United States Patent

[11] **3,626,234**

[72] Inventor **Werner Grimm**
Grossauheim, Germany
[21] Appl. No. **58,847**
[22] Filed **July 28, 1970**
[45] Patented **Dec. 7, 1971**
[73] Assignee **RSV Prazisionsmessgerate GmbH**
Hechendorf/Pilsensee, Germany
[32] Priority **Mar. 1, 1969**
[33] **Germany**
[31] **P 19 10 461.1**

[54] **GLOW DISCHARGE TUBE**
2 Claims, 3 Drawing Figs.

[52] **U.S. Cl.** **313/210,** 313/231, 313/237, 356/86
[51] **Int. Cl.** **G01j 3/12,** H01j 17/04, H01j 17/26
[50] **Field of Search** 356/86; 313/210, 231, 237

[56] **References Cited**
UNITED STATES PATENTS
3,543,077 11/1970 Grimm 313/210

Primary Examiner—John Kominski
Attorney—Singer, Stern & Carlberg

ABSTRACT: A glow discharge tube with a tubular anode extending into an annular cathode body has clamped against the outer face of the cathode a test body in the form of a thick circular disk which in the surface engaging the cathode body is provided with one or more bores arranged in a circular area opposite and smaller than the end of the tubular anode. The bore or bores are adapted to receive the samples of the material to be analyzed, such as short pieces of wires or wire-shaped metal bodies, the ends of which project just a little distance, if any, beyond the surface of the test body engaging the cathode body.

