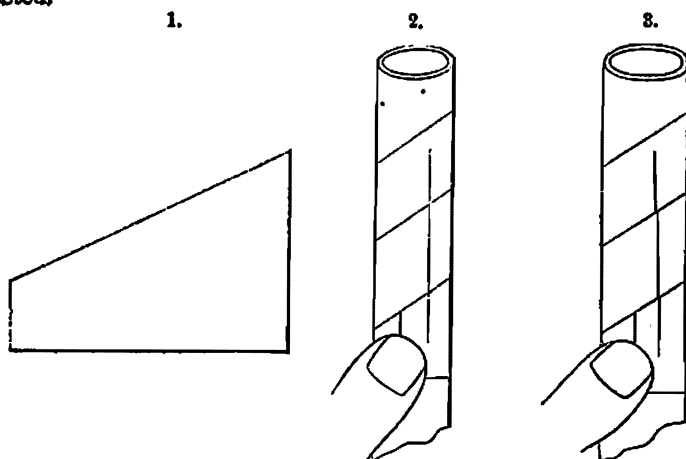


ART. L.—*On some new Manipulations*; by M. CAREY LEA.

I. GRADUATION OF BURETTES.

(1.) *Selection of Tube.*

ALTHOUGH no tube is sufficiently regular to dispense with subsequent exact calibration with a good balance, yet it is very desirable to have as near approach as possible to perfect uniformity, both in order to diminish the trouble of the weighings, and to a certain extent, that of the calculations, in actual use. In the following manner a variation in the diameter of the tube under examination, amounting to $\frac{1}{1250}$ or less can easily be detected.



A piece of thin glazed letter paper is cut to the shape represented in fig. 1, this is wound tightly round the tube, making the successive folds perfectly correspond at bottom, a card is held to the paper, and a thin line is drawn with a fine pointed pencil, passing over three folds of the paper as represented in fig. 2.

The paper is then loosened, slipped a few inches along the tube and tightened again, keeping the lower edge exactly even. A very small difference in the diameter of the tube will cause the line to appear broken, as represented in fig. 8, instead of straight. A displacement from end to end amounting to one $\frac{1}{16}$ of an inch is easily observable, and as this difference corresponds to a little over six diameters, it is clear that a difference in the mean diameter of $\frac{1}{16}$ of an inch or less, makes itself evident to the unassisted eye. With an ordinary lens, a difference of $\frac{1}{32}$ of an inch is easily distinguishable, corresponding to a difference of diameter of $\frac{1}{32}$ of an inch. When great accuracy is desired it is advisable to stretch the paper band strongly beforehand, whereby it acquires a very slight permanent increase of length, and is not further extended by the very moderate force requisite to wrap it tightly round the tube.

It is so easy in this manner to detect slight differences that I have never been able to find a piece of tube of a foot or more in length which would bear this test when applied with the utmost rigor. But by examining large quantities of tube, pieces can be found sufficiently good for graduation. These are to be cut out from the tube in which they are found, and the part which has been ascertained to be sufficiently regular, must be carefully marked that the graduation may not be carried beyond that portion.

(2.) *Graduation of the Burette.*

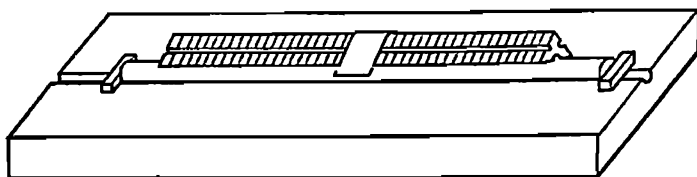
All modes of graduation are necessarily the same in principle, and consist in transferring divisions from a scale to the tube. Accurate three-edged engineer's scales are easily to be had, and are very convenient for this purpose. The glass tube is secured by cleats into a groove in a block of wood. And here I may remark that the directions usually given in respect to the groove in which the tube rests, are certainly mistaken in recommending the groove to be part of a circle of a diameter *greater* than that of the tube. Such an arrangement causes the tube to rest loosely on the bottom of the groove, and to rock easily from side to side, touching as it does, in one place only. But if the groove is that of a circle of diameter rather *less* than that of the tube, the latter rests on the edges of the groove, and remains steadily in its position.

