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EMERGENT STEM CORRECTION FOR THER-
MOMETERS IN CREOSOTE OIL
DISTILLATION FLASKS

BY

R. M. WILHELM, Laboratory Assistant
Bureau of Standards

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EMERGENT STEM CORRECTION FOR THERMOMETERS IN CREOSOTE OIL DISTILLATION FLASKS

By R. M. Wilhelm

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I. INTRODUCTION

In many industrial operations where mercurial thermometers are used, the form of the apparatus may prevent the placing of the thermometer so that its entire mercury column is immersed at the temperature of the bulb. Under such conditions the temperature of the stem in general differs from that of the bulb. Thermometers are commonly scaled to read correctly only when the entire mercury column is at the temperature of the bulb, and if used otherwise they will not indicate the true temperature of the bulb unless corrections are applied to their readings. A particular example is that of the use of thermometers in distillation flasks, where the

correction may amount to 15° or 20° ¹ under actual distillation conditions.

The magnitude of this correction depends for the greater part on the length and the temperature of the emergent stem (referring only to the part containing the mercury thread). One of the chief sources of error in the use of mercurial thermometers arises from uncertainties as to the temperature of this emergent stem, as it may have as its average almost any temperature between that of the bulb and that of the air in the room, depending upon the conditions under which the thermometer is used.

The emergent stem correction may be computed, provided certain conditions are known, such as average stem temperature, depth of immersion, and the kind of glass of which the thermometers are made. Thermometers tested under conditions of total immersion at the Bureau of Standards are furnished with emergent stem correction sheets which show by example how this correction may be determined, if the instrument is read partially immersed, by using a simple formula. But aside from the general dislike of the use of formulas in industrial operations, in some cases the factors involved (the emergent stem temperature in particular) are difficult to obtain accurately.

Thermometers are sometimes scaled for use or tested with the bulb and only a part of the stem immersed. Such thermometers read correctly, however, only when the temperature conditions under which the thermometers were graduated or tested correspond to those of use, and this is seldom the case. It is obvious that when a thermometer scaled for a given immersion is used at some different immersion its readings will generally be in error.

For some processes it is not of great importance that the thermometer readings should represent the actual temperature of the bulb so long as such processes are confined to a single laboratory and to uniform conditions; but whenever it becomes necessary to specify the conditions under which the processes are carried out, so that they can be repeated elsewhere, a knowledge of the true temperatures becomes necessary.

The purpose of this investigation was primarily to ascertain the magnitude of the emergent stem correction error which may occur

¹ All temperatures in this paper are expressed in degrees centigrade.

in the use of thermometers in the distillation of creosote, a question which has arisen at various times in connection with the testing of thermometers for use in distillation flasks.

Some work has already been done on the subject of emergent stem corrections of thermometers in distillation flasks. In a circular by Dean and Bateman,² of the United States Forest Service, on the distillation of coal-tar creosote, there is published a table of emergent stem corrections. Wiebe³ has also given emergent stem correction tables for thermometers used with glass and with metal distillation apparatus. These tables are considered more fully later.

II. APPARATUS AND METHOD

1. FLASKS AND THERMOMETERS USED

Four types of distillation flasks, as shown in Fig. 1, were used—the retort, recommended by the American Railway Engineering and Maintenance of Way Association; the Hempel flask, as used by the Forest Service; the Lunge side-neck flask, recommended by the Committee on Preservative Treatment of Poles and Cross Arms of the National Electric Light Association; and the ordinary 200-cc side-neck flask, with dimensions as follows: Diameter of bulb 8 cm, diameter of neck 2 cm, height of neck 15 cm, distance from bottom of outlet tube to top 8 cm. All the tests were made according to the specifications⁴ applying to the particular form of apparatus used, except where special experiments were made to determine the effect of variations from the specified procedure. A graduated stem continuous scale type of mercurial thermometer, 40 cm long, graduated into 1° intervals from 0° to 400° C, was used. The length of a 1° interval was 0.8 mm; the distance from middle of bulb to 0° mark was 2.5 cm. The thermometer was “filled under pressure” to prevent distillation of the mercury, and was well annealed to prevent a rise of the readings with continued heating.

