Aug. 6, 1946. G. L. ROYER ET AL 2,405,167

APPARATUS FOR FLUID TREATING UNDER CONTROLLED CONDITIONS

Filed Sept. 3, 1943 2 Sheets-Sheet 1

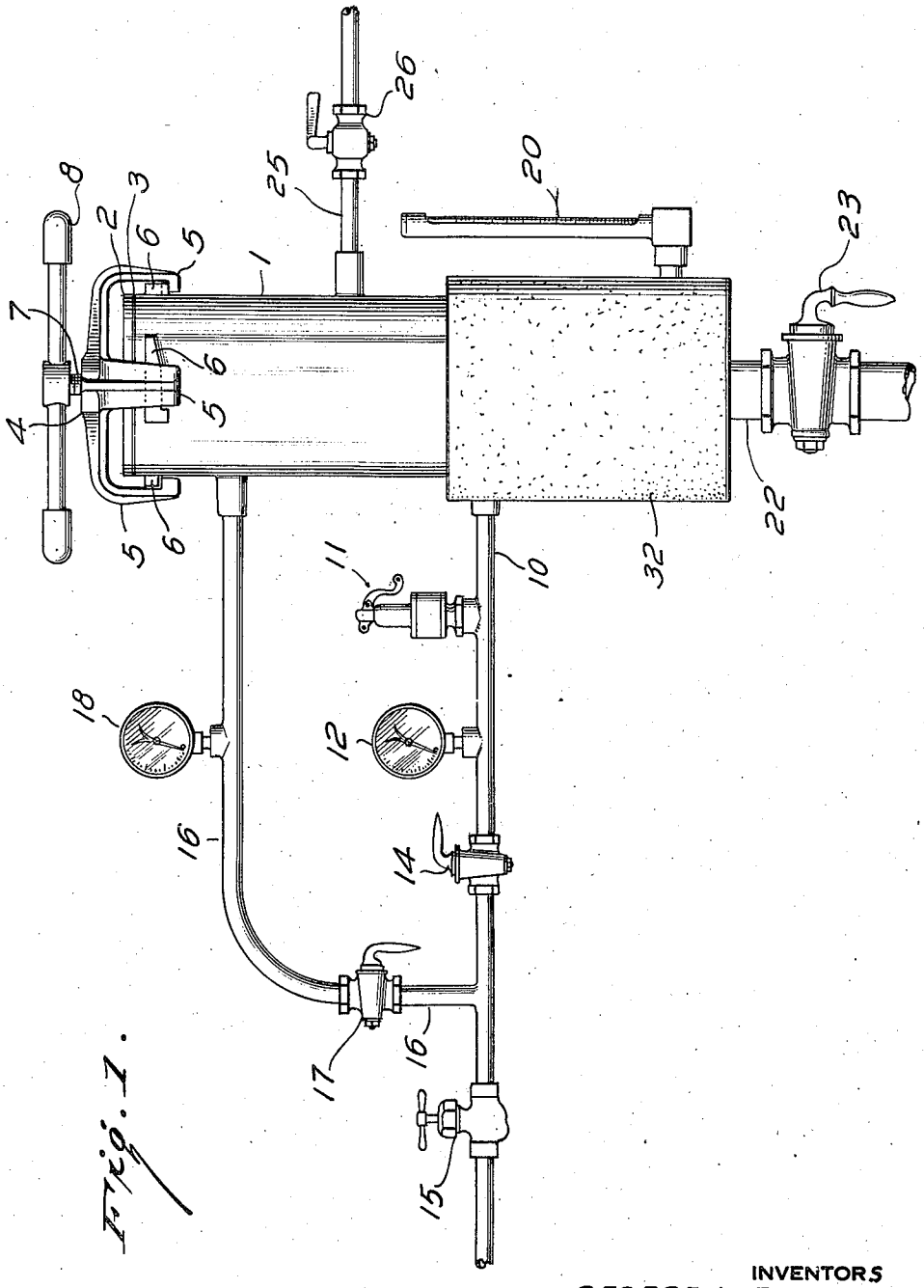

Fig. 1.

