The Cryogenic Laboratory of the University of Toronto.

By Prof. J. C. McLENNAN, F.R.S.

SHORTLY after the commencement of the War it became evident that if helium were available in sufficient quantities to replace hydrogen in naval and military airships, losses in life and equipment might be very greatly lessened.

It was known that there existed in America supplies of natural gas containing helium in varying amounts, and Sir Richard Threlfall, as a result of preliminary calculations that led him to believe that this helium could be extracted at a cost that would not be prohibitive, proposed that the Board of Invention and Research of the British Admiralty should undertake an investigation of the matter. As a result of this proposal the writer was asked by the Board to determine the helium content of the natural gases of Canada. This survey was carried out in the winter of 1915-16, and it was found that from 10,000,000 to 12,000,000 cubic feet of helium could be obtained per year from the natural gas of the Bow Island supply near Calgary, Alberta.

In the autumn of 1917 the Admiralty sanctioned proposals to proceed with an attempt to extract this helium, and in the summer of 1918, after exhaustive experiments had been made, a plant was designed for the purpose. This apparatus was constructed and installed at Calgary and was operated from September 1919 until April 1920. In the course of this operation of the plant, considerable supplies of helium of high purity were obtained and it was shown that the estimates of Sir Richard Threlfall as to the cost of production were amply verified.¹

During the winter of 1919-20 proposals were put forward by the writer to use the helium extracted at Calgary for scientific purposes. These met with approval, and financial grants were made for the liquefaction of helium by the Honorary Advisory Council for Scientific and Industrial Research of Canada, by the University of Toronto, and by the Carnegie Foundation for Research. Some apparatus was also loaned by the Admiralty and by the Air Ministry of Great Britain. With these grants special apparatus for liquefying air, hydrogen, and helium was constructed and its installation in the Physical Laboratory of the University of Toronto was completed towards the end of 1922. In the preliminary operation of the plant, special facilities in the way of power were provided by the Hydro-Electric Commission of Ontario and by the Hydro-Electric Commission of Toronto.

Helium was liquefied with the equipment for the first time on January 10 of this year, and the Cryogenic Division of the Physical Laboratory was formally opened on January 24, when demonstrations were given of the production of liquid air, liquid hydrogen, and liquid helium. Series of experiments were also shown illustrating the uses of these liquefied gases.

FIG. 1.—Hydrogen liquefier.

LIQUID AIR APPARATUS.

The apparatus constructed for the liquefaction of air consisted of a 40-kilowatt alternating current motor, a Norwalk compressor of the three-stage type, a water cooler, carbon dioxide purifying towers, and one of L'Air Liquide's machines having a capacity of producing 20 cubic metres of oxygen per hour. This machine was provided with valves which enabled one to isolate the rectification column from the oxygen heat exchanger, permitting the operation of the apparatus as a machine for liquefying air or as one for producing gaseous oxygen. The column was also

¹ Trans. of the Chem. Soc. vol. 117, p. 923, 1920.

provided with modifications for the extraction of the rare gases from the atmosphere. In operating this apparatus the air was compressed to 40 atmospheres, and in a series of tests it was found that about 300 kilograms of liquid air could be made per day. With such a supply of liquid air available, ample provision was made, it will be seen, for meeting the needs in regard to liquid air of all departments of the University.

LIQUID HYDROGEN APPARATUS.

The equipment for liquefying hydrogen included a four-stage belt-driven compressor built by the Burckhardt Engineering Works of Basle, Switzerland. Its cylinders were water-cooled, had a forced lubrication,

FIG. 2.—Hydrogen liquefier (as installed).

and were fitted with steel piston rings. The pistons were all in line and constituted one shaft. The gas was cooled after each compression by means of a number of heat exchangers immersed in a tank of running water. The compressor was constructed so as to prevent any loss of gas, and with this end in view, the piston rods were provided with special stuffing boxes in which the packing was sealed with oil contained in specially designed holders.

The space behind each piston as well as the safety valves was directly connected with a gasometer and through the latter to the intake of the compressor. The compressor had a capacity of 60 cubic metres of free gas per hour and required a motor of 30 kilowatts to operate it when delivering at 200 atmospheres pressure. Twenty litres of water per minute were disposed of by the heat exchangers.

The hydrogen liquefier is shown schematically in

Fig. 1 and as it was installed in the laboratory by Fig. 2. The regenerator coils indicated were similar to those used in the well-known Hampson apparatus for liquefying air. In operating the liquefier, hydrogen specially purified was compressed to 150-200 atmospheres and cooled to -205°C . by means of liquid air boiling under reduced pressure.

