March 7, 1950 J. R. BOWMAN 2,499,626

DIPOLE MOMENT INDICATOR AND CONTROL METHOD

Filed April 30, 1947 2 Sheets-Sheet 1

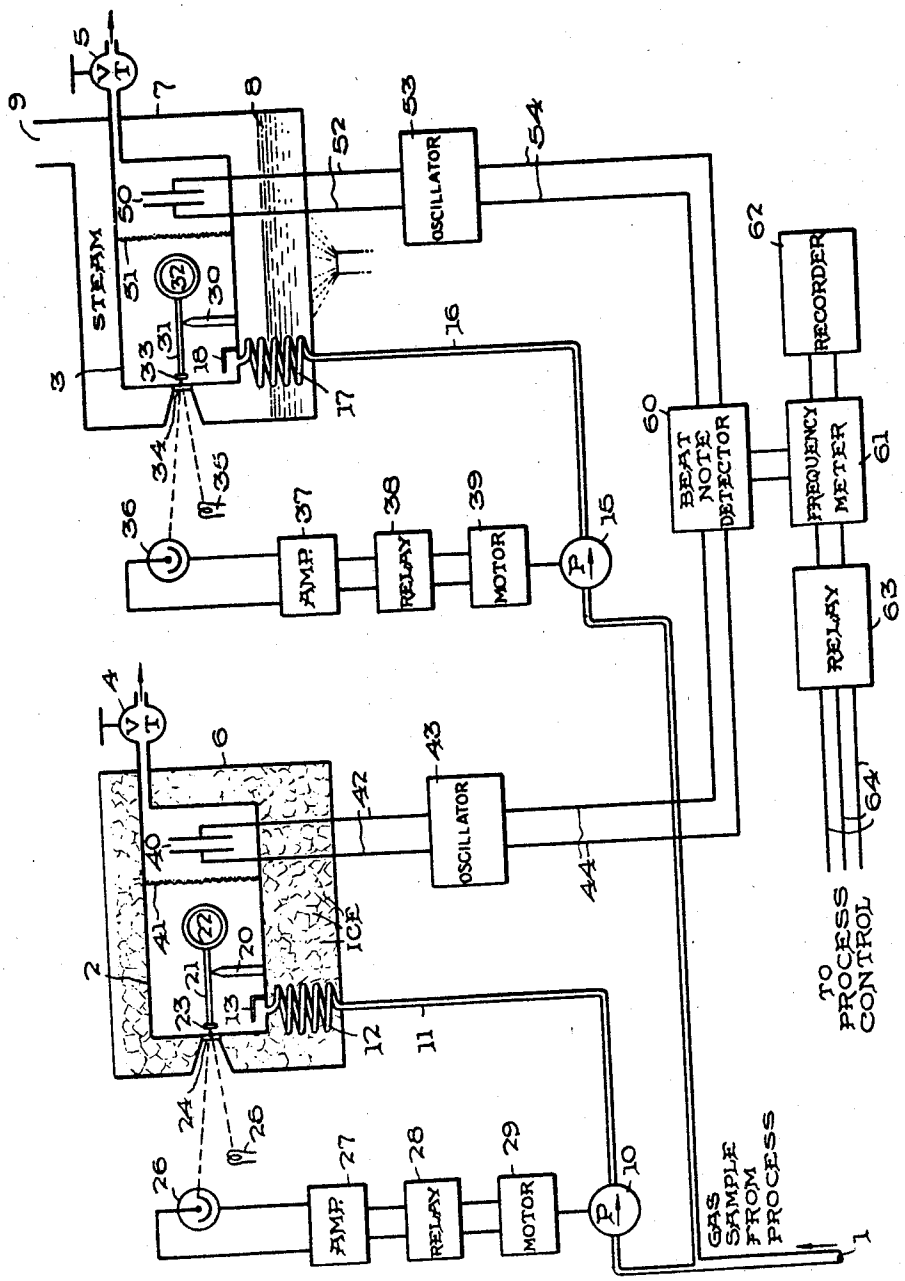


Fig. 1.

