Dr. H. KAMERLINGH ONNES *on the cryogenic laboratory at Leiden and on the production of very low temperatures.*

§ 1. For more than ten years I have bestowed all my available time upon an investigation of the manipulation of condensed gases in order to make physical experiments in liquid-baths of very low temperature possible. After much labour I have succeeded in my efforts and at present a section of the physical laboratory has been arranged for these operations only.

Only comparatively small means were at my disposal in this investigation. Of the whole of the subsidy, granted to the laboratory by Government, a small portion only could be employed for this purpose, especially of late. I always lacked sufficient assistance. The same instrument-maker who constructed the new apparatuses under my direction was also charged with the care of those already completed and helped me in handling them, when being used. So we were compelled every now and then to put aside the innovations already undertaken. For a short time only, at the beginning of my work, I was helped by a scientific assistant. Consequently the investigation could only progress with intervals of stagnation which sometimes did

much harm. It was only recently that I had a couple of instrument-maker's apprentices at my command, to whom I could entrust simple operations.

Even if more money and assistance had been at my disposal for this purpose, it would have been my aim to make experiments at low temperature possible with the simplest means. But in that case not so much time would have been lost by trying work with what was really insufficient.

In mentioning this, however, I must at the same time point out that in our country the State is almost the sole promoter of physical investigations and that in this case too, all the expenses were defrayed by the treasury.

So it is my duty to express my sincere thanks to the Government. Without its collaboration in extending and improving the physical laboratory at Leiden it would have been impossible for me to found a cryogenic laboratory there.

§ 2. I was induced to work with condensed gases by the study [1]) of VAN DER WAALS's law of corresponding states. It seemed highly desirable to me to scrutinize the isothermal lines of the permanent gases, especially of hydrogen (§ 9), at very low temperatures. And the idea of applying on their determination the new experimental method, introduced by PICTET's ingenious researches attracted me much. It was already in my inau-

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[1]) H. KAMERLINGH ONNES. General theory of the fluid state. Verh. Kon. Ak. v. Wet. Amsterdam, XXI, 1881 Beibl. z. Wiedem. Ann. 5, p. 718.

gural address in 1882 that, with a view to this, I called the pumps of CAILLETET and PICTET indispensable laboratory-instruments. As such they ought to be considered with still more right after the publication of WROBLEWSKI's and OLSZEWSKI's [1]) paper in 1883.

A first step in the investigation a sketch of which will be given here, was the purchase of a compressor devised by CAILLETET [2]) in 1883 for condensing any quantity of pure gas.

As CAILLETET's experiments on the decantation of liquid gas proved ethylene to be the most important substance for investigations on low temperatures, I began by repeating those experiments. As soon as ethylene was prepared and condensed in rather considerable quantities, I might proceed to a repetition of PICTET's experiments, in which I intended to employ ethylene in the second cycle.

The compression of the ethylene will be attended with great difficulties unless thorough care be taken in the preparation of the gas with regard to its purity. The method of preparing (passing alcohol-vapour through sulphuric acid) was continually improved by availing ourselves of our experience in subsequent preparations. Not until 1890 it was brought to such a degree of perfection that now each time when the gas is wanted we can be sure of a regular distillation of a gas, containing at least 99 pct of ethylene, in quantities exceeding 1,5 M^3. The distillation takes place in three apparatuses,connected

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[1]) WIEDEMANN's ann. XX. 1883.

[2]) Ann. de Chimie et de Physique 5e série, tome XXIX, 1883.

in parallel, having their purifying and drying-apparatus completely arranged for continuous use and provided with the necessary automatic safety- and regulating-contrivances. For the distillation itself glass flasks are used in which the temperature is carefully controlled and into which the gas, already prepared, returns of its own accord if the pressure in them diminishes.

In the first years of my investigation there was no question of ethylene being an article of commerce; afterwards I applied to the well-known manufacturer ORCHARD to have it prepared. The first order in 1892 was only executed after half a year and besides the gas contained no more than 97 pCt. of ethylene; a second order has not been executed to this day.

Consequently for the present it remains unavoidable to prepare this gas in the laboratory and as in the experiments a loss of ethylene may occur repeatedly, it is necessary to keep the apparatus ready for a regular preparation. This is very troublesome and moreover ethylene is very expensive.

Therefore in physical laboratories as they are in general, the apparatuses for the use of liquid ethylene as a cooling agent, must be so constructed that they require only a small total quantity of ethylene, that this is kept pure and can be completely re-collected. Hence there must always be circulation in perfectly closed and if possible exhausted apparatuses.

