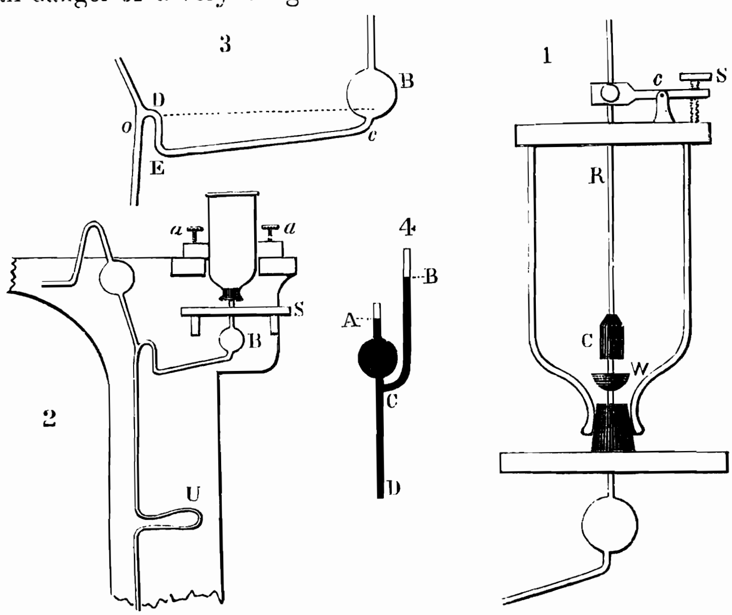
ART. XVI.—*On a method of obtaining and measuring very high Vacua with a modified form of Sprengel-pump;* by OGDEN N. ROOD, Professor of Physics in Columbia College.

IN the July number of this Journal for 1880, I gave a short account of certain changes in the Sprengel-pump by means of which far better vacuua could be obtained than had been previously possible. For example, the highest vacuum at that time known had been reached by Mr. Crooks, and was about $\frac{1}{17\ 000\ 000}$, while with my arrangement vacuua of $\frac{1}{100\ 000\ 000}$ were easily reached. In a notice that appeared in "Nature" for August, 1880, p. 375, it was stated that my improvements were not new, but had already been made in England four years previously. I have been unable to obtain a printed account of the English improvements, and am willing to assume that they are identical with my own; but, on the other hand, as for four years no particular result seems to have followed their introduction in England, I am reluctantly forced to the conclusion that their inventor and his customers, for that period of time, have remained quite in ignorance of the proper mode of utilizing them. Since then I have pushed the matter still farther, and have succeeded in obtaining with my apparatus vacuua as high as $\frac{1}{390\ 000\ 000}$, without finding that the limit of its action had been reached. The pump is simple in construction, inexpensive and, as I have proved by a large number of experiments, certain in action and easy of use: stopcocks and grease are dispensed with, and when the presence of a stopcock is really desirable its place is supplied by a movable column of mercury.

Reservoir.—An ordinary inverted bell-glass with a diameter of 100^{mm} and a total height of 205^{mm} forms the reservoir; its mouth is closed by a well-fitting cork through which passes the glass tube that forms one termination of the pump. The cork around tube and up to the edge of the former is painted with a flexible cement. The tube projects 40^{mm} into the mercury and passes through a little watch-glass-shaped piece of sheet-iron, W, figure 1, which prevents the small air bubbles that creep upward along the tube from reaching its open end; the little cup is firmly cemented in its place. The flow of the mercury is regulated by the steel rod and cylinder CR, figure 1. The bottom of the steel cylinder is filled out with a circular piece of pure india-rubber, properly cemented; this soon fits itself to the use required and answers admirably. The pressure of the cylinder on the end of the tube is regulated by the lever S, figure 1; this is attached to a circular board which again is firmly fastened over the open end of the bell-glass. It will be noticed that on turning the milled head S, the motion of the steel cylinder is not directly vertical, but that it tends to describe a circle with *c* as a center; the necessary play of the cylinder is however so small, that practically the experimenter does not become aware of this theoretical defect, so that the arrangement really gives entire satisfaction, and after it has been in use for a few days accurately controls the flow of the mercury. The glass cylinder is held in position, but not supported, by two wooden *adjustable* clamps *a a*, figure 2. The weight of the cylinder and mercury is supported by a shelf, S, figure 2, on which rests the cork of the cylinder; in this way all danger of a very disagreeable accident is avoided.