Before fastening the cleats, and while the tube is lying in the groove, touching its edges, a diamond pencil is drawn along the tube, the edge of the groove serving as a rule, and a line is made from one end of the space intended to be graduated to the other. The tube is then rotated on its axes a quarter of an inch, and another line is drawn parallel with the former. These lines serve

to limit the length of the single degrees, each fifth and tenth being drawn beyond them.

The cleats are then fastened, the three-edged rule is laid with one of its edges resting on the tube, and its ends also are secured by cleats (these last are not represented in the figure). A piece

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of thin sheet brass (such as is used for enveloping combustion tubes) is bent at a sharp angle, and its right hand edges cut to an exact right angle with the bend. This is rested on the upper edge of the ruler, extending across its front face and over the tube—its right hand edge serves to guide the diamond pencil in transferring the divisions. Twentieths of an inch, or millimeters are easily and exactly ruled in this way, and the burette is ready for calibration.

(3.) Calibration.

When a burette contains a portion of liquid, it is a matter of great nicety to determine the division or fraction of a division to which the surface of the liquid corresponds. To save the labor of holding the paper with the lower half blackened behind the tube, it is convenient to substitute card board, and to cut two parallel slits in it so as to form a band, which being slipped over the burette, the card maintains its own position. The use of paper blackened on the lower half, gives, according to Mohr, all the accuracy desirable. Bunsen, on the contrary, uses a cathetometer. That the former method is insufficient any one may satisfy himself by placing the card in position, and then moving his head slightly in a vertical direction when the black line which is assumed to mark the surface of the liquid, will be found to move also, and though the change is but small, it will be found that even the slightest movement of the eye produces a change in the position of the line which gives a difference in results easily detected by a good balance. Let us suppose that the observer notes the position of the black line before and after removing a portion of the liquid, he cannot be certain that his eye has occupied the same relative position in both cases, unless he takes due precautions. Every observer does not possess a cathetometer, and the following arrangement which is of extreme simplicity, will be found to answer every purpose.

A slip of wood is provided $\frac{1}{8}$ inch thick, $1\frac{1}{2}$ inches wide, and 2 feet long. A piece of card about 4 inches square has two parallel slits made in it at one end, one slit half an inch from the top, the other the same distance from the bottom. These slits are of such a length as just to permit the piece of wood to be passed through them, presenting the appearance represented in the margin.

To make a reading, the half black card previously spoken of is slipped over the burette, and the line of separation between the white and black is brought one millimeter below the black line which marks the surface of the liquid. The instrument represented in the margin, is then placed beside the burette, the lower end resting on the table, and the card is raised or lowered until the edge B exactly corresponds with the line of separation in the black and white card. The stick is then drawn toward the observer, the end B still resting on the table, and the observer places his eye so as to keep the line B in range with the line of separation, and makes the reading.

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Another precaution which I regard as essential, and which I have nowhere seen mentioned, is the following: The observer should place himself where he has a strong *side light*. The half black half white card must not be placed parallel to the eyes of the observer, but must be turned toward the light, so as to make an angle of 45° with the line of vision. In this way a strong light is reflected from the card and thrown through the burette in a manner greatly conducive to clearness of vision, and the black line which marks the surface acquires a peculiar sharpness.

I cannot better illustrate the necessity of these precautions, (especially the use of the little instrument above described, and which may be termed the *eye adjuster*) than by the comparison of the following results obtained without them and with them.

Distilled water corresponding to the divisions of a tube prepared in the manner above described was carefully weighed in an accurate balance.

1. Without the precautions.

Mean of first three trials,	2.023 grams.
" " second " "	1.974 "

2. With the precautions.

Mean of first three trials,	2.046 grams.
" " second " "	2.047 "
" " two more "	2.050 "

It will be seen that the trials without the precautions which I recommended gave results which were not only discordant with each other, but were *all wrong* and below the truth. With the

precautions, on the contrary, the mean of the first three differed from that of the second three by only a single milligram, or less than the sixtieth part of a drop.