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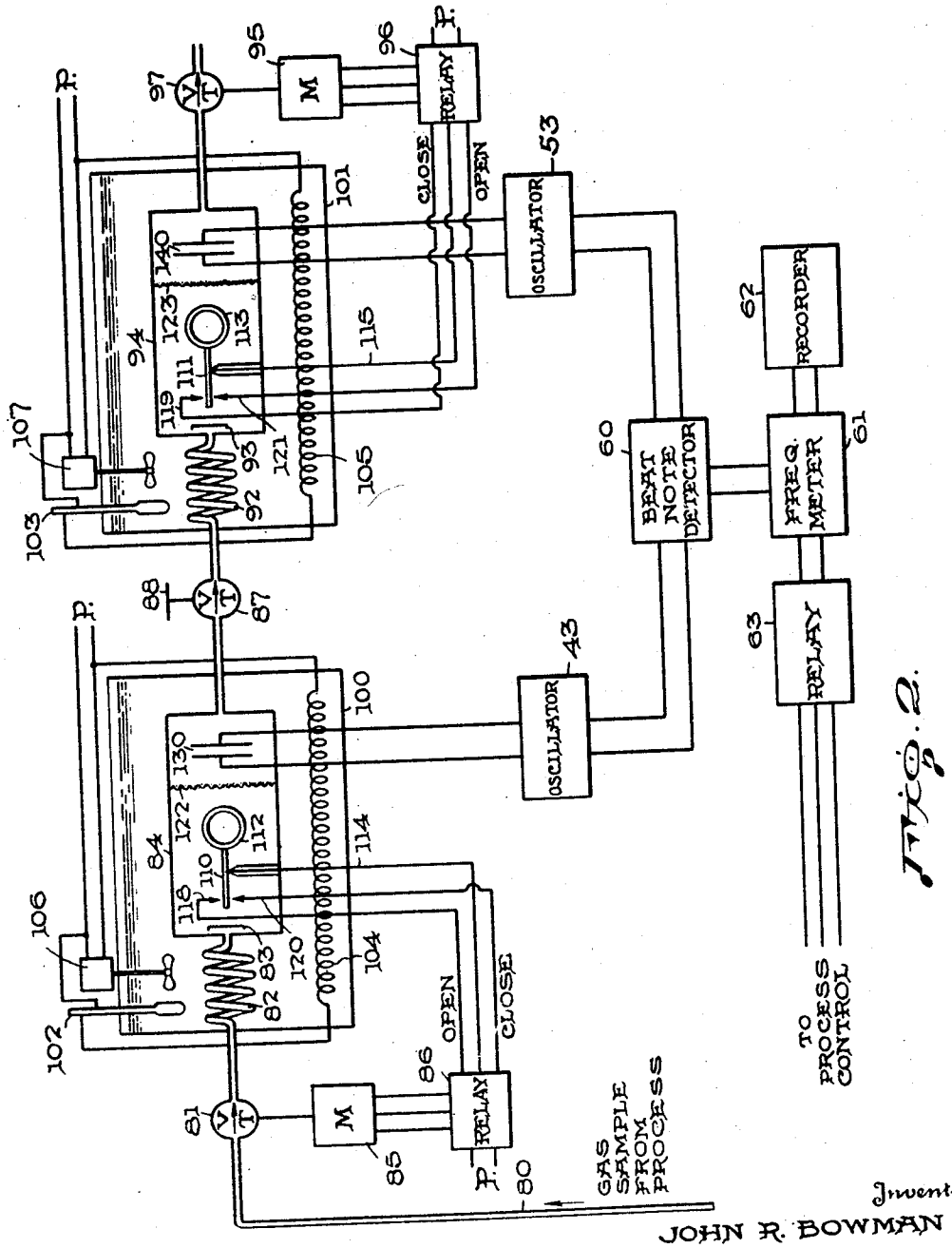


Fig. 2.

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UNITED STATES PATENT OFFICE

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DIPOLE MOMENT INDICATOR AND CONTROL METHOD

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7 Claims. (Cl. 175—183)

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This invention concerns apparatus for the determination and recording of dipole moment of gases and a method of process control based on dipole moment measurement.