These conditions are fulfilled by my apparatuses and with respect to the required quantity I venture to say that in my arrangement for condensing oxygen, the minimum has been attained. While in DEWAR's invest-

igation there is question about preparing large quantities of ethylene (in an account of a lecture no less than a hundredweight is mentioned [1]) which undoubtedly is only possible if powerful means can be disposed of, in my experiments with oxygen the quantity employed does not, as a rule, exceed 1,5 K.G. The quantity of oxygen disposable in the liquid state, which with the aid of these 1.5 K.G. can be maintained is from $^1/_4$ to $^1/_2$ Liter. This may be considered amply sufficient for most experiments.

The very complicated experiments referred to at the end of this communication (§ 10), require a quantity of about 6 K.G. of ethylene. The preparation of this quantity no longer requires any considerable exertion now, though the apparatuses are destined for the quantity required in the ordinary experiments.

§ 3. At first for compressing ethylene the above-mentioned CAILLETET-compressor was exclusively used. I know of no investigation of later date, in which the use of the mercury-compressor, invented by CAILLETET, is mentioned. WROBLEWSKI who had the opportunity of working with CAILLETET's apparatuses at Paris, has had a compressor constructed by SCHULTZ for his subsequent experiments. Neither was I at first pleased with this mercury forcing-pump, but the idea on which CAILLETET constructed this compressor is so exceedingly beautiful that I willingly spent a few years in order to work out the idea in an improved form.

The most important modification is that the upward

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[1] The Electrician, June 17, 1892, p. 169.

working plunger has been taken away from the barrel, that the packing at the bottom of the barrel has been removed and that the cylinder in which the gas is compressed has been made to communicate by an U-shaped tube, filled with mercury, with an erect forcing-cylinder. In this cylinder a plunger works downward and consequently the packing could be placed above the mercury. The mercury is separated from the packing by glycerine which at the same time serves for lubrication.

Then for the inconvenient oscillating inlet-cock has been substituted an exhaust-valve of peculiar construction which freely admits the gas into the barrel of the pump even at a very small difference of pressure though in the subsequent compression it must bear a pressure of 100 atmospheres without leakage.

And finally an excess of mercury in the pressure-tube is maintained by a small capillary tube with presicely regulating cock which forms a communication between the mercury in the pressure-tube and the mercury which is under higher pressure at the bottom of the pumpreservoir into which the gas is forced.

By these improvements and a few others of less importance a compressor is obtained upon which we can fully rely. The compressor can be worked by the hand or by a small electromotor and of course by any gearing. It can stand for a long time unemployed and yet is immediately ready again for use. As the gas only comes in contact with mercury or with solids, the apparatus is eminently suited for condensing gases which are chemically pure or costly or which are disposable in small quantities only. The improved forcingpump is a laboratory-

instrument completely adapted for these purposes. If only the quantity of gas which can be compressed in a given time, but not the loss, purity etc., were considered then one would prefer a commercial machine of a little higher price, which, moved by a sufficient force, does in the same time the tenfold or hundredfold of the work. But such commercial machines do not answer to the laboratory-requirements mentioned.

A description of this compressor was already promised in 1891 in Dr. STOEL's academical thesis [1]). I hope to give it soon with the necessary detailed drawings.

The instrument has since proved to be fully capable of all the laboratory-work in which it is not our object to condense large quantities in a short time. So it was, for instance, lately used by Dr. SIERTSEMA [2]) for compressing electrolytically prepared oxygen in his investigation of the dispersion of magnetic rotation in this gas and by Dr. KUENEN [3]) for compressing ethane in order to purify this gas by fractional distillation at a low temperature. In this method for obtaining pure gases — as far as I know [4]) applied for the first time

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[1]) Diss. Leiden, 1891. Measurements on the influence of the temperature on the viscosity of fluids between the boiling point and the critical state. Physik Revue I, p. 513. Communic. No. 2.

[2]) Versl. d. Afd. Natk. Kon. Akad. Wet. Amsterdam, 26 Jan. 1895. Communic. No. 15.

[3]) Investigation on mixtures of aethane and nitrous oxyde to be shortly published.

[4]) After the publication of this paper I found that the method of purifying gases by liquefaction and boiling was used as early as 1889 by prof. OLSZEWSKI. Bulletin internat. de l'Acad. des

in the physical laboratory of Leiden by Dr. KUENEN in his academical thesis [1]) (1892) — the described pump is very useful.

By supplying gas under higher pressure from an auxiliary compressor, the capacity is proportionally increased; of this contrivance we also avail ourselves frequently.

