

easily purified by crystallizing from benzene, or from benzene-ligroin. Melting-point 158.5° .

1. 0.1641 gram substance gave 0.1668 gram silver chloride.

2. 0.2115 gram substance gave 0.2127 gram silver chloride.

1. 0.2397 gram substance gave 0.4714 gram carbon dioxide and 0.0620 gram water.

2. 0.2484 gram substance gave 0.4891 gram carbon dioxide and 0.0619 gram water.

	Calculated for	Found.	
	$C_{19}H_{11}N_2O_2Cl_3$.	1.	2.
C	54.09	53.63	53.69
H	2.63	2.87	2.76
Cl	25.23	25.13	24.86

α -Benzoyl phenylhydrazino trichlorquinone is a bright red crystalline body, readily soluble in hot benzene and alcohol, from each of which it crystallizes in long rectangular prisms or in star-shaped groups of small prisms. It is sparingly soluble in ligroin, and practically insoluble in glacial acetic acid and dilute caustic soda solution. Its action with phenylhydrazine is similar to that of quinone, decomposition occurring with evolution of gas. By dissolving in concentrated sulphuric acid as well as by the action of alcoholic potash, the benzoyl group is split off, but some further reaction takes place forming a dark brown crystalline body. This is soluble in caustic soda and melts with decomposition at 198° . Its structure has not yet been determined.

OHIO STATE UNIVERSITY, JANUARY, 1900.

NOTE ON A METHOD OF STANDARDIZING WEIGHTS.

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ALTHOUGH nowadays it is possible to procure, for a comparatively small sum, sets of weights which are reasonably exact, it is recognized that for gravimetric work demanding any considerable degree of accuracy, corrections on account of occasional errors in the weights must be applied. It is obvious, moreover, that if the relative values of the weights in an inexpensive box are known, this set may serve a purpose which would otherwise demand a much more expensive one. For ten years every student in quantitative analysis at Harvard has been required to standardize his weights; and since the method

adopted has some peculiarities, not described in the usual directions for this purpose,¹ it is explained below. This method does not pretend to be especially original; the reason for its publication is simply a desire to advance the cause of accuracy.

According to our method the weights to be standardized are weighed wholly on one side of the balance, the comparison being made by substitution. This procedure, of course, eliminates a possible inequality in the length of the arms of the balance, which must otherwise be computed. A more important advantage is the fact that it also obviates the mental confusion resulting from the continual interchanging of weights between the opposite pans. Thus is avoided one of the common sources of error in the beginner's work.

It is, of course, necessary that all of the fractional weights should, taken together, constitute a gram; and because the milligram weights are never used, it is convenient to add an extra centigram weight from another set to supplement the other small weights. The different weights of the same denomination should be marked in a recognizable way, and should always be arranged in the same order in the box. The comparison usually begins with centigrams and proceeds upwards. One of the centigram weights is placed upon the *left-hand* scale-pan, and is balanced by any suitable tare, care being taken that the rider is not too near either end of its path.² The zero point of the balance need not have been taken in the first place. The swings of the balance with its centigram load are now carefully noted and then another centigram weight is substituted for the first. Obviously, the amount by which the rider must be moved to attain the same position of equilibrium gives at once the difference between the two weights; and even the neophyte could hardly err as to its sign. Of course, instead of moving the rider, the difference may be calculated from the change in the swings; or both swings and rider may be used. The first weight is then replaced upon

¹ For the usual method adopted by Kohlrausch, see Ostwald's Handbook of Phys. Chem. Measurements (Walker), p. 41.