This invention is based on the control of a chemical process in response to changes in the dipole moment of a gas. The apparatus involves the determination of the dipole moment of a sample or stream of gas, the result of the measurement being either recorded for reference or used in controlling a parameter of the process so that a desirable state of operation is maintained. Any process variable may be regarded as a parameter, and one or more such parameters which affect the gas stream may be brought under control.

The theory on which my invention is based is described in the book "Dielectric Constant and Molecular Structure" by Smythe (Chemical Catalogue Co., 1931) and also in the book "Polar Molecules" by Debye (Chemical Catalogue Co., 1929).

All molecules can be classified sharply as symmetrical or unsymmetrical. All members of the first group have no permanent dipole moment and theoretically all unsymmetrical molecules have a permanent residual dipole moment. Measurement of the dipole moment of a mixture of gases will, therefore, give specific indication of the concentration of unsymmetrical molecules in the mixtures. Thus, a measurement of the mean permanent dipole moment will provide a convenient and useful index of the composition of the mixture. While the method and apparatus here described determines the concentration of unsymmetrical molecules in the gaseous mixture, it is commonly found that only one component of the mixture is of such unsymmetrical character and the method and apparatus of this invention thereby permit determination of its concentration. If necessary an interfering unsymmetrical component may first be removed from the mixture of gases by chemical or physical separation before passing the mixture into the apparatus of this invention.

By way of example, in certain hydrocarbon distillation processes it becomes necessary to determine, for purposes of controlling the still, the concentration of butene-1 distinct from N-butane and in the presence of butene-2. In order to

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make such a determination, advantage may be taken of the fact that butene-1 is the only one of the hydrocarbons concerned that is unsymmetrical. It, therefore, is the only one that has a dipole moment different from zero. A value of 0.37×10^{-18} has been reported for the dipole moment of butene-1 (Smythe & Zahn, J. Am. Chem. Soc. 47, 1501 (1925)). In this process butene-1 is the only hydrocarbon concerned that is unsymmetrical and none of the other materials mentioned would interfere with the measurement of its concentration, because their dipole moments are zero.

The concentration of butene-1 may, by the use of the method and apparatus of this invention, be determined in a mixture of C_4 olefins. The application of my invention is, however, not restricted to the determination of butene-1 in these examples, but has broad application as may be seen from the following examples of polar and non-polar molecules.

I Polar	II Non-Polar
1-Butene	Butane
1,2-Butadiene	Propane
Isobutylene	2-Butene
Pentenes	1,4-Butadiene
Propylene	

My invention may therefore be used as well for determining the concentration of propylene, 1,2-butadiene, pentenes, or isobutylene if these occur alone in the product, or for determining the sum of their concentrations if these occur together. These compounds are mentioned as examples of polar or unsymmetrical compounds whose concentration may be determined by my invention but this list is not by way of limitation since many other such polar compounds are known.

In theory when an electrostatic field is applied to an atom or molecule, a dipole moment is induced in it. The magnitude of this induced moment for a given field intensity is dependent on the nature of the molecule and can be observed indirectly by its influence on the dielectric constant. If a condenser is insulated with a dielectric material between its plates, the magnitude of the susceptibility of the molecules of the dielectric to acquire a dipole moment affects the capac-

itance of the condenser. The capacitance will be proportional to the number of molecules between the plates, other things being constant, but since susceptibility to polarization (that is acquiring dipole moment under the influence of an electric field) is independent of temperature, the effect of temperature on the condenser will be through its effect on the density of the dielectric only. Thus, if a gas is placed between the plates of a charged condenser, the molecules will acquire a dipole moment and the capacitance of the condenser will be affected, this effect being attributed to the dielectric constant of the gas. This effect takes place in all gases whether symmetrical or unsymmetrical.

If the molecules of the gaseous dielectric are unsymmetrical, they will have a dipole moment of their own, in addition to the induced polarization. In the presence of an electric field, such molecules will be subject to orientation by the field so that the permanent dipole will tend to be aligned with the lines of electric force. The presence of this permanent dipole in the molecules between the condenser plates will add to the apparent capacitance of the condenser. The resulting orientation contribution to the capacitance will be dependent on the temperature, because the random thermal motion of the molecules will tend to destroy the orientation.