The compressed hydrogen passed successively through the coils L_1, L_1', L_2, L_3, L_4 , and L_5 . The coils L_1 and L_1' were arranged in parallel and the valve Z served to regulate the proportion of gas that went through each of them. This ensured the proper interchange of heat between the oncoming compressed gas and the outgoing low-pressure vapours. The coils L_1, L_2 , and L_5 were cooled by gaseous hydrogen returning to the gasometer from the expansion nozzle C_1 , and the coils L_1 and L_3 by the evaporated air drawn off by the vacuum pump. The coil L_4 was partly immersed in a bath of liquid air held in the flask M_1 .

The valve A served to admit more liquid air from the reserve supply whenever the indicator E_1 of the cork float E showed that it was required. To add to the efficiency of the liquefier, the expansion coil L_5 was provided with a close-fitting German silver envelope which when properly wrapped with flannel permitted a good junction to be effected between the inner wall of the silvered vacuum flask M_2 and the coil. This ensured that the expanded gas passed over the closely wound tubes of the coil and so brought about a good exchange of heat.

The liquid hydrogen as it formed passed through the opening in the bottom of the flask M_2 and was collected in the silvered flask M_3 . The float indicator D, D_1, D_2 , served to show the level of the liquid in this collecting flask. The weight D was connected with the thin German-silver float D_1 by means of a silk thread running over three pulleys D_2 provided with jewel mountings. The valves B and B_1 were used for drawing off the liquid. These were arranged so that they could be pre-cooled by cold gaseous hydrogen as it was returned to the gasometer. The stuffing boxes and screw thread of the valves B, B_1 , C and A were so arranged that they were not exposed to cooling and in this way the danger of a freeze-up was eliminated.

The insulation of the apparatus was specially studied. Vacuum flasks were used where possible, and wherever parts were cooled below the temperature of liquid air they were surrounded by an atmosphere of dry hydrogen or by a partial vacuum in order to avoid unnecessary condensation. All parts were constructed of German silver where it was an advantage to do so on account of its low thermal conductivity. The entire apparatus was packed in natural wool and enclosed in a thin brass case that was sealed except for the drying tubes H and H_1 . These tubes served to equalise the internal and external pressures on the case and at the same time prevented water vapour from entering and condensing inside. Fig. 1 shows plainly the arrangement for supporting the apparatus together with the scheme of the pipe connexions. Mercury traps J and J_1 served to protect the apparatus at all times from any sudden but moderate excess of pressure, while the large rubber safety valves G and G_1 served to accommodate any sudden but violent

increase of pressure such as might arise from the breaking of the flask M_3 .

In operating with the hydrogen liquefier it was found necessary to remove all gaseous impurities from the gas. The commercial hydrogen used was made electrolytically and was found to contain as much as 1.5 per cent. of oxygen and 0.1-0.3 per cent. of nitrogen. To purify this gas it was passed through a high pressure bomb filled with palladiumised asbestos. This bomb was heated electrically to about 400°C ., and at this temperature the palladium acted as a strong and robust catalyser. The water produced by the union of hydrogen with the oxygen present was taken up with caustic potash. The hydrogen obtained after this preliminary purification was again purified by passing it through a specially constructed apparatus provided with coils cooled with liquid hydrogen, but to make the liquid hydrogen for carrying out this purification it was necessary to operate the hydrogen liquefier with the hydrogen subjected to the preliminary purification only. A few litres only could be made in a run before stoppage occurred, and this was used to effect the final purification of a certain quantity of the gas.

By repeated operations of this character a supply of about 100 cubic metres of highly purified hydrogen was gradually accumulated, and with it long runs of the liquefier were made without any stoppage occurring. To conserve this original supply of pure hydrogen care had to be taken during a run to store up all gas from the vaporised hydrogen and to use residual supplies of liquid hydrogen to purify additional quantities of the gas so as to make up losses.

In liquefying hydrogen as well as helium, it was necessary in order to avoid losses so far as possible to operate in a closed cycle that included a gasometer, the compressor and the liquefier. In a number of actual runs with the apparatus described above, no difficulty was experienced in making from 10 to 15 litres of liquid hydrogen an hour, and in one particular run as much as 50 litres of liquid hydrogen was accumulated.

LIQUID HELIUM APPARATUS.

The helium used in the experiments was obtained from the natural gas of the Bow Island district near Calgary, Alberta, in the year 1919-20, and had been kept since then safely stored in steel cylinders at about 150 atmospheres pressure. An analysis by means of absorption with cocoanut charcoal showed the gas in different cylinders to be about 90-95 per cent. helium. The chief impurity was nitrogen, with a varying percentage of methane and other gases. Tests made by

chemical absorption and explosion methods gave no indication of hydrogen being present.

