Sept. 10, 1968 F. GENSER 3,400,747

LABORATORY FLASK EVAPORATOR

Filed June 22, 1964 2 Sheets-Sheet 1

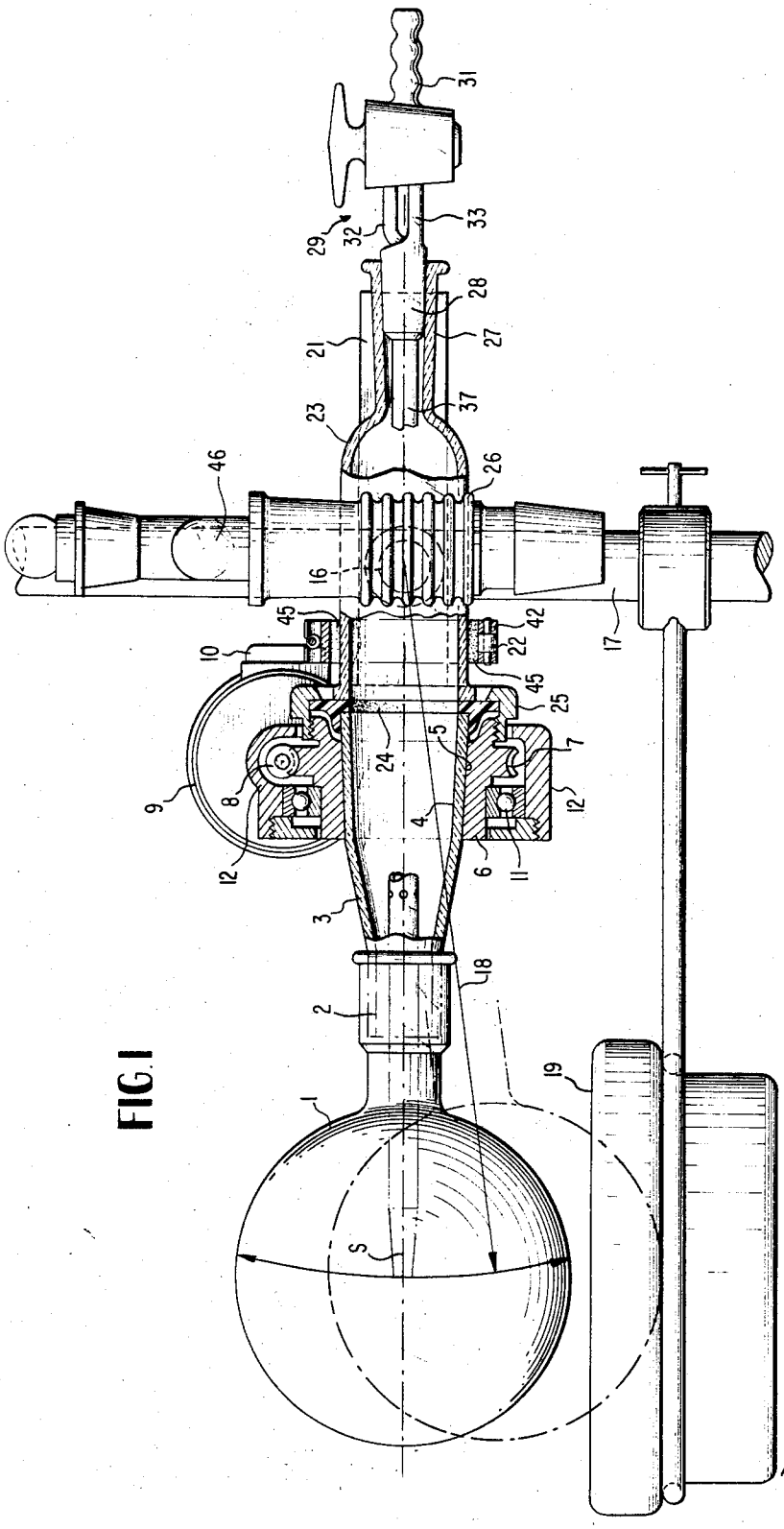


FIG. 1

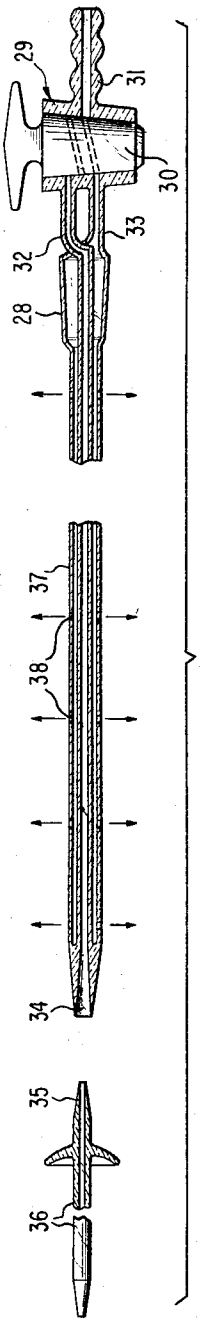


FIG. 4

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FIG. 2

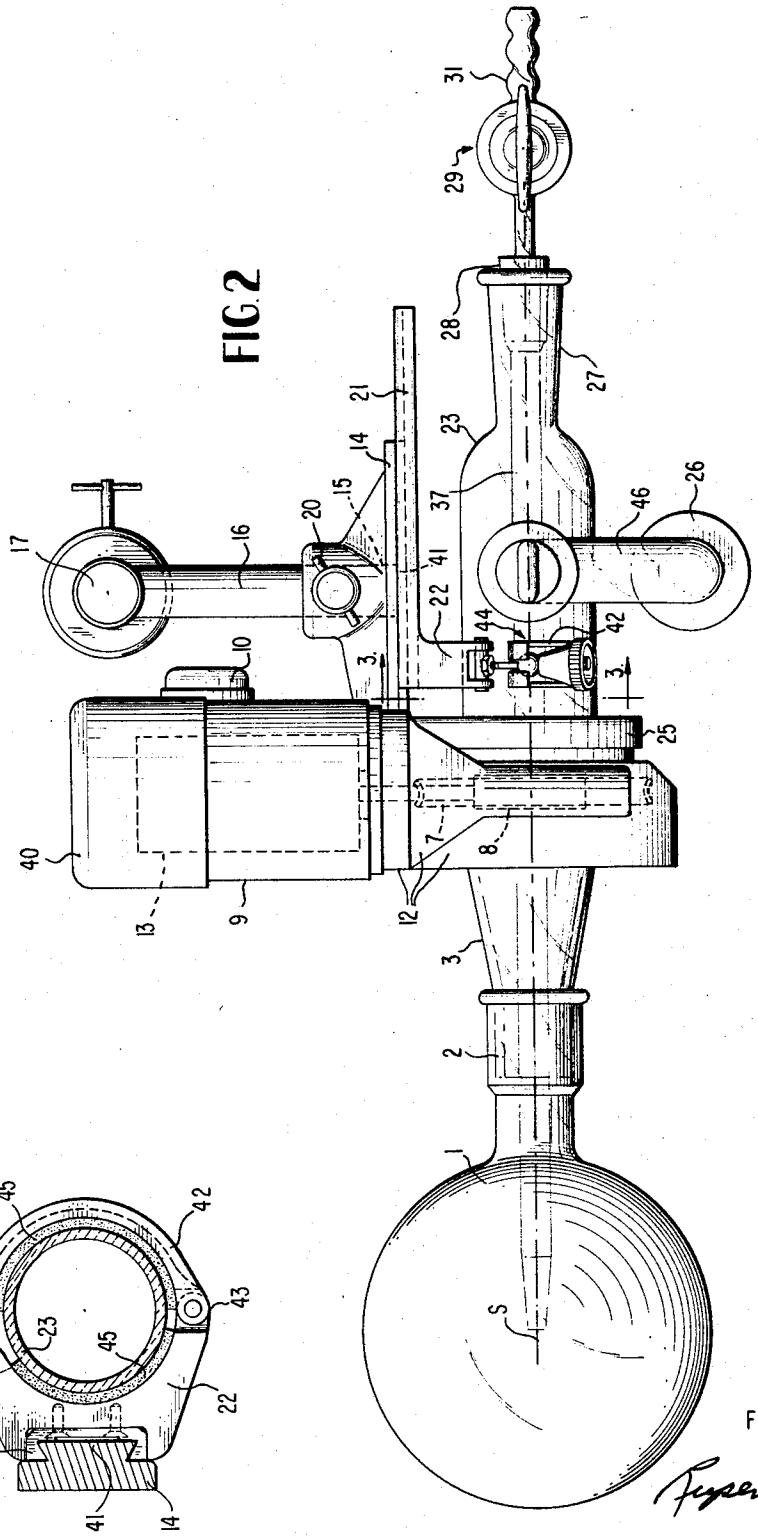


FIG. 3

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LABORATORY FLASK EVAPORATOR

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6 Claims. (Cl. 159—23)

ABSTRACT OF THE DISCLOSURE

A rotating flask evaporator device in which first and second hollow tube members are axially connected through a rotation joint. A flask is removably connected to the free end of the first hollow tube member and a gear is connected around the other end adjacent the rotation joint and includes a portion forming part of the joint. A motor is connected on an axis normal to the axis of the flask and hollow tube members to rotatably support the gear and also drive it to rotate the first hollow tube member and flask relative to the second hollow tube member, and the motor is also pivotally connected on a support means on an axis parallel with the motor axis and clampingly engages the first hollow tube member such that the flask, hollow tube members and motor are pivotal in a vertical arc. And a novel nozzle means is removably connected in the free end of the second hollow tube member and extends longitudinally interior of the tube members into the flask for conveying fluids thereto.

This invention relates broadly to laboratory evaporators and more particularly to rotating flask evaporators and a rotating vacuum joint therefor.