² A crude set of weights is, of course, the most convenient tare, and a 5 milligram weight may be kept on the left-hand pan so that the rider may assume a convenient position. The use of the *left-hand* scale-pan for the weights to be standardized renders a confusion of the sign of the correction less likely, because the rider is on the right. In this case, the *weights are the objects to be weighed*, and hence naturally take the left-hand position.

the left-hand pan, and if the swings correspond to the first observation it is reasonably certain that the balance has remained in a constant condition throughout the trial, and hence that the difference between the two weights has been accurately determined. In this way every weight is compared with every other weight of the same denomination, as well as with the combination of all the smaller weights. Thus are obtained a number of independent equations one less than the number of weights; and by assuming the value of any one of the weights the others may all be calculated.¹

We have found it most convenient for the purposes of calculation to make the temporary assumption that the first centigram weight is correct. From it by the simplest possible process of addition and subtraction may be built up quickly the values of all the other weights. While the values thus computed are wholly consistent among themselves, they are usually far too different from the face values of the weights for convenient use. The reason of this is because the assumed standard is so small a quantity. It is necessary then to translate these consistent values into other terms by dividing every value by the value of one of the larger weights, to be taken as the new and permanent standard.²

This division (which would demand six or seven place logarithm tables for the larger weights) is conveniently replaced, however, by a simple method depending upon the properties of small numbers in presence of large ones.³ It is quite sufficiently accurate to compare the value of each weight with the corresponding aliquot part of the value of the weight to be taken as a final standard. The difference between the actual value and the ideal value will give at once the correction to be applied to the weight.

¹ Of course, many extra equations, which are not independent of the others, are obtained by the literal fulfilment of the preceding directions. These extra equations may be used to verify the final results, if desired. The comparisons best suited for the calculation are given in a table on a following page.

² If it is desired to refer the whole box of weights to the international standard, it is, of course, necessary to include a weight which has been standardized at Washington in the series, and thus to find the value of the international gram in terms of the centigram chosen as the temporary basis. The aliquot parts or multiples of this value are then used precisely in the manner indicated further on. For all ordinary purposes, however, this is wholly unnecessary; and one of the 10-gram pieces of the box is a more convenient standard.

³ Nernst und Schoenflies Math. Behand. der Naturwiss., p. 303 (1893).

Of course, if the weight is not heavy enough this correction carries a *minus* sign, and must be *subtracted* from the result of any weighing in which that weight is used; for the deficiency necessitates the addition of extra weight on the rider-arm, and hence the sum of the face-values of all the weights used is too large. Long experience has shown that the sign of the final correction is the most insidious cause of error in the whole process; for the beginner always thinks that if a given weight is too light, its correction should be *added*.

The table below presents all the data and results of a sample standardization, as well as all the calculation which need be written down by anyone possessing even a moderate ability to add figures mentally. In the first column the weights are named by their face-values, which are enclosed in parentheses in order to show that they do not signify true grams. In the second column are given the results of the mutual comparison of these weights copied from a note-book in which every detail of each weighing was recorded. The third column gives the actual values of the weights based upon the first centigram weight; these values are obtained by simply adding together the appropriate preceding values in the third column and the last minute fractional weight enumerated in the second column. The aliquot parts of the value for the 10-gram piece, which is now to be taken as the permanent standard, are recorded in the fourth column, while the corrections sought, obtained by simply subtracting the numbers in the fourth column from those in the third, are given in the last vertical row.

Nominal values.	Data obtained by substitution method.	Preliminary values (actual).	Aliquot parts of 10.0768 (ideal). ¹	Corrections in milligrams (actual minus ideal). ¹
	Grams.	Grams.	Grams.	
(0.01) = Standard of comparison		Standard	0.01002	—0.02
(0.01') = (0.01)	+ 0.00006	0.01006	0.01002	+0.04
(0.01'') = (0.01')	— 0.00001	0.01005	0.01002	+0.03

¹ Owing to neglected fractions the figures in the last column, when added together, are sometimes slightly discordant with those given in the second column. This is inevitable; of course such corrections should always be calculated to one decimal place beyond the figure which one wishes to have exact.