For a generalized mixture comprising both symmetrical and unsymmetrical molecules, the dielectric constant observed will be made up of two parts. The first part is dependent on density of the gas and its chemical constitution only. Both symmetrical and unsymmetrical molecules contribute toward this part of the dielectric constant, but this part of the dielectric constant is not a function of temperature. The second part of the dielectric constant is that contributed by the permanent dipole moment of the unsymmetrical molecules present in the mixture. This part will depend also on density and chemical constitution but, in addition, will depend on the temperature. It decreases as the temperature increases. Consequently measurement of the dielectric constant at two temperatures and at the same density will distinguish symmetrical from unsymmetrical molecules in a mixture of gases.

My invention contemplates a method and apparatus for measuring the relative dielectric constants of a gas taken at two temperatures and the same density, and further contemplates using variations in such relative dielectric constant to control the process producing the gas.

It is accordingly an object of my invention to provide apparatus for the determination of dipole moment of a gaseous mixture.

Another object of my invention is to provide apparatus for continuously measuring and recording the dipole moment of a gaseous mixture.

Another object of my invention is to provide an apparatus whereby the concentration of unsymmetrical molecules may be determined in a mixture of gases.

Another object of my invention is to provide an apparatus for measuring the dielectric constant of a gas at constant density.

Another object of my invention is to provide an apparatus for continuously determining the dielectric constant of a gas at two temperatures and at substantially the same density.

Another object of my invention is to provide an apparatus for controlling a chemical process so as to maintain constant the dipole moment of its gaseous output.

A further object of my invention is to provide a method of continuously measuring and recording the dipole moment of a gaseous mixture.

Another object of my invention is to provide a method by which a chemical process may be controlled so as to maintain constant the concentration of unsymmetrical molecules in its gaseous output.

These and other objects of my invention are attained as set forth in this specification in which the drawings form a part, and in which:

Figure 1 is a diagram showing one embodiment of my invention, and

Figure 2 is a diagram showing a second embodiment of my invention.

These embodiments of my invention may be used to determine the dipole moment of a gaseous mixture by measuring the change in dielectric constant of the gas between two temperatures but at the same density, the change of dielectric constant being observed by comparing two condensers of which the gas at the two temperatures respectively forms the dielectric.

Referring to Figure 1, a sample of the gas from the process is withdrawn through pipe 1 and is fed into two cells, 2 and 3 which are held at different constant temperatures, the gas pressure in the two cells being adjusted so as to maintain in them the same constant density. The cells 2 and 3 may have outlet valves 4 and 5, by means of which the gas flowing through the cells may be throttled or regulated to a convenient small value. The valves 4 and 5 may be adjusted by hand to allow approximately equal rates of flow of gas through the respective cells.

The cells 2 and 3 are maintained at constant but different temperatures by placing them in constant temperature baths 6 and 7. By way of example, bath 6 is shown filled with crushed melting ice entirely surrounding the cell 2. Also by way of example, bath 7 may contain boiling water 8, the vapor from which entirely surrounds the cell 3, and passes off freely to the atmosphere through vent 9. By such means the cells 2 and 3 may be maintained at 0° C. and 100° C., respectively. Other temperatures may of course be used, provided only that they be kept constant.

Gas from sample tube 1 passes through pump 10 to tube 11 and through a coil 12 immersed in the crushed ice contained in the constant temperature bath 6. Outlet into cell 2 may have a baffle 13 to avoid setting up excessive convection currents. Simultaneously, gas from the sample tube 1 is also passed through pump 15, through tube 16 and coil 17 past baffle 18 and into cell 3.

