Physics. — "*Methods and apparatus used in the cryogenic laboratory. III. Baths of very uniform and constant low temperatures in the cryostat.*" Communication N°. 83 from the physical laboratory at Leiden by Prof. H. KAMERLINGH ONNES.

(Communicated in the meeting of December 27, 1902.)

§ 1. By means of the cryostat described in § 8, Comm. 14. Dec. '94, and § 3, Comm. 51. Sept. '99 we can obtain a bath of liquefied gas which is shut off from the atmosphere and boils at ordinary or diminished pressure. In such a bath the temperature is sufficiently uniform and constant for many experiments and measurements. If we use almost pure gases and if the evaporated gas is regularly recondensed by means of a compression apparatus, which as described in Comms. 14. Dec. '94, 53. Sept. '99 and 54. Jan. '00, does not contaminate the gas, the bath may be maintained as long as we wish. The operations in the bath itself as well as the addition of the liquefied gas can be watched through the observing glasses. Vacuum glasses are not required so that similar cryostats may be constructed for measuring apparatus of any dimensions. Before long we shall describe a cryostat where the gas apparatus and the bath are more independent.

I was led to describe the form of the cryostat, as it occurs in Comm. 51, through the communication of the results for the di-electric constants of liquid gases. (Comm. 52 Oct. '99), for which measurements only the temperatures of — 90° C. or — 182° C. were required. For other measurements, however, a measuring apparatus, once immersed in the cryostat, has been used at the whole range of temperatures between, — 23° C. (boiling point of methyl chloride at ordinary pressure) and — 210° C. (nitrogen at reduced pressure), given by methylchloride nitrous-oxide, ethylene, methane, oxygen and nitrogen as they were successively admitted into the cryostat.

For a long time improvements have been made in this cryostat by means of which we can attain a much greater uniformity and constancy in the temperature, while retaining the afore-mentioned advantages. A description of these alterations has now become necessary in order to judge of the accuracy of the temperature readings in the results from various measurements where we have availed ourselves of these improvements. These measurements will be treated in the next communications. Among others I mention here those bearing upon the isothermals of diatomic gases (Comms. 69 March '01

and 78, March '02) and the comparison between the platinum resistance thermometer and the hydrogen thermometer (Comm. 77 Febr. '02) In this description, as in Comm. 51. Sept. '99, it seems to me desirable to illustrate the use of the cryostat by means of a special example. We will consider the comparison of the hydrogen thermometer with the resistance thermometer where also a thermo-element had been immersed in the bath.

Plate I shows the cryostat and some of the auxiliary apparatus to scale, the connections are represented schematically. It has been drawn on a smaller scale than plate I of Comm. 51 Sept. '99, (which should be consulted together with the one now given) but it will suffice to give a survey of the whole arrangement and to show some of the alterations. While the details of the unmodified parts can be studied on plate I of Comm. 51, plate II of the present Communication shows the details of the parts enclosed by the dot-dash-line of plate I, as far as they are required for consideration of the new arrangements. The connection of the apparatus shown in Pl. I with the gas circulation can be seen in Pl. IV Comm. 51. The comparison of the platinum thermometer p and the hydrogen thermometer Th and their connections to the other pieces of the apparatus are given in Comm. 77 Febr. '02 § 3. For the comparison of the thermo-element Θ I am as yet obliged to refer to the very rough diagram of 1896 (Pl. I of Comm. 27 Mai and June '96). The communication, however, of some results for which the temperatures have been determined by means of a thermo-element will soon call for a description of the recent considerable improvements in the use of the thermo-elements.

On plates I and II a correction thermometer ζ, which is entirely independent of the cryostat, will be seen besides the three measuring apparatus mentioned above. It serves in our case to indicate the mean temperature of the capillary of the hydrogen thermometer, or in general, the mean temperature of similar pieces of measuring apparatus occupying the same part of the cryostat. For this purpose two spirals of platinum wire are wound round a glass rod, the one for that part of the rod, where the temperature varies slowly ζ_2, the other for that part where the temperature varies rapidly ζ_1. By means of the leads ζ_{00}, connected to the places of contact ζ_{11}, ζ_{12} and ζ_{21} and emerging through the tube ζ_{01}, we can determine the resistance of these spirals.

§ 2. First we shall mention some small changes in the cryostat of Comm. 51 which have no relation to the question of keeping the temperature constant and uniform.

The jet of liquefied gas let in at a (plate I) is directed, by means of the cock h_6 and the filter f, against a glass wall from which it streams along the delivery spout D_1 into the bath, here a double beaker B_{01} B_{02} (Pls. I and II), placed in the beakers B_1, B_2, B_3 of Pl. I Comm. 51. The cock and filter form part of a cover which as described in Comm. 51, may be removed together with S_1 and S_3 from the cryostat and may also be replaced by a syphon or a capillary with a cock outside the cryostat. The spreading of the jet over the wall may be watched through the windows V_1, and the height of the liquid in the bath through the windows V_2. The filter f serves principally to prevent opaque dust from the lead (oxide of copper etc.) from depositing just at the place where the jet touches the glass. In many cases, however, it happened in spite of the care taken in purification, that the liquefied gas itself, while evaporating under reduced pressure in the cryostat, had deposited a substance, formerly dissolved in it but solid at the lower temperature, thus rendering the bath opaque. Therefore, differing from Comm. 51, a glass beaker C_1 (Pls. I and II) with numerous openings in the bottom C_{10} (Pl. II) and containing some glass wool was suspended by the regenerator spiral b (Pl. I Comm. 51). This filter may be lifted from the cryostat together with the piece S_6.