Vacuum-bulb.—Leaving the reservoir, the mercury enters the vacuum-bulb B, figure 2, where it parts with most of its air and moisture; this bulb also serves to catch the air that creeps into the pump from the reservoir, even when there is no flow of mercury; its diameter is 27^{mm}. The shape and inclination of the tube attached to this bulb is by no means a matter of indifference; accordingly figure 3 is a separate drawing of it; the tube should be so bent that a horizontal line drawn from the proper level of the mercury in the bulb passes through the point *o*, where the drops of mercury break off. The length of the tube EC should be 150^{mm}, that of the tube ED 45^{mm}; the bore of this tube is about the same as that of the fall-tube.

Fall-tube and bends.—The bore of the fall-tube in the pump now used by me is 1·78^{mm}; its length above the bends (U, figure 2) is 310^{mm}; below the bends the length is 815^{mm}. The bends constitute a fluid valve that prevents the air from returning into the pump; beside this, the play of the mercury in them greatly facilitates the passage of the air downward. The top of the mercury column representing the existing barometric pressure should be about 25^{mm} below the bends when the pump is in action. This is easily regulated by an adjustable shelf, which is also employed to fill the bends with mercury when a measurement is taken or when the pump is at rest. On the shelf is a tube, 160^{mm} high and 20^{mm} in diameter, into which the end of the fall-tube dips; its side has a circular perforation into which fits a small cork with a little tube bent at right angles. With the hard end of a file and a few drops of turpentine the perforation can be easily made and shaped in a few minutes. By revolving the little bent tube through 180° the flow of the mercury can be temporarily suspended when it is desirable to change the vessel that catches it.

Gauge.—For the purpose of measuring the vacua I have used an arrangement similar to McLeod's gauge, fig. 4; it has, however, some peculiarities. The tube destined to contain the compressed air has a diameter of 1·35^{mm}, as ascertained by a compound microscope; it is not fused at its upper extremity, but closed by a fine glass rod that fits into it as accurately as may be, the end of the rod being ground flat and true. This rod is introduced into the tube, and while the latter is gently heated a very small portion of the cement described below is allowed to enter by capillary attraction, but not to extend beyond the end of the rod, the operation being watched by a lens. The rod is used for the purpose of obtaining the compressed air in the form of a cylinder and also to allow cleansing of the tube when necessary. The capacity of the gauge-sphere was obtained by filling it with mercury; its external diameter was sixty millimeters; for measuring very high vacua this is somewhat small and makes the probable errors rather large; I would advise the use of a gauge-sphere of about twice as great capacity. The tube CB, figure 4, has the same bore as the measuring tube in order to avoid corrections for capillarity. The tube of the gauge CD is not connected with an india-rubber tube, as is usual, but dips into mercury contained in a cylinder 340^{mm} high, 58^{mm} in diameter, which can be raised and lowered at pleasure. This is best accomplished by the use of a set of boxes of various thicknesses, made for the purpose and supplemented by several sheets of cardboard and even of writing-paper. These have been found to answer well and enable the experimenter to graduate with a nicety the pressure to which the gas is exposed during measurement. By employing a cylinder filled with mercury instead of the usual caoutchouc tubing small bubbles of air are prevented from entering the gauge along with the mercury. An adjustable brace or support is used which prevents accident to the cylinder when the pump is inclined for the purpose of pumping out the vacuum-bulb. The maximum pressure that can be employed in the gauge used by me is 100^{mm}.

All the tubing of the pump is supported at a distance of about 55^{mm} from the wood-work; this is effected by the use of simple adjustable supports and adjustable clamps; the latter have proved a great convenience. The object is to gain the ability to heat with a Bunsen burner all parts of the pump without burning the wood-work. Where glass and wood necessarily come in contact the wood is protected by metal or simply painted with a saturated solution of alum. The glass portions of the pump I have contrived to anneal completely by the simple means mentioned below. If the glass is not annealed it is certain to crack when subjected to heat, thus causing vexation and loss of time. The mercury was purified by the same method that was used by W. Siemens (Pogg. Annalen, vol. cx, p. 20), that is, by a little strong sulphuric acid to which a few drops of nitric acid had been added; it was dried by pouring it repeatedly from one hot dry vessel to another, by filtering it while quite wârm, the drying being completed finally by the action of the pump itself. All the measurements were made by a fine cathetometer which was constructed for me by William Grunow; see this Journal, Jan., 1874, p. 23. It was provided with a well-corrected object-glass having a focal length of 200^{mm}, and as used by me gave a magnifying power of 16 diameters.

