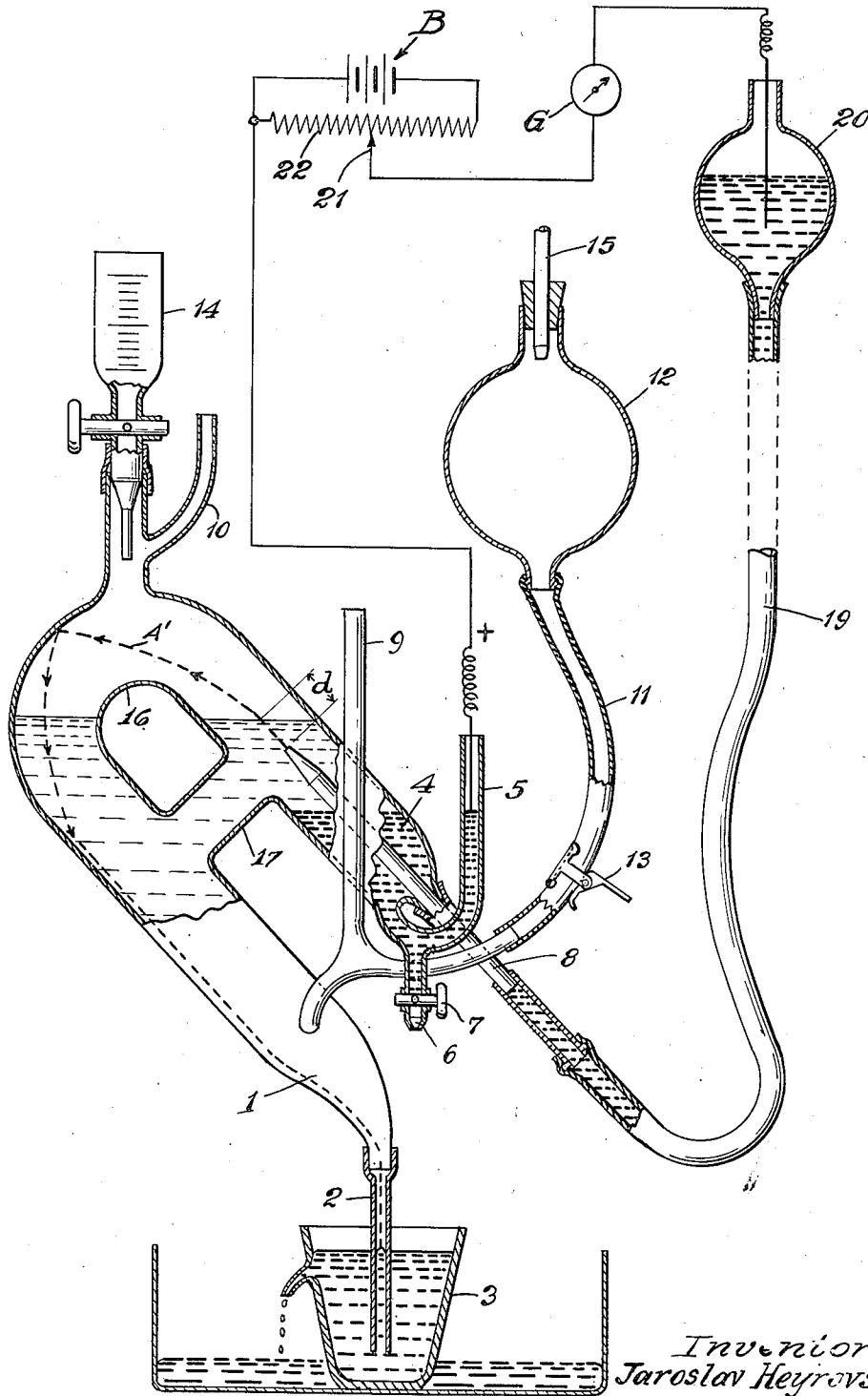


March 14, 1950

J. HEYROVSKY
APPARATUS FOR ELECTROANALYSIS

2,500,284

Filed Oct. 1, 1947



Inventor
Jaroslav Heyrovský
by Singer, Ehler, Stern & Carlberg
Attorneys

UNITED STATES PATENT OFFICE

2,500,284

APPARATUS FOR ELECTROANALYSIS

Jaroslav Heyrovský, Prague, Czechoslovakia, assignor to Zbrojovka Brno Narodni Podnik (also named Brno Arms Factory, National Corporation), Prague, Czechoslovakia, a corporation of Czechoslovakia

Application October 1, 1947, Serial No. 777,224
In Czechoslovakia October 2, 1946

4 Claims. (Cl. 204—195)

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This invention relates to apparatus for electroanalysis particularly to the type using a capillary mercury electrode.