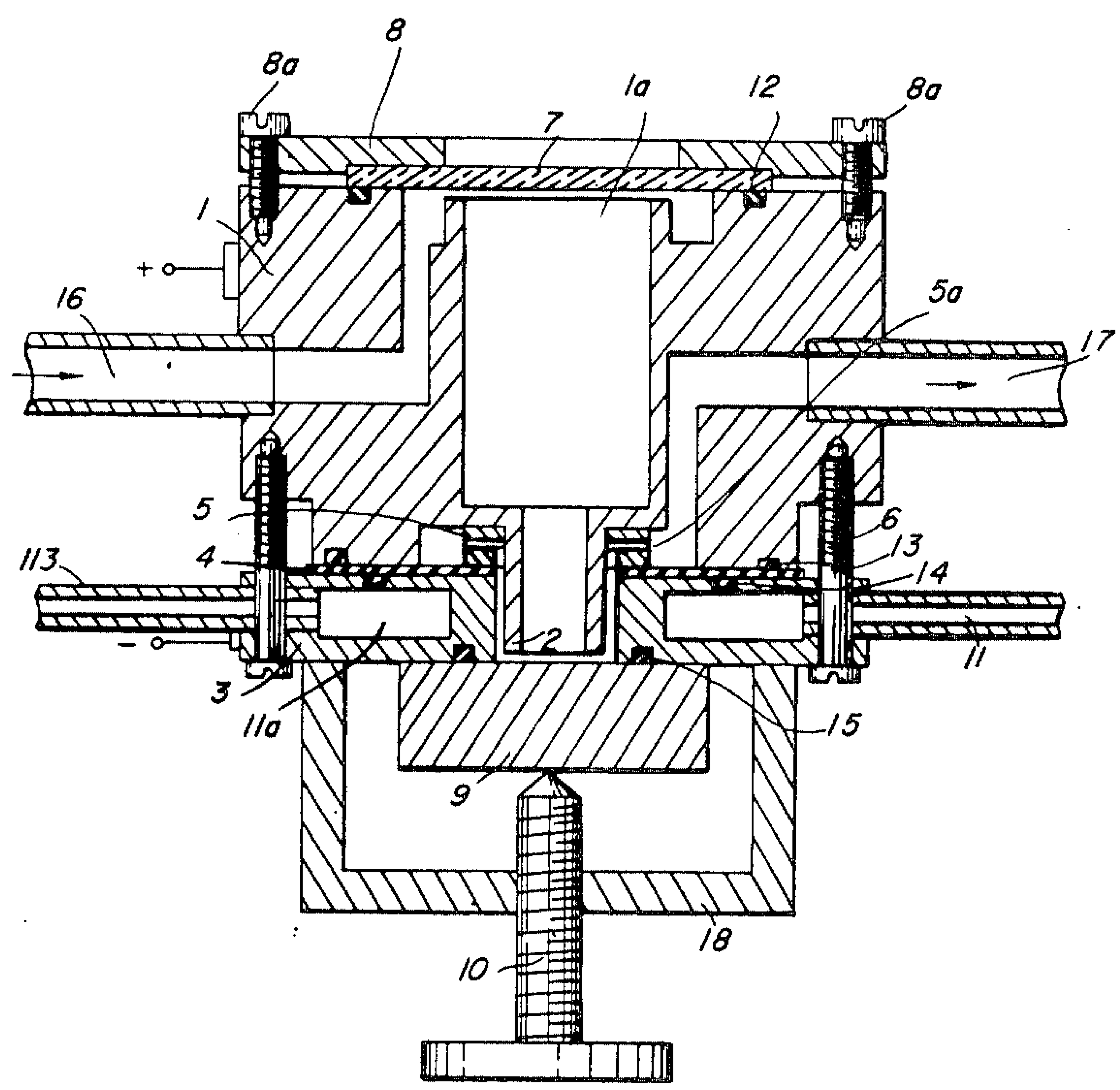


Fig.1

