Physics. — *Methods and apparatus used in the cryogenic Laboratory. XXIII. A horizontal cryostat for the measurement of magnetic susceptibilities at low temperatures.* By E. C. WIERSMA and H. R. WOLTJER. (Communication N⁰. 201*c* of the Physical Laboratory at Leiden). (Communicated by Prof. W. J. DE HAAS).

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§ 1. *Introduction.* One of the methods generally used for the measurement of magnetic susceptibilities depends on the determination of the forces exerted on the substance by an inhomogeneous magnetic field. In previous experiments the arrangement was such that these forces were directed vertically. The substance under investigation was attached to a vertical rod. The weight of the system formed by the rod and the substance was balanced by floats immersed in mercury. The substance was placed above or below the centre of an electromagnet and the magnetic attraction or repulsion acting along the vertical is measured either by additional weights [1]) on the floating system or by the interaction of suitably arranged coils [2]). This system, however, would drift away under the influence of the slightest asymmetry. This difficulty was overcome by guiding the system in its up and down motion by means of two flat spiral springs. The unavoidable stiffness in vertical direction of these springs destined to prevent horizontal displacements limited the sensitivity. The greatest accuracy obtained by us was about 7 mg. As an increase in the sensitivity was needed, especially for work on the susceptibility of oxygen at high densities, an attempt was made to adapt for low temperature work the method used by WEISS and his collaborators [3]), in which the force is exerted in a horizontal direction.

Owing to the difficulty of constructing a cryostat for liquid cooling agents for the purpose mentioned, it was decided to make use of cold gases. By this method also the difficulty of the changing hydrostatic pressure of the liquid during the measurements is obviated. [4])

This method of cooling has the additional advantage that it can be used over a wide temperature range, down to nearly 20° K. if cold hydrogen gas is the cooling agent and moreover for those temperatures where no liquids are available. The range between 20° and 65° K.

[1]) E. OOSTERHUIS, Leiden Comm. N⁰. 139*b*.

[2]) H. KAMERLINGH ONNES and A. PERRIER, Leiden Comm. N⁰. 139*a*.

[3]) P. WEISS and G. FOËX, J. Phys. **5** (1911) pp. 1, 275, 744, 895; G. FOËX and R. FORRER, J. Phys. **7**, (1926), p. 180.

[4]) This difficulty has since been overcome in another manner; see W. J. DE HAAS, E. C. WIERSMA and W. CAPEL, Leiden, Comm. N⁰. 201*b*.

(boiling point of hydrogen and freezing point of nitrogen) is of particular importance, as it is in this range that certain anhydrous chlorides undergo the transition to the condition which exists at liquid hydrogen temperatures when the magnetic susceptibility is dependent on the field strength.[1])

Cryostats working on this principle satisfying a high demand for constancy of temperature have been in use for some time.[2])

In the present case it was necessary that the parts lying between the poles of the electromagnet should be of small dimensions. On the other hand, however, a temperature constant to 0.1 to 0.05° was sufficient.

An apparatus has now been built making use of the method of WEISS in conjunction with a vapour cryostat for maintaining the substance under investigation at a low temperature. As this apparatus has been found to work satisfactorily a short description of it is given.

§ 2. *General Considerations.* The small tube containing the substance under investigation was fastened to one end — the right hand end — of a glass tube — the carrier — hung horizontally from two pairs of wires, each pair in the form of a *V*. (fig. 1). The left hand end carried a coil. At either end of this coil, coaxial with but free from it, were placed two fixed coils. By regulating strength of the current flowing in the correct direction through these coils it was possible to balance the force exerted on the substance by the field of an electromagnet, and thus to determine the susceptibility.

By means of a sensitive arrangement of mirrors[3]) it was possible to determine when the carrier was in its null position. To protect this device, the carrier must not be subjected to shocks or large displacements. For this reason the coils in series with a set of rheostats W_2, W_3, W_4, were connected in parallel with the electromagnet, and this system again in series with a further rheostat W_1 (fig. 2). The resistance of W_2 was first so chosen that for weak magnetic fields there was approximate equilibrium. The current strength was then increased with the rheostat W_1 when the equilibrium was but slightly disturbed[4]) and could be restored at any moment with the resistances W_3 and W_4, and finally adjusted when the current through the electromagnet had reached the desired value.