Manipulation.—The necessary connections are effected with a cement made by melting Burgundy pitch with three or four per cent of gutta percha. It is indispensable that the cement when cold should be so hard as completely to resist taking any impression from the finger nail, otherwise it is certain to yield gradually and finally to give rise to leaks. The connecting tubes are selected so as to fit as closely as possible, and after being put into position are heated to the proper amount, when the edges are touched with a fragment of cold cement which enters by capillary attraction and forms a transparent joint that can from time to time be examined with a lens for the colors of thin plates, which always precede a leak. Joints of this kind have been in use by me for two months at a time without showing a trace of leakage, and the evidence gathered in another series of unfinished experiments goes to show that no appreciable amount of vapor is furnished by the resinous compound, which, I may add, is never used until it has been repeatedly melted. As drying material I prefer caustic potash that has been in fusion just before its introduction into the drying tube; during the process of exhaustion it can from time to time be heated nearly to the melting point; if actually fused in the drying tube the latter almost invariably cracks. The pump in the first instance is to be inclined at an angle of about 10 degrees, the tube of the gauge being supported by a semicircular piece of thick paste-board fitted with two corks into the top of the cylinder. This seemingly awkward proceeding has in no case been attended with the slightest accident, and owing to the presence of the four leveling-screws the pump when righted returns, as shown by the telescope of the cathetometer, almost exactly to its original place. In the inclined position the exhaustion of the vacuum-bulb is accomplished along with that of the rest of the pump. The exhaustion of the vacuum-bulb when once effected can be preserved to a great extent for use in future work, merely by allowing mercury from the reservoir to flow in a rapid stream at the time that air is allowed to reënter the pump. During the first process of exhaustion the tube of the gauge is kept hot by moving to and fro a Bunsen burner, and is in this way freed from those portions of air and moisture that are not too firmly attached. After a time the vacuum bulb ceases to deliver bubbles of air; it and the attached tube are now to be heated with a moving Bunsen-burner, when it will be found to furnish for 15 or 20 minutes a large quantity of bubbles mainly of vapor of water. After their production ceases the pump is righted and the exhaustion carried farther. In spite of a couple of careful experiments with the cathetometer I have not succeeded in measuring the vacuum in the vacuum-bulb, but judge from indications, that is about as high as that obtained in an ordinary Geissler pump. Meanwhile the various parts of the pump can be heated with a moving Bunsen-burner to detach air and moisture, the cement being protected by wet lamp-wicking. In one experiment I measured the amount of air that was detached from the walls of the pump by heating them for 10 minutes somewhat above 100° C., and found that it was $\frac{1}{1000000}$ of the air originally present. I have also noticed that a still larger amount of air is detached by electric discharges. This coincides with an observation of E. Bessel-Hagen in his interesting article on a new form of Töpler's mercury-pump (Annalen der Physik und Chemie, 1881, vol. xii). Even when potash is used a small amount of moisture always collects in the bends of the fall-tube; this is readily removed by a Bunsen-burner; the tension of the vapor being greatly increased, it passes far down the fall-tube in large bubbles and is condensed. Without this precaution I have found it impossible to obtain a vacuum higher than $\frac{1}{25\,000\,000}$; in point of fact the bends should always be heated when a high exhaustion is undertaken even if the pump has been standing well exhausted for a week; the heat should of course never be applied at a late stage of the exhaustion. Conversely, I have often by the aid of heat completely and quickly removed quite large quantities of the vapor of water that had been purposely introduced. The exhaustion of the vacuum-bulb is of course somewhat injured by the act of using the pump and also by standing for several days, so that it has been usual with me before undertaking a high exhaustion to incline the pump and reëxhaust for 20 minutes; I have however obtained very high vacua without using this precaution.

During the process of exhaustion not more than one-half of the mercury in the reservoir is allowed to run out, otherwise when it is returned bubbles of air are apt to find their way into the vacuum-bulb. In order to secure its quiet entrance it is poured into a silk bag provided with several holes. When the reservoir is first filled its walls for a day or two appear to furnish air that enters the vacuum-bulb; this action, however, soon sinks to a minimum and then the leakage remains quite constant for months together.