Electroanalysis apparatus with the dropping mercury electrodes are known, in which mercury drops out, or under greater pressure streams down from the vertically placed capillary. This type of apparatus has certain disadvantages and is especially unsuitable for quantitative analytical purposes, since it is essential that the mercury electrode should maintain a constant surface area. The vertical jet of mercury splits up into many droplets and this makes the surface insteady and indefinite. This invention removes this disadvantage by letting the mercury jet stream from the capillary under pressure at an angle to the surface out of the solution to be analyzed and then directing the stream of mercury sidewise so it does not rebound onto the surface of the solution near the electrode. This adjustment gives a steady and constant surface of the mercury jet, since the jet leaves the solution before splitting up into droplets.

In accordance with this invention there is provided apparatus for electroanalysis including a vessel having a mercury capillary electrode under pressure and inclined so that a mercury jet is projected through the solution, obliquely to its surface and conducted sidewise, so that the mercury does not fall back to the surface of the solution near the electrode.

The invention will now be described with reference to the accompanying drawing.

The vessel has substantially the shape of the letter A whose main axis is arranged inclined with respect to the horizontal and comprises two substantially parallel tubular arms 1 and 4 connected at their upper ends by a curved tubular portion 16 and connected between their ends by a tubular cross arm 17. The mercury layer, forming one electrode, e. g. the anode, is placed in the lower portion of the higher placed arm 4 of the vessel. To this arm an outlet 6 with a tap 7 is fused on and a tube 5 through which the mercury is introduced into the arm 4. The other lower placed arm 1 of the A-shaped vessel has attached to its lower end an indiarubber tube 2, which is immersed into a crucible 3 filled with mercury. This crucible has near its upper edge a hole, through which the mercury flows out thereby keeping a constant level of mercury in the crucible and consequently also a constant level of the solution in the arm 4. The solution to be analyzed is introduced into the vessel through the side-tube 55

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9 and rests thus on the mercury in the arm 4 as well as on the mercury in the crucible 3.

In the arm 4 is placed the capillary tube 8, through which mercury streams out traversing the solution to be analyzed and constituting the other electrode, e. g. the cathode. The capillary tube 8 is at an angle to the surface of the solution and its tip is placed a certain distance d under the surface of this solution, but extends above the level of the mercury in the arm 4. The mercury jet is thus projected at an angle, say 45° , to the surface of the solution, passes the solution without splitting and hits the upper wall of the vessel as indicated by the broken line A'. Here the mercury rebounds into the arm 1, without hitting the surface in the arm 4 around the electrode, and flows down into the crucible 3, where it escapes through the opening. Since the level of the mercury in this crucible is constant, it is evident that also the distanced between the surface of the solution and the tip of the capillary tube 8 remains substantially constant. However, if for some reason this distance is changed, the original distance is easily restored by the addition of mercury through the side-tube 5 or by letting some mercury out through the tap 7 and tube 6. Such a change may happen e. g. when an inert gas (like nitrogen) is introduced into the vessel with the solution by means of the tube 9 to escape through the tube 10; hereby small drops of the solution may be splashed by the bubbles of the gas onto the walls inside the vessel. In this case it is necessary to add a little mercury through the side-tube 5 in order to raise the level of the solution. It may also happen that some drops of mercury fall on to the mercury surface in the arm 4; and in such a case the same amount of mercury has to be let out through the tap 7.

