July 9, 1974 M. CZUHA, JR 3,823,082

MOISTURE ANALYSIS APPARATUS.

Filed Feb. 23, 1973 2 Sheets-Sheet 1

FIG. 1

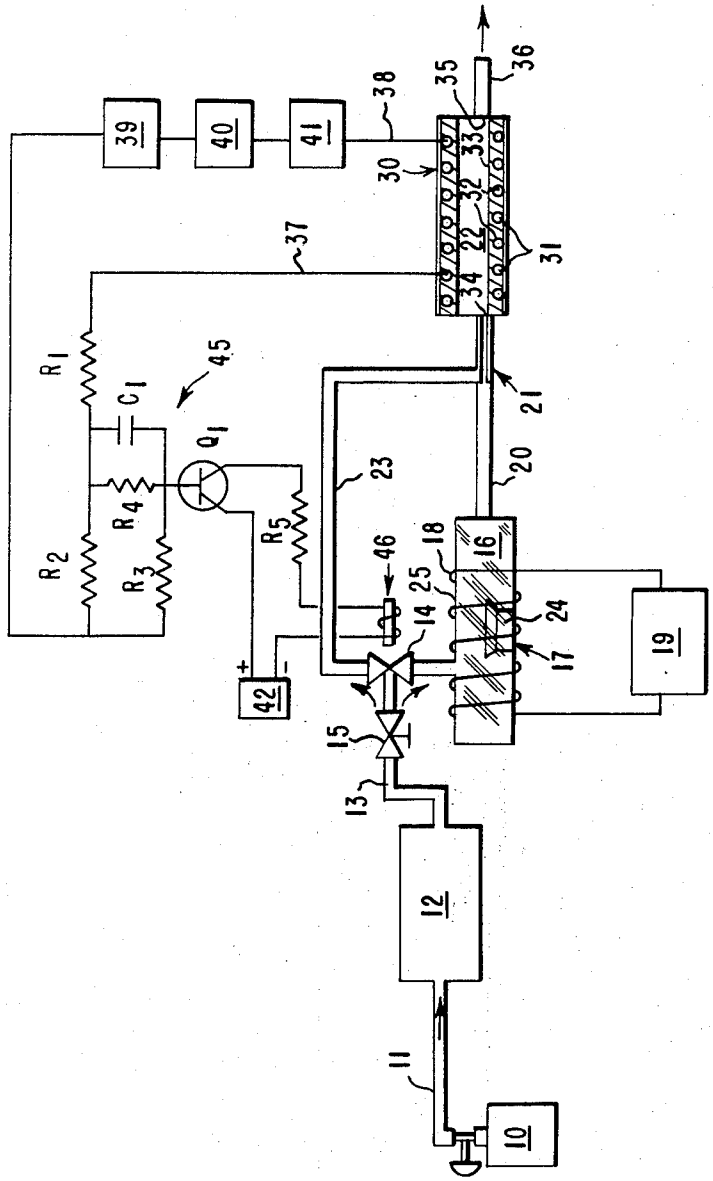
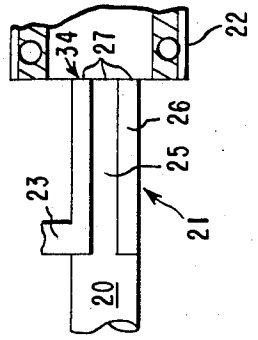


FIG. 2



MOISTURE ANALYSIS APPARATUS.