Measurement of the vacuum.—The cylinder into which the gauge-tube dips is first elevated by a box sufficiently thick merely to close the gauge, afterwards boxes are placed under it sufficient to elevate the mercury to the base of the measuring tube; when the mercury has reached this point, thin boards and card-boards are added till a suitable pressure is obtained. The length of the enclosed cylinder of air is then measured with the cathetometer, also the height of the mercurial "meniscus," and the difference of the heights of the mercurial columns in A and B, figure 4. To obtain a second measure an assistant removes some of the boxes and the cylinder is lowered by hand three or four centimeters and then replaced in its original position. In measuring really high vacua, it is well to begin with this process of lowering and raising the cylinder, and to repeat it five or six times before taking readings. It seems as though the mercury in the tube B supplies to the glass a coating of air that allows it to move more freely; at all events it is certain that ordinarily the readings of B become regular, only after the mercury has been allowed to play up and down the tube a number of times. This applies particularly to vacua as high as $\frac{1}{50\,000\,000}$ and to pressures of five millimeters and under. It is advantageous in making measurements to employ large pressures and small volumes; the correct working of the gauge can from time to time be tested by varying the relations of these to each other. This I did quite elaborately, and proved that such constant errors as exist, are small, compared with inevitable accidental errors, as for example that there was no measurable correction for capillarity, that the calculated volume of the "meniscus" was correct, etc. It is essential in making a measurement that the temperature of the room should change as little as possible, and that the temperature of the mercury in the cylinder should be at least nearly that of the air near the gauge-sphere. The computation is made as follows:

n=height of the cylinder enclosing the air;
c=a factor which multiplied by n converts it into cubic millimeters;
S=cubic contents of the meniscus;
d=difference of level between A and B, fig. 4;
=the pressure the air is under;
N=the cubic contents of the guage in millimeters;
x=a fraction expressing the degree of exhaustion obtained: then

$$x = \frac{1}{\dfrac{N\dfrac{760}{d}}{nc - S}}$$

It will be noticed that the measurements are independent of the actual height of the barometer, and if several readings are taken continuously, the result will not be sensibly affected by a simultaneous change of the barometer. Almost all the readings were taken at a temperature of about 20° C., and in the present state of the work corrections for temperature may be considered a superfluous refinement.

Gauge correction.—It is necessary to apply to the results thus obtained a correction which becomes very important when high vacua are measured. It was found in an early stage of the experiments that the mercury in the act of entering the highly exhausted gauge, gave out invariably a certain amount of air which of course was measured along with the residuum that properly belonged there; hence to obtain the true vacuum it is necessary to subtract the volume of this air from nc. By a series of experiments I ascertained that the amount of air introduced by the mercury in the acts of entering and leaving the gauge was sensibly constant for six of these single operations (or for three of these double operations), when they followed each other immediately. The correction accordingly is made as follows: the vacuum is first measured as described above, then by withdrawing all the boxes except the lowest, the mercury is allowed to fall so as nearly to empty the gauge; it is then made again to fill the gauge, and these operations are repeated until they amount in all to six; finally the volume and pressure are a second time measured. Assuming the pressure to remain constant, or that the volumes are reduced to the same pressure,

v=the original volume;
v'=the final volume;
V'=volume of air introduced by the first entry of the mercury;
V=corrected volume; then

$$V' = \frac{v' - v}{6}$$

$$V = v - \frac{v' - v}{6}$$

It will be noticed that it is assumed in this formula that the same amount of air is introduced into the gauge in the acts of entry and exit; in the act of entering in point of fact more fresh mercury is exposed to the action of the vacuum than in the act exit, which might possibly make the true gauge-correction rather larger than that given by the formula. It has been found that when the pump is in constant use the gauge-correction gradually diminishes from day to day: in other words, the air is gradually pumped out of the gauge-mercury. Thus on December 21st, the amount of air entering with the mercury corresponded to an exhaustion of

$\frac{1}{27\,308\,805}$ ......Dec. 21st.

$\frac{1}{38\,806\,688}$ ......Dec. 29th.

$\frac{1}{78\,125\,000}$ ......Jan. 15th.

$\frac{1}{83\,333\,333}$ ......Jan. 23d.

$\frac{1}{128\,834\,063}$ ......Feb. 1st.

$\frac{1}{226\,757\,400}$ ......Feb. 9th.