To keep up with the demand in the industry the chemical and related industries are increasingly requiring laboratory apparatus for research and routine work which is suitable for raising yields, shortening operating times, and carrying out continuous processes. In order to maintain the amount of equipment in a laboratory as low as possible, so as not to crowd the working area, new apparatus must insure greater efficiency and must be capable of more universal applications of use in the laboratory, as well as maintaining simplicity in manipulation.

The processes that are continually gaining importance and requiring new apparatus developments are those of careful concentration and distillation in a vacuum, which processes are largely carried out by means of thin-layer evaporation. It is well known in the art that in a rotating vessel a surface film is formed on the wall by the liquid in the vessel. The evaporating surface is therefore greatly enlarged compared with the surface of the liquid when the vessel is in stationary position. A vacuum-evaporation apparatus also has the advantages over other type evaporation apparatus of operating without boiling capillaries or boiling stones, and it reduces the distillation time by a substantial amount.

A rotating vacuum-evaporating system therefore operates at relatively low temperatures with a saving of both material and time. Thermolabile materials and those sensitive to oxygen, e.g. vitamins, hormones, enzymes, aromatic substances, alkaloids, peptides, unsaturated compounds and substances tending to polymerize or condense can often only be concentrated by means of modern thin-layer evaporators.

It has been found that flask evaporators are the only type of evaporating system which have thus far been successful for evaporating liquid on a large scale. However, the size of the flasks is limited in the usual commercial rotating evaporators to approximately four liters. Also with evaporators presently in use the distillation output

is frequently influenced by obstructed leads, coolers which are too small and reflux before the condensation plant.

With the rotating flask evaporator of the present invention, for the first time distillation or concentration, respectively, can be carried out between traces and semi-technical deposits. It is thus an object of the present invention to provide a construction of rotating flask evaporator which is relatively inexpensive and renders unnecessary the necessity of constructing the flasks of metal as required with prior art apparatus, even for producing substantial outputs.

Another object of the invention is to provide a construction of laboratory evaporator which is adaptable for numerous fields of application in chemistry, pharmacy, chemical medicine, physics and other related fields.

Another object of the invention is to provide a construction of rotating flask evaporator which permits the use of ordinary, relatively inexpensive glass flasks.

A further object of the invention is to provide a novel construction of rotating joint for laboratory flask evaporators capable of maintaining a vacuum seal between two members, while one member is subjected to rotation.

Still a further object of the invention is to provide a construction of rotating flask evaporator in which breaking stresses are eliminated from the rotating glass parts, and in which the sizes of the flasks may be substantially larger than in existing type evaporator apparatus.