The preliminary purification of the helium was effected by cooling it at a pressure of 150 atmospheres to -205°C . by means of liquid air boiling under reduced pressure. Under these conditions a large percentage of the impurity was condensed and drawn off. This partially purified helium was passed at high pressure first through a bomb filled with copper oxide and palladiumised asbestos maintained at a temperature of 400°C ., and then through heavy copper tubes filled

FIG. 3.—Helium liquefier.

with cocoanut charcoal and immersed in liquid air. This cycle of purification proved to be satisfactory, for during the liquefaction process there was no evidence at any time of any blocking of the expansion valve of the liquefier or of the very small capillary tubes that made up the expansion coil.

In the design and construction of the helium liquefier, special attention was given to problems connected with the heat capacity and heat insulation of the various parts of the apparatus. The liquefier is shown diagrammatically in Fig. 3 and the manner in which it was installed in the laboratory is shown in Fig. 4.

In the operation of the liquefier the manner in which the helium entered the apparatus is shown in the

diagram. It passed successively through the coils D_1 , D_2 and D_3 , D_4 arranged in parallel. It then entered

Fig. 4.—Helium liquefier (as installed).

the coils P_1 and P_2 also in parallel, and afterwards passed successively through the coils P_3 , P_4 and P_5 . The coils D_2 , D_4 , P_1 and P_3 were cooled by the cold hydrogen vapour as it was drawn off by the hydrogen vacuum pump, and the coils D_1 , D_3 , P_2 and P_3 by the expanded helium that issued from the region about the expansion valve on the way to the gasometer. The coil P_4 served for the final pre-cooling of the compressed helium and was immersed in liquid hydrogen boiling under a pressure of 6 cm. of mercury. A trap T was provided, by means of which the gas was freed from the last traces of oil or water vapour from the compressor. The tubes B_1 were made of copper and were filled with coconut charcoal. They were cooled with liquid air during the liquefaction process with a view of absorbing any gaseous contamination introduced during the operation of the cycle. The level of the liquid hydrogen in the refrigerator surrounding the coil P_4 was determined by means of copper-constantan thermo-couples, and alternatively by helium

gas thermometers with reservoirs at M and M_2 , that were connected with a mercury manometer by fine steel tubing G_2 .

The liquid hydrogen from large vacuum-surrounded metal containers was first transferred to the unsilvered flask F_1 , that was protected by an outer silvered flask F_2 containing liquid air. This flask F_2 was provided with two unsilvered vertical observation strips, one on either side, so that the level of the liquid hydrogen in F_1 could be seen directly. The valve C_1 controlled the intake of the liquid hydrogen from F_1 to the refrigerator, and the valve C_2 with its corresponding spindle controlled the expansion nozzle at the bottom of the coil P_5 . The efficiency of the regeneration properties of the expansion coil P_5 was assured by fitting closely over it a very thin german-silver envelope soldered at X to the bottom of the german-silver liquid hydrogen container. With this arrangement the expanded helium was forced to go through the interstices of the expansion coil in order to enter the holes H in the tube surrounding the expansion valve spindle.

The temperature of the region beneath the expansion nozzle was determined with a helium gas thermometer provided with a german-silver reservoir at M_3 and a connecting steel capillary tube G_1 . The protecting vacuum flask F_3 was provided with a specially designed siphon tube P . This tube was double-walled and was protected by silvering and by an intervening vacuum in the same manner as a Dewar flask. The flask F_4 could be made either totally silvered or partially silvered with a plain portion at the bottom. In the latter case it was protected by a plain vacuum flask containing liquid hydrogen, and this in turn by a plain vacuum flask containing liquid air.

FIG. 5.—Metal container for liquid air.

These figures illustrate the types of metallic vacuum Dewar Flasks found useful in handling large quantities of liquid air and liquid hydrogen. They were made of polished spun copper. In assembling them, extreme precautions, it was found, had to be taken to remove not only the air but also all water vapour from the space between the spherical surfaces. A container of 25 litres capacity when well constructed did not lose so much as a kilogram of liquid air per day.

FIG. 6.—Metal container for liquid hydrogen.

In constructing the hydrogen and helium liquefiers great care was taken to see that all the complicated

tubing was free from holes and much time was consumed in the work of eliminating leaks. The whole apparatus was, however, completed towards the end of 1922 and, as stated, was used early in January of this year for the production of liquid helium. The helium was compressed with an enclosed Whitehead torpedo compressor, and the liquefier was found to give the best results when run at a pressure of only 40 atmospheres.

Before attempting to make liquid helium all parts of the liquefier were cooled as low as possible with liquid air, the piping being cooled by circulating through it helium that had been previously cooled to liquid air temperature. When this precaution was taken, it was found that liquid helium could readily be made with a moderate amount of liquid hydrogen

supplied to the refrigerator surrounding the coil P_4 . In our experiments less than 10 litres of liquid hydrogen sufficed to produce more than a litre of liquid helium.