Inside of cell 2 may be placed an Edwards float balance shown diagrammatically as suspended on fulcrum 20. Such an Edwards float balance (see Technological Paper No. 89 of the U. S. Bureau of Standards, 1917) comprises a balance beam 21 having at one end a thin walled hollow sealed "float" bulb 22. The other end of the balance arm 21 has a small counterweight and a small mirror 23. The balance is adjusted so that it balances at approximately horizontal position. It is apparent that the buoyant effect of the gas contained in cell 2 surrounding bulb 22 will affect the balance position which may thereby be used as an index of the gas density or used to control this density. To the latter end a window 24 placed in the cell 2 adjacent to the mirror 23 permits light from a lamp 25 to fall on mirror 23 and be reflected to photocell 26. By well-known arrangement of optical parts, such as lenses and diaphragms, which does not form a part of this

invention, the photocell may be used to control the pressure of gas in cell 2 so as to maintain a constant balance point of the Edwards balance and thereby maintain the density of the gas in cell 2 at a constant value. The current from photocell 26 may be fed into amplifier 27 and by means of relay 28 be used to control the speed of motor 29 which drives pump 10 in a well known manner. The system is arranged so that when a decrease of density occurs due to flow of gas through valve 4 and consequent falling of the bulb 22, the photocell control system is effective to speed up the pump 10 thereby increasing the pressure and density in cell 2. The gas in cell 2 may thereby be maintained at constant density as it flows through the cell.

A similar system is effective to maintain flow of gas at constant density through cell 3. The cell 3 is equipped similar to cell 2 with an Edwards float balance mounted on fulcrum 30 and a beam 31, bulb 32, mirror 33, the latter reflecting light from lamp 35 through window 34 on to photocell 36, which acts through amplifier 37 and relay 38 to control motor 39 which, in turn, drives pump 15 so as to hold the position of the Edwards balance at a constant point and thereby effect constant density in the cell 3.

Other means of maintaining constant density than by control of the pump speed may alternatively be used. For example, the pump may be operated at constant speed and the relays 28, 38 used to open or close the outlet valves 4, 5, respectively. Inasmuch as the gas in cells 2 and 3 are maintained at constant temperatures, respectively, density control is obtained by any means which effects a change in pressure.

The density of gas in the cells 2 and 3 may be adjusted to very nearly the same value by the use of Edwards floats 22 and 32 of the same size and otherwise similar Edwards balance parts. Adjustment to very nearly the same density is required and may be attained by temporarily putting both cells 2 and 3 at the same temperature and pressure (e. g. atmospheric) and adjusting riders on the balances 21 and 31 to set both balances at their null points with respect to their respective photocells.

Inside of cell 2 is mounted condenser 40 which is connected through leads 42 to an oscillator 43 the latter being regulated in well-known manner to be independent of load and other variation, so that its frequency is a function only of the capacitance of condenser 40. In order to protect the sensitive Edwards balance from any electrostatic effects produced by the condenser 40, a Faraday screen 41 may be placed between the condenser and the balance, this screen allowing free passage of gas. The condenser 40 may be a precision "air" condenser. It must be of high precision and independent of effects other than the dielectric constant of the gas between its plates, but its capacitance may be small, for example 100 or 200 micromicrofarads. The drift in capacitance of condenser 40 should be less than a few parts per million. The condenser may be designed for zero temperature coefficient and since it is inside of cell 2 and thereby held at constant temperature, no difficulty is experienced from temperature effects. Condenser 40 should have good insulation and it may be equipped with guard rings (not shown) or other features customarily provided for precision capacity measurements.

Inside of cell 3 there is a similar condenser 50 shielded from the Edwards balance by screen 51

and connected by leads 52 to oscillator 53. Condenser 50 should have substantially the same capacity as condenser 40 when it has the same dielectric between its plates as 40. Oscillator 53 is designed similar to 43 to be independent in frequency from load effects, supply voltage variations and other factors in well-known manner, so that its frequency depends only on the capacitance of condenser 50.

The selection of frequency for the oscillators 43 and 53 should be such that the beat frequency when both cells contain pure butene-1, for example, is about 10,000 cycles. The oscillator 43 is connected by leads 44 and oscillator 53 by leads 54 to a beat note detector 60. Detector 60 is of conventional design and may deliver its output to a convenient frequency meter 61 whose output may be recorded on recorder 62 and, in addition, used to operate the supervisory relay 63 which controls the power through leads 64 for controlling one or more parameters of the chemical process supplying the gas sampled by tube 1. Alternately, recorder 62 may be of the controller type and contain relay 63 integral therewith.