Other and further objects of the invention reside in the structure of the rotating joint and its associated clamp, as set forth more fully in the specification hereinafter following by reference to the accompanying drawings, in which:

FIG. 1 is a side elevational view partly in vertical section of the rotating flask evaporator device of the invention;

FIG. 2 is a top plan view of the device of FIG. 1;

FIG. 3 is an enlarged cross-sectional view taken substantially along the line 3—3 of FIG. 2; and

FIG. 4 is a longitudinal sectional view of the feed and wash nozzle used in connection with the evaporator device.

The invention is directed to a rotating flask evaporator in which the working flask is rotated about its axis of symmetry and is further freely pivotable about an axis normal thereto. The free pivotability of the working flask about an axis normal to its axis of symmetry makes it possible to easily adjust the flask, of any size, with its liquid filling to any horizontal position, so it can rest freely on a waterbath device for support thereby, so that the weight of the flask and liquid therein are not supported by the rotating apparatus. A working flask which floats on a waterbath device can be rotated almost effortlessly and requires little force for its drive. Even for a working flask holding, for example, 20 liters, a driving motor of at most 55 watts is sufficient to rotate the filled flask, of which not even half the current is used for the rotation of the working flask. The rotating working flask is packed against a stationary part of the evaporator unit rotating apparatus by a packing sleeve, consisting for example of polyfluoroethylene with an addition of glass dust. But this packing, required to maintain the vacuum seal, has at the same time the advantage that the heat produced by friction between the rotating glass member and the packing reheats the vapor issuing outwardly from the working flask and thus prevents it from condensing and returning to the working flask before reaching the condenser. In special cases the frictional heat given off by the packing sleeve may even become so great that solid residues which normally should deposit only in the working flask also deposit in the glass adapter between the working flask and condenser. But this is easily remedied by surrounding the feed pipe, which supplies further liquid to be evaporated to the working

flask, by a pipe with a plurality of nozzles along the length thereof, whereby the newly supplied liquid issues from the nozzles to first dissolve the residues in the glass adapter, by manipulation of a two-way cock on the feed pipe which directs the liquid either through the nozzles or through an end aperture directly into the working flask.

The considerable savings of working time that is obtained by the device of the invention is evident, for example, from the fact that in a working flask which executes about 60 revolutions per minute, 200 cc. of water are completely distilled in 25 minutes, while under otherwise equal operating conditions, but with a stationary working flask, about 85 minutes are required for this distillation process.

Referring to the drawing in greater detail, the working flask **1**, of standard design, any required size, and preferably constructed of glass, with axis of symmetry S, is connected tightly on the standard ground core **2** of a glass adapter **3** whose conical part **4** is inserted in the conical bore **5** of a supporting ring **6** having a worm wheel rim **7** about the periphery. A drive worm **8**, which is fastened on the drive shaft of a small electric motor **9**, having on-off switch **10**, engages the worm wheel rim **7** to impart rotative movement to ring **6**, adapter **3** and flask **1** when the motor is energized. The supporting ring **6** is mounted for rotation in housing **12** by ball bearing **11**. At the same time motor **9** and a condenser **13** are connected stationary with housing **12**.

A bearing plate or connecting member **14** is also connected stationary with housing **12** and may be integrally cast with the same and is provided with a bore **15**, extending at substantially right angles to the symmetry axis S of the working flask **1**. Bore **15** is adapted to receive a fixed horizontal support shaft **16** which is an extension of upstanding support post **17**, and which is adjustably connected thereto, as indicated, so that it may be clamped in position at any desired horizontal level along the post. Support shaft **16** fits within bore **15** so that connecting member **14**, housing **12** and motor **9** may be freely rotated or pivoted about the support shaft and clamped in any desired position relative to the shaft by means of detachable clamp screw **20** carried by connecting member **14** and which extends into clamping engagement with shaft **16**. A waterbath device **19** carrying a quantity of liquid is disposed beneath working flask **1** and by the adjustment of clamping screw **20**, flask **1**, and housing **12**, and its associated members, may be pivoted in a vertical plane to any desired position along an arc governed by radius **18**, until flask **1** floats freely on the liquid surface of the waterbath device **19**, so that the waterbath supports the weight of the flask and its contents. This removes the weight of flask **1** and its contents from glass adapter **3** and the rotating joint mechanism, thus removing the breaking stresses from the glass parts which would normally be present if the waterbath did not assume the task of supporting the flask and its contents. Clamp screw **20** serves to hold the working flask **1** in any desired slanting position independently of the waterbath.