§ 4. Let us now, after this digression, return to the state of the investigation in 1885 when it was resolved to purchase the long wished for conjugated pumps, necessary for the repetition of PICTET's experiments. With more right than from PICTET's experiment, the possibility of pouring out oxygen in the manner as CAILLETET had poured out ethylene, could be argued from the formerly-mentioned beautiful investigation of WROBLEWSKI and OLSZEWSKI. So from that moment the next aim of my investigation became: to make oxygen circulate according to PICTET's method and to use it for experiments in the same way as WROBLEWSKI and OLSZEWSKI, in imitation of CAILLETET, had taught us to use ethylene.

The possession of the conjugated pumps and of the CAILLETET compressor brought this aim within my reach. While I was already negotiating about the conjugated

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Sciences de Cracovie. Jan. 1882, p. XXVII (No abstract in the Beiblätter).

[1]) Diss. Leiden, 1892. Measurements concerning the surface of v. d. WAALS for mixtures of carbonic acid and methyl chloride. Versl. en Med. d. Afd. Natk. Kon. Akad. v. Wet. 29 April, 1892.Communic. No. 4.

pumps, WROBLEWSKI's investigation was published in the Wiener Sitzungsberichte of 1885. At that time WROBLEWSKI still worked with no more than a few cubic centimeters of liquid oxygen and also pointed out as the »Methode der Zukunft:" [1]) »Der wesentliche Schritt vorwärts welcher in Hinsicht der Erweiterung der Methode zu thun wäre, ist sie so abzuändern dass man im Stande wäre den Sauerstoff so zu giessen, wie man heutzutage das Aethylen giesst Die Sache wird aber meiner Ueberzeugung nach nur dann mit Erfolg durchzuführen sein, wenn man zu den PICTET'schen Methoden zurückkommen wird und durch den Kreislauf von mehreren verflüssigten Gasen eine Cascade von den Temperaturen herstellen wird, von denen die letzte Stufe der Strom des flüssigen Sauerstoffs bildet."

In 1886 the above-mentioned pumps one of which was destined for a circulation of sulphurous acid, the other for an ethylene-cycle, could be put into action.

However we were far removed from having obtained with the possession of the pumps a regular circulation of condensed and evaporating gases.

Those pumps were nothing but ordinary technical engines and not calculated to answer to the high requirements of a physical laboratory.

In a laboratory they must be ready for immediate use each time, when it is desired to make an experiment. Even after having been unemployed for a comparatively long time, they may not show the slightest

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[1]) Wiener Sitzungsberichte XCI p. 710.

leakage or other disturbances. They must be constructed with a view to their being handled and taken care of by people only accustomed to physical laboratory work. Besides, for various operations accessory apparatuses are wanted and had to be constructed.

It took much time to free all pieces from smaller or greater leaks and defects, to lay perfectly tight packings, to make suitable conduits, to make cocks which do not get fixed by the cold, for which purpose such with cork-packings proved the best, to devise gauge-tubes showing the level of the condensed gas and filtering-apparatuses for protecting the cocks. Much that is an article of trade now was not yet known then and had consequently to be made, which was very troublesome. And moreover there had to be acquired practice in all sorts of unusual work.

It is quite another thing to have such an instrument in regular use as an ice-machine or to make experiments with it at very low temperatures. In the latter circumstances their use is attended with much more danger than in the regular manufactory-business and in a college this danger has all the more to be avoided. So there had to be made special arrangements for this purpose. The whole first cycle forms one closed metallic body which is everywhere of amply sufficient strength to bear the maximum-pressure of the liquid gas contained in it at the temperature of the room. Wherever the walls are cooled to low temperatures they are made of copper.

Having all this ready I had as yet made little progress. It is true that as early as 1887 I prepared liquid ethy-

lene in an apparatus similar to that used by PICTET for condensing carbonic acid, viz. in a condenser cooled by evaporating sulphurous acid, but firstly sulphurous acid proved after all to give much trouble when used in a laboratory, and secondly the PICTET refrigerators which I had hoped would have been serviceable at least in the first circulation, proved to be even more impracticable than one might already have expected from the drawing.

Even in the first circulation it was necessary to put above the refrigerator a regenerator, in which the ethylene to be condensed, in passing through a long coil, was preliminarily cooled by the cold vapours given out by the refrigerator.

This regenerator served at the same time as a precaution against a sudden priming of the condensed gas from the refrigerator to the pump.

As for the sulphurous acid, for this poisonous gas methyl-chloride was substituted. Against using methylchloride in the first cycle there is the less objection as in the second cycle large quantities of ethylene are necessarily present.