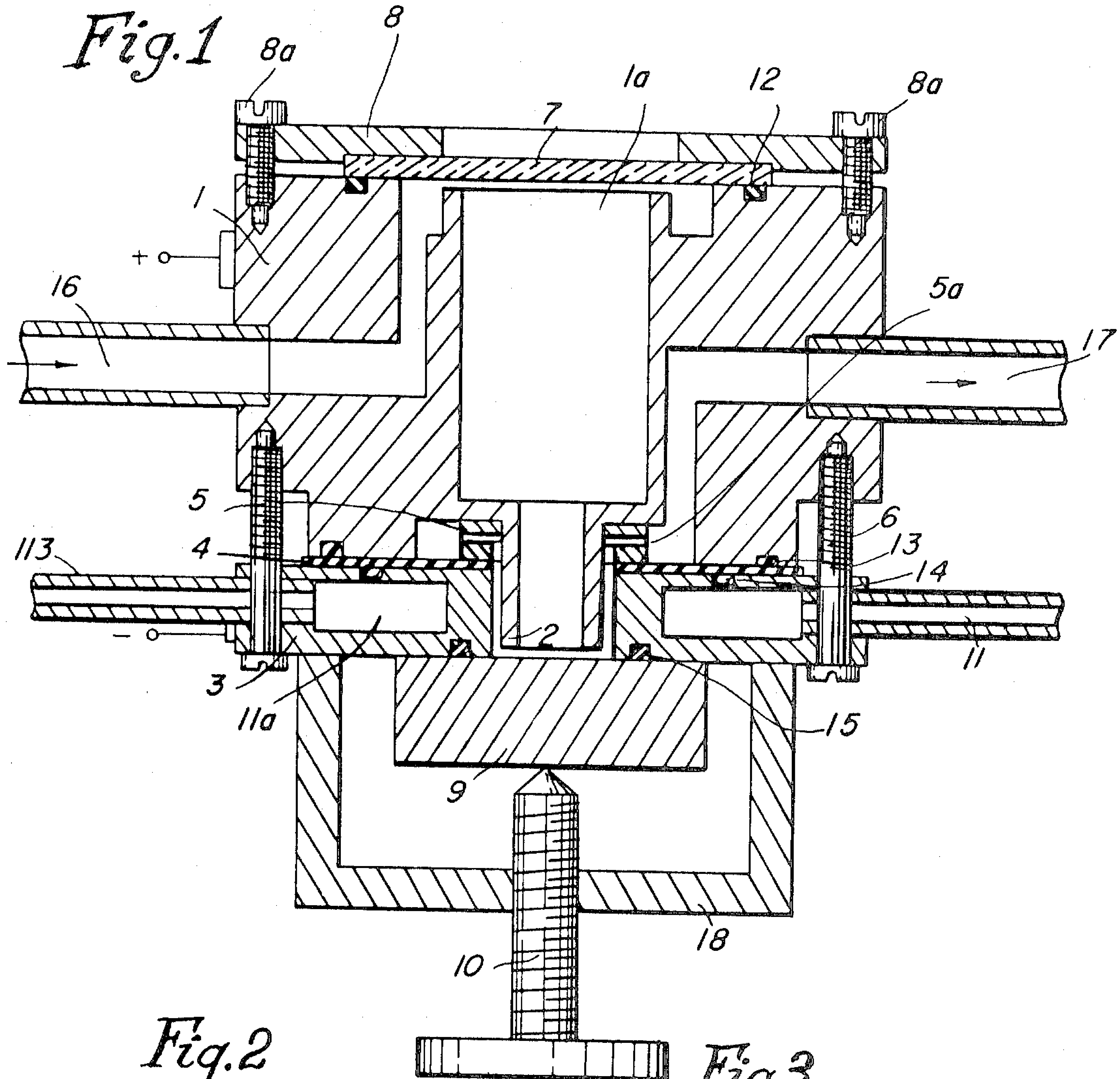


Fig.2

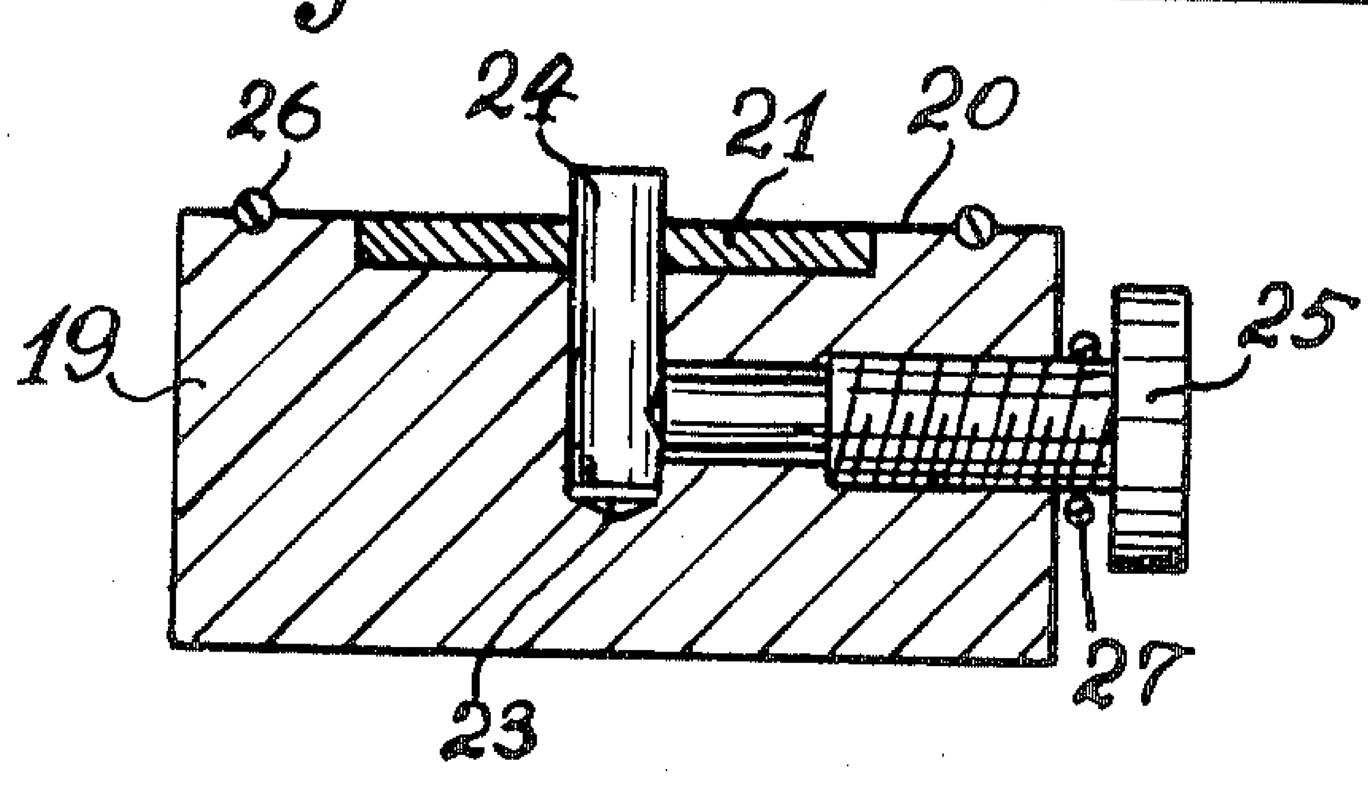
