FREEZE DRYING METHOD AND APPARATUS
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This invention relates to high vacuum drying operations and in particular to an improved method for determining the practical end point of freeze drying operations.
Freeze drying methods are used to dry temperature sensitive materials such as biologicals (antibiotics, sera, injectable vitamins, etc.), food products, and pharmaceutical products other than biologicals. In freeze drying methods, the material to be dried is frozen before applying vacuum and heat in a vacuum chamber. A continuous vacuum is maintained by means of a vacuum pump connected to the chamber. A condenser is generally used to remove the sublimated water vapor. As soon as all of the ice is removed from the material being dried, a higher temperature and lower pressure may be applied to the material without concern over melting the material being processed or causing the dry product to blow about the interior of the drier. If the applied heat is increased while the material being dried still contains solid ice, the appearance, flavor, or biological characteristics of the material may be destroyed. Of course, the permissible temperatures which may be applied to the material containing ice varies according to the material being dried.
For maximum efficiency of the drying operation, it is desirable that the temperature of the material being dried be increased as soon as all of the ice contained therein is sublimated. Simultaneously, it may be desirable to reduce the pressure within the drying chamber to aid in removing the last remaining amount of moisture. With temperature sensitive materials, such as enzymes or proteins, it may be desirable to maintain the temperature constant throughout the entire drying operation and reduce the pressure when all of the ice has been sublimated. A problem arises in accurately determining this point so that the temperature can be increased as rapidly and safely as possible and/or the pressure decreased. Thermocouples are generally used to indicate the temperature of the material being dried but are not entirely satisfactory. Thermocouples measure local temperatures and in a vacuum, relatively small interspaces act as excellent heat insulators so that good thermal contact with the material being dried is difficult to obtain. The problem is more acute where the material is being dried in a multitude of containers; for example, the drying of drugs contained in many small vials. In such a case, the temperature can be checked only at a relatively few points, often resulting in atypical measurements. In addition, as drying proceeds, the ice gradually recedes into the interior of the material being dried so that the thermocouple junction may emerge from the ice before all of the ice has actually been removed from the material. It can thus be seen that it is very important and yet quite difficult, in freeze drying operations, to accurately determine the instant at which the last traces of ice are removed from the material so that drying at a higher temperature and lower pressure can be initiated.
The present invention overcomes the noted difficulties and provides a method and apparatus for accurately determining the practical end point of a high vacuum drying operation, such as freeze drying, where water vapor is transported through a vacuum atmosphere. The method may also be utilized in other high vacuum drying operations where a solvent with a molecular weight higher than that of air must be removed from the material to be dried.
The method of the present invention consists of observing the pressure indications of two separate and dissimilar electrical vacuum pressure measuring devices placed in the vacuum chamber. By proper selection and utilization of control means in conjunction with the vacuum pressure measuring devices, the drying operation can be made automatic, permitting it to proceed unattended.
Two gauges are utilized; one an ionization type gauge of the kind described in U.S. Patent No. 2,497,213, and the other a thermocouple type gauge. In the practice of the method, it is essential that the two gauges be calibrated accurately for a vacuum atmosphere of air. The operation of each type of gauge is based upon different physical properties used in measuring the absolute pressure. Low molecular weight vapors such as water with a molecular weight of 18, as compared with air having an average molecular weight of 29, cause an ionization type gauge to read lower than for an equivalent pressure of air. Conversely, low molecular weight vapors have higher thermal conductivities and cause thermocouple type gauges to read higher than for an equivalent pressure of air. During the early part of a high vacuum drying operation, the two gauges differ in readings—the ionization type gauge reading lower than the thermocouple type gauge. The ratio of the readings may be as low as 1 to 2 if the vacuum system has minute air leakage. When all of the ice has been sublimated and essentially only air remains in the vacuum atmosphere, the gauges will have corresponding readings since they are originally calibrated accurately for a vacuum atmosphere of air. Thus, the end point of the drying operation, at which time all of the ice has been sublimated, can be determined accurately and the drying operation to remove the water bound by adsorption can be commenced immediately in order to obtain the desired residual moisture content. If desired, automatic control of the drying operation can be effected by automatic actuation of a vacuum control valve and a programmed heating cycle.
The practice of the method and apparatus used in practicing the method will be better understood upon consideration of the following description with reference to the following drawings, in which:
FIGURE 1 is a graph showing the pressure readings obtained as a function of time in the evaporation of ice;
FIGURE 2 is a graph showing the pressure readings obtained as a function of time when erythromycin phosphate is the material being dried;
FIGURE 3 is an elevational view of an apparatus embodying the present invention;
FIGURE 4 is a schematic representation in cross-section of an apparatus used in putting into practice the method of the invention.
With particular reference to the drawings, FIGURE 1 shows thermocouple vacuum pressure gauge readings (curve I) and ionization type vacuum gauge readings of curve II in the evaporation of ice utilizing the present invention. In a thermocouple type gauge, as described in High Vacuum Technique by Yarwood, the thermo-E.M.F. developed is recorded by a sensitive galvanometer. The temperature attained by the junction depends on the conductivity, and hence on the pressure. Vapors of lower molecular weight than air, for example water vapor, have higher thermal conductivities and cause thermocouple type vacuum gauges to read higher than for an equivalent pressure of air. Ionization type vacuum gauges, as described in U.S. Patent No. 2,497,213 or Vacuum Technique by Dushman, will read for an equivalent pressure of air—when measuring the pressure of low molecular weight vapors. In utilizing the
method of the present invention, the two gauges are first calibrated accurately for a vacuum atmosphere of air. Referring to FIGURE 1, it can be seen that in the initial stages of the evaporation of ice, when large amounts of water vapor are present in the drying chamber, the pressure acting on the two gauges is essentially the same. As evaporation of the ice proceeds and the quantity of water vapor in the drying chamber diminishes, the readings of the two gauges gradually approach each other in magnitude. At point E₁, all of the ice has been sublimated and the atmosphere remains in the vacuum atmosphere. The two gauges, having initially been calibrated for a vacuum atmosphere of air will thus have corresponding readings as illustrated in FIGURE 1. The effective end point of the drying operation is thus easily and accurately determined.

