

- [54] **APPARATUS FOR CARRYING OUT CHEMICAL REACTIONS, WITH FORMATION OF PHOTOREACTION PRECIPITATIONS**
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- [58] Field of Search..... 250/43; 204/193

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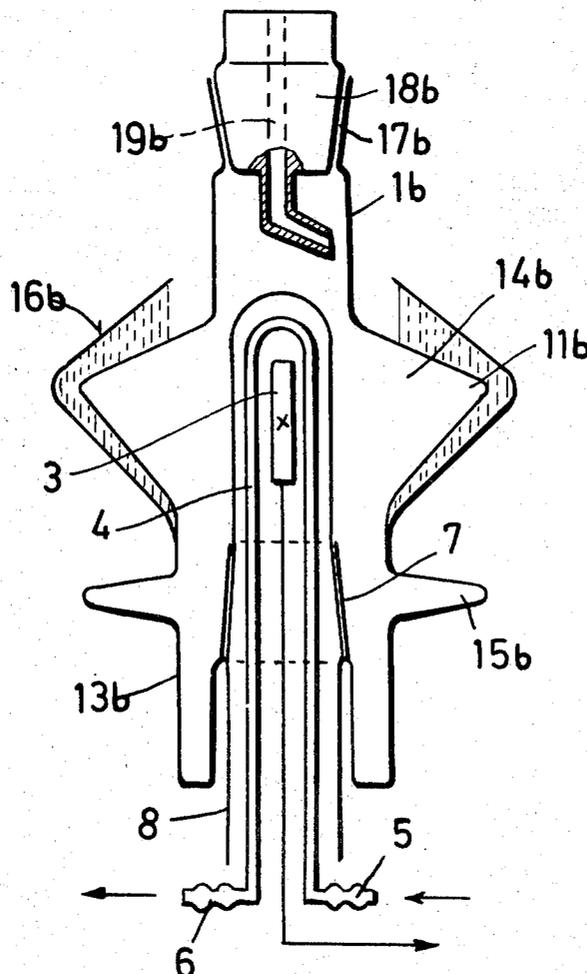
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[57] **ABSTRACT**

An assembly for effecting chemical reactions in solutions by radiation comprises a means providing a source of radiation and a reaction vessel rotatably mounted therearound. The reaction vessel is used to contain the solution to be treated by radiation. The vessel includes a bulge portion which extends radially outwardly for holding the solution at a location for exposure to the radiation. When a precipitate is formed upon exposure to the radiation during the rotation of the vessel, the precipitate is maintained at the furthest point from the radiation source in the bulge portion so that clear solution will be maintained near the source of radiation. Several specific embodiments of the assembly are disclosed.