The two pairs of wires supporting the carrier were used as leads for

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1) H. R. WOLTJER, Leiden, Comm. N⁰. 173*b*.
H. R. WOLTJER and E. C. WIERSMA, Leiden, Comm. N⁰. 201*a*.

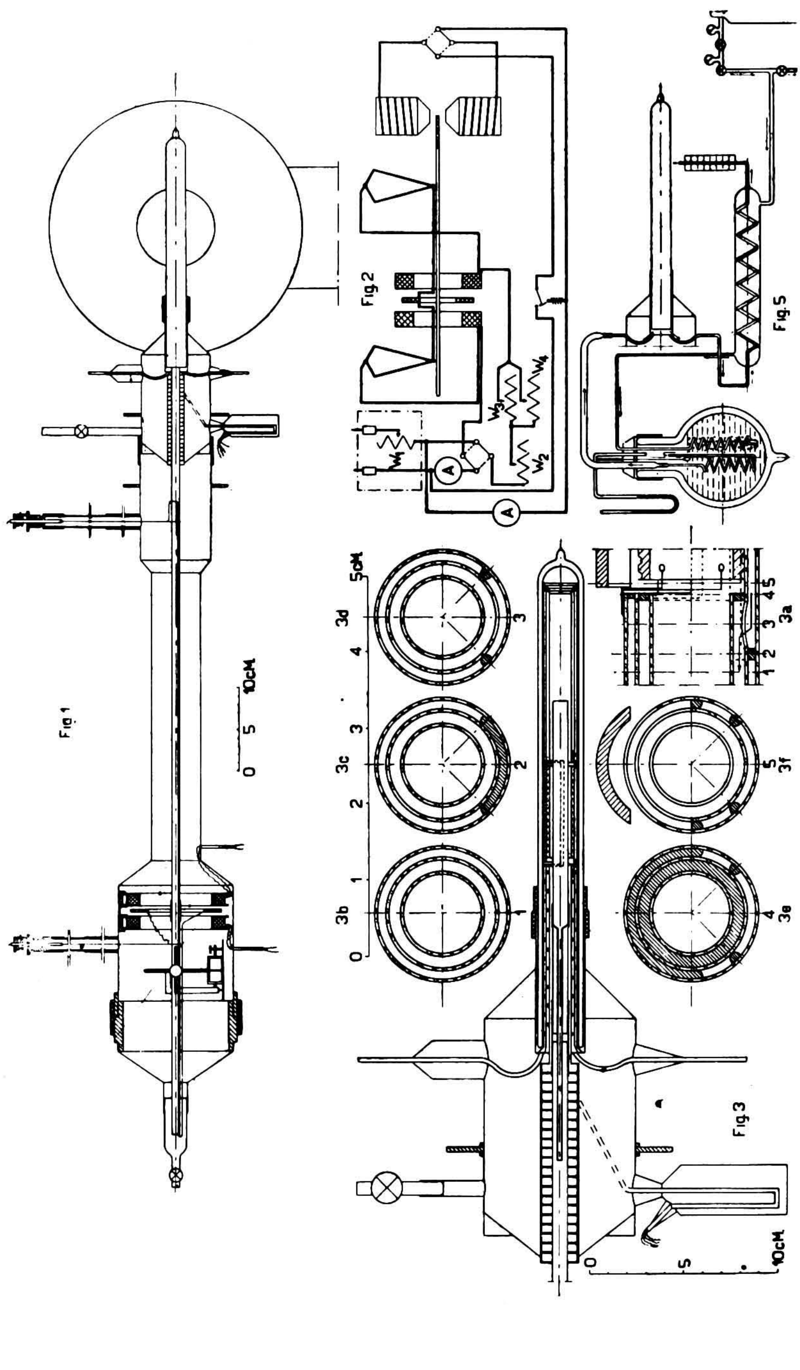
2) C. A. CROMMELIN, Leiden, Comm. Suppl. N⁰. 45, § 11.

3) This is a copy of the arrangement described by KOPP, see W. KOPP, Diss., Zürich 1919.