² Forest Service Circular 112.

³ Petroleum Jahrgang, VII, 1912.

⁴ American Ry. Eng. and Maintenance of Way Assn., 1905, pp. 4-14. National Electric Light Assn. Report on Preservative Treatment of Poles and Cross Arms, 1911, p. 21. Forest Service Circular 98, pp. 8-10. Forest Service Circular 112, p. 37.

FIG. 1.—Types of distillation flasks used and positions of thermometers

2. METHOD USED TO DETERMINE EMERGENT STEM CORRECTION

For measuring the temperature of the stem, use was made of a number of special "thread" thermometers made up and calibrated for this purpose, and auxiliary thermometers for the part of the stem not covered by the thread thermometers. With these thermometers the mean temperature of the emergent mercury column over its entire length could be very closely ascertained. The use of these "thread" thermometers is discussed in detail in a Bureau publication.⁵

Most of the apparatus and the creosote oil were furnished to the Bureau by S. R. Church, of the Barrett Manufacturing Co.

III. RESULTS

1. EMERGENT STEM CORRECTION TABLES

Average emergent stem corrections, applicable for the particular type of thermometer as used in the different distillation flasks, were determined for readings taken on the thermometer at every 20° from 200° up to 350°. The emergent stem corrections found are given in Table 1.

TABLE 1

Stem Corrections for the Thermometer ^a in the Four Types of Flasks

Reading	Retort	Hempel flask	Lunge flask	Ordinary flask
Degrees	Degrees	Degrees	Degrees	Degrees
200	+ 4.8	+ 4.7	+ 5.2	+ 4.7
220	+ 5.8	+ 5.0	+ 6.1	+ 5.0
240	+ 7.1	+ 6.1	+ 7.2	+ 5.4
260	+ 8.5	+ 7.5	+ 8.4	+ 6.6
280	+10.0	+ 8.5	+ 9.7	+ 8.5
300	+11.8	+10.5	+11.5	+10.5
320	+13.7	+12.4	+13.5	+12.5
340	+15.7	+14.4	+15.8	+14.5
350	+16.8	+15.5	+17.1	+15.5

^a These corrections apply only for a thermometer similar to that used in these tests.

The corrections are given to tenths of a degree, but an inspection of the curves plotted for the different runs shows that for the same reading the emergent stem correction may differ by over a degree from one curve to another. For this reason the corrections may not be reliable to better than a degree.

⁵ E. Buckingham, Bulletin Bureau of Standards, 8, p. 239.

The agreement between the emergent stem corrections observed in different runs is next best in the Hempel flask and worst in the

FIG. 4.—Stem corrections using Lunge flask with trap

ordinary side-neck flask, although the irregularity of several of the curves in this case was due to moisture in the oil and to increas-

FIG. 5.—Stem corrections using ordinary side-neck distillation flask

ing the size of the flame. The constancy of the emergent stem corrections in the retort should not be taken as a recommendation

of this form of apparatus, since there are apparently some very decided disadvantages in its use.⁶

The true mean temperature of the emergent stem of thermometers in distillation flasks is not easily obtainable. If it could be assumed that the temperature of that part of the thermometer stem below the cork were uniform and the same as that of the bulb, the emergent stem could be considered as that part within and above the cork. The mean temperature of this part of the stem could then be approximately obtained by hanging a small thermometer with its bulb somewhat below the middle of the emergent mercury thread. The emergent stem corrections computed from readings of a thermometer with its bulb about two-thirds way down the emergent mercury thread have been plotted for three of the flasks and the curves thus obtained are shown on the same figures (2, 3, and 5) with the true emergent stem correction curves. It may be seen that, except for the Hempel flask, the emergent stem corrections obtained by the two methods are not in agreement. As would be expected, the corrections obtained by neglecting the part of the stem below the cork are, with the exception mentioned, too low, since they do not take account of the temperature conditions below the cork. Thus, in the ordinary flask, the emergent stem corrections, using the stem temperature as indicated by a thermometer hung with its bulb about two-thirds way down the length of the emergent column, are from 1° to 2.5° less than the actual emergent stem corrections. In the Hempel flask, however, the emergent stem corrections computed in this manner agree very closely with the actual emergent stem corrections.