14 Claims, 5 Drawing Figures



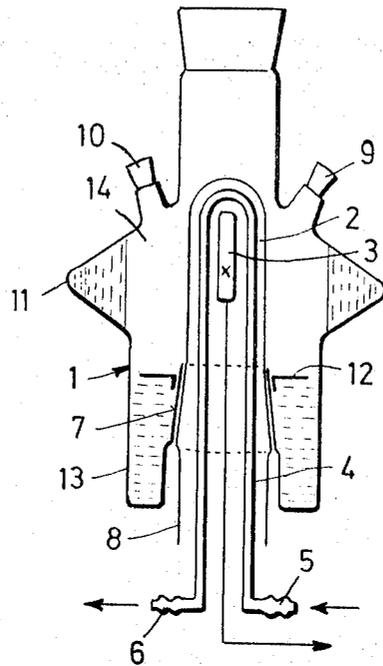


FIG. 1

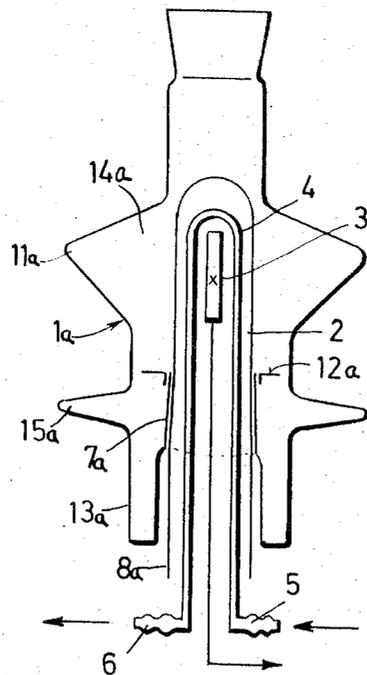


FIG. 2

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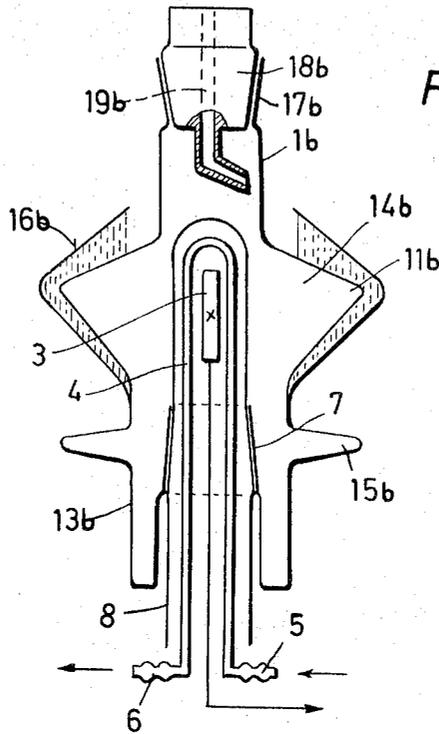


FIG. 3

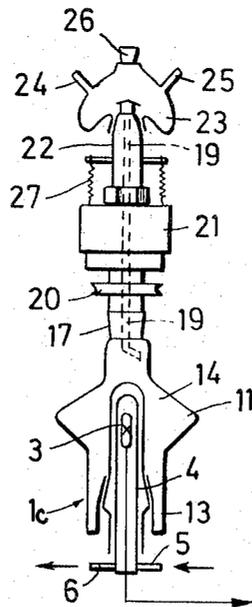
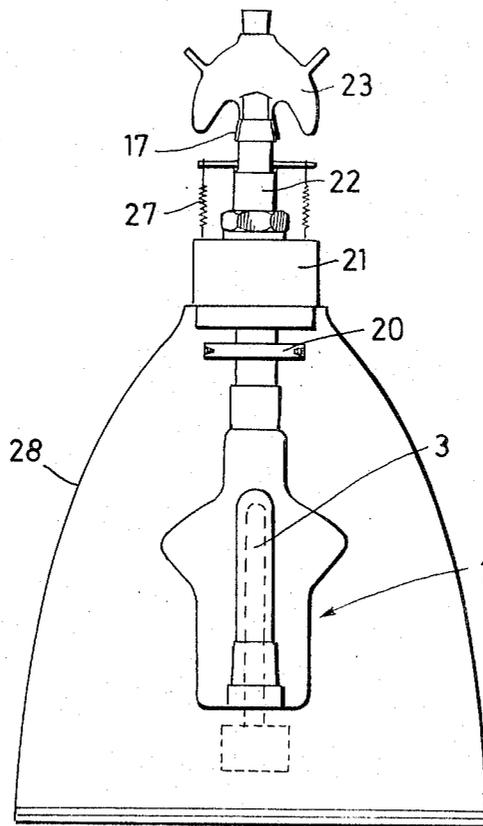


FIG. 4

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FIG. 5



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APPARATUS FOR CARRYING OUT CHEMICAL REACTIONS, WITH FORMATION OF PHOTOREACTION PRECIPITATIONS

BACKGROUND OF THE INVENTION

Apparatus for carrying out chemical reactions by radiation are generally well known. Such prior art apparatus includes a vessel in which a plurality of components is subjected to radiation exposure and subsequent precipitates are formed due to the chemical reaction initiated by the exposure. A source of radiation is arranged in the middle of the vessel which contains the solution consisting of the components to be treated. These prior art vessels are cylindrical and have an annular cross section. A so-called lamp hood is disposed in the center of the vessel. An illumination or radiation source such as a quartz burner is disposed in the lamp hood which is provided with a water cooling system.

The solution is disposed in the stationary, annular vessel and subjected to a stirring action by any suitable means. With an immersion lamp apparatus such as the quartz burner placed within a lamp hood, there is a rapid separation of the reactant precipitate initially effected within the solution. The formation of the precipitate is most intensive immediately upon the radiation surface of the cooled immersion lamp hood. The immediate formation of the precipitate leads to the development of a dense coating on the lamp hood within the vessel. The dense coating or deposit becomes increasingly dark in color in the layers closer to the source of radiation. Consequently, the reaction which must be effected by the radiation begins to increasingly slow down. It is only in the stirring region that the development of the dense covering or coating can be suppressed for a relatively long time.