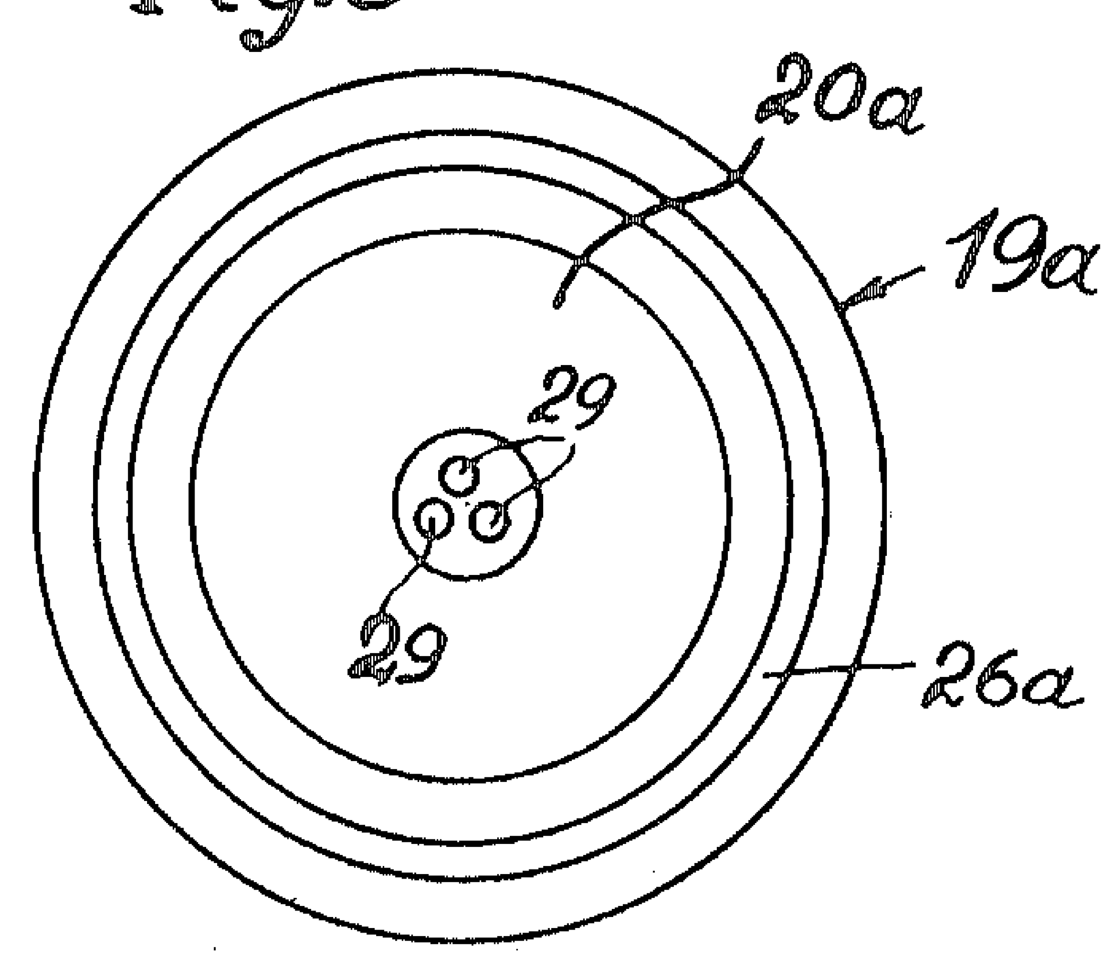