Filed Feb. 23, 1973 2 Sheets-Sheet 2

FIG. 4

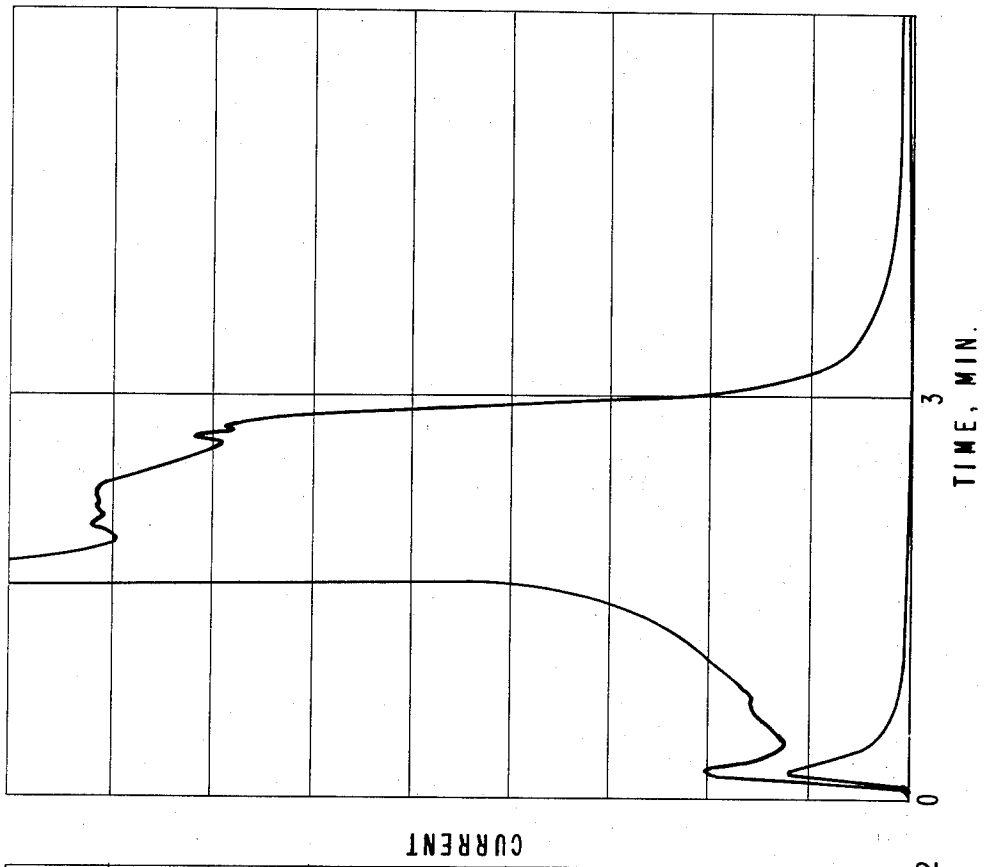
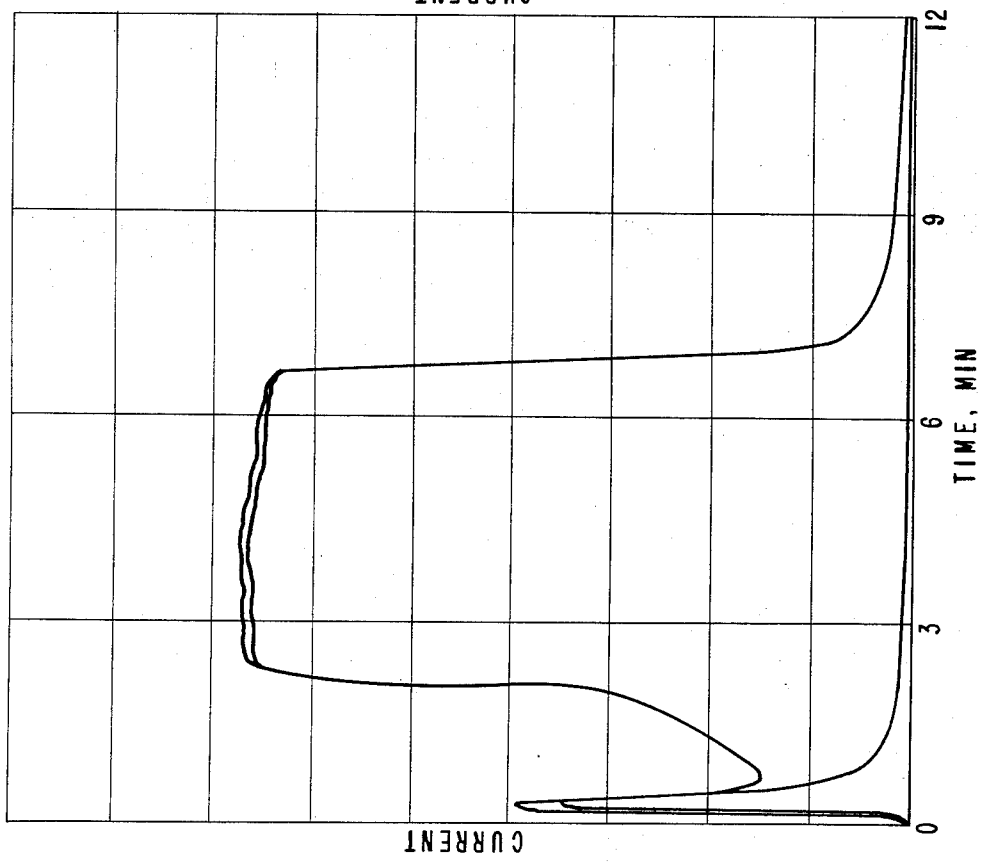