3. COMPARISON OF EMERGENT STEM CORRECTION TABLES

A comparison of the emergent stem corrections given in Table 1 with those given by Dean and Bateman, Table 2, and by Wiebe, Table 3 (column 2), show considerable disagreement. Table 3 gives corrections considerably larger than those in Table 1, while Table 2 gives smaller corrections.

Dean and Bateman probably used a thermometer graduated from 200° up, with an enlargement in the stem below the 200°

⁶ Alleman, Forest Service Circular 98, p. 8. Dean and Bateman, Forest Service Circular 112, p. 35.

mark. Wiebe does not state the type of thermometer used in his investigation, but it probably was one of the inclosed scale type (einschluss). The Engler flask used by Wiebe is smaller than the flasks used in this investigation.

The corrections in Table 3, column 3, were obtained by Wiebe for a thermometer used in a metal distillation apparatus. The question of emergent stem corrections in metal distillation apparatus was not taken up in this investigation, as this type of flask is little used in this country.

TABLE 2

Dean and Bateman's Corrections for the Emergent Thermometer Stem in 500-cc Side Neck Flask

Thermometer reading	Stem temperature	Degrees emergent	Correction	Corrected reading
Degrees	Degrees	Degrees	Degrees	Degrees
210	42.0	10	+0.3	210.3
220	43.5	20	.6	220.6
230	45.0	30	.9	230.9
240	47.0	40	1.2	241.2
250	48.5	50	1.6	251.6
260	50.0	60	2.0	262.0
270	52.0	70	2.4	272.4
280	53.5	80	2.9	282.9
290	55.0	90	3.4	293.4
300	57.0	100	3.9	303.9
310	58.5	110	4.4	314.4
320	60.0	120	5.0	325.0

TABLE 3

Wiebe's Corrections for the Emergent Thermometer Stem in Engler Flask and Metal Apparatus

Temperature	Stem correction—		Temperature	Stem correction—	
	In glass apparatus	In metal apparatus		In glass apparatus	In metal apparatus
Degrees	Degrees	Degrees	Degrees	Degrees	Degrees
60	+0.8	+0.2	200	7.2	4.3
80	1.6	.5	220	8.7	5.4
100	2.3	.9	240	10.3	6.6
120	3.1	1.4	260	12.2	8.0
140	3.9	1.9	280	14.1	9.3
160	4.9	2.6	300	16.3	10.6
180	5.9	3.4	320	18.8	11.9

Wiebe states that the results of distillation tests made with the metal and the glass apparatus are not comparable, owing to the fact that the vapor in the two types of apparatus is at different temperatures for the same temperature of the liquid. An example is given in which the temperature of the liquid was 178° in each type of apparatus, while the temperature of the vapor was 150° in the metal apparatus, but only 136° in the glass apparatus. Wiebe concludes that even though the readings in the two apparatus are corrected, the fractions at the lower temperature will be different and hence a standard method of distillation is necessary.

Tables of stem corrections have very little value unless it is certain that the thermometers used and the conditions under which the tests are made are similar to those for which the tables were computed.

IV. REASONS FOR VARIATIONS IN STEM CORRECTIONS

Using any one type of flask, differences in the emergent stem correction for a given reading of the thermometer may be caused by the following: (1) Unsuitable thermometers; (2) variations in rate of rise of temperature.

1. UNSUITABLE THERMOMETERS

Thermometers having an enlargement in the capillary between the ice point and the scale so that the graduations shall begin at a higher temperature, say 200° , should not be used in distillation flasks, unless the conditions are such that the temperature of the enlargement is the same as that of the bulb.

Although with such a thermometer the emergent stem correction is smaller, its variations may be larger, due to variations in the temperature of the enlargement under different conditions of use.