The precautions dictated by the use of this gas which forms with air an explosive mixture, as: heating of the room by steam, illumination by electric glow-lamps, a powerful ventilator, etc., also take away any danger when using methylchloride.

By choosing methylchloride and ethylene as the first two evaporating liquids, another idea of CAILLETET to whose genius we are so much indebted in this kind of investigations, has been realized; an idea which

was suggested f. i. in his communication of 1885 [1]).

Along with what has been mentioned in §§ 2 and 3, the next years following on 1887 were devoted to the achievement of the ethylene cycle. Besides difficulties of the same kind as those already summed up for the first circulation, among which should be mentioned that the ethylene had to be freed from the fine cloud of lubricating-oil which it carries along from the pumps, here quite other difficulties of a more scientific character presented themselves.

The ethylene had to be poured down into a metal vessel, which was still considered next to impossible by WROBLEWSKI [2]) and in this vessel it had to boil in a vacuum without causing any danger even in the case of imaginable mistakes or unexpected occurrences. A tube in the form of a coil immersed in that liquid could then serve for continually receiving oxygen, condensing it and letting the liquid oxygen flow away [3].)

To effect this the evaporated ethylene had to be

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[1]) Comptes rendus, tome C. p. 1033.

[2]) Sitz. Ber. Wien XCI. p. 670.

[3]) After the publication of this paper I learned that after PICTET, prof. DEWAR has made as early as 1886 an apparatus wholly of metal, being an arrangement of copper coils in which liquid oxygen was made and decanted. Though the apparatus was constructed on a small scale and was not used for collecting liquid oxygen I wish to point out this anticipation affecting a part of my idea. The description of the apparatus being found in an article on meteorites (Proc. Roy. Instit. 1887) and no abstract having been published in the Beiblätter it escaped my notice.

pumped away, condensed and poured again in this vessel. Especially the construction of this ethylene boiling-flask occupied me a very long time. In June 1890 the drawing was finished by me in its final form but the apparatus was not ready before 1891.

The complete ethylene-cycle forms again one metallic body in which the ethylene is moved round by the pump and which, if the circulation is not used, is entirely filled with ethylene under atmospheric pressure with the exception of a welded kettle in which the rest of the ethylene is kept in store under a pressure of 15 atmospheres.

The boiling-flask into which the liquid ethylene is poured when the circulation is in function, forms one whole with a large kettle of a capacity of 600 Litres into which the whole quantity of liquid ethylene might, if necessary, distil without exceeding the pressure which the apparatuses can bear. The pump takes the ethylene from this large safety-reservoir and compresses it into the high-pressure-reservoir, just mentioned, or through a return-valve which prevents the ethylene from flowing back, into an oil-arrester which can bear the maximum-pressure which the ethylene can attain at the temperature of the room. From the oil-arrester it is conveyed in a coil through the methylchloride-regenerator to the methylchloride condenser. Even if the ethylene thrown back into the oil-arrester passed there into the spheroidal state, neither this oil-arrester nor the condensation-coil, connected to it, would burst. By a regulating-cock which may be adjusted with great precision from the outside without conduc-

tion of heat, the ethylene can flow into the boiling-flask after having passed through a filtering-apparatus and check-valves which like other details as drying-apparatuses etc. cannot be considered here more closely. Both the pump and the oil-arrester and condensation-coil can discharge themselves through safety-valves into the large safety-reservoir, so that no part of the circulation can become exposed to a higher pressure than for which it has been calculated. Even after the addition of the ethylene which was kept in store this remains the case. In this way the possibility of accidents by confining a large quantity of this dangerous gas in a limited space is completely prevented. The purity of the gas is under continual control while pressure- and exhaust-gauges indicate the momentary state of the system.

The whole apparatus may be left to itself for months and then with the necessary precautions be brought into action within an hour's time. Only if the circulation is going on for a long time without interruption, some ethylene is lost by the quivering motion at those joints which are under high pressure. The ethylene lost is again supplied from the store. By a cock the circulation can be brought into communication with large bags of double india-rubber with intermedial tinfoil into which the ethylene can flow under the ordinary pressure. To the ethylene-condenser and to the large safety-reservoir other (glass) boiling-apparatuses for ethylene can be occasionally connected which are employed in protecting-cases of gauze and plate-glass, but for the rest in much the same way as by WROBLEWSKI and OLSZEWSKI and which

f. i. served for Dr. E. DE VRIES's academical thesis [1]).