FIG. 3



United States Patent Office 3,823,082 Patented July 9, 1974

3,823,082

MOISTURE ANALYSIS APPARATUS

Michael Czuha, Jr., San Gabriel, Calif., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del.

Filed Feb. 23, 1973, Ser. No. 335,116
Int. Cl. G01n 27/46
U.S. Cl. 204—195 W 5 Claims

ABSTRACT OF THE DISCLOSURE

A moisture detection apparatus, of the electrolytic cell type, in which the moisture released from a moisture source is transported to the cell by a flow of dry gas. The use of a fast acting valving means permits the gas flowing to the cell to be alternated between moisture laden gas and dry gas, and the use of a nozzle by-pass located at the input orifice of the cell permits the change from moisture laden gas to dry gas entering the cell to occur directly at the input to the cell.

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to the field of moisture analysis. More particularly, it relates to a moisture measuring apparatus, of the electrolytic cell variety, and to a method for improving its response time.

2. Background of the Invention

The determination of small amounts of water in moisture containing sources, particularly solid materials, has long been a troublesome analytical problem. Recently electrolytic cells have become widely used where quantitative or qualitative analysis of the moisture in a moisture source is desired. A typical electrolytic cell comprises a cavity containing a pair of electrodes, in spaced apart relationship, and a film of hygroscopic material, such as phosphorus pentoxide, deposited on the electrodes and in the region between the electrodes. A suitable voltage is applied to the two electrodes and when the hygroscopic material becomes conductive, such as would occur when it absorbs moisture, an electrolytic path is created between the electrodes. As moisture is absorbed by the hygroscopic material, the material becomes conductive, a current flows between the electrodes in the region of conductivity, and the water absorbed by the hygroscopic material is electrolyzed to hydrogen and oxygen. The hygroscopic material is thereby continuously regenerated and the electric current which flows through it is an accurate measure of the amount of moisture absorbed. Since the cell operates in accordance with Faraday's Law, it is inherently a very sensitive and simple apparatus.

The response of the electrolytic cell is rapid. The associated equipment should, therefore, be designed to operate as fast as possible so that the analysis can be carried out in as short a time as possible. It is also desirable to increase the sensitivity of the measurement by introducing as much moisture into the cell as possible so that the electrolytic current generated is as large as possible. These desires, however, have generally been frustrated by the fact that the electrolytic cell can be overloaded. When a sample containing water is heated, particularly a sample containing water of hydration, the water is driven off the sample at an extremely rapid rate, once the threshhold temperature of water release is reached. The length of the electrolytic cell is dictated by practical considerations, and, with a given cell length, there is a limit to the amount of water which the cell can accommodate in a given time. If too much water is introduced into the cell, the electrodes will short out, and if too high a temperature is used to drive off the water, the coating may sublime.

