SCIENTIFIC APPARATUS AND LABORATORY METHODS

A HIGH TEMPERATURE ELECTRIC FURNACE AND A MICRO-ADAPTATION

HOME-MADE electric furnaces are so much in vogue in American university research laboratories that a style of high temperature electric furnace which we have used with considerable satisfaction may be of interest to others.

For temperatures up to 900° or 1,000° C. a very inexpensive and practical electric furnace can be made by winding nichrome wire upon a grooved alundum core and suitably insulating it with two or three inches of magnesia or asbestos. For control a Thordarson A.C. regulator in series with the furnace has been found very satisfactory. It is desirable to have an ammeter also in the hook-up in series. Using number 17 (B&S gauge) nichrome wire a furnace core three inches in diameter can be held at 900° C. on about 10 or 12 amperes with three inches of asbestos packing. Or if wound in parallel with two strands of number 17 (B&S gauge) nichrome wire about 16 or 17 amperes will give the same temperature more rapidly. An ammeter, the scale of which reads to 10 amperes, is quite adequate for this type of work, making possible a highly satisfactory control for the lower temperatures. Such a meter may be shunted to read higher amperages.

For temperatures above 950° C. another heating element should be used. Platinum or one of the platinum metals is popularly selected. Since platinum has been shown to lose weight at the higher temperatures, around 1,500° C., we have resorted to the following expedient to cut down overload on the platinum heating element. An alundum crucible is wound with platinum wire and the whole is imbedded in a block of alundum cement. This is then introduced into the nichrome wound furnace. The mix is placed in a platinum crucible, which in turn is placed in the alundum crucible and covered with an alundum disc. But even with these precautions, it is necessary to proceed carefully to avoid the risk of burning out the platinum element at the higher temperatures.

Unless a rather large melt is wanted, a simple carbon resistance furnace is much preferable, as it is faster and requires less careful operation. Its main limitation is that only a small melt can be made, unless a very high amperage line and control unit are at hand. The furnace described below uses a crucible the size of one's little finger and of any convenient length, made out of an ordinary arc lamp carbon. It has been used on a maximum of 60 amperes A.C. 110 V. and is controlled by two 3 K.V.A., A.C. regulators. For this amperage the temperatures which we have been able to reach have been limited to 2,300° C. measured with a Leeds and Northrup optical pyrometer calibrated by the Bureau of Standards. Theoretically with higher amperage much higher temperatures should be possible, and with higher amperage larger crucibles could be used.

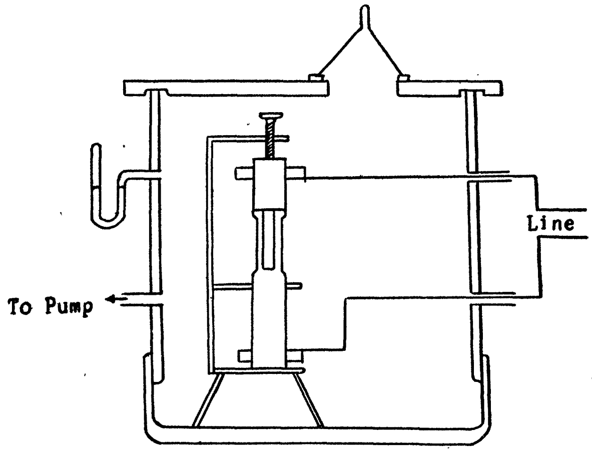

FIG. 1

The furnace itself (Fig. 1) operates under vacuum. It consists of a metal jacket cut from a piece of 8-inch steam pipe, threaded at one end, on which a cap has been tightly fitted. The other end was turned in a lathe to make it true, and on a metal plate a groove was turned to fit the open end of the pipe. Into this groove is fitted a rubber gasket, smeared on both sides with stopcock grease. In the top of the plate a two-inch hole gives a view of the interior. Over this hole is fitted a glass funnel with a similar gasket contact. Into the sides of the pipe have been fitted several $\frac{3}{8}$-inch pipes, two of which serve to introduce the lead wires, one for the off take to a Cenco Hyvac pump, and one for a monometer gauge. Others are for special purposes. All seals are made with De Khotinsky cement. The crucible support within the furnace is made of "transite" board; the crucible—an arc lamp carbon drilled out at one end for the desired depth—is fastened into a collar by means of a set screw. The collar in turn is fastened to one of the leads. Another carbon, solid, similarly fastened into a collar and connected to the other lead, is placed on top of the loaded crucible carbon and is pressed into place by an adjustable spring. The crucible itself then offers the greatest resistance in the circuit, since its cross-section shows the smallest amount of conductor. It is therefore its own heating element.