§ 5. The aforesaid ethylene-boiling-flask with condensation-coil is made of copper. The walls are very thin. They are strengthened by bars and rings so that they can bear exhaustion or pressure. Among other contrivances the condensation-coil is provided with a safety-cap so that an explosion in the boiling-flask may be considered as an impossibility. In the wider lower part of the boiling-flask, the cylindrical room of 22 cM. diameter in which the boiling takes place, the liquid ethylene surrounds about 10 spiral windings of the condensation-coil. These have a cooling-surface of 0.15 M^2 and a capacity of about 300 cM^3. Above the broad surface of the boiling ethylene we find a tapering conical room of 25 cM. height for bridling the priming of the ethylene. In this room numerous windings with a cooling-surface of 0.15 M^2 utilize particularly the cooling-effect of all splashed drops of liquid ethylene. Farther upward we meet with a regenerating-room in which the cold ethylene-vapours pass between windings of the coil of 0.2 M^2 cooling surface. The passage from the regenerating-room to the strong metal mouthpiece is formed by a brass tube the walls of which are very thin but strengthened by rings. This neck has been soldered to the other metallic parts but

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[1]) E. C. DE VRIES. Diss. Leiden 1893. Mesures relatives à l'influence de la température sur l'ascension capillaire de l'éther sulfurique depuis la température critique de ce liquide jusqu'au point d'ébullition de l'éthylène. Arch. Neerl. XXVIII. Communic. No. 6.

can supply only an insignificant quantity of heat by conduction. The main part of the boiling-flask, is not suspended to this weak neck but to a wooden support of light but strong structure firmly fastened to the strong metal mouthpiece to which the neck also has been soldered.

The mentioned mouthpiece is screwed to a beam of a frame and can in the usual way be joined to the large safety-reservoir by copper tubings. Near the mouthpiece we find the handle of the non-conductive regulating-cock for the supply of ethylene (to be adjusted by the ear) and a glass tube, in which a pointer indicates the level of the liquid surface in the flask.

In the construction we have tried to obtain the most favourable ratio between the condensing surface, the quantity of metal to be cooled, the required cooling-liquid, the supplied condensed liquid and the surface which conducts heat from the outside. Moreover we wished to avail ourselves completely of the cold ethylene-vapour.

Much care has been taken to prevent, as far as possible, condensation of water-vapour and supply of heat by convection, conduction and radiation. In this respect I succeeded sufficiently by enveloping the flask in successive layers of cells coated with felt and filled with wool and by well pasting each layer with cotton and paper and varnishing these.

Within a couple of hours it is possible to pass into the boiling-flask more than a litre of liquid ethylene and make this to boil under a pressure of two or three centimeters. In this case the regenerator works in such

a way that the escaping ethylene leaves the boiling-flask at nearly the ordinary temperature.

The solution of the problems, presenting themselves in this circulation, has been very carefully studied. I wished, namely, to obtain a type for working with few kilos of condensed gases and few horsepowers which enables us to use without danger liquid ethylene and liquid oxygen in the laboratory and secondly to arrange several cycles for a temperature-cascade. If we wish to give a still greater cooling-surface to the condensation-coil, the boiling-flask has a downward annular continuation. If more cycles are employed, some of their boiling flasks can be constructed concentrically. (See § 10).

§ 6. Instead of condensing the ethylene by means of the condenser immersed in methylchloride, I also made use of a coil which is put into a ring-shaped basin kept filled with solid carbonic acid.

When using ordinary commercial carbonic acid the continuous drawing off of large quantities from the bottles offers great difficulties.

Experiments with carefully dried carbonic acid proved me that the obstruction of the cock must nearly always be ascribed to the circumstance that the liquid gas contains water. At my request the Dutch Kaenolite and Carbonic-acid-Company at Rotterdam has undertaken the preparation of dry carbonic acid by distilling the ordinary commercial substance over quick-lime. I strongly recommend this carbonic acid for all laboratory-purposes; it is possible to empty within a few minutes a cylinder filled with ten kilograms of it.

If we wish to dispose of the methylchloride-circulation

for another purpose, and if therefore we replace it for cooling the ethylene condenser by solid carbonic acid in the manner just explained, we want considerable quantities of this latter substance every hour, even then if we can prepare the solid carbonic acid beforehand in sufficient quantity, not by letting the gas flow out of the reservoirs at the ordinary temperature but by cooling it in the methylchloride-cycle. Only in the case that a large carbonic-acid ice-machine is at hand or that a friendly manufactory of liquid carbonic acid would procure the solid acid at the cost-price, the cooling by solid carbonic acid could be permanently applied. Hence it will, as a rule, be necessary to cool the ethylene-condenser by a circulation, for which we chose a methylchloridecycle making it further so easy to work at all temperatures between —20° and —70°.

