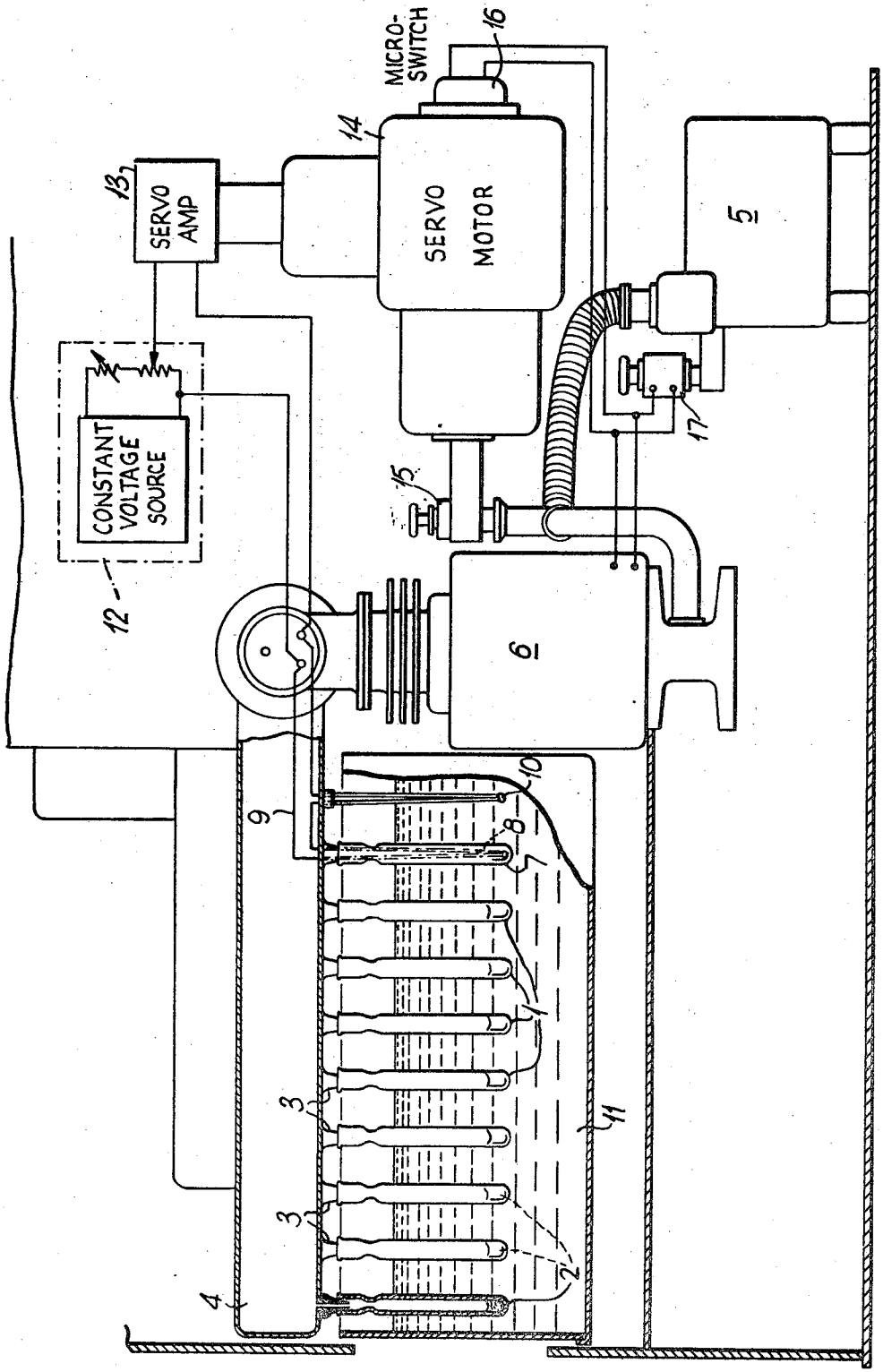
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ISOTHERMAL DRYING APPARATUS AND METHOD

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ISOTHERMAL DRYING APPARATUS AND METHOD

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ABSTRACT OF THE DISCLOSURE

A method of drying specimens under isothermal conditions in which receptables containing the specimens are uniformly heated to a predetermined temperature, the receptacles are partially evacuated and the total pressure in the receptacles is varied to maintain the loss of heat from the specimens due to evaporative cooling substantially equal to the rate at which heat is supplied to the specimen. When the desiccation of the specimen is completed, the receptacles are evacuated to a high vacuum and hermeticaly sealed.

There is also disclosed an apparatus for performing the method.

A common method of preserving specimens of materials is to dry them and then seal them into receptacles which are stored until the specimens are required.