4) The force between the coils is proportional to the squared current strength and this holds sufficiently well also for the force exerted by the field on the (para-magnetic) substances since, for the current used by the magnet, the field is approximately proportional to the current strength. Naturally the accuracy could be increased by measuring in the region of the saturation field, but the magnet would then become much too hot.

the current through the movable coil. The length of the wires could be adjusted by screws running in packing tubes until the carrier hung freely in the cryostat.

The whole system of carrier, supporting wires and mirrors was contained in an air tight enclosure, to the right hand end of which the cryostat was attached.



§ 3. *The Cryostat.* (Fig. 3). The most important part of this consists of three concentric new silver tubes [1]) (Fig. 4) [2]). Cold vapour from liquified gas (air or hydrogen) is circulated through the sheaths between the tubes, called the inner and outer circulation spaces. The cold gas has been given a spiral motion in the left hand part of the system. For this reason a spiral of copper wire, about 1.5 mm thick was soldered unto the inner tube (internal diameter 10 mm, external diameter 11 mm). The whole was turned on a lathe to exactly 14 mm ext. diameter, and placed inside a new silver tube of 14 mm ext. diameter. In a similar manner a copper wire about 1 mm thick had been soldered unto this second tube and the whole turned down to 17 mms.

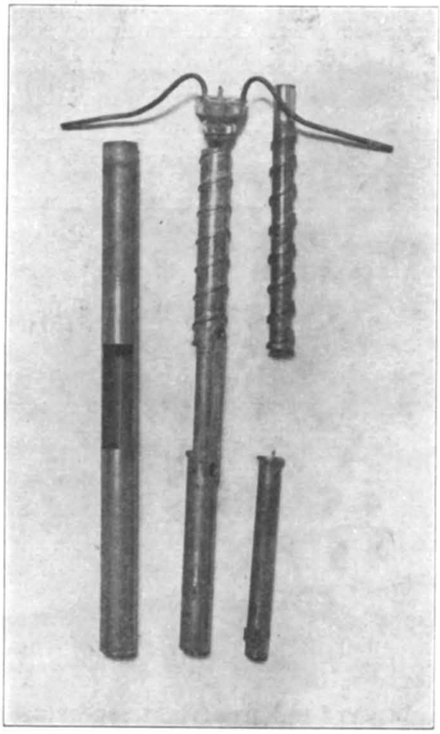


Fig. 4.

These tubes were placed inside a vacuum glass of 19 mm. internal and 24 mm external diameter. The length of the tubes was calculated for the symmetrical type of investigation tubes. [3]) In these the form of the glass is symmetrical and the plane of symmetry passes through the centre of the field of the pole pieces. The forces on the glass are eliminated but the length is increased. After the experimental tube had been suitably connected to the carrier, small covers were soldered to the right hand ends of the inner and outer new silver tubes. The circulation space was thus closed off from that containing the substance under investigation.

It was feared that there might be considerable flow of heat from the hot to the cold spaces, causing strong convection currents which would disturb the carrier. This difficulty was overcome in two ways. Firstly, the dimensions were made such that the space round the carrier was as small as did not interfere with its free movement. The internal diameter

[1]) It would have been preferable to have used a material such as copper for the right hand part to obtain a more even temperature distribution. This however would have offered great technical difficulties. New silver also had the advantage that accurately worked tubes of suitable sizes were obtainable An important point in the choice of the material is the rate of conduction of cold from the gas, but little is known on this subject.

[2]) The small projections, to be seen in the photograph above the middle tube and the under part of the right hand tube, are due to the strings used to fasten the tubes on the paper for photography.

[3]) H. KAMERLINGH ONNES and A. PERRIER, l.c.

of the inner tube was 10 mms, the external diameter of the experimental tube 8 mms. Secondly, the space between the cold and hot parts was occupied for a length of about 11 cms by a system of baffles in the form of fibre rings placed two or three mms apart. The expectation that convection currents would lose their kinetic energy through the formation of vortices between these baffles was later found to be justifiable (see below.)

It was of great importance that the temperature of the substance under investigation should be as much identical as possible with that indicated by the platinum resistance thermometer. The porcelain former, on which the resistance wire was wound, closely surrounded the investigation tube, having the same internal diameter as the inner new silver tube. It had an external diameter slightly less than the internal diameter of the middle tube.