In FIGURE 2, the freeze drying operation was carried out in accordance with the present invention, using erythromycin phosphate as the material being dried. The thermocouple vacuum gauge pressure readings are indicated by curve III, and the ionization type vacuum gauge pressure readings are shown by curve IV. Four contain- ers, each containing about 550 milliliters of frozen, heavy paste concentrate of erythromycin phosphate were placed in a vacuum drying chamber. The drying cycle began at approximately 50 μg. Hg, as determined by the ionization type vacuum gauge pressure. The pressure was then increased to about 300 μg. Hg; and after an operating period, as described above, the pressure was reduced to about 500 μg. Hg. The increases in pressure early in the drying operation were primarily for the purpose of decreasing the overall drying period and to obtain as frequent data points as possible. In practice, the drying operation can be conducted at a constant pressure until the major portion of the moisture has been removed, at which time the pressure can be decreased to remove the last remaining amount of moisture. Drying was continued under these conditions, with the moisture content of the erythromycin phosphate gradually being reduced, as indicated by the declining portion of curve III. At point E₂, the erythromycin phosphate is essentially dry and the two gauges indicate corresponding readings. In order to remove adsorbed moisture, it is desirable to reduce the pressure in the drying chamber at the end of the cycle. The result of the foregoing action is that the point E₃ was reached. After the point E₂, the pressure in the drying chamber was reduced from about 500 μg. Hg to 100 μg. Hg, resulting in points E₃ and E₄, respectively. Subsequently, the operating pressure was adjusted down to the ultimate vacuum for the system, and at point E₅, the cycle was terminated. Since after point E₁ is reached, the atmosphere in the drying chamber is essentially air, the two gauge readings are substantially the same. Six hundred sixteen grams of dry, moisture-free erythromycin phosphate in the form of a firm pulverizable cake was recovered.