In addition to the above disadvantages, the solution itself is made cloudy on the whole because of the general precipitation initiated by exposing the solution to radiation. The cloudiness impairs the continuation of the chemical reaction in those parts of the solution which are still to be treated. Several procedures have been followed such as repeated interruption of the radiation exposure, filtration of the solution and careful cleansing of the lamp hood to provide some assistance in obtaining the desired results in using the prior art immersion lamp apparatus. However, these prior art operations have to be carried out under an inert atmosphere which requires additional equipment and involves a comparatively long time.

A simple scraping of the precipitation from the lamp hood by mechanical means does not lead to any substantial improvement. The resultant suspension leads to a great reduction in the optical transmissivity or radiation within the solution. Exposure of a stationary solution to radiation such as light also does not provide any technical advance. The suspended fraction or precipitate from the solution sinks very slowly to the bottom. However, the crystals or precipitate formed directly on the irradiation surface of the cool lamp shaft are not flushed away and therefore form a dense coating that grows very quickly. This dense coating is especially formed on surfaces of high luminous density where light radiation is used in effecting the chemical reaction. Due to these disadvantages, the yield in the chemical operation is not very high.

PURPOSE OF THE INVENTION

The primary object of this invention is to provide an apparatus for effecting chemical reaction by radiation in which a reliable and quick reaction can always be carried out in a very simple manner.

Another object of this invention is to provide an apparatus for effecting such chemical reactions and prohibiting the deleterious effect on the chemical reaction made by a buildup of the precipitate on parts of the apparatus.

SUMMARY OF THE INVENTION

The apparatus as disclosed herein includes a vessel that is rotatably mounted around a source of radiation. The vessel is annular and contains the solution to be treated by any type of radiation such as light, gamma rays, soft x-rays and the like. The vessel includes a bulge portion which extends radially outwardly for holding the solution at a location for exposure to the radiation whereby when a precipitate is formed upon said exposure during the rotation of the vessel, the precipitate is maintained at the furthestmost point from the radiation source in the bulge portion so that clear solution will be maintained near the source of radiation. In a more specific embodiment of the invention, the bulge portion is annular and has a cross-sectional shape of a trough or triangular form.

In a specific embodiment of this invention, a vessel is provided with a lower collecting space and a reaction chamber. The bulge portion forms a part of the reaction chamber. When the vessel is rotated, the solution to be treated ascends from the lower collecting chamber into the reaction chamber and passes into the bulge portion. The centrifugal force created by the rotating vessel causes any precipitate formed in the solution upon exposure to the radiation to move to the furthestmost point from the radiation source in the bulge portion. On the other hand, the portion of the solution which has not yet been subjected to the radiation remains as a clear solution and therefore is exposed to the unimpeded radiation from the source such as a quartz burner, mercury arc lamp, soft x-rays, gamma rays and the like.

Another feature in this specific embodiment provides an annular disk member used as an upper cover for the vessel collecting chamber. The disk is located below the bulge portion and serves as a protection against light by association with the outwardly blackened collecting chamber. A gap is formed between the outermost periphery of the annular disk and the inside of the outer wall of the vessel. The suspended precipitate and solution moves in a sufficiently thin film through the annular gap. In a further embodiment, the collecting chamber may include another bulge portion for the purpose of collecting any precipitates formed in the reaction chamber.

A further embodiment of the assembly made in accordance with this invention includes a cooling jacket which encloses the bulge portion in the reaction chamber.

BRIEF DESCRIPTION OF DRAWINGS

Other objects of this invention will appear in the following description and appended claims, reference being made to the accompanying drawings forming a part of the specification wherein like reference charac-

ters designate corresponding parts in the several views.

FIG. 1 is a sectional diagrammatic view of a first embodiment of an apparatus made in accordance with this invention,

FIG. 2 is a sectional diagrammatic view of a second embodiment of an apparatus made in accordance with this invention,

FIG. 3 is a sectional diagrammatic view of a third embodiment of an apparatus made in accordance with this invention,

FIG. 4 is a diagrammatic view of an illuminating centrifuge with a driving component and made in accordance with this invention, and

FIG. 5 is a diagrammatic view of a rotary photoreactor made in accordance with this invention and having a protective hood.