Space for the thermometer was obtained by cutting away the new-silver tubes over the length of the former. A complete section was cut out from the inner tube so that this actually consisted of two parts. Half sections were cut from the other two tubes. A part of the outer circulation space, shut off by a closed loop of copperwire fixed to the middle tube, was reserved (see fig. 4) for the gas flowing from the right to the left hand part of the inner space.

The path of the cold gas through this part of the circulation space can be seen from the cross sections shown in figs. *3a*—*3f* taken in conjunction with fig. 4. The thermometer was covered with a copper lid in the form of a half cylindermantle to ensure uniformity of temperature. The leads for the resistance thermometer ran between the vacuum glass and the outer new silver tube and then through a separate enclosure cooled by liquid air. Heat conduction along the leads was thereby prevented.

§ 4. *Cooling.* In the present research, for which this cryostat was primarily designed and in which no temperatures below 154° K. were needed, cold air was used as cooling agent. Air from a cylinder (Fig. 5) was bubbled through liquid air in a large reservoir (5 litres capacity) in order to cause evaporation. The cold vapour was further cooled by liquid air and then led through a DEWAR tube into the cryostat. As the air leaving the cryostat was still cold, it was passed through the spiral of a glass condenser. In this way it served to cool the air from the cylinder, which was passed through the jacket of the condenser before reaching the 5 Liter reservoir. This somewhat primitive regeneration was found to work efficiently, the used air being brought up to room temperature. No moisture condensed on the flowmeter, unless at very high velocities.

The rough adjustment of the rate of flow was made with the reducing valve of the pressure cylinder and the fine adjustment with a valve in a parallel tube. In practice the rate of flow was not used for the adjustment as much as the pressure in the large reservoir. This was read on a long, open oil manometer, and was regulated according to the directions of the observer controlling the temperature. So long as there was no obstruction, the pressure corresponded unequivocally to the velocity but could be read more accurately. No trouble with obstructions was experienced if care was taken, when the apparatus was not in use, to keep the 5 Litre reservoir filled with liquid air and the exit of the circulation space closed. The latter was thus kept perfectly dry.

§ 5. *Details.* The temperature difference between the surroundings of the cryostat and the internal cold part was in the first place dependent on the rate of flow of the gas, and could be considered proportional to it so long as the temperature was not too low. The change in temperature of the liquid air was of secondary importance. Table I gives the relation between the absolute temperature T reduced on $T = 291°$ K

TABLE I.

v	T
100 L/hour	254° K.
200	216
300	179
400	150
500	139
600	134

as temperature of the surroundings and the rate of flow v to a first approximation. From this it was also possible to obtain a qualitative idea of the thermal insulation. Thus, to maintain a temperature difference of 60 degrees a rate of flow of 160 Litres (*NPT*) per hour was necessary. Assuming that the air entered the cryostat as cold as possible (say 83° K) left the condenser at room temperature (say 291° K), and warmed up at constant pressure ($c_p = 0{,}25$), then $160 \text{ L} = 160 \times 1{,}3 \times 10^3$ gms gained about

$$160 \times 1{,}3 \times 0{,}25 \times (291 - 83) = 1{,}08 \times 10^4 \text{ cals/hour}.$$

There was therefore a thermal leak of $\dfrac{1.08 \times 10^4}{60} = 180$ cal/hour for one degree temperature difference.

Actually the leak was smaller on account of the higher initial, and the lower final temperature of the cold air. About a 100 cals. are necessary to vaporise 1 gm of air and bring it to room temperature at constant pressure. With a leak similar to that discussed above, $180 \times 208/100 = 374$ gm or 467 cm^3 of air at 83° K must be vaporised and brought to room temperature per hour. The level of liquid air in a cryostat of cross section 47 cm^2 and having the same insulation as the vapour cryostat would therefore fall at ten cms/hour. Bearing in mind the open connection between the cold and the hot spaces this result is not un-satisfactory.

Table I could be used also as an aid in adjusting the temperature.

Fig. 6*a* and 6*b* show the variation of the temperature during an experiment on two occasions.