This problem has been recognized for some time, and several solutions to it have been suggested. In particular, M. W. Bell has disclosed, in U.S. Pat. 3,146,181, the advantages of using an induction heater to heat the moisture source and the use of an electric feedback circuit to monitor the electrolytic current in the cell and control the amount of power supplied to the induction heater in inverse relation to the electrolytic current. While this approach can be used to prevent the electrolytic cell from overloading, the system responds very slowly to this control technique.

In the present invention, a dry gas is used to transport the moisture released from the moisture source to the electrolytic cell. Rather than attempt to prevent overloading of the electrolytic cell by controlling the oven temperature, a second source of dry gas is provided for the electrolytic cell. By use of valving means, the flow of gas to the electrolytic cell can be alternated between moisture laden gas and dry gas. When the electrolytic current from the cell indicates that the cell is becoming overloaded, the valve can be adjusted to purge the system with dry gas. To take advantage of the rapid response time of the eltctrolytic cell, a fast acting valve is used. It has been found that if the valve is located near the input orifice of the electrolytic cell, moisture holdup and leakage of the valve seals will cause erratic fluctuation in the measurement. On the other hand, it has also been found that if the change between the moisture laden gas and the dry gas takes place at any other position than directly at the input orifice of the electrolytic cell, erratic fluctuation of the output of the electrolytic cell will also occur.

SUMMARY OF THE INVENTION

To overcome these and other difficulties, the present invention provides an improvement in an apparatus for determining the moisture present in a moisture source, of the type comprising: an electrolytic cell, in the form of a cavity, having an input orifice and an output orifice and containing a pair of spaced apart electrodes with a body of hygroscopic material interconnecting the electrodes; an enclosure to contain the moisture source; means for releasing moisture from the moisture source into the enclosure; transport means for transporting the moisture release from the moisture source from the enclosure to the cavity, said moisture cell comprising a dry gas supply, a first duct connecting the gas supply to the enclosure and a second duct connecting the enclosure to the cell; and means, coupled to the cell, for impressing a potential across the cell electrodes to electrolyze the moisture absorbed by the hygroscopic material, the electrolytic current flowing between the electrodes through the hygroscopic material being related to the quantity of moisture electrolyzed in the cell.

The improvement is to be found in that fact that the apparatus further comprises a nozzle by-pass located in the second duct immediately adjacent to the input orifice of the cell so that moisture laden gas can be introduced through the nozzle by-pass directly into the cell, a third duct connecting the dry gas supply to the nozzle by-pass so that dry gas can be introduced through the nozzle by-pass directly in the cell, and a fast acting valve means located in the first and third ducts for alternating the flow of gas to the cell between moisture laden gas and dry gas. The nozzle by-pass is connected so that the change from moisture laden gas to dry gas entering the cell occurs at the input to the cell.

The apparatus may also comprise electrical feedback means coupled between the cell and valving means to regulate the amount of moisture laden gas conveyed to the cell in inverse relation to the electrolytic current flowing through the cell. In the preferred embodiment, the valving

means is a single three way solenoid valve, one leg of which is connected to dry gas supply, a second leg which is connected to the enclosure, and a third leg to which is connected to the third duct so that dry gas can either be fed directly into the nozzle by-pass or to the nozzle by-pass through the enclosure.

BRIEF DESCRIPTION OF THE FIGURES

The invention can best be described with reference to the following figures in which

FIG. 1 is a schematic illustration of one embodiment of the present invention;

FIG. 2 is an enlarged view of the nozzle by-pass shown in FIG. 1;

FIG. 3 is a plot of the current output, I, from the electrolytic cell as a function of time for a sample analyzed using the apparatus shown in FIG. 1; and

FIG. 4 is a plot of the current output of the electrolytic cell as a function of time, for a sample analyzed on an apparatus similar to that shown in FIG. 1, but without the nozzle by-pass and with the valving means positioned in place of the nozzle by-pass.