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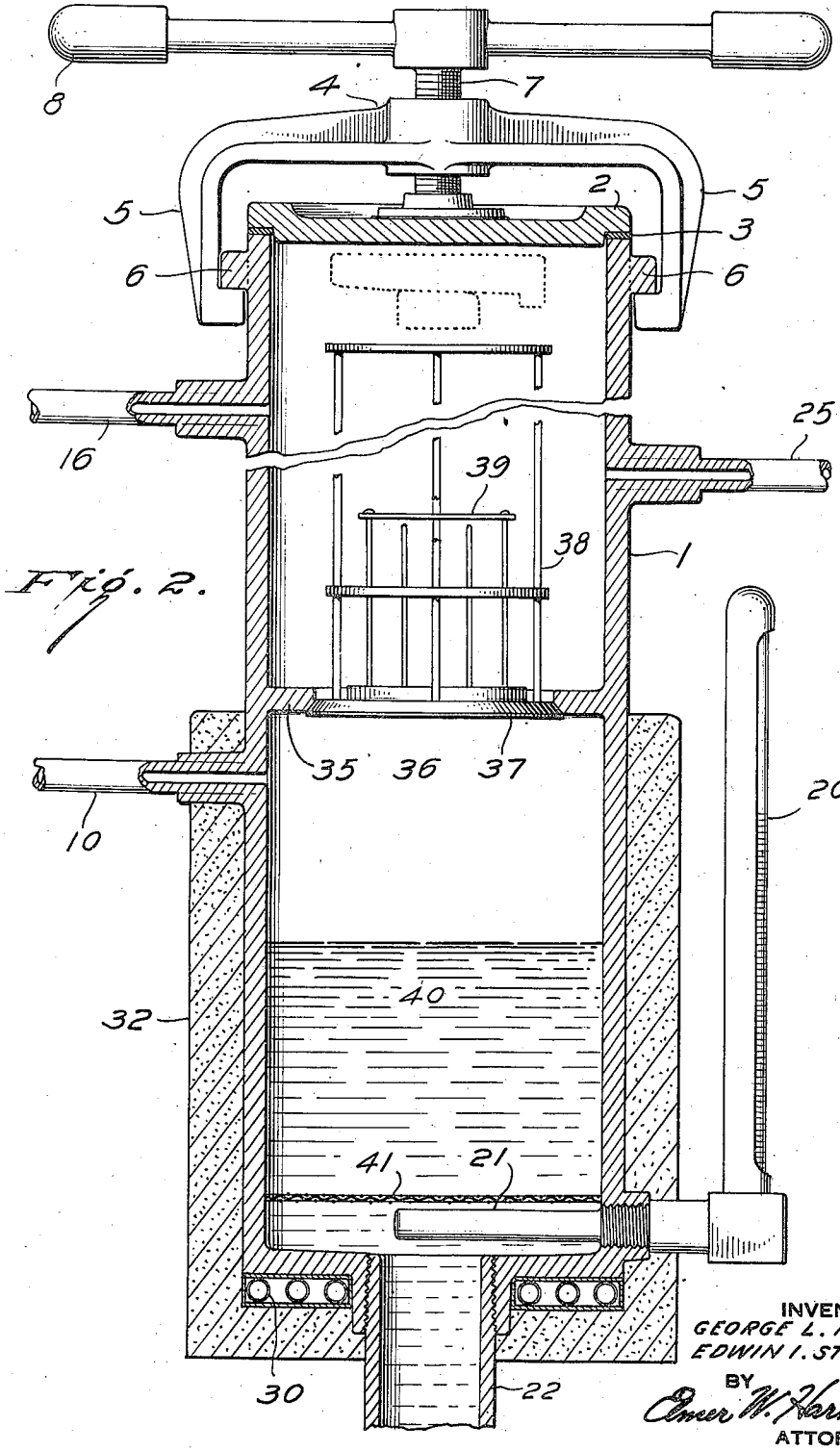

Fig. 2.

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APPARATUS FOR FLUID TREATING UNDER CONTROLLED CONDITIONS

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5 Claims. (Cl. 68—3)

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This invention relates to an apparatus for the treatment of a material with a liquid under controlled temperature and pressure conditions. More particularly, the apparatus is capable of establishing instantaneous contact between a sample to be treated and a liquid at a predetermined temperature, while a positive or negative pressure is maintained in the atmosphere above the liquid.

Particularly for the study of various chemical reactions at elevated temperatures and pressures, an apparatus capable of quickly contacting a sample and a liquid under pre-determined conditions has been needed. In addition it is also desirable that the apparatus be adapted to permit quickly removing the liquid from contact with the sample. Preferably too, some provision should be made for washing any residual liquid retained by the sample therefrom. Such an apparatus should be capable of accomplishing these objects, and at the same time should be relatively simple in structure, easily operated, and easily cleaned.

It is, therefore, the object of the present invention to provide such an apparatus, capable of accomplishing these desired objects. According to the present invention, such an apparatus has been constructed and has proved highly useful in conducting studies on rates of reaction at elevated temperatures and pressures.

One form of such an apparatus is shown in the accompanying drawings in which:

Figure 1 is a general vertical view showing the apparatus and the arrangement of its external connections, and

Figure 2 is a vertical section through the reaction chamber showing the internal arrangement and connections.

With reference to Figure 1, the apparatus is seen to comprise a reaction vessel 1 closed by cover 2. Cover 2 is seated on gasket 3 and is held in place by means of clamp 4, comprising a plurality of hooks 5 adapted to engage lugs 6 on the exterior of the reaction vessel. Clamp 4, and therefore cover 2, is tightened by means of screw 7 and handle 8.