DESCRIPTION OF SPECIFIC EMBODIMENTS

More specifically, a first embodiment of an apparatus made in accordance with this invention is shown in FIG. 1 and includes a vessel, generally designated 1, which is rotatably mounted on a light filter hood 8. A hollow chamber 2 is formed in the center of the vessel 1 within the filter hood 8. An illumination source 3 is disposed within the hollow chamber 2. The vessel 1 may be made of glass, metal or the like. The illumination source 3 may comprise the quartz burner, a mercury high-pressure lamp, or any other type of radiation used to effect chemical precipitations. The vessel 1 has a suitable conical socket portion 7 which is contiguous to the light filter mounting hood 8. It is possible that the vessel 1 may be seated in fixed relationship with respect to the hood 8 so that the entire combination may be rotated by a suitable drive means. However, it is also possible that the vessel 1 may be in sliding relationship with the light filter mounting hood 8 within the conical socket 7 so that it may be rotated about the hood 8 which would be mounted in a stationary position.

In each of the specific embodiments disclosed herein, a closed cooling system including a fluid-conducting jacket 4 is provided to effect cooling of the illumination source 3. The fluid-conducting jacket 4 includes a supply inlet 5 and discharge outlet 6. The cooling fluid is circulated by a circulating pump throughout the jacket 4 and in contact with a heat exchanger (not shown). The cooling fluid should be free from material which would form deposits in the cooling jacket 4 in the vicinity or region of the light source 3. Distilled water is advantageously used during operation of the specific embodiments disclosed herein. Tap water usually contains iron hydroxides which would deposit out onto the inner walls of the cooling jacket 4 in the region of the light source 3.

The closed cooling system offers an additional advantage in that it may be used as a light filter as well as perform the cooling function for the light source 3. Where there is no passing filter glass available for the construction of the exchangeable light filter hood 8, a circulating light filter solution can be used which is able to pass. Under most circumstances, the replaceable mounting hood 8 performs the important function of the light filter. The hood 8 can be made of quartz or any of the many different frequency costly light filter materials available in the prior art. The use of one and the same rotational member or vessel 1 permits the universal use of same for photochemical reactions effected in different spectral regions. This can be accomplished

by simply using various types of hoods 8 which are made out of the different filter materials available. There is an obvious advantage in the use of such exchangeable or replaceable filter hoods 8. If the replaceable light filter mounting hood 8 were provided, the entire rotational vessel or member 1 would have to be made of the desired light filter material. This would obviously increase the cost of the desired apparatus by a substantial amount.

Filling pipes or spouts 9 and 10 are used for introducing the components of the solution to be treated. The rotatably mounted vessel 1 includes a bulge portion 11 located at the height of the illumination source 3. The annular bulge 11 may be made in the form of a trough and have a triangular or any concave shape as seen in cross-section. The lower portion of the vessel 1 includes a cylindrical collecting chamber 13. An annular light protection cover disk 12 may be arranged between the annular bulge portion 11 and the cylindrical collecting chamber 13. The disk 12 is composed of material that prevents the solution contained in the chamber 13 from being illuminated by the radiation source 3. Disk 12 is connected to the inner wall of the vessel 1 and an annular gap is left between the disk 12 and the inside of the outer wall of the vessel 1.

When the vessel 1 is rotated, the solution therein ascends from the cylindrical collecting chamber 13 through the annular gap located between the disk 12 and outside wall and into the photoreaction chamber 14. The annular bulge 11 forms a portion of the reaction chamber 14 and provides an additional horizontal dimension thereto. That is, the bulge portion 11 extends radially outwardly for holding solution adjacent the radiation source 3 while the vessel 1 is rotating and precipitate is being formed. As is evident from the drawings and disclosure, the bulge portion 11 has sufficient depth with respect to the radiation source 3 that the precipitate is maintained at the furthestmost point from the radiation source 3 and the clear solution is maintained near the radiation source 3. The light protection disk 12 protects the solution within the cylindrical collecting chamber 13 from direct incidence of any radiation from the reaction chamber 14. The disk 12 does not become wet during the operation of the vessel 1 because the solution media passes through the annular gap between the disk 12 and the outer wall. Again, the movement of the solution from the chamber 13 into the reaction chamber 14 is effected through a centrifugal force established by the rotation of the vessel 1. Drive means may be located at positions 17b and 18b as shown in FIG. 3 or any other suitable outer position of the vessel.