2. VARIATIONS IN RATE OF RISE OF TEMPERATURE

The rate of rise can be controlled fairly well, and as is seen from the curves (particularly Fig. 4), the faster the rate of rise, the smaller the emergent stem correction. This is due to the fact that for a faster rate of rise the flask and the air above it become hotter, due to the larger flame used. Care should therefore be taken that the rate of rise is controlled according to specifications.

V. EFFECT OF RADIATION AND OF CONDENSED VAPOR ON TEMPERATURE MEASUREMENTS

While discussing variations due to emergent stem corrections it is not out of place to call attention to other causes of variation in the indications of the thermometer, such as (a) heating of the bulb by radiation from the hotter oil and (b) cooling of the bulb due to radiation to colder objects and to the condensing oil which runs down over the bulb.

The magnitude of the error due to radiation from the oil and hot flask is difficult to ascertain, and is probably negligible except where the bulb and stem are close to the oil. It seems desirable for this reason to have the thermometer bulb at some distance above the level of the oil.

Table 4 shows the magnitude of the cooling effects for one type of flask. Two thermometers were read simultaneously, one being shielded, the other not. A perforated conical aluminum shield, having a length about equal to that of the bulb and fitting tightly around the stem just above the bulb, was used. The difference between the indications of the shielded and the unshielded thermometers amounted to as much as 3° . This experiment shows the cooling effect on the bulb only; the effect on the stem is entirely negligible. Varying amounts of condensing oil vapor will cause variations in the readings for the same temperature of the vapor. It is probably inconvenient in industrial work to eliminate this source of error in the present type of distillation flask.

TABLE 4

Effect of Radiation and Condensed Vapors on Thermometer Reading

Readings	Difference between thermometer readings, neither thermometer shielded	Difference between readings, one thermometer shielded	Effect of shielding
Degrees	Degrees	Degrees	Degrees
200	+1.0	0.0	+1.0
225	.0	-3.0	+3.0
250	.0	-2.5	+2.5
275	+ .2	-3.0	+3.2
300	+ .5	-2.5	+3.0
325	- .5	-3.0	+2.5
350	-1.2	-3.0	+1.8

VI. EFFECT OF ERROR IN TEMPERATURE MEASUREMENT ON FRACTIONS DISTILLED

It is evident, since the amount of distillate increases with increasing temperature, that, using the same sample of oil, if at one time a fraction is taken off at a certain temperature and at another time this fraction is taken off at a different temperature, the results in the two cases will not agree.

The specifications by which distillation tests are governed state that the fractions shall be taken off at certain specified temperatures, which are to be read off on a thermometer the type of which is also specified. From the previous discussion of the subject of emergent stem corrections it is obvious that unless this correction is taken account of the results obtained by assuming that the reading on the thermometer represents the specified temperature will depend not only upon the reading of the thermometer, but also upon the conditions of stem temperature prevailing at the time of test. On the other hand, if the specified temperatures are taken to mean the temperature of the bulb of the thermometer, and fractions are taken off at readings on the thermometer which represent the specified temperature of the bulb, the results obtained will not agree with those obtained by taking off the fractions at uncorrected readings.

It was at first intended to run a series of distillations under these two conditions and to compare the results thus obtained; later it was decided that a very good idea of the magnitude of the disagreement between the two methods could be obtained from the data on percentage distillates published by the National Electric Light Association in their report of June 2, 1911. From the data a curve was drawn representing the percentage distillates for the Lunge flask corresponding to various thermometer readings. Using this curve, two series of percentage distillates were taken off. In the first case the specified temperatures were taken to mean thermometer readings; the fractions were therefore taken off at readings 170° , 205° , 245° , etc. (Table 5); in the second case the specified temperatures were taken to mean bulb temperatures and the fractions were taken off at readings 166° , 200° , 238° , etc. (Table 6), these readings being those which correspond to bulb temperatures of 170° , 205° , 245° , etc.

TABLE 5

Fractions	I to	II to	III to	IV to	V to	VI to	VII above
Points specified	170°	205°	245°	270°	320°	360°	360°
Per cent distillate	1.4	3.6	54.0	67.6	82.5	92.1	99.5

TABLE 6

Fractions	I to	II to	III to	IV to	V to	VI to	VII above
Points specified	170°	205°	245°	270°	320°	360°	360°
Readings on thermometer to give specified temperature of bulb	^a 166°	200°	238°	262°	308°	344°	344°
Per cent distillate	^a 1.0	3.2	46.8	63.5	79.4	88.0	99.5

^a Extrapolations.