With the arrangement as described in Comm. 51 all the gas, formed after the liquid leaves the cock, goes in the direction indicated by the arrows on Pl. I Comm. 51. With the arrangement as described here, however, the gas which is formed while the bath is being filled follows, in the main a different direction to that which afterwards evaporates from the bath. In fact, differing from Comm. 51, a valve D_{110} with a spring D_{12} has been added, which almost closes the opening of the delivery spout D_1 for gas, but allows liquid to flow through a very narrow opening D_{111} along the gutter D_{13}. The first considerable quantities flowing from the cock, serve to cool all the beakers and the whole cryostat in the way indicated in Comm. 51 (the arrows of plate I might be borrowed from plate I of Comm. 51), unless the supply becomes so great that the valve D_{111} is opened and the gas also flows out through the opening R_{10} in the ring R_1, plate II. The gas which later evaporates from the beaker B_{01}, finds the valve D_{111} closed and escapes only through the opening R_{10}, along the way indicated by the arrows on plate II, so that it serves only to screen the immediate neighbourhood of the bath from external heat.

The difference in form between the rings R_3 and R_4 on plate II and those on plate I Comm. 51 is very slight. This follows from the wish to use the parts that served in the experiments, referred to in

Comm. 51, as much as possible in the arrangement of the measuring apparatus considered here. Formerly the bath could be excentrically mounted with reference to the tube F_1 whereas this time a central mounting was desirable. The existing dimensions of parts of the apparatus have also had the result that in the experiments described here the bath must be placed a little too high with regard to the observing glasses V_2, which might easily have been avoided if we had been perfectly free in our construction.

The glass ring R_5, not occurring in the arrangement of Comm. 51, serves still better to screen the bath from external heat. Like the other beakers and glass cylinders B_1, B_2, B_3, B_4, B_{01}, B_{02}, it is silvered inside and outside, leaving open, however, vertical strips nearly corresponding in width with the resistance thermometer p.

The conical rim B_{01} lies loose on the beaker B_{01}. When the liquid boils up, it streams back to B_{01} along the wall of the funnel; if, however, B_{01} is filled to the brim and more liquid is poured in, this superfluous liquid flows over into the beaker B_{02}, which also is filled before a measurement is made. If an intense cooling of the neighbourhood of the bath is required, the beakers B_1, B_2, B_3 must also be filled. It should be remembered, however, that if this is done, the evaporation at low pressure, as long as liquid remains in the outer beakers, requires a powerful vacuumpump.

The bath itself only evaporates slowly. Instead of the double beakers B_{01} B_{02} we might take a vacuum glass in order to diminish the evaporation as has sometimes been done (comp. § 3). But it is not always easy to obtain vacuum glasses of the required dimensions and internally finished with the accuracy necessary for the proper working of the stirring apparatus. Moreover one will not be inclined to immerse delicate measuring apparatus in the bath before one is sufficiently certain that the vacuum glass will not burst as such of greater dimensions sometimes do.

§ 3. To make clear the purpose of the arrangements to be described in the next sections, it seems to me that the following particularisations will be useful. First of all the temperature gradient in the bath. Even when the liquid boils regularly we find that in the lower layers, as a result of the hydrostatic pressure, the temperature exceeds that of the upper layers. If, as often happens with greatly diminished pressures when boiling is not produced artificially, only evaporation at the surface occurs instead of boiling, the temperature in the upper layers of the bath may fall considerably below that of the lower. If then the liquid suddenly boils up, which always happens whenever

we do not stir vigorously, an unexpected change takes place in the distribution of the temperature in the bath and hence in the temperature of any measuring apparatus placed in it. In measurements of the kind considered here, we cannot allow such irregularities and fluctuations in the temperature of the bath, either as to time or place.

Of the various methods of preventing this sudden ebullition, the simplest is the generation of small bubbles of gas by means of the heat of a short resistance (boiling thread). If, however, there are ignitible gases among those successively introduced into the apparatus and if consequently an explosive mixture with air might be formed, this method is not without danger.

To bring about ebullition a current of gas is often led through the liquid, which, however, has the disadvantage of contaminating the evaporated gas. To avoid this difficulty I have led through the bath a current of the gas itself. This means was applied for instance to avoid the retardation in boiling in the vacuum vessel mentioned at the end of § 2, and also in order to cause a strong stirring in the bath by means of the current of gasbubbles. But this means also presents many difficulties, mostly arising from condensation phenomena in the delivery tube, or higher temperature of the gasbubbles; I therefore, preferred, the arrangement as described in § 4.

If the cryostat is used as it was intended to be in Comm. 51, the requirements for very accurate measurements would not be fulfilled, even though a uniform temperature throughout the bath was attained. There still remains a systematic regular rise of the temperature, because the gas used is never perfectly pure and the more permanent part evaporates first. In cases where measuring apparatus require longer to adopt the temperature of the bath than the time in which the temperature changes the amount permitted by the accuracy of the observation, we cannot reach more accurate results without additional means.