The solution to be analyzed is introduced into the A-shaped vessel up to the level shown in the drawing. The mercury body in the arm 4 serves as anode and the mercury jet supplied under pressure by the tube 19 from the reservoir 20 forms the cathode. The voltage between these two electrodes is increased by sliding the contact 21 along the wire resistance 22, the ends of which are connected to opposite terminals of the battery B. The variations in current caused by the increasing voltage are indicated by the galvanometer G. The electroanalysis which is carried out with the streaming mercury electrode surpasses in quantitative and qualitative determinations the sensitivity and accuracy of the ordinary polarographic analysis which is an analysis performed with the dropping mercury electrode. Thus traces of lead

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or cadmium in concentration as small as $2.5 \times 10^{-5}m$. (i. e. 1 part per 200,000 parts of water) which produce in ordinary polarographic analysis with the greatest sensitivity of the galvanometer waves about 12 millimeters high, will produce with the streaming electrode waves which are 4 centimeters high with a sensitivity reduced to $1/30th$. The streaming electrode is thus 100 times more sensitive than the dropping electrode.

In the qualitative sense the use of the streaming mercury electrode offers the advantage of distinguishing between thallium and lead, cadmium and indium, which is not possible with the dropping mercury electrode. When connected to a cathode ray oscillograph the streaming mercury electrode yields figures which permit the determination, both qualitatively and quantitatively, of organic isomers such as the ortho-, meta-, and para-nitrophenols or nicotinic, isonicotinic and picolinic acids.

The new vessel is also adapted for carrying out polarometric titrations in the atmosphere of an inert gas. In that case a burette 14 is fixed to the top of the A-shaped vessel. After each addition from the burette the solution in the vessel is stirred by the streams of the inert gas and the level of the solution is restored by letting out some of the solution by means of the clamp 13 into the rubber tube 11 or glass-bulb 12. After the next addition of the burette, the solution has to be expelled from the bulb 12 and tube 11 by a stream of the gas passed through 15 into the vessel and stirred.

Then a part of the solution is again let out into the bulb 12 to restore the original level of the solution in the vessel; the exact adjustment may be effected by adding or letting out some of the mercury contained in the arm 4.

What I claim as my invention is:

1. An apparatus for electroanalysis including an A-shaped vessel having two substantially parallel tubular arms connected at the upper ends by a curved tubular portion and connected between their ends by a tubular cross arm, said vessel being positioned with its main axis inclined to the horizontal so that the lower portion of one of said substantially parallel tubular arms is positioned higher than the other, a mercury body in the lower portion of the higher placed tubular arm and forming one electrode, a capillary tube extending axially into the lower portion of said last named arm and extending through said mercury body to a point above the surface thereof but below the surface of the solution in the vessel to be analyzed, a conduit connected to the lower end of said capillary tube for supplying mercury under pressure to said capillary tube from the exterior of said vessel the pressure causing said last named mercury to be discharged in the form of a jet through said solution and into

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the upper curved portion of the vessel from which it is deflected by the wall of said curved portion into the other tubular arm, said jet forming the second electrode, and means for discharging the mercury deflected into the other lower placed tubular arm from the lower end of the latter.

2. An apparatus for electroanalysis including an A-shaped vessel having two substantially parallel tubular arms connected at the upper ends by a curved tubular portion and connected between their ends by a tubular cross arm, said vessel being positioned with its main axis inclined to the horizontal so that the lower portion of one of said substantially parallel tubular arms is positioned higher than the other, a mercury body in the lower portion of the higher placed tubular arm and forming one electrode, a capillary tube extending axially into the lower portion of said last named arm and extending through said mercury body to a point above the surface thereof but below the surface of the solution in the vessel to be analyzed, means for supplying mercury under pressure to said capillary tube and to discharge said mercury in the form of a jet through said solution and into the upper curved portion of the vessel from which it is deflected by the wall of said curved portion into the other tubular arm, said jet forming the second electrode, said last named means including a discharge tube attached to the lowermost end of said lower placed tubular arm, and a crucible filled with mercury into which the lower end of said discharge tube extends, said crucible being provided with means for maintaining the surface of mercury therein at a constant level.

3. An apparatus for electroanalysis is claimed in claim 1, including a side tube connected to the lower end of the higher placed arm of said A-shaped body for supplying said body of mercury in said higher placed tubular arm of said A-shaped body with small and finely adjustable additional amounts of mercury.

4. An apparatus for electroanalysis as claimed in claim 3, including means connected to the lowest portion of said side tube for removing from said body of mercury in said higher placed tubular arm of said A-shaped body small amounts of mercury in finely adjustable quantities.

JAROSLAV HEYROVSKÝ.

REFERENCES CITED

The following references are of record in the file of this patent:

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Number	Name	Date
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