Nominal values.	Data obtained by substitution method.	Preliminary values (actual).	Aliquot parts of 10.01768 (ideal).	Corrections in milligrams (actual minus ideal). ¹
	Grams.	Grams.	Grams.	
(0.02) = (0.01) + (0.01')	-0.00001	0.02005	0.02004	+0.01
(0.05) = (0.02) + etc.	--0.00007	0.05009	0.05009	±0.00
(0.1) = (0.05) + etc.	-0.00006	0.10019	0.10018	+0.01
(0.1') = (0.1)	+0.00001	0.10020	0.10018	+0.02
(0.2) = (0.1) + (0.1')	-0.00004	0.20035	0.20035	±0.00
(0.5) = (0.2) + etc.	-0.00011	0.50088	0.50088	±0.00
(1) = (0.5) + etc.	-0.00004	1.00183	1.00177	+0.06
(1') = (1)	-0.00002	1.00181	1.00177	+0.04
(1'') = (1)	-0.00006	1.00177	1.00177	±0.00
(2) = (1') + (1'')	+0.00025	2.00383	2.00354	+0.29
(5) = (2) + etc.	-0.00040	5.00884	5.00884	±0.00
(10) = (5) + etc.	-0.00040	10.01768	10.01768	Standard.
etc.		etc.	etc.	etc.

It is convenient to prepare from the individual corrections a card exhibiting at once the corrections corresponding to the usual combinations of weights from 1 to 99 centigrams and from 1 gram upwards. This card minimizes both the labor of applying the corrections and the danger of possible error in the process. It is hardly worth while to print here an example of such a card, but the method of application may be illustrated. The page of the note-book is ruled with two parallel vertical lines, which contain the two corrections found in the card. The upper correction corresponds in each case to the whole grams, and the lower to the fractional weights.

	Observed weight. Grams.	Cor. mg.	Corrected weight. Grams.
Weight of crucible + substance	19.3105	{ +0.39 -0.01	19.3109
“ “ “ alone	16.9916	{ +0.06 +0.06	16.9917
Weight of substance			2.3192

Thus the increased accuracy is gained with very little sacrifice of time or mental labor. It is usually more convenient to adjust the rider by filing to exactly 12.0 (or 10.0) milligrams rather than to apply a correction for this also.

¹ See foot-note, p. 147.

The method of comparing the actual value with an ideal one is also a convenience in standardizing burettes by means of the Ostwald "calibrator." In the original description of this process it is assumed that the calibrator delivers exactly an integral number of cubic centimeters;¹ but if only a few instruments are to be calibrated it is both troublesome and expensive to secure such a precise instrument. We have found it convenient to use a calibrator of any size, and to compare in parallel columns its multiples with the actual readings of the burette. The capacity of this calibrator is most conveniently obtained in the following manner. Suppose that as a mean of several comparisons it has been found that sixteen fillings of the calibrator correspond to 49.53 cc. on a given burette. Grease is the most serious foe to accuracy in this process. The burette is now refilled, and exactly this amount of pure water is run out into a weighed flask, with all the precautions which would be used in an actual titration. The weight of the water gives by appropriate calculation² the true volume of sixteen fillings of the calibrator. Suppose this was found to be 49.44 cc.; then the volume of the calibrator as it is actually used in a calibration must be $\frac{49.44}{16} =$

3.090 cc. The differences between the successive readings of the burette and the successive numbers 3.09, 6.18, 9.27, . . . etc., give at once the errors of the graduation of the tube at these intervals. These differences or corrections may be plotted on a diagram in which the ordinates are volumes and the abscissas corrections. The correction to be applied for 50 cc. is obviously -0.09 cc. Here again the sign is somewhat perplexing to the inexperienced.

These simple methods have stood the test of years of use in our first course upon quantitative analysis, and their practicability under these circumstances shows that reasonable accuracy in weights and measures is within the easy reach of all.

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¹ Ostwald: Handbook of Phys. Chem. Measurements (Walker), p. 87 (1894).

² This Journal, 21, 527 (1899).