The present invention also provides an apparatus which is especially well suited for putting into practice the foregoing described method. The essential feature of the apparatus consists in a vacuum drying chamber being provided with two separate and dissimilar vacuum pressure measuring devices as previously described. The two devices may be used for visual and manual monitoring of a freeze drying operation in accordance with the present invention. If desired, by appropriate means, the drying operation can be made entirely automatic. An example of a suitable automatic system utilizing the two dissimilar vacuum pressure measuring devices is as follows. An ionization type vacuum gauge pressure is equipped with a device for measuring and transmitting device for operating a pressure control valve such as a vacuum control air bleed or a butterfly type damper valve at the suction side of the vacuum pump to maintain a constant operating pressure within the drying chamber. A thermocouple type vacuum pressure gauge is equipped with a sensing control and transmitting device set at the same point as the ionization type gauge pressure control read- ing. Such a system will permit the drying operation to proceed unattended at a constant heating medium temperature until the thermocouple type gauge sensing device actuates the set point and transmits a signal, electrically or pneumatically, to a heating device such as a thermostatically controlled immersion type electrical heater in the heating medium circulating system. Likewise, at the set point, the thermocouple type gauge sensing and transmitting device actuates the ionization type gauge control device to actuate the vacuum control air bleed; for example, to close it and thereby decrease the pressure for the more complete removal of the small remaining amount of moisture. At this time, the major portion of the ice is removed from the product and a higher temperature and/or lower pressure may be applied without concern over melting the material being processed or causing the dry product to blow about the interior of the drying chamber.

The improved apparatus as represented in FIGURES 3 and 4. Within the vacuum drying chamber 10 is placed a container 11 in which air is confined the material 12 to be dried. In the illustrated embodiment, a support 13 is provided to hold the container 11 and the material 12 to be dried. Heat can be supplied to the material 12 by any suitable means. As illustrated, a circulating fluid heating system 14 is utilized. A heating fluid 15 is circulated through the material 12 to effect drying. A fluid 16 is used to heat the fluid 15 in the heating system 18 to desired constant heating temperature. An auxiliary heater 19 is utilized in conjunction with the thermocouple type gauge pressure sensing control and transmitting device 20 to increase the heating temperature at the desired point in the drying cycle. A sensing control and transmitting device 21 in the manner described. A thermocouple type gauge pressure sensing control and transmitting device 22 is employed in conjunction with the ionization type gauge 23 to actuate the heating means 19 at the desired point in the drying cycle, as previously described. Likewise, a pressure sensing control and transmitting device 24 is employed in conjunction with the ionization type gauge 25 to actuate the vacuum pressure control valve 26 located at the vacuum side of the suction pump 27. The function of this pressure sensing control and transmitting device is to operate the vacuum control valve 26 in order to maintain the desired operating pressure. When the thermocouple type gauge 23 reaches the set point, at which time the major portion of the moisture has been removed from the material being dried, the pressure sensing control and transmitting device 20 will deactivate the pressure sensing control and transmitting device 25, utilized in conjunction with the ionization type gauge 24, to activate the vacuum pressure control valve 26. In this way, the pressure control valve 26, which may be a vacuum control air bleed for example, is closed at the appropriate time to thereby decrease the pressure in the vacuum drying chamber 10 and aid in removing the small amount of adsorbed moisture which remains in the material 12 to be dried. When drying temperature-sensitive materials at such a temperature such that the material temperature can be maintained constant throughout the run. In such a case, the thermocouple gauge 23 and pressure sensing control valve 26 can be used solely to reduce the pressure within the drying chamber at the desired point in the drying cycle, in the manner described.
A condenser 28 is provided to condense the water vapor withdrawn from the material 12. The condenser 28 may be in the form of a cooling coil and may be located in an adjoining chamber rather than in the vacuum drying chamber 16. Valves 29 may be located at various points for displacement of the drying chamber atmosphere, draining condensate, or other purposes.

As illustrated in FIGURE 3, a thermocouple type gauge 23 and an ionization type gauge 24 may be employed for visual and manual control of a freeze drying operation. Readings of the two gauges may be observed and when the readings are substantially the same, indicating that the major portion of the ice has been removed from the material being dried, the pressure control valve 26 may be closed to reduce the pressure in the vacuum drying chamber 10 and thereby facilitate the removal of the last amount of moisture remaining in the material. The heating temperature may be safely increased at this time.