Another feature of this invention is directed to the specific manner in which the rotation of the vessel 1 is effected. This rotation is advantageously controlled intermittently by the use of a time switch. For example, the centrifuge vessel 1 may be driven or rotated for a period of 60 seconds and then stopped for an interval of about 3 seconds. This rotating and stopping cycle may continue for over an extended period of time. During the rotation of vessel 1, any precipitate formed through the action of the radiation of this solution will be collected in the annular bulge 11. Any clear solution remaining in the chamber 14 will be closer to the illumination source 3 than the precipitation. Any reaction precipitate formed within the solution will immediately

pass due to the centrifugal force into the outer portion of space in the annular bulge 11.

When the rotation of the vessel 1 is stopped during the 3 second interval or any other interval of time, the solution and the precipitation located in the reaction chamber 14 will move downwardly into the collecting chamber 13. The reduced rotation of the vessel 1 will cause the effect of the centrifugal force to be lessened or completely stopped. The solution and precipitate will pass through the annular gap between the outer vessel wall and the light protecting hood disk 12. When rotation of the vessel 1 is restarted, the solution is transferred back into the reaction chamber 14. Substantially all of the radiation reaction precipitate which has descended into the collecting chamber 13 during the stopping interval remains there. Any precipitate that is newly formed within the chamber 14 by exposure to light or other type of radiation is immediately accumulated in the pointed, outer space of the annular bulge 11 as shown in this embodiment. It has been found that approximately two-thirds of the depth of the layer of solution within the bulge 11 always remains free from clouding or turbidity. Consequently, the action of the light or radiation source is not impeded by the layer of solution.

A further feature of this invention is to vary the illumination steps by altering the height of the cylindrical radiation surface of the solution rotated with the chamber 14. The alteration in the height of the rotating surface can be selected within wide limits. This is achieved through the use of correspondingly widened or constricted opening angles of the annular bulge 11 within the construction of the vessel 1. The widening of the opening angle with the annular bulge in the reaction chamber 14 can sometimes be kept very large. In this instance, it is possible to use several light or radiation sources arranged annularly on a circle. The radiation from these light sources may be focused onto a circular, water cooled metallic reflector and then into the region of the rotating solution surface which is formed in the chamber 14 as a cylindrical sheath. On the other hand, the illumination or exposure steps can be reduced within wide limits by constricting the opening angle of the annular bulge 11 within the chamber 14. This latter type of structure will be advantageously used with radiation-reactions which proceed quickly. Additionally, the structure would be usable in instances where it was desired to recognize the final point of a reaction within a solution.

Another embodiment of an apparatus made in accordance with this invention is shown in FIG. 2. The rotatably mounted vessel 1a includes an annular bulge 15a disposed within the collecting chamber 14a. The light protection disk 12a performs the same function as the disk 12 in the embodiment as shown in FIG. 1. The annular bulge 15a is very similar but smaller than the annular bulge 11a. The purpose of the annular bulge 15a is to collect any of the reaction precipitate which has moved downwardly into the collecting chamber 13a after having been formed in the annular bulge 11a. With the precipitate being captured by the annular bulge 15a when the vessel 1a is rotated after a stopping interval, the solution is able to enter into the reaction chamber 14a in a completely clear form. In other words, the annular bulge 15a constitutes a trough in which the precipitate or any other solid material is collected when the solution in the collecting chamber 13a

begins to ascend under the action of the centrifugal force during the rotation of the vessel 1a. This result is due to the greater specific gravity of the precipitate which causes it to migrate into the trough or annular bulge 15a. The end point of the radiation-reaction process is achieved at that exposure stage when precipitate no longer forms while the solution is subjected to the radiation from the illumination source 3.

Another feature of this invention with respect to the annular bulge 15a is that it may have its external wall blackened to prevent the passage of light or other radiation. If a wall forming the annular bulge 15a is opaque to the passage of light or other radiation, it is possible to collect therein a light sensitive photo-reaction precipitate without having adverse reaction formed therein. In this instance, the wall of the bulge 15a may be coated by the application of a quick-drying black "Teroson"-rubber composition. With this type of coating, the annular bulge 15a becomes a dark-room centrifugal separator in association with the light protection hood disk 12.