I wish to take this opportunity of acknowledging my indebtedness to Prof. Kamerlingh Onnes of Leyden, the pioneer and outstanding authority in research at liquid helium temperatures. He not only assisted me very materially through correspondence and conversation, but also furnished me with drawings of the installation at Leyden.

It is hoped that with the cryogenic equipment now available at the University of Toronto, a series of low-temperature researches will be organised shortly for workers who for any reason may not find it convenient to go to Leyden to carry out investigations.

Rickets in Vienna.

A NUMBER of summary publications have made readily available the rapid advance in our knowledge of rickets in the last few years since Mellanby in 1918 brought forward serious evidence implicating a deficiency of fat-soluble vitamin A, and Hulschnisky in 1919 showed that the bone-lesions in children could be cured by ultra-violet light, and McCollum and his co-workers in 1921 demonstrated that the disease could be conveniently produced in rats by defective diets. Last year the Medical Research Council published the survey by Prof. Korenchevsky¹ of the experimental aspects and Dr. J. L. Dick² brought out a useful book on the human disease and its history. More recently an admirable survey of the whole question by Prof. E. A. Park³ has appeared, and there has now been added a full account of the results of the expedition under Dr. Harriette Chick,⁴ sent in 1919 to Vienna by the Lister Institute and the Medical Research Council, to study deficiency diseases under the conditions of almost experimental accuracy and precision afforded by the generous hospitality of Prof. v. Pirquet's Kinderklinik.

The report shows, beyond any reasonable doubt, that the incidence of rickets may be determined by diet, and that vitamin A plays an important part; that it may be prevented and cured by cod-liver oil; that it may be cured by sunshine or the rays from a mercury-vapour lamp, and that a diet which in summer is adequate for young infants may, in winter gloom, permit its development. From the practical point of view, the facts provide most of what the sanitarian needs: a proper supply of cod-liver oil, or its equivalent in vitamin A, and of sunshine, or its equivalent in ultra-violet light, will prevent rickets, and a deficiency of one may be made good by a larger supply of the other. What is at present unknown is how much vitamin A in the more customary forms of milk and green vegetables is wanted to give the same result as teaspoonfuls of the far more potent cod-liver oil. But there is no

longer any excuse for there being two schools of thought disputing for a hygienic and a dietetic ætiology respectively: as usually happens in such controversies, it turns out that both parties are right.

In the larger matter of the circumstances which condition the proper and regular growth of bone the results are of great interest. Granting an adequate supply of the necessary materials—and of these calcium and phosphorus are the most obvious, and their importance has already been examined by direct experiment—vitamin A is necessary: with enough of this, rats grow satisfactorily in the dark (Goldblatt and Soames, *Biochemical Journal*, vol. xvii., 1923, p. 294). Ultra-violet light of about 300 μ has much the same effect, and it was at first supposed, rather naturally, that it operated by causing a photo-synthesis of vitamin A. But rats on a diet grossly deficient in vitamin will grow normally under the influence of ultra-violet light only for a time: in the end, if no vitamin A is provided in the food, growth ceases, and the animals go downhill. Evidently light enables the animal to make the most economical use of such store of vitamin as it may have in its body or of any small amounts it may receive in its food: light can only partly replace vitamin, and if there is abundance of vitamin, light has no favourable influence on growth. In the same way vitamin makes a short supply of calcium or phosphorus go further, so that, while any of the three may be a limiting factor, up to a certain point of deficiency it is the sum (or product) of calcium phosphate and vitamin which is the effective determinant. Light *per se* is not a limiting factor, but may become the determinant under conditions of defect in the others.

Obvious as is the effect of ultra-violet light on the naked human skin, it is a little difficult to believe that it can act directly on the general body surface of hairy animals such as rats: "man is naked," as Richard Owen remarks, "and is the only terrestrial mammal in that predicament." It is, therefore, satisfactory to find it shown that air irradiated by the mercury vapour lamp is effective in promoting growth in rats, as Kestner showed it was in hastening the regeneration of blood lost by hæmorrhage: such air, in the absence of ultra-violet light itself, will also cure rickets in children. It does not seem to be known as yet whether radiation of the body surface with exclusion of radiated air

¹ Medical Research Council Special Report Series, No. 71. The Aetiology and Pathology of Rickets from an experimental point of view. Pp. 172 + 18 plates. (London: H.M. Stationery Office, 1922.) 4s. net.

² London: W. Heinemann.

³ *Physiological Review*, vol. iii., 1923, p. 106.

⁴ Medical Research Council Special Report Series, No. 77. Studies of Rickets in Vienna 1919-22 (Report to the Accessory Food Factors Committee appointed jointly by the Medical Research Council and the Lister Institute). Pp. 203 + 14 plates. (London: H.M. Stationery Office, 1923.) 7s. 6d. net.