$\frac{1}{232\,828\,800}$ ......Feb. 19th.

$\frac{1}{388\,200\,000}$ ......March 7th.

That this diminution is not due to the air being gradually withdrawn from the walls of the gauge or from the gauge-tube, is shown by the fact that during its progress the pump was several times taken to pieces, and the portions in question exposed to the atmosphere without affecting the nature or extent of the change that was going on. I also made one experiment which proves that the gauge-correction does not increase sensibly, when the exhausted pump and gauge are allowed to stand unused for twenty days.

Rate of the pump's work.—It is quite important to know the rate of the pump at different degrees of exhaustion, for the purpose of enabling the experimenter to produce a definite exhaustion with facility; also if its maximum rate is known and the minimum rate of leakage, it becomes possible to calculate the highest vacuum attainable with the instrument. Examples are given in the tables below: the total capacity was about 100,000 cubic mm.

Time.	Exhaustion.	Ratio.
	$\frac{1}{78\,511}$	
10 minutes		$1 : \frac{1}{3·53}$
	$\frac{1}{276\,980}$	
10 minutes		$1 : \frac{1}{6·10}$
	$\frac{1}{1\,687\,140}$	
10 minutes		$1 : \frac{1}{4·15}$
	$\frac{1}{7\,002\,000}$	

Upon another occasion the following rates and exhaustions were obtained:

Time.	Exhaustion.	Rate.
	$\frac{1}{7\,812\,500}$	
10 minutes		$1 : \frac{1}{3·18}$
	$\frac{1}{24\,875\,620}$	
10 minutes		$1 : \frac{1}{2·69}$
	$\frac{1}{67\,024\,090}$	
10 minutes		$1 : \frac{1}{1·22}$
	$\frac{1}{81\,760\,810}$	
10 minutes		$1 : \frac{1}{1·67}$
	$\frac{1}{136\,986\,300}$	
10 minutes		$1 : \frac{1}{1·23}$
	$\frac{1}{170\,648\,500}$	

The *irregular* variations in the rates are due to the mode in which the flow of the mercury was in each case regulated.

Leakage.—We come now to one of the most important elements in the production of high vacua. After the air is detached from the walls of the pump the leakage becomes and remains nearly constant. I give below a table of leakages, the pump being in each case in a condition suitable for the production of a very high vacuum:

Duration of the experiment.	Leakage per hour in cubic mm., press. 760^{mm}.
$18\frac{1}{2}$ hours	·000853
27 hours	·001565
$26\frac{1}{2}$ hours	·000791
20 hours	·000842
19 hours	·000951
19 hours	·001857
7 days	·001700
7 days	·001574
Average	·001266

I endeavored to locate this leakage, and proved that one-quarter of it is due to air that enters the gauge from the top of its column of mercury, thus:

Duration of the experiment.	Gauge-leakage per hour in cubic mm., press. 760^{mm}.
18 hours	·0002299
7 days	·0004093
7 days	·0003464
Average	·0003285

This renders it very probable that the remaining three-quarters are due to air given off from the mercury at B, fig. 4, from that in the bends and at the entrance of the fall-tube *o*, fig. 3.

Farther on some evidence will be given that renders it probable that the leakage of the pump when in action is about four times as great as the total leakage in a state of rest.

The gauge, when arranged for measurement of gauge-leakage, really constitutes a barometer, and a calculation shows that the leakage would amount to 2·877 cubic millimeters per year press. 760^{mm}. If this air were contained in a cylinder 90^{mm} long and 15^{mm} in diameter it would exert a pressure of $·14^{mm}$. To this I may add that in one experiment I allowed the gauge for seven days to remain completely filled with mercury and then measured the leakage into it. This was such as would in a year amount to ·488 cubic millimeters press. 760^{mm}, and in a cylinder of the above dimensions would exert a pressure of $·0233^{mm}$.

Reliability of results; highest vacuum.

The following are samples of the results obtained. In one case sixteen readings were taken in groups of four with the following result:

Exhaustion.

$$\frac{1}{74\,219\,139}$$

$$\frac{1}{78\,533\,454}$$

$$\frac{1}{79\,017{\cdot}272}$$

$$\frac{1}{68\,503\,182}$$

$$\text{Mean} \quad \frac{1}{74\,853\,449}$$

Calculating the probable error of the mean with reference to the above four results it is found to be 2·28 per cent of the quantity involved.