The operation is as follows: After loading the crucible make sure that the upper carbon is in good contact with the top of the crucible to avoid arcing. Adjust the metal plate and gasket in proper position on top of the furnace, and suitably cover the window opening. Start the vacuum pump and bear down upon the furnace top for a short time until it takes hold. Allow about fifteen minutes for pumping before turning the current on the crucible. The temperature of the crucible rises very fast following the ammeter quite closely. In fact, for careful melts we have found it desirable for one man to follow the crucible temperature with an optical pyrometer while another reads the milliammeter and adjusts the controls accordingly.

Crucibles so made are inexpensive and need not be used more than once. Such a furnace will not function if there are leaks in the system, as the crucible burns readily under such conditions. Some burning is unavoidable, owing to the release of absorbed air on heating. Due to this burning we have not been able to hold a melt at a high temperature for a prolonged period of time. In the selection of carbons to be used for crucibles, soft carbons are preferable to hard carbons.

A modification of this type of furnace has been used to determine melting points with moderate accuracy. Drill a hole in the side of an arc lamp carbon and place the material to be melted in the hole. Attach the lead wires to the ends of the carbon and support it on something such as transite board. The whole may then be mounted on a microscope stage and the melting observed while another observer reads the temperature with an optical pyrometer. Since this is not done in a vacuum the carbon burns rather rapidly but there is ample time for careful and deliberate measurements.

Others may have used furnaces similar to those described here. This description is offered at the suggestion of a visitor who expressed surprise that the higher temperatures could be reached so easily, and who believed that there were others who would be interested in these methods.

R. C. EMMONS

UNIVERSITY OF WISCONSIN

SPECIAL ARTICLES

THE STRUCTURE OF GLUCOSE

THE remarkable difference in stability and properties of the pyranose and furanose ring forms, respectively, of carbohydrates and polysaccharides points to some marked fundamental difference in their structure. This point of view receives added emphasis from the lack of a similar striking difference in properties between the five- and six-membered carbon rings.

The old von Baeyer strained ring hypothesis is no longer acceptable in the light of Ruzicka's synthesis of higher-membered lactone and ketone rings. There now appears to be definite evidence for the belief that while in the case of a pentamethylene strainless ring all the carbon atoms lie in one plane, in the case of hexamethylene, as first pointed out by Sachse and Mohr, a strainless ring is only possible if all the atoms are *not* assumed to lie in one plane; in other words, are united as a "puckered" strainless ring (A) or (A′).

It has been tacitly assumed, without any proof, however, that replacement of a carbon atom by oxygen does not influence the valence direction of the carbon atoms, and that the oxygen functions in this respect similar to carbon, namely, with an assumed normal valence angle of 109° 28′.

While it is true that certain X-ray data point to the tetrahedral character of the oxygen atom, the more recent work of Debye, of Sänger and of Williams serves to show that the valence angle of an oxygen atom attached to two carbon atoms, as in ethyl ether, is 32°, in other words, corresponds to that of the oxygen atom when linked with two atoms of hydrogen.

If now this latter value of 32° be taken as the *normal* carbon-oxygen-carbon valency angle, certain very interesting results follow.

For example, it is found that a ring system consisting of five carbon atoms and one oxygen forms a "puckered," strainless, pyranose ring (B). In this the carbon atoms are joined to one another at a (theoretical) valence angle of 109° 28′ and lie in one plane. The –C–O–C linkages with an oxygen valence angle of 32° subtend an angle of 109° 28′ with the plane of the carbon atoms.

On the assumption that in the furanose form (C) all the atoms lie in one plane, and that the *normal* –C–O–C– valence units form a much smaller oxygen valence angle than the system –C–C–C– (32° as compared with 109° 28′) such a ring must represent a strained system and one which should show a marked tendency to undergo ring scission, and ready conversion, under the influence of mild physical and chemical agents into the "puckered," strainless, pyranose ring type. That this must be the case is evident from the simple calculation, $360° - 3(109°\ 28') = 31°\ 36'$, or the approximate angle suggested above for –C–O–C– linkage. It is not necessary to assume that the structure is that of a regular pentagon, inasmuch as the –C–O–C– linkage may represent some value smaller than that assumed in the regular figure.

The theory appears to offer an explanation of many abnormalities occurring between the various isomeric ring structures of carbohydrates and polysaccharides.