§ 7. But let us return to the ethylene-cycle. Not before 1892 the whole circulation was in so complete function that liquid oxygen could be drawn off from the interior coil of the boiling-flask into the glass apparatuses prepared in the mean time.

Already WROBLEWSKI had convinced himself of the possibility of transferring a few cubic centimeters of liquid oxygen from his condensation-tube into another apparatus. In 1890 OLSZEWSKI [1]) had made an important step farther in this direction by substituting for the glass tube which he had used with WROBLEWSKI for condensing oxygen a wider steel one. By means of a small tube in the bottom he poured down from it for

[1]) Bull. intern. de l'Acad. des Sciences de Cracovie, 1890, p. 176.

the first time liquid oxygen. In the subsequent years he used liquid oxygen baths for most important investigations [1]). But for all this the extension of the method such as I tried to find and as it was also wished for by WROBLEWSKI, was not yet arrived at.

Again, in 1891 PICTET [2]) had given a description of the magnificent manufactory for the use of artificial cold, established by this leader in the domain of the liquefaction of the permanent gases at Berlin after 14 years' efforts. „Depuis 1887," he writes, „j'étais hanté par le désir constant d'établir sur une plus large échelle un laboratoire à basses températures .... j'ai été forcé d'attendre et de travailler pour me donner la satisfaction, la grande jouissance de réaliser mon rève."

No less than 50 horse-powers is the power of the steam-engine at his command for his pumps and he believes that „pour opérer normalement et travailler experimentalement avec de l'air atmosphérique liquide il faut disposer d'une force d'au moins 30 à 40 chevaux vapeur, actionnant 6 a 7 compresseurs [3])

In my experiments 6 or 8 H.P. are required if we wish to reach low temperatures rapidly; in regular

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[1]) After the publication of this paper a detailed description of prof. OLSZEWSKI's work appeared Phil. Mag. Febr. 1895 from which we learn, that in the steel cylinder there was prepared 100 cM. liquid oxygen in 1890 and 200 cM. in 1891. The liquid oxygen baths obtained when this quantity was poured down from the condenser and which prof. OLSZEWSKI used in his beautiful researches did not attain 50 cM.

[2]) Verh. der physik. Gesellsch. Berlin, 24 April 1891.

[3]) C. R. t. CXIV p. 1245.

action the compressors do not require so much. I was highly astonished to see that PICTET founds his idea about the necessity of great motive power on a new insight into the transport of heat at low temperatures, while my boilingflask had been very sufficiently protected indeed at —130°.

Moreover it did not appear that PICTET had occupied himself with collecting large quantities of liquid oxygen in glass apparatuses suitable for experiments. In a subsequent communication [1]), PICTET even calls —50° and —165° „températures que nous pouvons considérer comme limite de la zone utile des recherches dans notre laboratoire."

On the 17th of June 1892 condensed oxygen was for the first time drawn from the condensation-spiral of my boiling-flask and collected in a glass vessel suitable for experiments at the ordinary atmospherical pressure.

The quantity collected was but very small indeed — only 20 cM^3 — when the apparatus broke; but that in this way a sufficient quantity could be obtained and kept and that the dimensions of the apparatuses were well chosen was sufficiently proved.

The reparation and improvement of the various contrivances, serving for this purpose in such a way that no oxygen could be lost, lasted till December 1893, when I succeeded for the first time in collecting a quantity of $^1/_4$ Litre of liquid oxygen in a glass without pressure and could show it for hours to several scientific friends. The methylchloride-pump being on that occasion

[1]) C. R. 10 Dec. '94.

destined for pumping away the oxygen at low pressure, the carbonic-acid-refrigerator of § 6 did service then.

In the mean time in 1892 DEWAR's memorable experiments had been published which first solved the problem in such a splendid manner and enabled him to make with his colleagues LIVEING and FLEMING a series of extremely difficult measurements in large quantities of liquid oxygen. Yet I have the satisfaction to have independently and in my own way struggled against the same scientific difficulties as this famous scientist and to have founded at Leiden too the means of working with liquid oxygen in a laboratory which for the rest is also well-arranged.

Since December 1893 I have been again engaged in making various slight improvements, so that we can now say that we can obtain with sufficient speed and without considerable expenses a liquid-bath of more than a quarter of a litre of oxygen under the ordinary or under reduced pressure and that this can be kept disposable for any time with a small quantity of circulating oxygen. I had the honour to show a glass of liquid oxygen fit for experiments under these conditions to the President of the Physical Section in May 1894.

Now the work is so far advanced that I shall be happy to show the same thing to every one interested.