To show that the latter results were of no fortuitous exactitude, I may mention other instances. The distilled water corresponding with a given space was weighed *six* times and the mean found was 2.090 grams. Subsequently it was deemed advisable to submit this to a second verification, and the mean of *nine* trials gave 2.091. To the above two cases I may add the following:

Table No. 3, mean of first 4 weighings,	2.054,	of 4 more,	2.0515
" " 4, " " " 3 "	2.387,	" 4 "	2.362
" " 5, " " " 3 "	2.365,	" 3 "	2.370
" " 6, " " " 2 "	3.355,	" 2 "	3.551
" " 7, " " " 2 "	3.577,	" 2 "	3.577
" " 8, " " " 2 "	2.3605,	" 2 "	2.3630

Comparing the first and second columns, that is, the first set of determinations, with the second we find that the maximum error was .005, and the minimum .0, or exact correspondence. The average of all the cases was 3.8 milligrams, or about one-twentieth of a drop. It would be difficult in burette analysis to obtain greater accuracy than this, for if we are using, for example, ten per cent solutions, the maximum error would reach half a milligram of the reagent used, and the average error would be one-third of a milligram.

I might greatly extend this table of verifications, but I think what I have cited will be sufficient to show that the very simple precautions which I here propose, viz., the position of the burette and card relatively to the light, and still more, the use of an eye-adjuster consisting of a slip of wood and sliding card are sufficient to carry us to the extreme limits of accuracy which the burette is capable of affording. It also shows that the burette is entitled to great confidence where carefully used and when the reactions which mark the termination of the operation are perfectly distinct and sharp.

II. INVERSE FILTRATION.

Large quantities of mixed solid and liquid matter may be filtered with a funnel of the smallest dimensions very speedily and conveniently, by proceeding in the following manner. A piece of stout muslin is strained over the mouth of a small funnel, and tied securely at the neck, taking care that the string covers all the folds perfectly. A piece of india-rubber tube is then passed over the open end of the stem of the funnel: this piece may either be several feet in length, or it may be shorter, and the difference be made up by inserting a glass tube. The funnel and tube are then filled with water, the open end of the

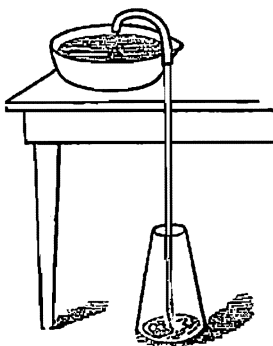
tube is closed with the finger and the funnel is quickly inverted in the vessel containing the mixture to be filtered. The other end of the tube hangs down into a convenient vessel placed on the floor. If it is desirable that water shall not be added to the mixture, the funnel is inverted empty, and the air is drawn out by a pipette inserted into the open end of the india rubber tube.

It is evident that this arrangement is a combination of filter and siphon, and the pressure of the column of water in the longer leg of the siphon expedites the operation very much and leaves the solid portions much drier than ordinary. As the liquid begins to be exhausted, the solid portions are to be gathered round the funnel with a spatula. When the filtrate ceases to run, the funnel is left full:—in order that this may not return upon the solid matter, the funnel is lifted out of the vessel and the broad end quickly turned uppermost, when the contents of the funnel flow down the tube.

When a precipitate is to be well washed this mode is evidently not applicable, although a tolerable washing is perfectly practicable. But when liquid and solid matters are to be quickly separated on a large scale, it is very useful. When masses of small crystals strongly retaining the mother water, are to be freed from it, this may be done more quickly and more thoroughly than by ordinary filtration. Many other cases will readily suggest themselves. For example, when potash has been boiled with lime to render it caustic in large vessels, it is usually drawn off with a siphon into bottles and left twelve to twenty-four hours to settle, and then must be carefully decanted. It is far less trouble to filter it in the above manner in the act of removing it by a siphon. And so with many of the rough operations which occasionally present themselves in the laboratory, and which are upon a scale rather exceeding the capacities of ordinary filtering funnels.

In the separation of crystals from the mother water, muslin will generally give a clear filtrate. In other cases it is necessary to place a piece of filtering paper inside the muslin. The paper must of course be of size sufficient to be secured by the twine at the neck of the funnel. But the force with which the column of water acts upon the muslin over the mouth of the funnel, draws it to a concave shape, and sometimes breaks the paper. To avoid this, the paper may be folded so as to fit the *inside* of the funnel, turning the edges over and securing them as before de-

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scribed. To do this requires a little dexterity; the diameter of the paper must be six or seven times that of the mouth of the funnel, it must be folded across in two rectangular directions in the ordinary way, then opened and reversed and from opposite points in the edges folded some distance past the middle. The funnel is then passed into it and the edges are passed round the neck of the funnel, the muslin is next placed over the paper and the whole is secured round the neck with cord.