Slightly below the vertical center of the vessel, pressure inlet pipe 10 is connected to the reaction vessel 1. Pipe 10 carries a safety valve 11 of conventional design, a pressure gage 12 and a shut-off valve 14. Conduit 10 is also connected to some source of fluid under pressure, which being conventional and forming no part of the present invention is not shown. Conduit 10 is also provided with a shut-off valve 15 by means

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of which the supply of fluid under pressure may be shut off from the entire apparatus.

With respect to the source of fluid under pressure, any of many modifications may be made to serve the purposes of the present invention. The fluid may be either a gas such as air, nitrogen, hydrogen or the like or may be a vapor such as steam. However, at the pressure under which the operation of the present device is contemplated, steam will ordinarily be fully saturated and perhaps more properly designated as a gas. The pressure supply may be a tank or cylinder from which compressed gas is delivered, either with or without a reducing valve. Again, a compressor directly connected through a suitable reducing valve may be used. If steam is used, the source may be directly connected to the generator. Ordinarily, air is the fluid which would be used and the most convenient source of pressure is usually a compressor operating through a reducing valve.

Between valves 14, and 15, by-pass conduit 16 is connected to conduit 10. By-pass conduit 16 is also connected to the reaction chamber 1 at a point in the upper half of the reaction vessel. Conduit 16 also carries a shut-off valve 17 and a pressure gage 18. By this arrangement, the operator may introduce fluid, independently and at different pressures, into either the top or bottom of the reaction vessel.

Since for the purposes of the present invention it is of primary importance that the operator know the exact temperature of the liquid in the reaction chamber 1, the latter is ordinarily equipped with a thermometer 20. However, the thermometer may be readily replaced by some equivalent device such as a thermocouple or the like.

Also of primary importance in the operation of the device of the present invention is the necessity for being able to quickly remove the liquid at the designated time. For this purpose the reaction vessel is equipped with a relatively large bottom outlet 22 which is normally closed by shut-off valve 23 during the dyeing operation. Discharge may be made into any suitable disposal system. Because of the pressure in the vessel, discharge is very rapid.

It is also important that the material being dyed be promptly cooled in order that the reaction may stop when the liquid is discharged. Otherwise, dyeing would continue to some indeterminate extent. To accomplish this object the apparatus is equipped with water inlet 25, the flow in which is controlled by valve 26. Provision

of the water inlet also is of assistance in stopping the dyeing action at the predetermined point by washing out any residual dye liquor which may be retained by the material.

Figure 2 shows the interior details of the reaction vessel. In order to bring liquid 40 in the reaction vessel to the desired temperature, heating coil 30 is provided in the bottom of the reaction vessel. While these are represented in Figure 2 as electrical resistance coils, the actual source of heat may take any other desired form. For example, steam coils or electrical induction heating may be used without departing from the scope of the present invention. The location of the thermometer 20 with its bulb 21 immersed in the liquid immediately above the heating coil is also shown. A grill 40, or other equivalent support for the disk 37, when the latter is immersed in the liquid is provided. This grill serves the double purpose of protecting thermometer bulb 21 and preventing disk 37 from closing the opening into the discharge conduit 22. While not absolutely necessary to do so, it has also been found well to lag the lower part of the apparatus, as by means of the insulating cover 32.

Approximately midway the height of the reaction chamber is located an annular collar 35. This collar is beveled along its lower inner periphery in order to make a pressure tight fit with the beveled upper periphery of a pressure disk 37. When gas or other fluid under pressure is admitted under the disk through conduit 10, disk 37 is held against collar 35. By this arrangement, fluid under pressure admitted to the lower half of the reaction vessel not only controls boiling of the liquid, but also when the pressure in the lower part of the vessel exceeds that in the upper, the two are separated from each other by disk 37.

Mounted on disk 37 is a cage of stainless steel or some other corrosion resistant rods 38. This cage is open at the top and a skeletal frame 39, also of some corrosion resistant material, upon which the sample of material to be treated may be wrapped is inserted therethrough and supported on the upper surface of the disk 37. In this way the sample is held completely out of contact with the liquid in the lower half of the reaction vessel.

The operation of the device is believed to be apparent from its description. The liquid to be tested, ordinarily after being preheated, is placed in the lower half of the reaction vessel and heat is turned on. Disk 37 is lifted into place against collar 35 by any convenient means such as a hooked wire and sufficient pressure is built up through the inlet from conduit 10 to hold the disk in place. A sample to be treated is then mounted on the spindle 39, or an equivalent holder, and set within the cage on the upper surface of the disk 37. Cover 2 and gasket 7 are then set in place on the top of the apparatus and fastened by clamp 4.