Fig.3



GLOW DISCHARGE TUBE

The invention relates to a glow discharge tube for the analysis of wires and wire-shaped metal bodies and comprises a further development and improvement of the inventor's prior U.S. Pat. application Ser. No. 735,304, filed June 7, 1968.

In this prior patent application is disclosed a glow discharge tube for qualitative and quantitative spectral analysis in which the anode body on its end facing the cathode is provided with a nipple and that a test body of electric conductive material is pressed against the cathode body, whereby the test body comprise entirely or partly of a substance to be analyzed.

The surface of the test body has to be plane or almost plane and therefore in such an arrangement it is not possible to analyze wires or wire-shaped bodies owing to their curved or round shape.

It is an object of the invention to adapt a glow discharge tube as disclosed in the prior patent application Ser. No. 735,304 for the analysis of wires and wire-shaped bodies, such as twisted wires, or strips produced by wires passed through flat rollers, such an analysis is to take place with a high accuracy and within a very short time.

In accordance with the invention this object is attained by pressing against the cathode body a test body provided with at least one bore for the reception of the body to be analyzed and that the side facing the anode comprises an electric conductive material which is difficult to vaporize and is not analyzed with the wire and has a plane or almost a plane surface, whereby the cross section of the bore is adapted to fit the cross section of the body to be analyzed, the condition being that between the wall of the bore and the perimeter of the inserted body the smallest possible play or clearance be produced. The clearance in the bore should be not more than 0.15 mm. and preferably should be less than 0.10 mm. The end or peak of the body to be analyzed and facing the anode should have a clearance within +2.0 mm. and −1.0 mm., preferably +1.0 mm. to −0 mm. from the level in which the edge of the test body facing the anode is disposed.

Another object of the invention is to employ a test body provided with a plurality of bores for the reception of bodies to be analyzed, whereby these bores are arranged within a range whose diameter is at least 0.50 mm. smaller than the diameter of the bore of the anode nipple, so that this range is directly in alignment with the cross section of the bore in the anode nipple.

The drawing illustrates by way of example a few embodiments of the invention.

Referring to the drawing:

FIG. 1 illustrates diagrammatically an axial sectional view of the glow discharge tube of the invention;

FIG. 2 illustrates diagrammatically an axial sectional view of the wire holding test body of the invention, and

FIG. 3 illustrates in a top plan view a wire holding test body for a plurality of wires to be analyzed.

Referring to FIG. 1 of the drawing, the housing of the glow discharge tube consists substantially of a hollow anode body 1 provided with a downwardly extending anode nipple 2 and an annular cathode body 3 which concentrically surrounds with clearance the anode nipple 2. For electrical insulation, there is disposed between the lower face of the anode body 1 and the upper face of the cathode body a disc 4 of 0.2 mm. thickness made of nonconducting material such as Teflon. The disc 4 is pressed by a ring 5 consisting of copper beryllicum and mounted on the nipple 2 against the upper face of the cathode body 3. The ring 5 is provided with six radial bores 5*a* of 1.5 mm. diameter. Screws 6 made of electrically nonconducting material for instance Novatex hold the anode— and cathode-bodies together. A circular disc 7 made of optically plane-polished quartz glass, is connected by means of an annular plate 8 and screws 8*a* to the upper end of the anode body and thereby close the discharge tube on the upper spectrographical end. The test body 9 to be analyzed forms the bottom of the discharge tube and is pressed by means of a tommy screw 10 tightly against the lower end of the cathode body 3. The screw 10 is mounted in a bracket 18 which may be attached to the cathode body 3.