§ 4. We now pass on to the description of the arrangements which form the subject of this communication. The uniform temperature in the bath is obtained by stirring. The stirring apparatus is placed concentrically to the bath, thus leaving room in the most profitable way for the measuring apparatus. From this space the stirring apparatus (as in Comm. 27 May and June '96 Pl. III) is separated by a protecting cylinder ξ_0 (comp. the figure to the left of plate I). The upper ring χ_{01} is provided with small valves χ_{04} covering openings of the same form. If the stirring apparatus moves in the cylindrical space between ξ_0 and B_{02} the valves shut up

during the upward movement and open during the downward movement. The upward movement is brought about by means of the thin wires χ_1, the downward movement by the weight of the stirring apparatus itself which for this purpose is weighted with the heavy ring χ_{02} by means of the rods χ_{03}. As yet a more rapid motion of the stirring apparatus than this method affords has not been required; if wanted a construction with small rods instead of threads would be necessary. The valves are hinged on bent pins χ_{041}. The complete section of the stirrer to the right of plate II shows the valves shut, the section of χ_{01} at the top shows them open. When the stirring apparatus is moved up and down and the bubbles of vapour escape the movements of the valves resemble those of the fins of fishes.

It is very important that the up and down motion of the ring should be perfectly perpendicular and that the protecting cylinder ξ_1 and the beaker B_{02} should have a perfectly vertical position for, to make the valves work properly, only a narrow space can be left between the stirrer and the cylindrical walls. The cylinder ξ_0 is enclosed between two rings provided with grooves ξ_1 and ξ_2, of which the upper is connected with the ring ξ_5 by means of glass tubes. Through the operation of the spring ξ_{31} and the arch ξ_{30}, this ring is pressed against the ring ζ_6 on to which the beaker B_{02} with a ground upper rim is fastened by means of cords. To this ring ξ_6 the hooks ξ_7 are also fastened, against which the upper rim of the beaker B_{01} is also pressed by means of cords. In this way a cylindrical space is reserved for the pumping motion of the stirrer.

In order to admit the measuring apparatus it was advisable to leave free the whole space offered by tube F_1, which is equal to that in the bath available for a measuring apparatus. To this end the threads χ_1, formed of very thin silk cords enclosed in steel wire are led through 3 openings E_{61} in the cover E of the bath and then over a pulley axis χ_2 with three grooves to a connecting piece χ_3, which is moved by a single thread passing over the pulleys χ_4 and χ_5. The cord must be moved from outside the case and the case must remain perfectly air-tight. This is obtained by passing the cord through an india rubber tube χ_{61}, which at χ_{60} fits hermetically on to the cover of the cryostat and in which the thread χ_{62} is also hermetically fixed. A thin steel wire is wound spirally round the india rubber tube. In this way the walls of the tube offer sufficient resistance to the atmospheric pressure to prevent them from collapsing when low pressure exists in the cryostat, while at the same time

they remain elastic enough to permit the movements of the cord. A regular up and down motion of the stirring apparatus is secured by the wheel χ_7.

§ 5. A constant temperature is attained by continually adjusting the pressure, at which the liquid in the bath evaporates, to the indications of a resistance thermometer p placed concentrically in the bath. A sensitive thermometer forms an inherent part of the cryostat under consideration when it is to be used for very constant temperatures and the dimensions allowing a resistance thermometer to be introduced, the latter has been chosen as the most trustworthy. Its inner diameter controls the greatest cross section of the measuring apparatus which can be immersed in the bath, and therefore, as in our case, it must correspond to that of the tube F_1. The construction of this thermometer has been described in detail by B. MEILINK (Comm. 77 Febr. '02) with a view to a comparison between it and the hydrogen thermometer referred to above. The leads pass through the openings R_{30}, R_{40} of the ebonite rings R_3 and R_4, and then through the stopper into the tube T_{11}. On the plates I and II they are indicated by the same letters as on the plate of Comm. 77.

When the bath has reached the required temperature the galvanometer in the WHEATSTONE's bridge, which serves to measure the resistance of p, is adjusted to zero by introducing suitable resistances. As soon as the deviations of the galvanometer make it necessary, a sign is given to the assistant, charged with the regulation of the pressure in the cryostat, who then raises or diminishes the pressure, whereby the temperature in the bath rises or falls. The great volume of the cryostat is here very useful in checking oscillations in pressure. The arrangements required for the regulation of pressure are shown in plate I, the separate pieces of apparatus to scale and the connections schematically. (Comp. Comm. 51 Sept. '99, pl. IV). The assistant uses the oil manometer X_9, which is connected to the cryostat by X_1 and X_2 (comp. pl. II Comm. 51) and the cock X_{90}, the cock X_{91} being open. If we shut the cock X_{91} the motion of the oil enables us to very accurately watch the variations of the pressure in the cryostat by means of the difference between the pressure in it and of the quantity of gas temporarily shut off in the reservoir X_{92}. If through some cause or other the variations of pressure increase considerably, or if we want to stop the regulation, or to proceed to another pressure, the oil is prevented from running over by our opening the cock X_{91}. The pressure in the cryostat is varied by more or less opening the fine cocks Y_{43} and Y_{42} of the regulation

tube Y_{41}. Two cases are to be distinguished here. With operations at ordinary pressure it will be sufficient to adjust the cryostat at a pressure a little higher than that of the atmosphere and to either connect the cock Y_{42} with a gasholder *Gaz.* or to disconnect them, as the occasion demands. As soon as the pressure passes a certain limit settled for the cryostat, the gas escapes from the cryostat through the large safety apparatus. For operations at reduced pressure, the cryostat, after the pressure has been sufficiently lowered by means of the exhaustpump of the circulation *Ech.* 1, is disconnected from the latter and connected by means of the cock Y_{43} to the exhaustpump *Ech.* 2., and is then reduced to a lower pressure. Obviously we can sometimes avail ourselves for this latter operation of the same exhaustpump as used with *Ech.* 1. The evaporation will proceed more gradually when a connection is made to a reservoir at reduced pressure *Vac.*, plate. If a reservoir of large volume is used we can even work without an exhaustpump, which may be valuable when it is necessary to avoid vibration for the measurements. Thus with the bath of nitrogen under diminished pressure the auxiliary compressor of Comm. 54 Jan. '00 plate VII was connected near *Ech.* 1 to the gaslead and the vessel of 5 m³ mentioned above (comp. Comm. 14 Dec. '94 § 10) served as vacuum reservoir, after being exhausted through Y_{36} and Y_{37} by means of a BURCKHARDT vacuumpump, connected to the gaslead at *Ech.* 2. This vacuumpump will be described later.