By operating oscillators 43 and 53 at relatively high frequency, it is possible to detect minute changes in the relative capacity of condensers 40 and 50. This may be done as above described, in the manner of the well-known ultra-micrometer principle. Thus with the two cells 2 and 3 maintained at constant density and different temperatures, any variation in the constitution of the gas sample from tube 1 which does not change the concentration of unsymmetrical molecules will affect condensers 40 and 50 in exactly the same way and, therefore, produce no change in the beat note or indication of frequency meter 61. However, if the concentration of unsymmetrical molecules changes, then condensers 40 and 50 will change by different amounts, giving rise to a variation in the beat note between oscillators 43 and 53. Such a change will affect the indication of frequency meter 61 and this may react on supervisory relay 63 to operate the necsary process controls to correct the condition.

Figure 2 shows another embodiment of my invention. In Figure 2 the gas is indicated to be withdrawn from the process through pipe 80 and passed through a throttling or needle valve 81 into heating coil 82 and thence past baffle 83 into cell 84. The opening of throttling valve 81 is controlled by a reversible motor 85 under the supervision of relay 86. Outlet from cell 84 is through hand-controlled needle valve 87 whose setting may be adjusted by means of handle 88. The gas then flows into heating coil 92 and past baffle 93 into cell 94 from which it exhausts through throttling valve 97 whose opening is controlled by motor 95 under the supervision of relay 96. It is assumed that the gas sample obtained from the process is under relatively high pressure, but if this is not the case pipe 80 may be supplied from the process through a pump and pressure accmulator (not shown) so that there is always a steady supply of sample gas available at pipe 80 and the gas will flow through the two cells 84 and 94 at a rate controlled by the setting of valve 87. In order for gas flow to take place in the direction indicated, the cell 84 is thermostated at a higher temperature than cell 94. Inasmuch as the densities in the two cells 84 and 96 will be maintained the same, as will be described, and the cell 84 is at a higher temperature, it follows that the gas therein is also at a

higher pressure than that in cell 94. Valve 97 may exhaust to the atmosphere or the gas may be returned to the process by means of a pump, not shown. The cells 84 and 94 and the coils 82 and 92 for preheating the gas are respectively maintained in constant temperature bath tanks 100 and 101 which are filled with oil or other liquid. The temperatures of constant temperature baths 100 and 101 are controlled by means of thermostats indicated diagrammatically at 102 and 103 and which control electric heating power supplied to coils 104 and 105, in customary manner, the electric power being supplied over the wires marked P. Each thermostated constant temperature bath also has a stirrer 106 and 107 as is customary in constant temperature baths of this type. In this manner the gas is maintained in cells 84 and 94 at different known constant temperatures as it flows through the respective cells. The temperature of the respective cells 84 and 94 may be measured with thermometers in the bath in well known manner. Temperature regulation should be to within 1° or better and this is easily accomplished by well known thermostatic means.

In order to control the densities of the gas in cell 84 an Edwards balance 110 is mounted therein having float 112. The beam of the Edwards balance is connected through wire 114 to the relay 86. Cooperating stationary contacts 118 and 120 are also connected to relay 86. The balance point of the Edwards balance thus may vary between contacts 118 and 120 controlling thereby the position of relay 86 which controls the power supplied to reversible motor 85 for controlling the throttle opening of valve 81. The system is so arranged so that if the density in cell 84 falls and float 112 falls, contact 118 is made and the relay 86 actuates motor 85 to open valve 81 somewhat further. If the density in cell 84 becomes too high thereby raising float 112 circuit is completed through contact 120 which operates to close valve 81 and restrict the flow of gas into cell 84.

In a similar manner, cell 94 has Edwards balance 111 having float 113 and stationary contacts 119 and 121. The center contact of the balance is conencted through wire 115 to relay 96 as also are contacts 119 and 121. Relay 96 supervises the operation of motor 95 which acts to open or close valve 97. If the density of gas in cell 94 is too high, thereby raising float 113, circuit will be made through contact 121, which acts to open valve 97 somewhat further. On the other hand, if the density of gas in cell 94 is too low, float 113 will drop and close circuit through 119 which acts to close the valve 97 and thereby restrict the outflow of gas from the cell. Motors 85 and 95 are supplied through relays 86 and 96 with power through leads marked P. These motors may be equipped with limit switches so that their respective valves operate only between predetermined limits of opening. The respective Edwards balances 110 and 111 may be made very sensitive so that the density of gas in the cells is maintained very nearly constant. The balances may be adjusted to produce equal densities in the two cells in the same manner as described for accomplishing this in Figure 1.