§ 8. The manner in which I used liquid oxygen for experiments diverges at one important point from DEWAR's, not to speak of the arrangement of his cycles which I don't know. It will be remembered how DEWAR, in his lecture at the Royal Institution (on the 10th of June '92) the report of which the newspapers spread over the whole earth, passed the liquid oxygen

in vacuum glasses from hand to hand. With my method such a thing is not to be obtained directly. But the construction of the splendid apparatuses, devised by DEWAR, requires much care and after having been used they lose something of their excellence. So it may be of importance to show that also without vacuum glasses liquid oxygen can be used for experiments in rather considerable quantities.

The apparatuses used by me, which I shall for simplicity's sake call boiling-glasses, remind us in many respects of WROBLEWSKI's and OLSZEWSKI's apparatuses and most of all of those which WROBLEWSKI described in 1885 and in which he employed an artifice already used by DEWAR in 1884, viz. to protect the cooled liquid by its own vapour. In fact I had in my mind from the very beginning to unite the beautiful work of CAILLETET, WROBLEWSKI, OLSZEWSKI on one hand and that of PICTET on the other.

The resemblance is such that the glass into which the liquid oxygen is collected, has partly the same diameter and shape as WROBLESKI's glass. But upwards it is widened into an eccentric bowl in order to check the vortices occasioned by the issuing and the rapid evaporation of the jet and in order to render priming harmless. In the double copper lid of this bowl are two glass tubes. One is a continuation of the experimental tube, and has about the same width. It serves for plunging apparatuses into the experimental tube with which it forms PICTET's »puits frigorifique." In the second double tube beside it is the valve for drawing off oxygen.

This cock is moved from the outside by an insulating handle in a glass tube in much the same way as the regulating-cock of the ethylene boiling-flask. Together with this cock the well insulated tube which supplies the liquid oxygen is placed in the boiling-glass. The jet of filtered oxygen is directed against the interior wall of a glass tube of a somewhat smaller diameter than the interior side-tube in which it is freely suspended. At the upper part this jet-receiver is almost closed by coils of the oxygen-supply-tube; at the bottom it ends in a nozzle of very thin copper through which the oxygen flows into the bowl-shaped room. Between the jet-receiver and the interior side-tube the oxygen-vapour escapes and then circulates round the whole apparatus between the double side-tube and other double walls. The experimental tube hangs in a small thin glass vessel near the bottom of which the evaporated oxygen is delivered.

The whole boiling-glass is surrounded by a thin copper case which I shall call the boiling-case. The evaporated oxygen issuing into this case is drawn away at the top of the boiling-case which has been so constructed that it can also be evacuated. The oxygen is received into the compression-pump under normal or reduced pressure; finally, the whole quantity present can escape through a safety-valve into bags of double india-rubber with intermedial tinfoil. The boiling-case is composed of three annular parts, carefully coated at the inside with felt covered at the interior with rings of nickel-paper in order to diminish the radiation. Over the bottom and the lid india-rubber covers are stretched, giving passage to the various tubes. They form herme-

tical seals and serve at the same time as large safety-caps. For it is true, what DEWAR says as recently as Febr. '94: The prosecution of researches at temperatures approaching the zero of absolute temperature is attended with difficulties and dangers of no ordinary kind [1]).

In the case four windows have been made, placed two by two opposite each other, one serving for the illumination, while the other is used for the observation. The upper pair serves for the observation of the jet and for the corresponding regulation of the cock (taking into account the indications of the manometers). The lower couple is oblong and enables us to follow the experiments in the liquid oxygen bath. The spy-windows consist of successive compartments, through the foremost of which, if necessary, hot air, dried by phosphorus pentoxide, can be sent.

Simpler but in some respects of the same construction was the boiling glass with case in which Dr. DE VRIES, when preparing his academical thesis in my laboratory, measured the capillary ascent of ethyloxide at the temperature of boiling ethylene.

One can follow the phenomena in the boiling-glass described now for hours and so sharply that f. i. the height at which liquid oxygen rose in a capillary tube could be observed in this way.

As soon as the apparatus has been sufficiently cooled, the oxygen evaporates from the surface; the troublesome boiling in the liquid, also mentioned by OLSZEWSKI and which DEWAR especially wanted to prevent by

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[1]) Proc. Roy Instit.

using vacuumglasses, does not occur in this method.