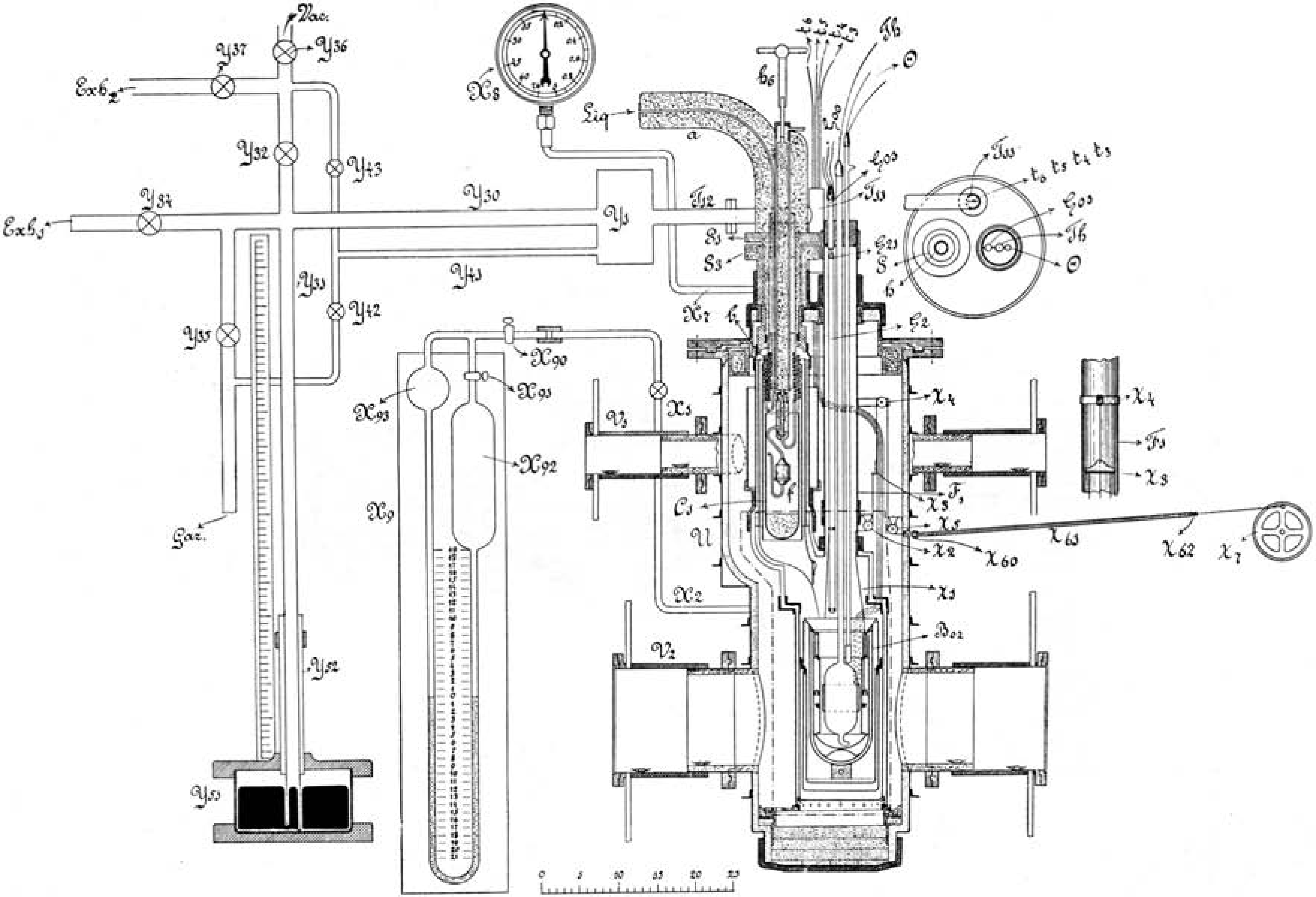
In a few words we shall indicate the method which we usually follow in order to get a wellfilled bath at diminished-pressure. First the double beaker B_{01}, B_{02}, or several beakers B_1, B_2, B_3 are filled at ordinary pressure, then we begin to slowly exhaust through Y_{34}; all other cocks being shut by means of the pump, generally used for the circulation *Ech.* 1; while boiling is prevented by rapidly moving the stirring apparatus described in § 4. When the required pressure is reached the cryostat is to be connected to the great reservoir *Vac.* at the same pressure. If this cannot be done we hardly ever succeed in admitting through the cock h_6 the yet required quantity of liquid slowly enough to keep the pressure in the cryostat free from undesirably large fluctuations or even to avoid with the help of Y_{36} momentarily returning of it to nearly its ordinary value. Therefore, if a change of temperature for some time is allowed, it is in that case better to shut Y_{34} before more liquid is added and to connect the cryostat through Y_{35} to the gasholder. As long as the beaker B_{02} is not full the gas leaving the cryostat is allowed to pass through Y_{35} into the gasholder. If the beaker B_{02} is full, which is shown by

the rise of the level in B_{01}, we once more begin to diminish the pressure (Y_{32} shut, Y_{34} open) which process generally takes some time. Then more liquid is admitted as before and if necessary this process is repeated several times. If the beaker is sufficiently filled at the desired reduced pressure we begin to regulate the pressure with the duly exhausted vacuum reservoir as described above.