DETAILED DESCRIPTION OF THE INVENTION

Referring to FIG. 1, a gas supply tank **10** is shown with a connection **11** leading to a dryer **12**. First duct **13** leads from the dryer **12** to a valving system **14** through a regulator **15**. In the embodiment shown, valving system **14** is a three way solenoid valve; one leg of which is connected to the dryer **12** through the first duct. A second leg of valve **14** is connected to an enclosure **16** which forms part of the oven used to heat and release moisture from a moisture source. Inside the enclosure **16** a support structure **17** is located. This support structure consists of a pedestal **24** and a container **25** supported by the pedestal, in which the sample to be analyzed, i.e., the moisture source, is placed. Heater **18** and power supply **19** for the heater, form the rest of the means for releasing moisture from the moisture source. Enclosure **16** is connected to electrolytic cell **22** through a second duct **20** and nozzle by-pass **21**.

The third leg of solenoid valve **14** is connected directly to the nozzle by-pass **21** through a third duct **23**. By manipulating valve **14**, the gas flow supply to electrolytic cell **22** can be alternated between moisture laden gas and dry gas. When valve **14** is positioned to provide a path between dryer **12** and electrolytic cell **22** through enclosure **16**, then the flow of dry gas through enclosure **16** serves to transport moisture released from the moisture source from enclosure **16** into the electrolytic cell. When valve **14** is positioned to connect dryer **12** with duct **23**, the dry gas is provided directly to the electrolytic cell.

The oven used to heat the moisture source can be of any suitable type. In the embodiment illustrated, however, the enclosure is a glass enclosure, the heater is in the form of a resistance wire **18** wound around the glass, and the tray or container **25** is in the form of a small platinum boat which will respond rapidly to changes in the output of coil **18**.

There are many types of electrolytic or conductivity cells and the choice of which to use depends on the circumstances. In the embodiment illustrated, the electrolytic cell **22** comprises a hollow tube **30** containing a pair of electrodes **31** and **32** in the form of helices disposed within the tube **30** in spaced apart relation to one another. The electrodes **31** and **32** are fabricated from fine platinum wire or other suitable electrode material. A film of hydroscopic material **33**, such as phosphorus pentoxide, is deposited on the coils on the interior of the tube completely embedding the electrodes and bridging the gap between them. The surface of the hygroscopic material is exposed to the interior of the tube **30**. The electrolytic cell has an input orifice **34** and an output orifice **35**. The nozzle by-pass **21** is connected to the input orifice **34** of the electrolytic cell. In the embodiment illustrated in FIG. 2, the nozzle by-pass **21** is formed by restricting duct **20** into a nozzle and providing an annular duct **26** formed concentric with nozzle **25**. Both the nozzle **25** and the annular duct **26** terminate at the input orifice **34** to the electrolytic cell so that the mixing port **27** for moisture laden gas from duct **20** and dry gas from duct **23** is located at the input orifice to the cell. The cell is also provided with an outlet duct **36** attached to outlet orifice **35** so that the gas entering the cell and the gas generated in electrolysis can be vented from the cell.

A pair of leads for electrodes **37** and **38** provide a power circuit to the cell. A direct current voltage supply **39** is provided in conductor **38**. The output lead or conductor **38** from the cell communicates with a zero control **41**, a current integrator **40** and a cell power supply **39**. The cell power supply is provided to supply the voltage across electrodes **31** and **32**, and the cell current integrator is provided to measure the current flowing between the conductors, through the hygroscopic material. An electrical feedback circuit **45** is connected between the electrolytic cell **22** and solenoid valve **14**. A separate power supply **42** for the solenoid is also provided. This feedback circuit is of conventional design. The values of the components are given in Table I.

TABLE I

Resistors		Capacitor	
Designation	Value (ohms)	Designation	Value (microfarads)
R^1	39	C_1	1,000
R^2	270		
R^3	5,100	Transistor	
R^4	5,100	Designation	Type
R^5	1,000	Q_1	2N 1188

The feedback circuit monitors the electrolytic current generated by the electrolytic cell and activates solenoid valve **14** to switch from a position where moisture laden gas is introduced into the cell to a position where dry gas is introduced into the cell, when the electrolytic current reaches a value indicating an overloaded electrolytic cell. Simply stated, an increase in the electrolytic current in the cell above the turn on point of the transistor Q_1 will cause transistor Q_1 to conduct and activate solenoid **46** to switch solenoid valve **14** to the point where dry gas introduced through duct **23** enters the electrolytic cell.