A higher vacuum measured in the same way gave the fol lowing results:

$$\frac{1}{146\,198\,800}$$

$$\frac{1}{175\,131\,300}$$

$$\frac{1}{204\,081\,600}$$

$$\frac{1}{201\,207\,200}$$

The mean is $\frac{1}{178\,411\,934}$, with a probable error of 5·42 per cent of the quantity involved. I give now an extreme case; only five single readings were taken; these corresponded to the following exhaustions:

$$\frac{1}{379\,219\,500}$$

$$\frac{1}{371\,057\,265}$$

$$\frac{1}{250\,941\,040}$$

$$\frac{1}{424\,088\,232}$$

$$\frac{1}{691\,082\,540}$$

The mean value is $\frac{1}{381\ 100\ 000}$, with a probable error of 10·36 per cent of the quantity involved. Upon other occasions I have obtained exhaustions of $\frac{1}{373\ 134\ 000}$ and $\frac{1}{388\ 200\ 000}$. Of course in these cases a gauge-correction was applied; the highest vacuum that I have ever obtained irrespective of a gauge-correction was $\frac{1}{190\ 392\ 150}$. In these cases and in general, potash was employed as the drying material; I have found it practical, however, to attain vacua as high as $\frac{1}{50\ 000\ 000}$ in the total absence of all such substances. The vapor of water which collects in bends must be removed from time to time with a Bunsen-burner while the pump is in action.

It is evident that the final condition of the pump is reached when as much air leaks in per unit of time as can be removed in the same interval. The total average leakage per ten minutes in the pump used by me, when at rest, was ·000211 cubic millimeters at press. 760^{mm}. Let us assume that the leakage when the pump is in action is four times as great as when at rest; then in each ten minutes ·000844 cubic millimeters press. 760^{mm} would enter; this corresponds in the pump used by me to an exhaustion of $\frac{1}{124\ 000\ 000}$; if the rate of the pump is such as to remove one-half of the air present in ten minutes, then the highest attainable exhaustion would be $\frac{1}{248\ 000\ 000}$. In the same way it may be shown that if six minutes are required for the removal of half the air the highest vacuum would be $\frac{1}{413\ 000\ 000}$ nearly, and rates even higher than this have been observed in my experiments. An arrangement of the vacuum-bulb whereby the entering drops of mercury would be exposed to the vacuum in an isolated condition for a somewhat longer time would doubtless enable the experimenter to obtain considerably higher vacua than those above given.

Exhaustions obtained with a plain Sprengel-pump.—I made a series of experiments with a plain Sprengel-pump without stopcocks, and arranged, as far as possible, like the instrument just described. The leakage per hour was as follows:

Duration of the experiment.	Leakage per hour in cubic mm. at press. 760^{mm}.
22 hours	·04563
2 days	·04520
2 days	·09210
4 days	·06428
Mean	·0618

Using the same reasoning as above we obtain the following table:

Time necessary for removal of ½ the air.	Greatest attainable exhaustion.
10 minutes	$\frac{1}{5\,000\,000}$
7·5 minutes	$\frac{1}{7\,000\,000}$
6·6 minutes	$\frac{1}{12\,000\,000}$

In point of fact the highest exhaustion I ever obtained with this pump was $\frac{1}{5\,000\,000}$; from which I infer that the leakage during action is considerably greater than four times that of the pump at rest. The general run of the experiments tends to show that the leakage of a plain Sprengel-pump, without stopcocks or grease, is, when in action, about 80 times as great as in the form used by me.

Note on annealing glass tubes.—It is quite necessary to anneal all those parts of the pump that are to be exposed to heat, otherwise they soon crack. I found by enclosing the glass in heavy iron tubes and exposing it for five hours to a temperature somewhat above that of melting zinc, and then allowing an hour or two for the cooling process, that the strong polarization figure which it displays in a polariscope was completely removed, and hence the glass annealed. A common gas-combustion furnace was used, the bends, etc., being suitably enclosed in heavy metal and heated over a common ten-fold Bunsen-burner. Thus far no accident has happened to the annealed glass, even when cold drops of mercury struck in rapid succession on portions heated considerably above 100° C.

I wish, in conclusion, to express my thanks to my assistant, Dr. Ihlseng, for the labor he has expended in making the large number of computations necessarily involved in work of this kind.

New York, June 10, 1881