§ 9. Apart from the just-mentioned capillary phenomena which are closely related to the determination of the temperature (See E. C. DE VRIES, thesis p. 43) [1]), I hope I shall shortly be able to give a communication concerning the isothermal lines of hydrogen at extremely low temperatures. To the investigation of these isothermals the last three years of WROBLEWSKI's life were devoted; he did not succeed however in obtaining reliable data of the isothermals at — 180° (in boiling oxygen) from which f. i. the critical temperature of hydrogen might be deduced. I thought it in many respects of high importance (see § 2) to take up this investigation. As early as '93 I made measurements to this end by means of a hydrogen-thermometer with a reservoir of 30 c.M.^{3} while the copper-German-silver thermo-electric couple used by WROBLEWSKI has also been studied again. The determination of the isothermals (or of VAN DER WAALS's ψ-surface) [2]) for mixtures of permanent-gases with hydrogen is so closely related to this investigation that it forms almost a part of it.

If to pour out hydrogen requires more powerful apparatuses than now already in possession of the cryogenic laboratory, for this quantitative investigation of isothermals which can be considered as preliminary to the working with liquid hydrogen, as well as for numerous other researches, that which was brought about has proved to be amply sufficient.

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[1]) Comp. footnote p. 16 of this Communication.

[2]) Comp. footnote p. 10 of this Communication.

§ 10. Among the problems relating to the liquefaction of hydrogen we must rank the use of liquid methane as an intermediate link between liquid ethylene and liquid oxygen. Methane as a cooling-agent was already mentioned by CAILLETET in a pli cacheté of 1881 [1]); in 1884 DEWAR drew the attention to this very important aid for liquefying the more permanent gases; finally also WROBLEWSKI and OLSZEWSKI occupied themselves with methane (1884). Yet it has not been used in any investigation. One of the main difficulties is that most methods of preparation yield the gas mixed with large quantities of hydrogen so that it is consequently difficult to condense. Now in this respect the aforesaid (§ 3) process of fractional distillation promises good results. When the opportunity presented itself, I had a few cubic meters of methane prepared. The ordinary method of preparation was followed but with special care as to constancy of temperature.

In order to be able to intercalate a new cycle and to compress gases, as in this case the impure methane, in rather considerable quantities, without using the other compressors, I requested from H.E. the Minister of the Navy the loan of a BROTHERHOOD compressor, as are in use for launching torpedos. With great kindness this excellent instrument was put at my disposal by H. E., on condition to return it at the first intimation. It is repeatedly used for supplying in a short time large quantities of air compressed to 100 atmospheres; besides I provided it with the necessary

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[1]) C. R. t. 99. p. 213.

contrivances for compressing costly or dangerous gases and so it could also serve in the experiments on pouring oxygen for speedily restoring into reservoirs large quantities of escaped gas. The weight of this admirable instrument is only 90 kilo. It brings about 10 M^3 of gas to a pressure of 100 atm. in one hour. Methane compressed with this compressor was condensed in the condensation coil of the ethylene boiling-flask and poured into the same boiling-glass which serves for the storing of oxygen. The boiling-glass and boiling-case having been constructed to supply the least quantity of heat possible to the liquid, a spiral-wire through which an electric current was conveyed, was immersed in the liquid in order to make it boil. This wire can also serve for determining the latent heat. The liquid having been in ebullition for some time can be drawn away by a small tube reaching to the bottom of the boiling-glass and having been evaporated can be again compressed with the compressor for pure gas (§ 3). The operation taking much time, it was only applied to a small quantity and a judgment of the practicability of the method cannot be formed yet.

For cooling very much oxygen with liquid methane, which for this purpose may boil under the ordinary atmospheric pressure, a similar boiling-flask is destined as was described for the ethylene. In order to contain the numerous coil windings the boiling-room has a downward annular continuation and is surrounded by a vapour-jacket in the same way as the oxygen glass. The liquefaction of the required methane necessitates a condenser with large cooling-surface, consequently

a larger ethylene boiling-flask, now containing 6 K. G. of ethylene which commands the necessity of a larger safety-reservoir (to which purpose a welded kettle of 5 M^3 serves) and a greater displacing-capacity in the methylchloride-cycle or otherwise the insertion of a concentric nitrous-oxyde-cycle.

With these apparatuses at which was worked occasionally for a couple of years already, the whole motive power of the steam-engine which otherwise serves principally for the electric plant of the physical laboratory can, if necessary, be gradually used for experiments which require much liquid oxygen. I think it possible to pour out also liquid hydrogen then.

Finally it is an advantage of the present arrangement of the cryogenic laboratory that by the addition of new vacuum-pumps and compressors i. e. refrigerating machinery, such as can be had in the trade, and of the correspondingly required motive power the cascade can be extended to lower temperatures. The now founded installation being of a more scientific character than these additional circulations will then be completely adapted for working at the very lowest temperatures in the new cascade and so the cryogenic laboratory of the future may be obtained.