Plate III shows a couple of graphical representations of the variations of the temperature of the bath. The ordinates show the deflections on the scale of the galvanometer in centimeters. The abscissae represent the time in minutes; fig. 1 relates to a measurement in methane at ordinary pressure; a deflection on the scale of 1 c.m. corresponds to about 0.009 deg. (the open space in the figure indicates a magnetic disturbance). Fig. 2 refers to oxygen at a diminished pressure; here a deflection on the scale of 1 c.m. corresponds to 0.005 deg. They were borrowed from the measurements of MEILINK mentioned above.

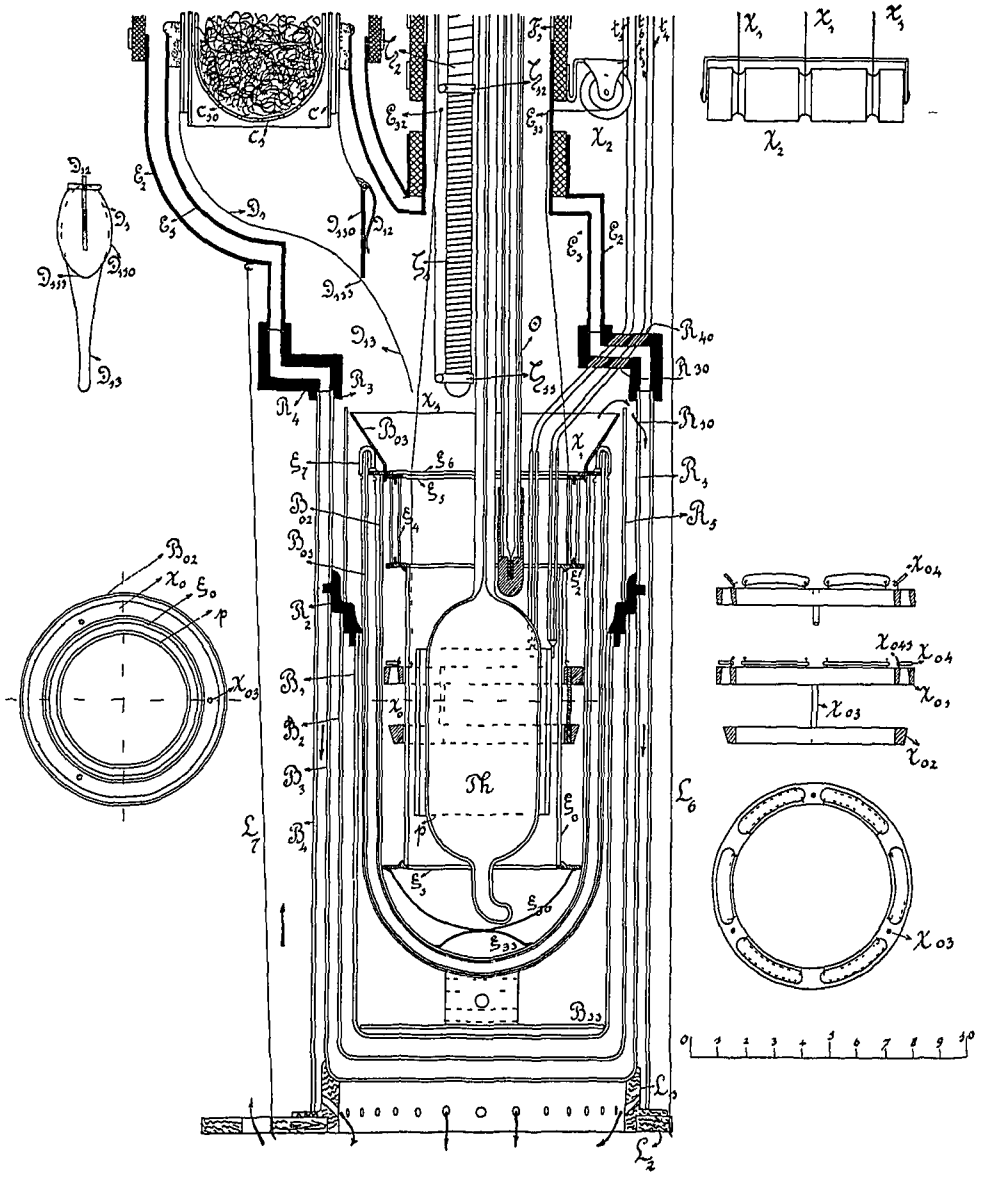
The temperature of the measurement is determined by the help of graphical representations, extending over the whole time of measurement, from which the portions reproduced on plate III have been taken. For this determination the readings of the galvanometer are noted down about twice every minute. By means of the planimeter we derive from the graphical representation obtained, the mean ordinate, which mean is considered as the temperature of the bath during the whole measurement.

(March 25, 1903).