It is to be understood that housing **12**, motor **9**, and the working flask may be pivotally connected to the upstanding support post **17** by means other than the connecting member **14** as shown. For instance, a collar **40** may be rotatably connected to the opposite end of motor **9** in housing **12** with a clamp screw, not shown, for maintaining collar **40** in any desired rotative relation to the motor housing to assume the same function of the pivot connection between support shaft **16** and connecting member **14**. Collar **40** may be provided with an extension not shown, having a vertical aperture or the like therethrough and an adjustable clamp for engaging the upstanding support post **17** so that the rotating joint can be adjusted to any horizontal level along the post.

Connecting member **14** is provided with a dovetail guide **41** along its front face which engages a cooperating dovetail member in holding slide **21** which is securely connected to connecting member **14** in any desired longitudinal position so as to pivot about the horizontal axis with members **14**, **12**, **9**, **3** and **1**. Holding slide **21** is provided with a releasable gripping shackle **22** on the end closest to housing **12** with one portion **42** of the shackle being pivotally connected through a bifurcated pivot joint **43** to the stationary portion of the shackle so as to engage about glass adapter member **23** to securely hold the same in the desired position. The movable shackle portion **42** is locked in the closed position, to the stationary shackle portion, by means of a pivot bar thumb screw locking device of normal design, shown generally at **44**. The inner diameter of shackle **22**, including the inner diameter of movable shackle portion **42** is provided with a rubber lining **45** so as to prevent breakage of glass adapter member **23**.

A packing ring **24** preferably constructed of polyfluoroethylene with the addition of glass dust is held securely against the end face of glass adapter **3** by means of a threaded ring **25** disposed in screw threaded engagement with one end of supporting ring **6**, such that ring **25** and packing ring **24** rotate with supporting ring **6**. Glass adapter member **23** is securely clamped by shackle **22** to holding slide **21** which is longitudinally adjustable to bring the open end face of glass adapter member **23** into engagement with packing ring **24** such that the packing ring is disposed between the end faces of adapters **3** and **23**. When motor **9** is energized there is sliding engagement between packing ring **24** and the end face of glass adapter member **23**. Glass adapter member **23** connects the rotating flask evaporator to the other parts of the evaporator system through the upstanding arm extension **46** which carries a bellows **26** for connecting the apparatus to the remainder of the system. Bellows **26** is preferably constructed of polytetrafluoroethylene. To prevent diffusion losses in the bellows **26** from becoming noticeably great, at a vacuum of less than 10^{-2} torr, metal, and in particular chromium, is vapor deposited on the outside thereof with the exception of the packing surfaces. The remainder of the evaporator, such as the condenser, which connects to the end of the bellows is not shown since many different types of apparatus may be utilized.

Glass adapter member **23**, which is positioned along the symmetry axis S is provided with a conical end opening **27**, at its opposite end, through which fluids are admitted to flask **1**. This opening is usually plugged in an appropriate manner. As previously mentioned in special cases solid residues deposit out of the vapor issuing out of flask **1** out onto adapter **3** and also onto the inner bore of glass adapter member **23**, although it is desired to have them deposit out only in the working flask. To remove these residues the feed and wash nozzle **29** is connected into the conical end opening **27**, and extends along the axis of symmetry to flask **1**. Conical portion **28** of the feed and wash nozzle **29** is disposed in sealing engagement with the opening **27**. Nozzle **29**, as shown more particularly in FIG. 4, consists of a two-way cock **30** with a hose connection **31** for connecting the nozzle to a supply of new liquid to be evaporated. A pair of branch lines **32** and **33** extend on the opposite side of cock **30** from hose connection **31** with line **32** extending longitudinally in a substantially straight line and terminating in a widened conical portion **34** on its free end which enables the nozzle device to receive the male cone connector **35** of a pipe extension **36** which may be used to extend from the terminating end of nozzle **29** to the center of the working flask **1**, depending upon the size of the working flask.