The drying is frequently carried out by a process known as "freeze drying" in which the specimen is either prefrozen and then placed in a vacuum chamber or placed directly in the chamber and frozen by evaporative cooling. The liquid contained in the specimen, which is usually water, is subsequently removed by the process of sublimation. The receptacle in which the specimen is contained is then hermetically sealed.

It has been shown, however, that cultures of microorganisms that have been dried at bacteriologically mesothermal temperatures have greater viabilities than those dried from the frozen condition.

Mesothermal conditions during drying can be achieved by partially immersing receptacles containing the cultures in a water bath, the temperature of which is maintained at about 20° C. Thermal energy supplied from the water bath allows the rate of evaporation of liquid from the sample to be increased at the same time as it maintains the mesothermal state. The increased rate of evaporation and drying, however, is inclined to fluctuate, thus making accurate control and reproducibility of the specimen temperature difficult to achieve.

According to the present invention there is provided a method of drying a specimen under isothermal conditions wherein a receptacle containing the specimen is heated to a predetermined temperature and the receptacle is partially evacuated, the total pressure within the receptacle being controlled in such a manner that the rate of loss of heat from the specimen is substantially equal to the rate at which heat is supplied to the specimen.

According to the invention there is also provided apparatus for drying specimens under isothermal conditions comprising means for uniformly heating a plurality of receptacles, each of the receptacles being adapted to contain a specimen to be dried, means for evacuating the receptacles, temperature sensing means for determining the temperature of at least one of the specimens, and means responsive to the temperature sensing means for controlling the total pressure within the receptacles in

such a manner that the rate of loss of heat from the specimen is substantially equal to the rate at which heat is supplied to the specimens from the heating means, whereby the etmperature of the specimen is maintained substantially at the predetermined value.

Preferably the heating means comprises a water-bath, and the means for controlling the total pressure in the receptacles comprises an adjustable valve for admitting air, or other permanent gas into a pipe through which the receptacles are evacuated, and which may be closed off when desiccation has been substantially completed.

The method also preferably includes the steps of evacuating the receptacles to a high vacuum after desiccation is substantially completed, and then sealing the receptacles.

Referring to the accompanying drawing which illustrates diagrammatically, by way of example, an isothermal drying apparatus embodying the invention, a number of ampoules **1**, which contain equal volumes of a material **2** which is to be dried, are attached by means of rubber nipples **3**, of a known kind which will not be described further, to a manifold **4** through which they are evacuated by a gas-ballasted rotary vacuum pump **5**, and an air-cooled oil diffusion pump **6**. A control ampoule **7** is identical with the ampoules **1** but contains a slightly larger amount of the material **2**. One junction **8** of a thermo-couple **9** is inserted into the control ampoule **7** and the other junction **10** is immersed in a water bath **11**, which surrounds the ampoules and is heated in a conventional manner to the temperature at which it is desired to carry out the drying operation. The E.M.F. generated by the thermo-couple **9** is fed in opposition to a control potential generated by a source **12**, and the resultant is fed into a null-balancing servo-amplifier **13**. The servo-amplifier **13** drives a servo-motor **14** that is linked to a needle-valve **15** which enabled air, or other gas, to be bled into the system. The arrangement is such that the needle valve **15** is opened when the temperature difference between the control ampoule **7** and the water bath **11** rises above a value corresponding to a preset control E.M.F. The conditions within the ampoules **1** parallel those in the control ampoule **7**. Thus as water vapour is removed from the ampoules **1** and **7** and the material **2** cools to a predetermined temperature, the needle valve **15** is opened and air, or other permanent gas, is leaked into the system. The effect of this is to prevent further cooling of the material. This is because the proportion of water vapour removed by the pump **5** is reduced, and therefore the water vapour pressure in the ampoules **1** and **7** rises; and as the rate of evaporation of a liquid from a surface is proportional to the water vapour pressure gradient at and above the surface, the rate of evaporation of water from the material **2**, and hence the cooling, falls off. The converse applies if the temperature of the control ampoule **7** rises above the predetermined value.

When the bulk of the water has been removed, the temperature of the material in the ampoules **1** begins to rise towards that of the water-bath **11**. This occurs in the control ampoule **7** slightly later than the other ampoules **1** because of the slightly greater amount of material it contains. Thus it is ensured that the material **2** in the ampoules **1** is nominally dried before the final stage of the process begins. As the temperature of the control ampoule **7** rises above the control temperature the needle valve **15** is driven to the fully shut position at which the needle valve mechanism operates a microswitch **16** which then actuates a mechanism, not shown, by means of which the manifold is moved to a position at which the ampoules **1** and **7** are removed from the water bath **11** and held in an approximately horizontal position. The microswitch **16** also operates a magnetic air valve **17** which shuts off the gas ballast to the rotary pump **5** enabling it to act as the

backing pump for the diffusion pump **6**, and after an interval governed by a delay means, not shown, that may include a pressure sensitive, or a temperature sensitive switch, energises the heater of the diffusion pump **6**.