The differences between the fractions using the two methods are given below.

TABLE 7

Fraction	I	II	III	IV	V	VI	VII
Differences in per cent distillates	0.4	0.4	7.2	4.1	3.1	4.1	0.0

The above figures must be taken as only illustrative of the effect of taking into account or disregarding emergent stem corrections and should not be used as a means for correcting results.

VII. A SUGGESTED METHOD OF TESTING THERMOMETERS USED IN DISTILLATION APPARATUS

The total correction which must be applied to the readings of a thermometer used in a distillation flask to reduce these readings to those corresponding to the temperature of the vapor may be considered as made up of three parts—(a) the scale correction, i. e., the correction to the reading of the thermometer which could be obtained by comparing its readings when totally immersed with the corrected readings of a standard thermometer; (b) the emergent stem correction, which, as heretofore explained,

is the correction due to the emergent stem; and (c) the corrections which should be applied to allow for effect of condensing oil and of radiation. Tables of emergent stem corrections have been previously given, and the application of these corrections to the thermometer readings would suffice to reduce the readings to true temperatures, if it could be assumed that the other corrections mentioned above were negligible. This is seldom the case, however, as the scale correction itself in some cases might exceed 5° . To provide a means whereby the total correction may be determined to an accuracy sufficient for most oil distillations—i. e., about 1° or 2° —the following method is suggested:

The method involves the boiling, in the distillation flask to be used, of certain substances of known "boiling points." For distillations above 200° the substances suggested are naphthalene, B. P. 218° , and anthracene, B. P. 340° . The thermometer to be tested is placed as for a distillation test and read with its bulb immersed in the vapors of these substances. The difference between the temperature of the vapor and the reading will be the total correction to the thermometer at these two points, provided the conditions correspond to those of an actual distillation, and if this correction could be considered as changing linearly a straight line drawn between the two corrections plotted on coordinate paper would represent the total corrections to the thermometer at intermediate points. It has been shown, however, that one of the factors in the total correction, viz, the emergent stem correction, does not change linearly with respect to temperatures. An inspection of the correction curves in Figs. 2, 3, 4, and 5 shows that the curves depart from a straight line by as much as 2° , and hence, if a greater accuracy than this is desired, an allowance for this curvature must be made. Fig. 6 shows the result of a calibration by this method of one of the thermometers used in this investigation. Its scale corrections were already known. Its reading in the vapor of naphthalene was 214° , in anthracene vapor 329° ; the total correction at the reading 214° was therefore $+4^{\circ}$, and at 329° , $+11^{\circ}$. The dotted line in Fig. 6 is a straight line drawn between the two corrections for readings at 214° and 329° . In drawing the solid line allowance has been

made for the fact that the emergent stem corrections do not change linearly. In this method the scale correction and the corrections mentioned on page 16 must be considered as changing linearly between the two readings, unless the scale corrections have been previously determined, in which case allowance may be made if these scale corrections do not change uniformly.

For the convenience of those who may use this method, a table of "deviation" corrections is given, namely, Table 8. These corrections were obtained by plotting a curve corresponding to the

FIG. 6.—Total correction curve. Thermometer used in ordinary side-neck distillation flask

average of the emergent stem corrections given in Table 1 for the four types of flasks and finding the amount that the curve thus obtained deviated from a straight line passing through readings corresponding to temperatures of 218° and 340°.

TABLE 8
Deviation Corrections

Readings.....	200°	220°	240°	260°	280°	300°	320°	340°	350°
Corrections.....	+0°.7	-0°.1	-0°.6	-0°.8	-0°.8	-0°.6	-0°.2	+0°.4	+0°.7

To find by this method, then, the total corrections to a thermometer in a distillation flask, first find the total corrections to

the thermometer for readings in naphthalene and anthracene, then from these two corrections construct a table or a plot representing a series of corrections changing linearly between 200° and 350° . Finally, to allow for the nonuniformity of the stem correction curve, add algebraically to the corrections found by linear interpolation, as indicated above, the deviation corrections given in Table 8, and the resulting corrections will be the total corrections to the thermometer, within certain limits of error which have already been discussed.