The other branch line **33** leads into a jacket **37** concentrically disposed around and spaced from inner pipe or line **32** with its terminating end fused with the end of inner pipe **32** so that no liquid may issue from the end of jacket **37**. A plurality of fine bores **38** are disposed along the length of jacket **37** and about the periphery thereof to direct jets of fluid against the inner walls of glass

adapters 3 and 23 when the two-way cock 30 is deposited in the position to direct fluid from connection 31 to branch line 33. Any deposits on the inside of glass adapters 3 and 23 are washed down into the working flask together with the liquid being newly introduced into the flask for evaporation. If no residues are present to be washed into the flask, then cock 30 is positioned to direct fluid from connector 31 through branch line 32 and out open end 34 into the flask or from there through pipe extension 36 into the flask.

The device of the invention is applicable to crystallization processes, or disintegrating substances in the flask by using the device as a spherical mill by placing glass beads in a strong glass flask with the substances to be disintegrated. The device is also applicable for drying and degassing oils and oily substances, hydration, reactions without agitators, suctioning-off acid vapors and poison gases, inorganic evaporating processes, etc.

While the invention has been described in certain preferred embodiments it is realized that modifications may be made, and it is to be understood that no limitations upon the invention are intended other than those which may be imposed by the scope of the appended claims.

I claim:

1. Evaporator apparatus comprising support means, flask means having a symmetry axis, first hollow adapter means connected at one end to said flask means and having a free end, motor means connected at substantially right angles to said hollow adapter means, clamping means connected to said motor means, gear means connected about said first hollow adapter adjacent the free end, a housing connected with said motor means rotatably supporting said gear means, said motor means including a rotation shaft connected to drive said gear means, second hollow adapter means secured to said motor means by said clamping means and having a free end positioned adjacent the free end of said first hollow adapter means, a packing ring connected between the free ends of said first and second hollow adapter means forming a rotation joint, means connecting said packing ring to said gear means for rotation therewith, and means freely pivotally connecting said motor means to said support means whereby said flask means and motor means are freely pivotable about an axis normal to the symmetry axis and said first hollow adapter means is rotatable relative to said second hollow adapter means.

2. Evaporator apparatus as set forth in claim 1 in which said gear means includes a threaded portion adjacent said free ends, said packing ring having a peripheral portion extending adjacent said threaded portion, and said means connecting said packing ring to said gear means comprising a threaded ring member engaging the peripheral portion and in threaded engagement with said threaded portion of said gear means.

3. Evaporator apparatus comprising flask means having a symmetry axis, first hollow adapter means connected at one end to said flask means, motor means connected to rotate said first hollow adapter means and said flask means about the symmetry axis, support means, means pivotally connecting said motor means to said support means whereby said flask means and motor means are pivotal about an axis normal to the symmetry axis, second hollow adapter means connected to said motor means whereby said first hollow adapter means is rotatable relative to said second hollow adapter means, and a pair of concentric fluid conduits forming a washing nozzle means adapted to convey fluid in a selected one of said conduits connected to said second hollow adapter means and extending longitudinally therethrough and through said first hollow adapter means to said flask means substantially along the symmetry axis.

4. Evaporator apparatus as set forth in claim 3 including rotatable valve means connected to one end of said pair of conduits, and a pair of passages in said rotatable valve means whereby on rotation of said valve means one of said passages is connected to a respective conduit to convey fluid therethrough.

5. Evaporator apparatus as set forth in claim 3 in which the inner concentric conduit is provided with an end opening and the outer conduit is provided with a plurality of nozzle apertures around the periphery thereof throughout its length.

6. In evaporator apparatus, a rotation joint comprising hollow adapter means having a free end adapted to receive an evaporation vessel, gear means connected about said hollow adapter means adjacent the opposite end, a housing, said gear means mounted for rotation in said housing, a stationary hollow adapter connected in axial alignment adjacent the said opposite end of said hollow adapter means, a packing ring axially connected between said hollow adapter means and said stationary hollow adapter and having an outwardly extending portion adjacent said gear means, means connected to said gear means and in contact with said outwardly extending portion about its periphery, and motor means connected to said housing and connected to rotate said gear means, packing ring and said hollow adapter means relative to said housing and stationary hollow adapter.

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