The structure as shown in FIG. 2 enables the complete separation between readily vaporizable, and easily decomposable reactants or solvents from the reaction solution which is treated by radiation in the chamber 14a. In addition, the structure may also be advantageously used where it is impossible to avoid completely thin, energy-dissipating coating having long exposure times. The maintenance of the separation of these materials prevents contamination of the solution itself by the development of additional, undesired inner filters. In certain instances, it may be advantageous to provide solid wipers to the structure as disclosed herein. It is further possible to lower the vapor pressure of the readily decomposable reactants by cooling or passing solvents to a degree which can be tolerated.

Another embodiment of an apparatus made in accordance with this invention is shown in FIG. 3. A cooling jacket 16b is disposed around the annular bulge 11b. The bulge of the cooling jacket 16b is adapted to the shape of a reaction chamber 14b. The configuration of jacket 16b prevents any spraying of the cooling medium during the rotation of the vessel 1b. The use of the cooling jacket 16b enables a temperature control of the photoreaction chamber 14b to be effected. The cooling jacket 16b is open at the top in this particular embodiment and becomes narrower from the top towards the middle and bottom. This construction permits the extraction and the feeding of the cooling medium into the jacket 16b. A cone-shaped opening structure 17b is provided at the upper end of the vessel 1b. A cone stopper 18b is fitted into the opening structure 17b. A supply pipe 19b is disposed within the cone structure 18b. Fluid media which is to be treated in the vessel 1b is introduced through the supply pipe 19b.

An embodiment of the invention which incorporates a driving mechanism for the rotation of an apparatus made in accordance with this invention is shown in FIG. 4. The drive mechanism includes a pulley 20 which may be driven by a V-belt or the like from an electric motor or the like, neither of which are shown. The drive mechanism also includes a ball bearing 21, a rotating packing 22 having a drip feed passage 19 which corresponds to the supply pipe 19b as shown in the embodiment of FIG. 3. The assembly includes a container 23 which includes different connections 24, 25 and 26. A compression spring suspension 27 serves

to produce a suitable sealing with the floating ball bearing 21 and the rotating packing 22.

The rotating vessel can also be designed for continuous light or radiation exposures in through-flow operation. For this purpose, another rotating packing for the discharge and supply line for the light source can be provided at the bottom of the cylindrical collecting portion. Continuous operation may take place in such a way that the material for exposure to light is supplied batch-wise. The supply is from the centrifuge head through the passage formerly provided for the dropping funnel and is retained in the rotational member until an automatic registration of the spectrum in the region of the clear irradiation zone signals the required conversion and initiates the stoppage of the rotation. The material being exposed would flow into a storage tank and the rotational member would once again be set in rotation, filled, etc.

The exposure centrifuge apparatus as described hereinabove can be used for carrying out many different photo reactions. With high speeds of rotation of the vessel 1, it is expedient to arrange the whole system between a protective hood 28 as shown in FIG. 5. It is understood that any source for radiation such as mercury high-pressure lamp rays, soft x-rays, gamma rays, ultraviolet, infrared elections, and the like may be used in conjunction with the apparatus of this invention. When employing gamma rays to effect precipitation in solutions sensitive to such gamma rays, the separation may be effected through intermittent rotational operation. In those instances where gamma rays are used, however, the collecting chamber 13, 13a, 13b, 15a, 15b and the light protective annular disks 12, 12a would have to be provided with a protection against the radiation. Such a protection may be obtained by using lead having suitably thick walls.

ADVANTAGES OF THE INVENTION

There are many advantages in the use of the invention disclosed herein. Use of the assembly made in accordance with this invention provides a non-impeded constant reformation of the photo-reaction precipitation process. This result is obtained because the annular bulge formed in the reacting chamber 14 separates the precipitate and maintains a clear solution through which the radiation source may penetrate for further reaction. In other words, a clear irradiation zone of the reaction solution rotating in the reaction space or chamber is constantly maintained. The reaction precipitate migrates into that portion of the reaction chamber which is furthest from the light source as soon as it is formed. Therefore, a quick, constant deposition of the precipitate produces a high yield in a very short time.