In this discharge tube the distance between the upper face of the test body 9 and lower end of the anode nipple 2 amounts to 0.2 mm.; the distance between the cathode body 3 and the anode nipple 2 is 0.25 mm; the axial length of the anode nipple 2 is 12 mm. the outer diameter of the nipple 2 is 10 mm. and the inner diameter of the nipple is 6 mm.

In order to quickly carry off the heat which is released during the discharge, the cathode body 3 is made of a good heat-conducting material, such as a copper-beryllium-alloy, and is provided with a water-cooling system 11, 11*a*, 11*b*. Four sealing rings 12, 13, 14, 15 insure a vacuum-tight closure of the discharge tube.

The carrier gas flows continuously through the gas inlet nipple 16 into the anode cavity 1*a* and is constantly withdrawn by an outlet nipple 17 attached to a pump so that in the cavity of the anode nipple 2 the required operating pressure is maintained; and as result of the dimensioning a predetermined pressure drop is established between this cavity and the radial bores 5*a* in the ring 5.

It has been found advantageous to utilize as a carrier gas an inert gas, for example argon, because with the latter, no band spectrum occurs. Preferably one operates with a gas pressure of particularly 2 to 20 torr.

As energization device comprising a source of direct current with a control range of 0.02 to 0.5 amperes and 3000 volts voltage is sufficient. For the current stabilization it is advisable to utilize a device with high inner resistance of about 10 kΩ. The positive terminal (+) is connected to the anode body 1 and the negative terminal (−) of the direct current source is connected to the cathode body 3, as shown or in any other suitable manner.

The glow discharge tube is secured with the anode body having a zero potential to a spectrograph, in such manner that their optical axis coincide.

With the glow discharge tube described in the foregoing by way of example, an exchange of the test bodies is possible within a period of about 20 seconds. A gas pressure of 2 to 20 torr and a current intensity of 0.02 to 0.5 amperes are sufficient in order to keep the annealing and exposure time below 60 seconds. With the aid of a photoelectric evaluation device, the result of the analysis may already be available after about 1.5 minutes.

The accuracy of the analysis obtained is considerably better than with the conventional optical spectralanalytic methods. Thus by means of the glow discharge tube in accordance with the invention, it is possible, for example, to determine nickel in a 50 percent nickel-iron-alloy to .6 percent while by means of a hollow cathode discharge tube, the same element may be determined in the same alloy only at about 1.5 percent.

The advantages attained by the invention, consist particularly therein, that in a routine way, substances without great expenditure of effort may be analyzed within a very short period of time and with greater accuracy than previously spectralanalytically, without requiring an exchangeable auxiliary electrode for the discharge tube.

FIG. 2 illustrates a test body constructed in accordance with the present invention. It comprises a cylindrical metal body 19 having a diameter of 40 mm. and a thickness of 20 mm. The upper surface 20 of this metal body facing the anode is provided with a circular recess into which is cemented an apertured disk 21 consisting of spectroscopic carbon and having a diameter of 25 mm. and a thickness of 2 mm. A central bore 23 in this body 19 has a depth of 12 mm. and is used for inserting therein the sample 24 which is adapted to be clamped in said bore 23 by a radially disposed clamping screw 25. Sealing rings 26 and 27, one on top of the body 19 and one extending around the body of the clamping screw 25 respectively, seal the sample 24 against the atmosphere in the operative position of the glow discharge tube. The clamping screw 25 in its clamping position assures that the sample 24 will be given the full cathode potential.

Sample holders as illustrated in FIG. 2 having a bore of 5 mm., 2.1 mm. and 1.5 mm. when having inserted therein wires of different thicknesses of a cobalt-nickel-iron-beryllium alloy are being pressed one after the other against the cathode body of the described glow discharge tube for producing an analysis of the wires.