In order to properly freeze dry a material, the ice therein must be sublimed. If the ice in the material is permitted to melt, the properties of the freeze dried material are not as good. With the method of the present invention, completion of the initial drying stage, at which point the major portion of the moisture has been removed from the material being dried, can be accurately determined so that initiation of the final drying stage can be started or the operation can be terminated, as desired.

Others can readily adapt the present invention in other specific forms by employing one or more of the novel features disclosed, or equivalents thereof. All such practice of the invention is considered to be a part hereof provided it falls within the scope of the appended claims.

What is claimed is:

1. A method of drying a temperature sensitive material in a vacuum chamber having in communication therewith a thermocouple type vacuum gauge and an ionization type vacuum gauge, comprising the steps of: freezing the material; placing the frozen material in the vacuum chamber; applying heat and vacuum to the vacuum chamber to remove moisture from the material being dried; calibrating the thermocouple type vacuum gauge and the ionization type vacuum gauge for a vacuum atmosphere of air; recording the thermocouple type vacuum gauge reading while conducting the drying operation; simultaneously recording the ionization type vacuum gauge reading; and applying additional heat and vacuum to the vacuum chamber when the thermocouple type vacuum gauge reading and the ionization type vacuum gauge reading are essentially the same, at which time the major portion of the moisture has been removed from the material being dried and the vacuum chamber atmosphere is substantially all air.

2. A method of drying a temperature sensitive material in a vacuum chamber having in communication therewith a thermocouple type vacuum gauge and an ionization type vacuum gauge, comprising the steps of: freezing the material; placing the frozen material in the vacuum chamber; calibrating the thermocouple type vacuum gauge and the ionization type vacuum gauge for a vacuum atmosphere of air; applying vacuum to the vacuum chamber to remove moisture from the material being dried; charting the thermocouple type vacuum gauge reading while conducting the drying operation; simultaneously charting the ionization type vacuum gauge reading; and terminating the drying operation when the thermocouple type vacuum gauge reading and the ionization type vacuum gauge reading are essentially the same; at which time the major portion of the moisture has been removed from the material being dried and the vacuum chamber atmosphere is substantially all air.

3. In a freeze drying operation conducted in a vacuum chamber having in communication therewith a thermocouple type vacuum gauge and an ionization type vacuum gauge, the method of determining when substantially all of the moisture has been removed from the material being dried, comprising the steps of: calibrating the thermocouple type vacuum gauge and the ionization type vacuum gauge for a vacuum atmosphere of air; simultaneously recording the pressure of the vapor within the vacuum chamber; simultaneously producing an impulse in the ionization type vacuum gauge; and terminating the drying operation when the respective impulses of the thermocouple type vacuum gauge and the ionization type vacuum gauge are essentially the same, at which time the vacuum chamber atmosphere is substantially all air and the major portion of the moisture has been removed from the material being dried.

4. In a freeze drying operation conducted in a vacuum chamber having in communication therewith a thermocouple type vacuum gauge and an ionization type vacuum gauge, the method of determining when substantially all of the moisture has been removed from the material being dried, comprising the steps of: calibrating the thermocouple type vacuum gauge and the ionization type vacuum gauge for a vacuum atmosphere of air; simultaneously recording the ionization type vacuum gauge reading and the thermocouple type vacuum gauge reading while conducting the drying operation; and terminating the drying operation when the thermocouple type vacuum gauge reading and the ionization type vacuum gauge reading are essentially the same, at which time the vacuum chamber atmosphere is substantially all air and the major portion of the moisture has been removed from the material being dried.

5. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; supporting means within said chamber for supporting the material to be dried; heating means for heating said material; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; thermocouple type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber; ionization type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber; and means for controlling vacuum pressure within said vacuum drying chamber; and a pressure sensing control and transmitting device responsive to the thermocouple type vacuum pressure measuring means to actuate and close the vacuum control means when the thermocouple and ionization type vacuum measuring means have substantially the same reading, whereby the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.

6. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; a support within said chamber for supporting the material to be dried; means coupled to said support for heating said support; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; thermocouple type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber; ionization type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber; and means for controlling vacuum pressure within said vacuum drying chamber; and a pressure sensing control and transmitting device responsive to the thermocouple type vacuum pressure measuring means to actuate and close the vacuum control means when the thermocouple and ionization type vacuum measuring means have substantially the same reading, whereby the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.
substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; vacuum control means for controlling the vacuum within said vacuum drying chamber; and a pressure sensing control and transmitting device responsive to the thermocouple and ionization type vacuum pressure measuring means to actuate and close the vacuum control means when the thermocouple and ionization type vacuum pressure measuring means have substantially the same reading, whereby the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.

7. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; supporting means within said chamber for supporting the material to be dried; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; thermocouple type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber; ionization type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; and a pressure sensing control and transmitting device responsive to the thermocouple and ionization type vacuum pressure measuring means to actuate the heating means when the thermocouple and ionization type vacuum pressure measuring means have substantially the same reading, whereby additional heat is automatically supplied when the major portion of the moisture has been removed from the material being dried.

8. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; a support within said chamber for supporting the material to be dried; heating means coupled to said shelf for heating said support; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; thermocouple type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber; ionization type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; vacuum control means in communication with the vacuum pressure measuring means and ionization type vacuum pressure measuring means to actuate the heating means when the vacuum pressure measuring means and ionization type vacuum pressure measuring means have substantially the same reading, whereby additional heat is automatically supplied and the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.

9. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; supporting means within said chamber for supporting the material to be dried; heating means within said chamber; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; thermocouple type vacuum pressure measuring means for measuring the pressure within said chamber; ionization type vacuum pressure measuring means for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; vacuum control means in communication with the vacuum pressure measuring means for controlling the vacuum within said vacuum drying chamber; pressure sensing control and transmitting means responsive to the thermocouple type vacuum pressure measuring means to actuate and close the vacuum control means, respectively, when the thermocouple and ionization type vacuum pressure measuring means have substantially the same reading, whereby additional heat is automatically supplied and the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.

10. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; a support within said chamber for supporting the material to be dried; means coupled to said support for heating said support; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; thermocouple type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means communicating with the chamber for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; vacuum control means in communication with the vacuum pressure measuring means and ionization type vacuum pressure measuring means to actuate the heating means when the vacuum pressure measuring means and ionization type vacuum pressure measuring means have substantially the same reading, whereby additional heat is automatically supplied and the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.

11. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; a support within said chamber for supporting the material to be dried; a circulating medium heating system coupled to said support for heating said support; a vacuum pump communicating with the vacuum drying chamber for evacuating said vacuum drying chamber; a condenser disposed within said chamber for condensing moisture withdrawn from the material being dried; a thermocouple type vacuum pressure gauge communicating with the chamber for measuring the pressure within said chamber; and an ionization type vacuum pressure gauge communicating with the chamber for measuring the pressure within said chamber, said thermocouple type vacuum pressure gauge and ionization type vacuum pressure gauge being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air.

12. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber; solar vacuum pressure measuring means for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; vacuum control means in communication with the vacuum pressure measuring means for controlling the vacuum within said vacuum drying chamber; pressure sensing control and transmitting means responsive to the thermocouple type vacuum pressure measuring means to actuate the heating means and close the vacuum control means, respectively, when the thermocouple and ionization type vacuum pressure measuring means have substantially the same reading, whereby additional heat is automatically supplied and the pressure within the drying chamber is automatically reduced when the major portion of the moisture has been removed from the material being dried.
chamber wherein the material to be dried is placed; supporting means for supporting the material to be dried; means coupled to said supporting means for heating the supporting means; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; said drying chamber having in communication therewith thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air; and vacuum control means for controlling the vacuum within said vacuum drying chamber.

13. A freeze drying apparatus for removing moisture from material to be dried, comprising: a vacuum drying chamber wherein the material to be dried is placed; heating means for heating said material; means for evacuating the vacuum drying chamber; a condenser for condensing moisture withdrawn from the material being dried; said drying chamber having in communication therewith thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means for measuring the pressure within said chamber, said thermocouple type vacuum pressure measuring means and ionization type vacuum pressure measuring means being calibrated for a vacuum atmosphere of air so that their pressure readings will be substantially the same when the major portion of the moisture has been removed from the material being dried and the vacuum drying chamber atmosphere is essentially air.

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