When the rotation of the vessel is stopped, the reaction precipitate descends from the annular bulge into a lower collecting space or chamber. If the rotation of the vessel is started again, the solution which has not been subjected to the reaction ascends into the reaction chamber formed in part by the annular bulge. The precipitate remains collected at the bottom of the vessel 1 due to the inertial force cause by the larger mass. The presence of a suspension in the solution being irradiated which would adversely affect the reaction is substantially prevented. Consequently, the apparatus made in accordance with this invention operates with a substantially better efficiency, more quickly and with a much higher yield. The apparatus as disclosed herein

is extremely versatile since it is designed for both continuous and intermittent centrifuging operations.

While the apparatus for carrying out chemical reactions, with formation of photoreaction precipitations has been shown and described in detail, it is obvious that this invention is not to be considered as being limited to the exact form disclosed, and that changes in detail and construction may be made therein within the scope of the invention, without departing from the spirit thereof.

Having thus set forth and disclosed the nature of this invention, what is claimed is:

1. An assembly for effecting chemical reactions in solutions by radiation comprising:

- a. means providing a source of radiation, and
- b. a vessel rotatably mounted around said source of radiation for containing a solution to be treated by radiation,
- c. said vessel includes a reaction chamber, a collecting chamber spaced vertically below the reaction chamber and a wall portion extending therebetween,
- d. said reaction chamber including a bulge portion which extends radially outwardly for holding solution adjacent the radiation source while the vessel is rotating and precipitate is being formed,
- e. said bulge portion having sufficient depth with respect to the radiation source so that the precipitate is maintained at the furthestmost point from the radiation source and the clear solution is maintained near the source of radiation,
- f. said wall portion being effective to allow passage of material between the bulge portion and the collecting chamber,
- g. and means for rotating said vessel around said source.

2. An assembly as defined in claim 1 wherein the bulge portion is annular and has a cross-sectional shape of a trough or triangular form.

3. An assembly as defined in claim 1 wherein the vessel includes an annular light protective disk disposed between the reaction chamber and the collecting chamber,

said annular light protective disk being spaced from the inside of the wall portion of the vessel to leave an annular gap therebetween for the passage of material including solution, precipitate and mixtures thereof between the reaction chamber and the collecting chamber.

4. An assembly as defined in claim 3 wherein the walls of the collecting chamber portion have a structural configuration that is impervious to light below the annular light protection disk.

5. An assembly as defined in claim 1 wherein the bulge portion is enclosed by a cooling jacket structure.

6. An assembly as defined in claim 1 wherein a second bulge portion is being located in the collecting chamber to contain precipitates which begin to move upwardly as the vessel rotates, said second bulge being smaller in cross-section than the first bulge portion which forms a part of the reaction chamber.

7. An assembly as defined in claim 1 wherein the radiation source comprises a plurality of light sources disposed in annular form and

at least one metallic reflector is disposed between the source and the solution being treated.

8. An assembly as defined in claim 1 wherein the radiation source provides electrons, soft x-rays or gamma rays.

9. An assembly as defined in claim 1 wherein a driving means is located a spaced distance from said vessel to effect rotational movement thereof.

10. An assembly as defined in claim 1 wherein the vessel includes an upper, central supply inlet and a lower discharge outlet disposed at the bottom thereof to remove collected materials from the collecting chamber.

11. An assembly as defined in claim 1 wherein a protective hood encloses the rotatably mounted vessel.

12. An assembly as defined in claim 1 wherein said vessel includes a hood member displaceably mounted over the radiation source to filter the radiation passing to the solution.

13. An assembly as defined in claim 1 wherein a fluid conducting jacket is disposed between the reaction chamber and the radiation source.

14. A vessel assembly for effecting chemical reac-

tions in solutions comprising:

a. an upper reaction chamber,

b. a radiation source,

c. a lower collecting chamber,

d. a wall portion extending between the upper and lower chambers to allow passage of material including solution, precipitate and mixtures thereof therebetween, and

e. means for rotating said chambers with respect to said source,

f. said reaction chamber and collecting chamber being formed around a center bore which is effective to contain the radiation source,

g. said reaction chamber including a bulge portion which extends radially outwardly for holding solution adjacent the radiation source while the vessel is rotating and precipitate is being formed,

h. said bulge portion having sufficient depth with respect to the radiation source so that the precipitate is maintained at the furthestmost point from the radiation source and the clear solution is maintained near the source of radiation.

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