When the cross section of the bore is so adjusted, with respect to the cross section of the sample **24** that the clearance does not exceed .15 mm., then practically no hollow cathode effect is obtained. If, however, the clearance is greater, then a part of the discharge will be drawn into this opening and does no longer contribute to the atomization and stimulation of the material of the sample. Preferably, a clearance of less than .1 mm. should be selected. To exclude a hollow cathode effect it is also necessary to clamp the sample to be analyzed in such a manner that its top or peak is not disposed more than 1 mm. below the upper surface of the disc **21** facing the anode. If this peak projects more than 2.0 mm. from the bore **23** the same is apt to melt during the discharging operation. It is particularly advantageous when the peak of the sample **24** on one hand is not arranged below the upper face of the disc **21** and on the other hand is disposed not more than 1.0 mm. above the upper plane of the disc **21**.

It is also possible to employ, according to FIG. **3**, a sample holder **19***a* provided with a plurality of bores **29**. Such a sample holder is recommended when the wires to be analyzed have a diameter of less than 1.50 mm. The employment of a plurality of samples assures that the discharge space will receive a sufficient quantity of atomized substance for the analyzing operation without creating a too high load on the sample material.

The sample holder **19***a* illustrated in FIG. **3** is provided with three bores **29** in its upper surface **20***a* having each a diameter of 0.7 mm. for receiving therein wire pieces each having a diameter of 0.5 mm. which are securely held in said bores **29** by their own resiliency and require no clamping screws. The sealing ring in the upper surface **20***a* is designated with **26***a*.

Wires having a diameter less than 0.5 mm. preferably are being twisted and then are inserted in the bores of the sample holder by observing the above mentioned limits.

If an internal standard is taken as a relative analyzing value, the diameter of the samples effect only the integration period and the measuring result is independent of the diameter of the sample. Even with thin wires an integration period of 60 seconds is sufficient. To obtain a high analyzing accuracy it is, for instance, possible to determine in a cobalt-nickel-iron beryllium wire having a thickness of 1.40 mm. by using nickel as internal standard the elements iron, beryllium and silicon within **40** seconds with a relative error of 1 percent.

The advantages of the mentioned glow discharge tube employing the sample holder of the invention resides particularly in the fact that by observing the mentioned additional requirements it is possible to directly analyze wires and wire-shaped bodies with a high accuracy and within a short period of time, whereby owing to the reference to an internal standard the measuring results are independent of the diameter of the samples.

What I claim is:

1. A glow discharge tube for the analysis of samples comprising wires and wire-shaped metal bodies, said tube comprising a sealed housing containing an inert gas at a glow discharge pressure therein, said housing further comprising a hollow anode body, an annular cathode body having one of its ends attached to, but insulated from, one end of said hollow anode body, a transparent closure plate closing the other end of said hollow anode body, and a test body closing the other end of said annular cathode body, said hollow cathode body having, at its end connected with said annular cathode body, a nipple which extends into the bore of said annular cathode body with concentric clearance and terminating a short distance from said test body which closes said annular cathode body, and means for pressing said test body tightly in engagement with said other end of said annular cathode body, wherein the improvement comprises the employment of a test body made of electrically conductive material which is difficult to atomize, said body being provided in its surface engaging said annular cathode body with at least one bore in axial alignment with said nipple for receiving the sample to be analyzed, the cross section of said bore being so selected with reference to the cross section of the sample to be analyzed that between the wall of the bore and the perimeter of said sample the smallest possible clearance is obtained, said clearance being not more than 0.15 mm, while the end face of the sample directed toward the anode nipple is arranged within the range of **2** mm above to 1 mm. below the surface of the test body engaging said annular cathode body.

2. A glow discharge tube according to claim **1**, in which said test body is provided with a plurality of bores, each one of which is adapted to receive a sample to be analyzed, said plurality of bores being arranged within a circular area which is somewhat less in diameter than the diameter of the bore in said nipple, said circular area being arranged in axial alignment with said nipple.

* * * * *