The ampoules are exacuated to a pressure of at least 10^{-3} torr, and the ampoules **1** are then sealed while under vacuum in a conventional manner by means of a high temperature flame.

The water bath **11** may be replaced by a heated, fluidised sand bath, This modification has the advantage that the ampoules **1** remain dry on the outside, reducing the risk of breakage during the sealine process: also should any of the ampoules **1** break during the first stage of the process, the danger of flooding of the vacuum system is eliminated.

In a simpler form of the apparatus, which is not illustrated, the process is completed after the initial drying by transferring the ampoules to a manifold on a secondary stage apparatus, and evacuating them by means of a two-stage rotary vacuum pump, before sealing them off as before.

In practice it may be found that for many purposes the apparatus will operate satisfactorily if the amount of material in the control ampoule is the same as that in the ampolues **1**.

We claim:

1. A method of drying a specimen under isothermal conditions comprising the operations of uniformly heating a receptacle containing said specimen to a predetermined temperature, partially evacuating said receptacle and controlling the total pressure within the receptacle in such a manner that the rate of evaporation of volatile material from said specimen is such that the rate of loss of heat from said specimen is substantially equal to the rate at which heat is supplied to said specimen whereby the temperature of said specimen is maintained substantially constant.

2. A method according to claim **1**, wherein said receptacle is finally evacuated to a high vacuum and hermetically sealed.

3. Apparatus for drying a specimen under isothermal conditions comprising; means for uniformly heating a plurality of receptacles to a predetermined temperature, each of said receptacles being adapted to contain a specimen to be dried; means for evacuating said receptacles; temperature-sensing means for determining the temperature of said specimens; means responsive to said temperature-sensing means; and means operable by said responsive means for controlling the total pressure within said receptacles in such a manner that the rate of loss of heat from said specimens due to the evaporation of volatile material from said specimens is substantially equal to the rate at which heat is supplied to said specimens from said heating means.

4. Apparatus according to claim **3** including means for evacuating said receptacles to a high vacuum when the desiccation of said specimens is completed.

5. Apparatus for drying a specimen under isothermal conditions comprising; means for uniformly heating a plurality of receptacles, each of said receptacles being adapted to contain a specimen to be dried; means for evacuating said receptacles; temperature-sensing means for determining the temperature of said specimens; means responsive to said temperature-sensing means; and means

operable by said responsive means for controlling the total pressure within said receptacle in such a manner that the rate of loss of heat from said specimens is substantially equal to the rate at which heat is supplied to said speciment from said heating means; said sensing means comprises a thermo-couple adapted to measure the temperature differential between a specimen contained in one of said receptacles and said heating means.

6. Apparatus according to claim **5** wherein said means responsive to said temperature-sensing means comprises a source of a variable control potential; a null balancing servo amplifier to which the potential generated by said thermo-couple and said control potential are applied in opposition; and a servo motor connected to said servo amplier and arranged to operate said controlling means.

7. Apparatus according to claim **3**, wherein said means for controlling the total pressure within said receptacles comprises an adjustable valve for bleeding a permanent gas into said evacuating means via said valve.

8. Apparatus for drying a specimen under isothermal conditions comprising; means for uniformly heating a plurality of receptacles, each of said receptacles being adapted to contain a speciment to be dried; means for evacuating said receptacles; temperature-sensing means for determining the temperature of said specimens; means responsive to said temperature-sensing means; and means operable by said responsive means for controlling the total pressure within said receptacle in such a manner that the rate of loss of heat from said specimens is substantially equal to the rate at which heat is supplied to said specimen from said heating means; the said heating means comprises a heated water bath.

9. Apparatus for drying a specimen under isothermal conditions comprising; means for uniformly heating a plurality of receptacles, each of said receptacles being adaped to contain a specimen to be dried; means for evacuating said receptacles, temperature-sensing means for determining the temperature of said specimens; means responsive to said temperature-sensing means; and means operable by said responsive means for controlling the total pressure within said receptacle in such a manner that the rate of loss of heat from said specimens is substantially equal to the rate at which heat is supplied to said specimen from said heating means; said heating means comprises a heated bed of fluidised solid material.

References Cited

UNITED STATES PATENTS

2,199,816	5/1940	Flosdorf	34—5
2,353,986	7/1944	Barr	34—5
2,994,132	8/1961	Newmann	34—92
3,077,036	2/1963	Newmann	34—5
3,145,562	8/1964	Hamilton	34—5
3,176,408	4/1965	Fuenteville	34—5
3,178,829	4/1965	Cox	34—5
3,192,643	7/1965	Rieutord	34—5
3,230,633	1/1966	Hamilton	34—92
3,259,991	7/1966	Illich	34—5

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