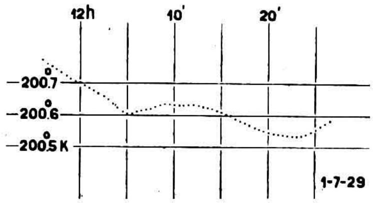


Fig. 6*a*.

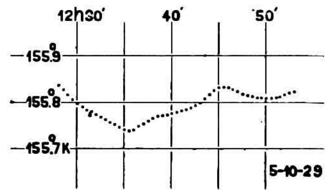


Fig. 6*b*.

With regard to the accuracy of the magnetic measurements the following remarks may be made. With the aid of the mirror system and a telescope the displacement was magnified rather less than 500 times, and since mms could be read with sufficient accuracy in the telescope, it was possible to reproduce the equilibrium position to about 0,002 mms by regulating the current through the coils. The corresponding force was naturally dependent to some extent on the weight of the investigation tube attached to the carrier, but was of the order of 0,2 mgm. As the forces are generally of the order of 1 gr. or more, the magnetic attraction could be compensated with an accuracy of at least 2 in 10.000. The experimental errors in the *measurement* of the force is a given field could, however, certainly amount to 6 ‰, since the ammeters in the circuits of the coils and the magnet were not certain to more than 1,5 ‰ and this error is squared in both readings. [1])

§ 6. *Testing of the Apparatus.* The extent to which the temperature of the substance under investigation was the same as that registered by the thermometer, was shown in the following ways. Firstly, when the temperature was allowed to pass through a minimum, the susceptibility passed through a maximum with a very short time lag. Secondly, control measurements were made with a tube containing gadolinum sulphate [2]) on different days, the temperatures being repeated and measurements made for rising and falling values.

The value of the current i (in milliamps) through the coils is proportional

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[1]) As the magnetic field is approximately proportional to the current. See note 4, p. 1047.

[2]) Used previously by H. KAMERLINGH ONNES and E. OOSTERHUIS, Leiden, Comm. N⁰. 129*b*, § 6.

to the susceptibility. In table II i^2 has been corrected for ammeter errors, for the empty carrier and for the anion, although these corrections do not amount to more than a few $^0/_{00}$.

TABLE II.

Date	10^{-2} T	10^{-2} i^2	10^{-4} $i^2 T$	i^2T (calc.—obs.) in $^0/_{00}$
27 June '29	287.9° K.	1457	4195	+ 2
„ „ „	233.2	1781	4153	— 8
29 „ „	288.7	1456	4203	+ 4
1 July „	227.3	1840	4182	— 1
	200.6	2075	4162	— 6
	170.7	2466	4209	+ 4
2 „ „	289.0	1452	4196	+ 2
	160.3	2604	4174	— 3
5 „ „	170.2	2474	4211	+ 6
6 „ „	289.4	1446	4183	+ 1

The last column gives the deviation from the mean of i^2T in $^0/_{00}$. As the uncertainty in the magnetic measurements can amount to some parts in thousand, there is no reason to doubt the accuracy of the temperature to 0.1° or 0.05° degree.

Calculation by least squares gave the most probable value of θ and C in the equation $i^2(T-\theta) = C$ as 0.4° and 4180 with probable errors of ± 1.5° and ± 30 respectively.

These determinations may also be used to calibrate the apparatus, The value of the force in mgms/milliamp^2 was found to be 6.75×10^{-3} from the following data: weight of cylinder of gadolinium sulphate, 2.897 gms., length 8.74 cms, field strengths at the ends of the cylinder (magnet current 15 amps.) 9179 and 842 gauss, Curie constant 2.016×10^{-2}.

§ 7. *Summary.* A cryo-magnetic apparatus has been built making use of the method of WEISS and his collaborators for the measurement of magnetic susceptibilities in conjunction with a vapour cryostat for maintaining the substance under investigation at a low temperature.

We wish to express our thanks to Prof. W. J. DE HAAS for the opportunity of carrying out this research, to Mr. L. OUWERKERK, technician 1st class, for the able construction of the cryostat and to Mr. C. W. COPPOOLSE, assistant of the Physical Laboratory, for his valuable help in the measurements.