Heating is continued until the liquid reaches the desired temperature, the pressure in the lower part of the vessel meanwhile being adjusted to the desired degree. When the desired temperature has been reached, valve 14 on conduit 10 is closed and valve 17 on the by-pass 16 is opened to admit pressure into the top part of the apparatus. When the pressure in the top part approximates that in the lower part, disk 37 is no longer supported by the pressure differential and falls to the grill 41 or some equivalent support, just above the thermometer bulb 21. In this way

the sample is brought at once into contact with hot liquid which by means of the present invention is at the desired temperature and pressure.

The fall of disk 37 can be easily heard and the operator thereby is able to start timing the duration for which the reaction is to be carried out. Since the entire vessel is pressure tight, once contact has been established between the material to be treated and the treating fluid, the apparatus may be disconnected from the fluid pressure supply by closing valves 15 and 17, valve 14 being normally closed at this stage of the operation.

Upon expiration of the predetermined time, the operator may stop the reaction at will. This is done almost instantaneously by opening valve 23 and allowing the treating liquid to flow out through the conduit 22 to a suitable receiver or a disposal system. At the same time, or immediately thereafter, the operator may if so desired open valve 26 and allow water for cooling and washing to flow through the apparatus, thus quickly reducing the temperature and removing the residual liquid held by the sample. After completing the washing and cooling operation, the cover may be removed and the sample withdrawn as by means of a hooked wire or any other suitable means.

It is obvious that many modifications in the operation of the apparatus may be readily made. Various combinations of pressure and fluid temperature may be used. The present apparatus also has an important added advantage in that if treatment under vacuum is desirable it may be readily carried out simply by attaching an evacuator to the conduit 10. Instead of building up a pressure in the lower half of the vessel to hold the disk 37 against collar 35, it is held in place by evacuating the upper part of the reaction vessel while allowing the lower part to remain at substantially atmospheric pressure. Contact between sample and liquid can then be made by evacuating the lower part of the chamber until both parts are at approximately the same relative pressure at which time the disk will fall.

While much of the discussion has been concerned with the dyeing of fabrics, the apparatus is applicable to many other operations. For example, it may be desirable to use the apparatus in carrying out the reaction between two liquids or a solid and a liquid in order that the reaction time may be readily controlled. In other situations, one of the reactants may be only fluid under high temperature and pressure conditions or may be a substance which is highly volatile except under suitable pressure. Another application is in the impregnation of solids with a material which is only sufficiently fluid for adequate penetration at high temperatures. Still another application is in those cases where it is desired to treat a solid with a material dissolved in a solvent which is itself injurious to the solid for a sufficiently short time to minimize the side reaction. Other possible applications will be readily apparent.

We claim:

1. In a pressure-treating device, adapted to contact a sample and fluid under controlled temperature and pressure conditions, a reaction vessel in combination with means for supplying heat to said vessel, movable means for separating said vessel into two separate superposed sections, external means for admitting fluid under pressure into the lower section whereby a pressure differential is created between said sections, means within the vessel adapted to hold said separating

means in operative position when the pressure differential exists between said sections, and external means for independently admitting sufficient fluid under pressure into the upper section to overcome said pressure differential, whereby said separating means is moved from operative position; said separating means comprising an impervious plate and said means for holding the separatory means in operative position comprising a shoulder on the inner surface of the reaction vessel, said shoulder being adapted to form a pressure tight fit with said plate.

2. An apparatus according to claim 1 in which the upper face of said plate carries a sample supporting means.

3. An apparatus according to claim 1 having an outlet of sufficient size to permit rapid removal of liquid from the reaction vessel and a means for admitting cooling and washing fluid to the interior of said reaction vessel.

4. In a pressure-treating device, adapted to contact a sample and a fluid under controlled temperature and pressure conditions; a reaction vessel in combination with movable means for separating said vessel into two separate sections, a material retaining means so mounted on said separating means that when said separating means is in operative position the material retaining means is enclosed within one of said sections; conduit means for independently admitting fluid under pressure into each of said sections; means within the vessel adapted to hold

said separating means in operative position when a pressure differential exists between said separated sections; and control means in said conduit means to independently regulate the pressure in each of said separate sections, whereby when the pressures in each of said sections are substantially equal said separating means is released from operative position.

5. In a pressure-treating device, adapted to contact a sample and a fluid under controlled temperature and pressure conditions, a reaction vessel in combination with means for supplying heat to said vessel, movable means for separating said vessel into two separate sections, a material holding means so mounted on said separating means that when said separating means is in operative position the sample holding means is enclosed within one of said sections; conduit means for independently admitting fluid under pressure into each of said sections, means within the vessel adapted to hold said separating means in operative position when a pressure differential exists between said separated sections; and control means in said conduit means to independently regulate the pressure differential in each of said separate sections, whereby when the pressures in each of said sections are substantially equal said separating means is released from operative position.

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