Each of the cells 84 and 94 have condensers 130 and 140 similar in all respects to those described in connection with Figure 1. The cells may also have Faraday screens 122 and 123, respectively, to shield the sensitive Edwards balance from electrostatic forces. The condensers may be connected, respectively, to oscillators 43 and 53 whose beat note is detected by detector 60 and fed into frequency meter 61 in a manner similar to that described in connection with Figure 1. Recorder 62 may be used to record the indication of meter 61. The deflection of meter 61 may also be used to control supervisory relay 63 which determines the operation of one or more controls affecting parameters of the process in order to control its efficient operation.

In order to adjust the apparatus of Figure 1 the cells 2 and 3 may be initially placed at the same temperature, for instance by filling both containers 6 and 7 temporarily with melting ice. The gas sampling line 1 may then be disconnected from the process and a known non-polar gas having no permanent dipole moment may be passed through the cells. Adjustment of the Edwards float balances 21 and 31 may then be made so that with gas in cells 2 and 3 at the same pressure (for example atmospheric), the Edwards floats are just on the verge of starting the pumps 10 and 15 respectively. At this point the beat note detector 60 and frequency meter 61 may be set at zero or some other convenient value. The gas sampling line 1 may then be re-connected to the process and the temperature difference between the two cells re-established. The Edwards float controls will thereupon maintain the densities in the two cells equal, and any difference in capacitance between condensers 40 and 50 as observed by deflections of the frequency meter 61 will indicate the presence and concentration of unsymmetrical gas having a permanent dipole moment. Calibration may be effected by passing through the system a known concentration of a gas of known dipole moment, for example butene-1 previously mentioned.

A similar adjustment may be used on Figure 2, the containers 100 and 101 being temporarily set to the same temperature and a known non-polar gas passed through the apparatus with all valves open so that equal pressure will exist in both cells 84 and 94. The Edwards float balances 110 and 111 may then be adjusted to their neutral position and the zero setting of frequency meter 61 noted or adjusted. Thereafter when the temperature difference is established between the cells 84 and 89 and an unknown gas sample passed through the system, the Edwards float balances will maintain equal densities in the two cells and the difference in capacitance of the condensers as reflected by variations in the frequency meter reading will indicate the presence and concentration of polar components in the gas stream. Calibration may be effected as explained with reference to Figure 1 by passing through the system a known concentration of a gas of known dipole moment.

Adjustment of the Edwards floats may obviously be made so that they balance at any desired densities by setting up these desired conditions on calibration. Furthermore, beat note adjustment for zero concentration of polar gas may be obtained by adjusting the frequency of either one of the oscillators 43 or 53, or by adjusting the zero of beat note detector 60 or of frequency meter 61 in well known manner.

While I have described apparatus of the ultra-micrometer principle for measuring the variation in relative capacity of the condensers 40 and 50, or 130 and 140, there are a number of other well known electrical means available for comparing the capacitances of the two condensers. With the ultra-micrometer method of comparing the capacities a concentration of a few tenths of a per cent of butene-1 may be detected in a mixture

of C_4 olefines. The condensers may, for example, be connected in a capacity bridge circuit whose balance variation is recorded and/or used to operate supervisory relays controlling the process. However, any known precision apparatus for measuring relative variation in capacitance of these condensers may be used in the practice of my invention, as for example, that of U. S. Patent No. 2,149,756 entitled Measuring apparatus and also other capacity type microgauge systems.

As an example of another application of my invention, it may be used for detecting carbon monoxide in air. Of the usual constituents of air (O_2, N_2, CO_2, H_2O, noble gases) only water vapor has a dipole moment. The air in which carbon monoxide is to be detected may therefore be dried by well known methods or auxiliary apparatus, and then tested by means of the apparatus of this invention. Since carbon monoxide has a dipole moment its presence is easily detected thereby in the dry air. The relay 63 may then be used to set off an alarm when the CO concentration becomes critical. In this connection, the apparatus may be useful around Fischer-Tropsch plants, furnaces, etc. to protect personnel and avoid explosive or otherwise dangerous concentrations of CO.