Vac.
Liq
a
Gaz.
0 5 10 15 20 25

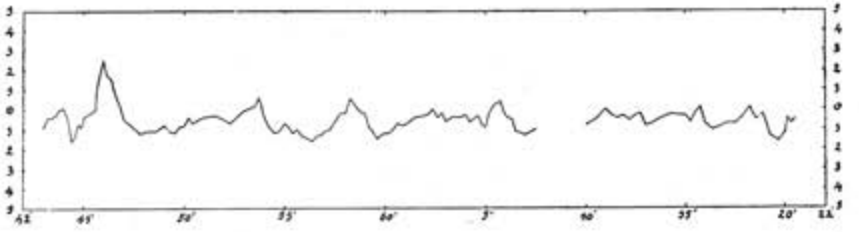
H. KAMERLINGH ONNES Methods and apparatus used in the Cryogenic Laboratory. III Baths of very uniform and constant low temperatures in the cryostat.

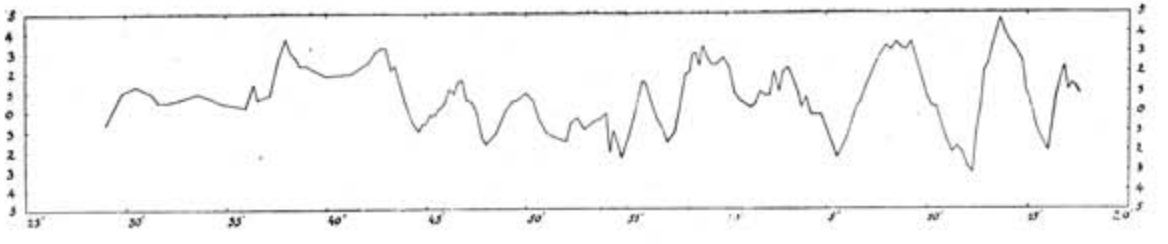
PLATE II



Proceedings Royal Acad Amsterdam Vol V

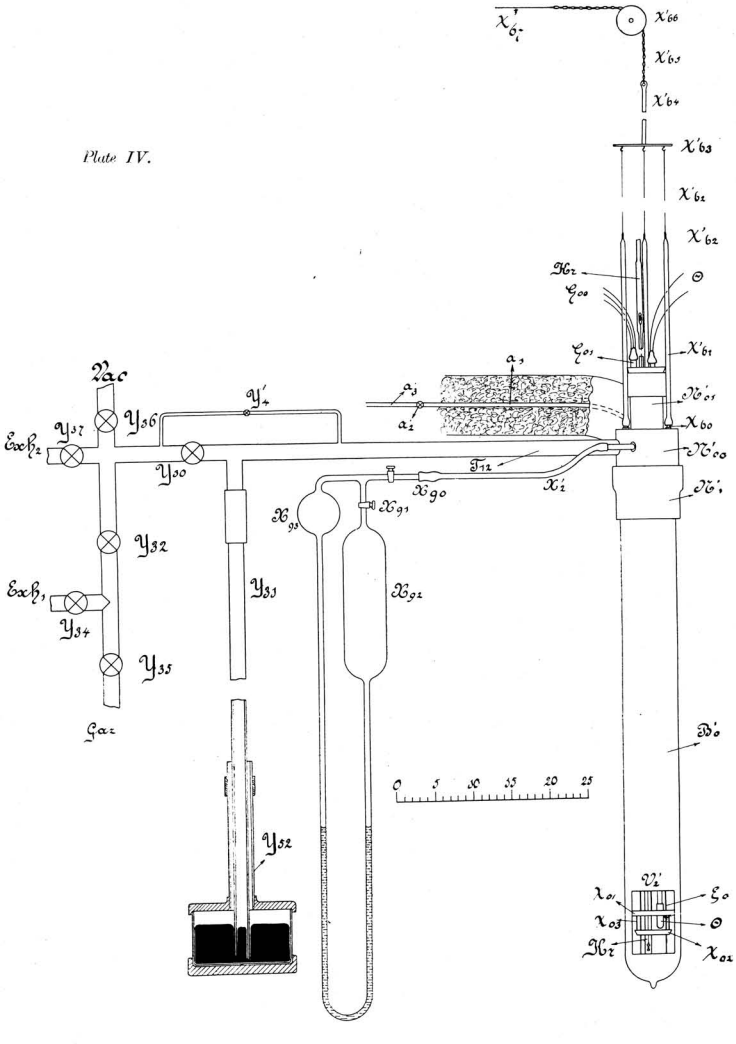
PLATE III.