FIG. 3 is a plot of the cell current as a function of time for a sample analyzed in the apparatus shown in FIG. 1. The lower curve is a blank, i.e., an experimental run in which the sample container **25** is empty. The upper curve represents the output of the electrolytic cell when the sample container holds 2.0 milligrams of water. The cell current rises rapidly to a point at which the electrical feedback circuit activates solenoid valve **14** to introduce dry gas rather than moisture laden gas into the electrolytic cell. When the current drops to the point where the electrical feedback circuit deactivates solenoid valve, moist air again is fed to the electrolytic cell. The double line at the top of FIG. 3 indicates oscillations between these two circumstances.

FIG. 4 shows the electrolytic cell output for an apparatus in which the nozzle by-pass has been removed and in which the valving system has been positioned close to input orifice of the electrolytic cell. If can be noted that the rise time is so fast that the cell overloads and the integrator cannot keep up with the count rate.

The current integrator **40** integrates the area under the curve shown in FIGS. 3 and 4 to provide a record of total current generated by the cell, during the period of time required to drive off all the moisture from the sample.

While the invention has been described above in conjunction with specific apparatus and in a specific appli-

cation, this is by way of example only and is not considered as a limitation of the scope of the invention.

What is claimed is:

1. In an apparatus, for determining the amount of moisture present in a moisture source, of the type comprising an electrolytic cell in the form of a cavity having an input and an output orifice and containing a pair of spaced apart electrodes with a body of hygroscopic material interconnecting them; an enclosure for containing said moisture source; means for releasing moisture from said moisture source into said enclosure; transport means for transporting the moisture released from said moisture source from said enclosure to the cavity in said cell, said transport means comprising a dry gas supply, a first duct connecting said dry gas supply to said enclosure and a second duct connecting said enclosure to said cell; and means coupled to said cell for impressing a potential across the cell electrodes to electrolyze the moisture absorbed by the hygroscopic material, the electrolysis current flowing between said electrodes through the hygroscopic material being related to the quantity of moisture electrolyzed in the cell; the improvement wherein said apparatus further comprises a nozzle by-pass located in said second duct immediately adjacent to the input orifice of said cell so that moisture laden gas can be introduced through said nozzle by-pass directly into said cell, a third duct connecting said dry gas supply to said nozzle by-pass so that dry gas can be introduced through said nozzle by-pass directly into said cell, and fast acting valving means located in said first and third ducts for alternating the flow of gas to said cell between moisture laden gas and dry gas, said nozzle by-pass being constructed so that the change from moisture laden to dry gas entering the cell occurs at the input to the cell.

2. The apparatus of Claim 1 further comprising an electrical feed back means coupled between said cell and said valving means to regulate the amount of moisture laden gas being conveyed to said cell in inverse relation to the electrolysis current flowing through the cell.

3. The apparatus of Claim 2 wherein said valving means is a single three way solenoid valve, one leg of which is connected to said dry gas supply, a second leg of which is connected to said enclosure, and a third leg of which is connected to said third duct so that dry gas can alternatively be fed either directly to said nozzle by-pass or to said nozzle by-pass through said enclosure.

4. The apparatus of Claim 2 further comprising current integrator means coupled to said cell to provide an indicattion of the total amount of moisture absorbed in the cell.

5. The apparatus of Claim 1 wherein said means for releasing moisture from said moisture source is an oven.

References Cited

UNITED STATES PATENTS

3,355,367	11/1967	Marsh	204—195 W
3,146,181	8/1964	Bell	204—195 W

TA-HSUNG TUNG, Primary Examiner

U.S. Cl. X.R.

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