Other modifications of my apparatus may be made without departing from the scope thereof. Thus, the compartments of cells 2, 3, 84, 94 may be rearranged so that the gas enters that part which contains the condenser and exhausts from that containing the Edwards balance. Baffles and other means of preventing eddy currents from affecting the balance are contemplated. The constant temperature baths may be insulated so that they remain at more constant temperature, and other obvious modifications may be made.

My invention may be used to control the process by which the gas stream is produced, the control being responsive to the dipole moment of the gas stream and operating on one or more appropriate process parameters. The term parameter of the process as used in the specification and appended claims may mean any process variable, such as temperature, pressure, flow rate, flow ratio, or other factor influencing the operation of the process and the quality of the material produced or the gas stream analyzed.

What I claim as my invention is:

1. Apparatus for determining the dipole moment of a gas which comprises means for maintaining a portion of the gas at a fixed known temperature, means for maintaining a second portion of the gas at a different fixed known temperature, means for maintaining the density of both said portions of the gas substantially equal and means for comparing the dielectric constant of said one portion with the dielectric constant of said second portion.

2. Apparatus for determining the dipole moment of a gas which comprises means for passing said gas at fixed density and known temperature between the plates of an electric condenser, means for passing said gas at the same density and different known temperature between the plates of a second similar electrical condenser and means for comparing the capacitances of said two condensers.

3. Apparatus for determining the dipole moment of a gas which comprises a pair of cells through which said gas is passed, means for maintaining each of said cells at different known temperatures, means for maintaining the gas in said cells at substantially equal densities, two similar electrical condensers in said cells respectively, said condensers having the gas as a part of their dielectric, and means for comparing the capacities of said condensers.

4. Apparatus for determining the dipole moment of a gas which comprises a pair of cells each containing said gas, float means in each of said cells, said floats having substantially equal displacements, means for detecting variations in buoyant force on each of said floats, means for adjusting the pressure in each of said cells to maintain said buoyant forces substantially equal, means for maintaining each of said cells and the gas therein at different temperatures, similar electrical condensers having the gas in the two cells respectively as a part of their dielectric, means for comparing the capacities of said condensers and means for exhibiting the variation in the relative capacities of said condensers.

5. Apparatus for determining the concentration of carbon monoxide in air which comprises means for sampling the air, means for drying said sample, means for passing said sample into two containers maintained at two fixed temperatures, respectively, similar Edwards float balances in each of said containers and means responsive to movement of Edwards float balances to vary the pressure in said respective containers so as to maintain substantially equal densities therein, similar electrical condensers in each of said containers a portion of the air in said containers forming the dielectric between the condenser plates, means for detecting minute variations in the difference between the capacitance of said condensers and means for indicating said difference.

6. The method of controlling the character of the gas produced by a process which comprises passing at least a portion of the gas into a cell, maintaining said cell and the gas therein at one temperature, adjusting the pressure in said cell to attain a fixed gas density, passing at least a portion of the gas into a second cell, maintaining said second cell and the gas therein at a second temperature, adjusting the pressure in said second cell to attain the same density as in the first cell, comparing the dielectric constants of the gas in the two cells, actuating a relay in response to variations in the relative dielectric constants of the gas in the two cells and causing said relay to operate means controlling a parameter of the process.

7. In apparatus for controlling a parameter of a process in response to variations in a characteristic of a gaseous product of said process, the improvement which comprises means for sampling a gaseous product of the process, means for passing said sample into two containers, means for maintaining said containers and their contents at two different fixed temperatures respectively, similar Edwards float balance means in each of said containers, means responsive to movement of said Edwards float balances to vary the gas pressure in said respective containers so as to maintain substantially equal densities therein, similar electrical condensers in each of said containers a portion of the gas in said containers forming the dielectric between the condenser plates, an electrostatic shield between the condensers and the Edwards float balance in each container, and means for detecting variations in the difference between the capacitances of said condensers in response to which a parameter of

the process may be controlled, whereby control is effected in response to the dipole moment of said sample.

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