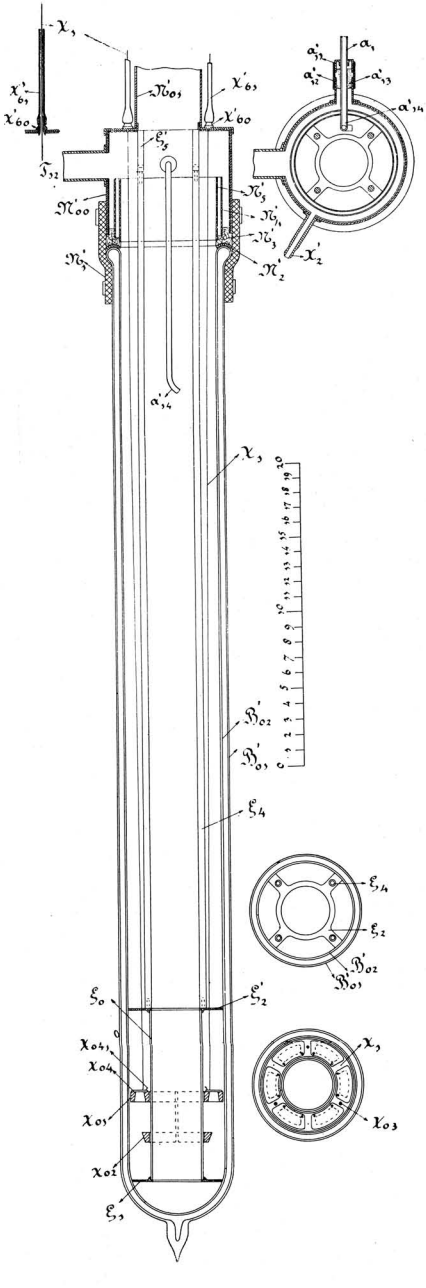




H. KAMERLINGH ONNES. **Methods and apparatus used in the Cryogenic Laboratory. III. Baths of very uniform and constant low temperature** (continued). **A Cryostat of modified form for apparatus of small dimensions.**

Plate IV.





H. KAMERLINGH ONNES. Methods and apparatus used in the Cryogenic Laboratory. IV. Permanent bath of liquid nitrogen.

Plate VI.

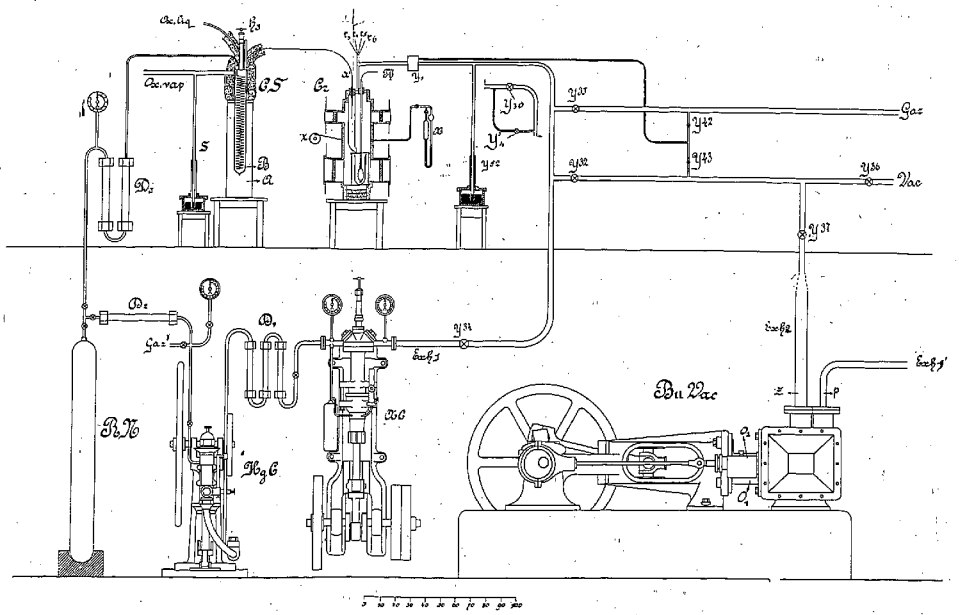
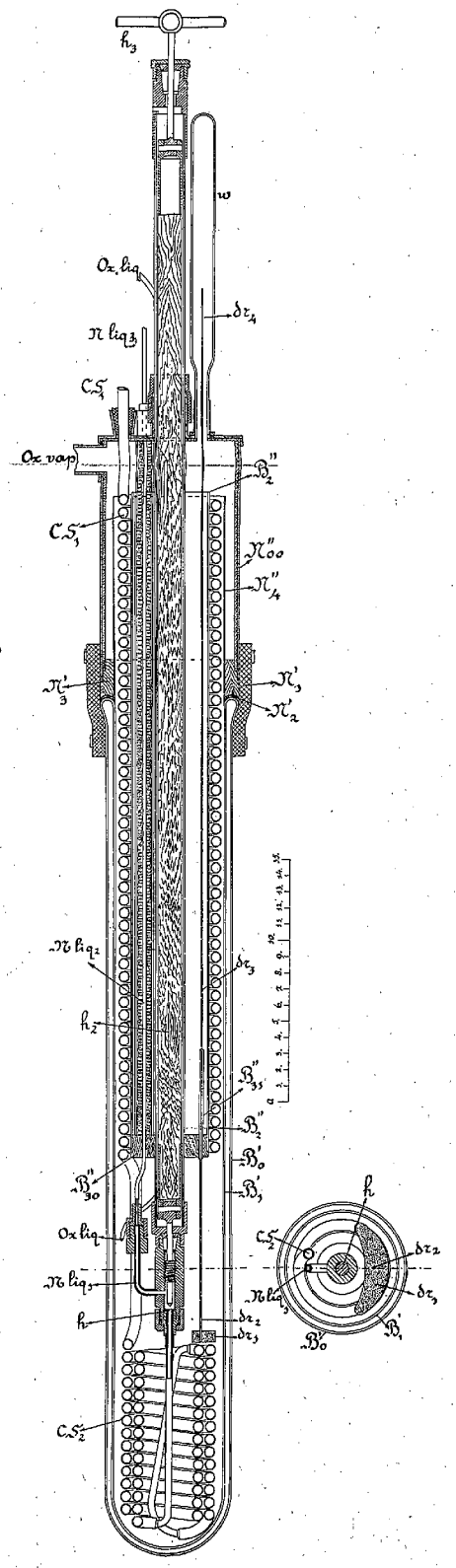


Plate VII.



