic substance and the gaseous reactant pass to the nozzles from outside the condensation chamber.

19. Apparatus as claimed in claim 18 read as appended to claim 17, wherein the said motor is a sleeve-gapped electric motor mounted partially on the said central shaft and partially on an evacuated surrounding housing portion.

20. Apparatus as claimed in claim 18 or 19, including housing wall portions surrounding parts of the said central shaft and defining therearound respective chambers through which the gaseous isotopic substance and the gaseous reactant

are passed to the said channels when the apparatus is in use.

21. Apparatus as claimed in any one of claims 14 to 20, wherein the said means for separating out a product of the chemical reaction include a vaporizing chamber arranged for receiving such collected solid particles from the condensation chamber and provided with capacitor plates for connection to a high-frequency generator, to subject the received particles to capacitive high-frequency heating, the vapourising chamber being connected to fractional condensation means.

22. Apparatus as claimed in claim 21, wherein there are two such vapourising chambers, arranged to receive such particles alternately, so that vapourisation of material from one of those chambers can be effected whilst the other is being supplied with such particles.

23. Apparatus as claimed in either of claims 21 and 22 when they are read as appended to claim 16, wherein the said selective excitation means are arranged to irradiate the said particles in the or each vapourising chamber.

24. Apparatus as claimed in any one of claims 14 to 23, wherein the selective excitation means comprises at least one laser device.

25. Apparatus for the preferential removal of one isotope component as compared with a second isotope component from a gaseous isotopic substance, substantially as hereinbefore described with reference to Figure 1, or Figures 2 and 3, of the accompanying drawings.

26. A method as claimed in any one of claims 1 to 12, using apparatus as claimed in any one of claims 15 to 24.

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2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheet 1

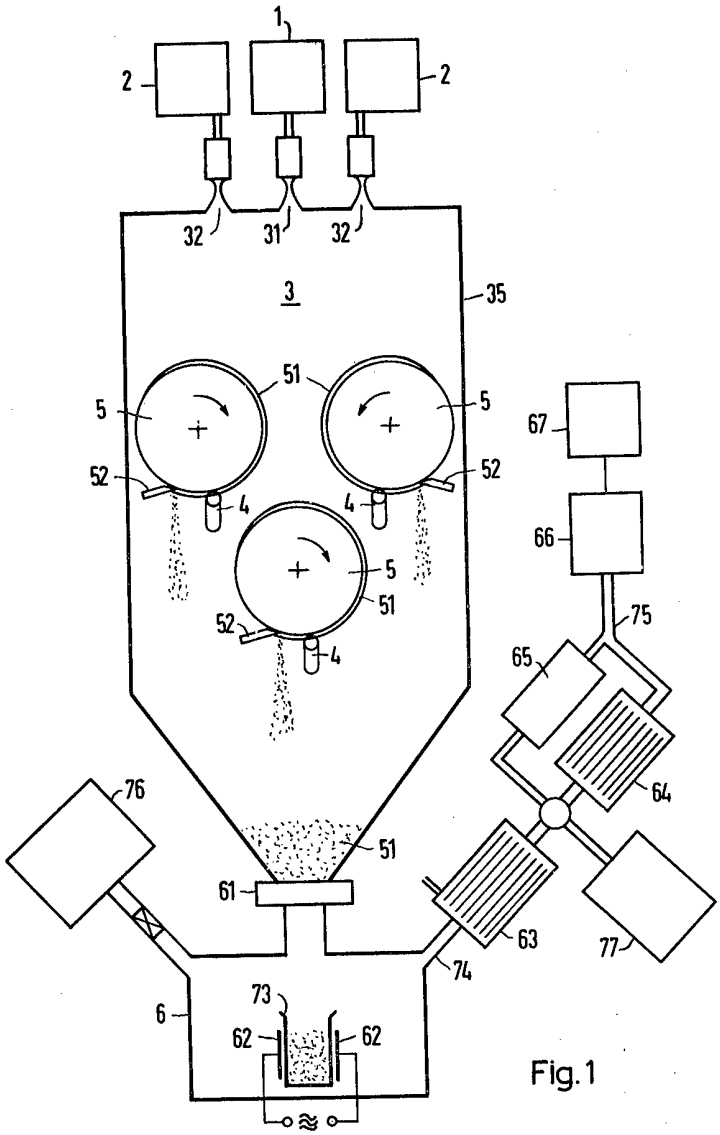

Fig.1

1579003 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

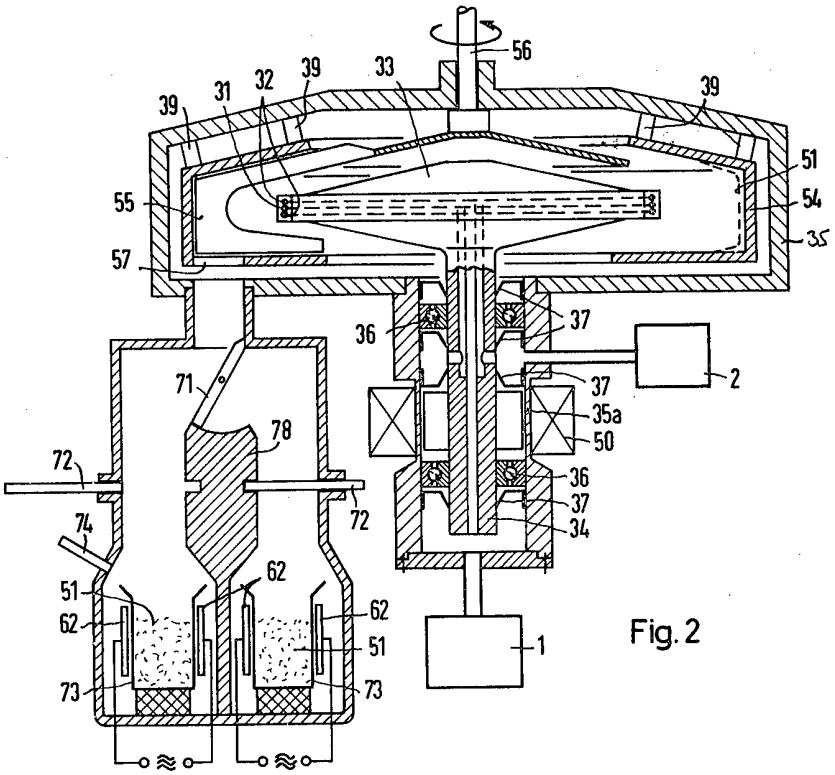

Fig. 2

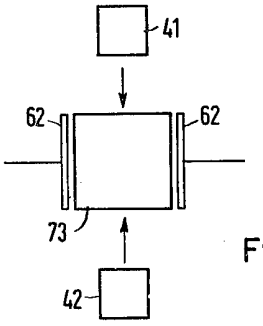

Fig. 3