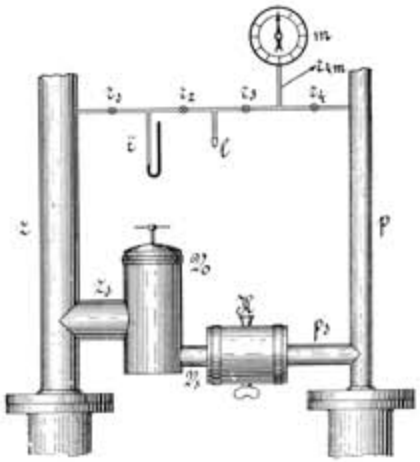


Fig. 4.

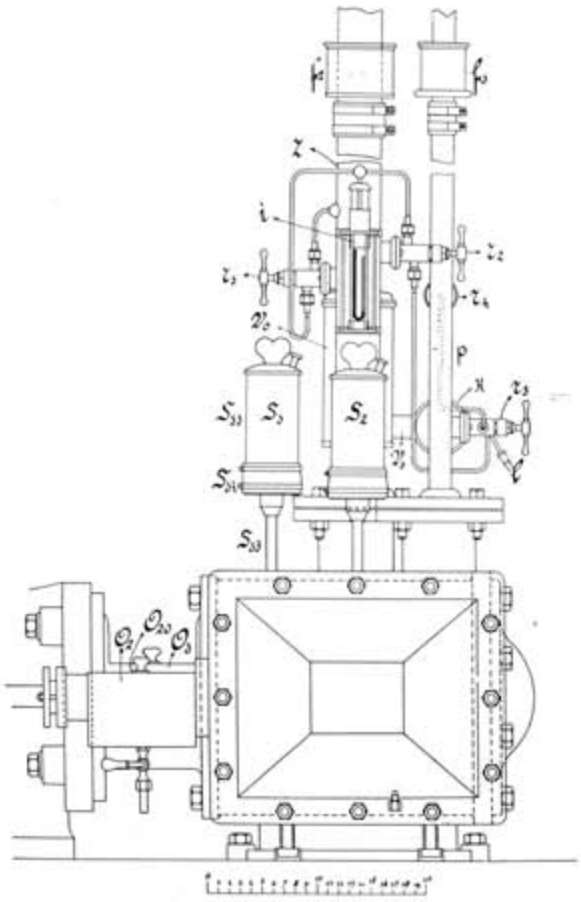


Fig. 1.

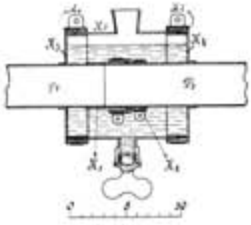


Fig. 5.

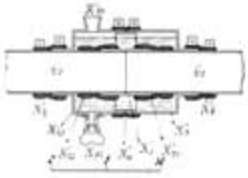


Fig. 8.

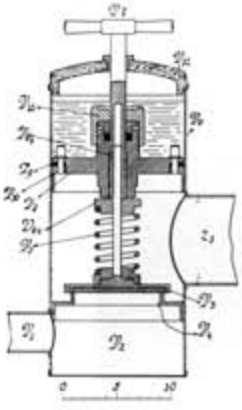


Fig. 6.

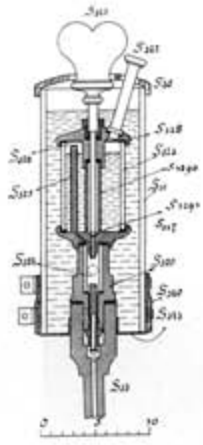


Fig. 7.

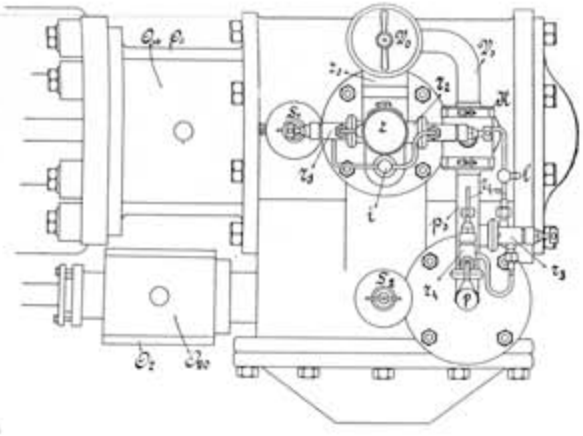


Fig. 2.

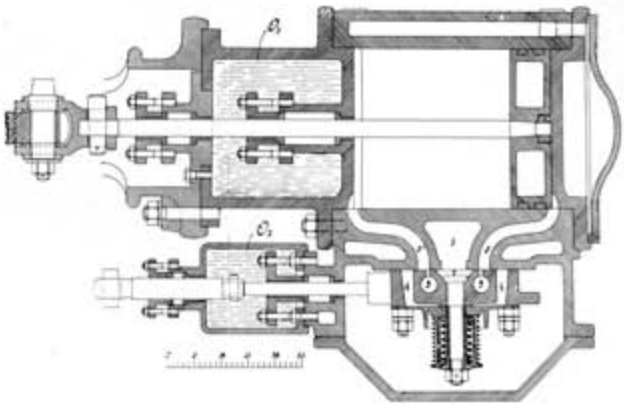


Fig. 3