In boiling either naphthalene or anthracene the condensed substance will solidify in the outlet tube and close it, and, except in the retort, the vapor will then rise in the neck of the flask. If the flame is hot enough, the condensation line of the vapor will eventually reach the top of the neck of the flask.

The height of the vapor of naphthalene and anthracene in the neck of the flask should be the same as it is in the creosote-oil distillation; but as in the latter case the condensation line is not sharp, special experiments were made to determine just where the condensation line with naphthalene and anthracene should be in order that the above condition should be satisfied. These experiments showed that the thermometer should be read in naphthalene and anthracene when the line of condensation reached a point about 3 cm above the bottom of the outlet tube (in the retort the top is about 3 cm above the bottom of the outlet tube).

The following directions should be observed in determining the total corrections of a distillation thermometer by measurements in the vapors of boiling naphthalene and anthracene:

1. The flask must be shielded and the thermometer placed according to the specifications for distilling creosote oil with the type of flask used.
2. The substance (naphthalene or anthracene) must be melted and boiling commenced slowly, and the thermometer must be read when the condensation line reaches a point 3 cm above the bottom of the outlet tube.
3. Care must be taken that there is an opening in the cork to insure that there shall be no excess of pressure inside the flask.

The method was not applied to the Hempel flask with beads in the neck. There may be some difficulty in this case owing to the

clogging of the beads by the solidified anthracene, necessitating special heating of the neck of the flask.

VIII. THE BOILING POINT OF ANTHRACENE

The boiling point of naphthalene has been accurately determined. The boiling point of anthracene was not known accurately. A series of observations showed that in the glass flasks the temperature of the vapor is 340° at standard atmospheric pressure. Investigation showed that the ordinary purified yellow anthracene could be used in obtaining the boiling point of the substance. A limited number of experiments showed no significant difference between the boiling point of the yellow variety and the most highly purified white material. The temperature was measured with both a mercurial thermometer and a thermocouple. The mean gave 340° as the temperature of the vapor of anthracene boiling at standard atmospheric pressure and it can probably be depended upon to 1° . The temperature of boiling substances is influenced by changes in atmospheric pressure. The temperature of boiling water increases approximately $0^{\circ}.04$ per millimeter increase of pressure, naphthalene $0^{\circ}.05$ per millimeter, and anthracene $0^{\circ}.07$ per millimeter. This last figure is only approximate and was computed from the known relations between boiling points and pressures. It can be seen that in the case of naphthalene and anthracene a range of variation of a degree in the temperature of the vapor is possibly due to variations in the local barometric pressure. However, if a barometer is not at hand, the correction can be assumed to be negligible under normal weather conditions for localities not more than a few hundred feet above sea level.

IX. SUMMARY

Emergent stem correction errors in creosote oil distillation flasks are discussed, and corrections applicable to a particular type of thermometer when used in any one of four different types of distillation flasks were determined. Tables of corrections are given applicable to these four types of flasks.

The emergent stem correction may amount to as much as 17° at 350° .

The emergent stem corrections applicable to the different types of flasks do not differ much from each other for similar thermometers read at the same temperature, the greatest difference being 2° . This is one reason why the published results of distillation tests made with different types of flasks agree fairly well. From the standpoint of temperature measurement and in particular emergent stem correction there is therefore little to choose between the different types of flasks.

The most desirable type of mercurial thermometer to use is probably the continuous-scale graduated-stem type divided into degrees, the length of a degree interval being about 0.8 mm. The thermometer should be "filled under pressure" to prevent distillation of the mercury, should be well annealed to prevent a rise of the readings with continued heating, and, if possible, should have its scale corrections determined by a standardizing laboratory. The undesirability of using a thermometer with an enlargement in the stem between the bulb and the scale has been discussed. Thermometers could be scaled to read correctly when immersed to the point at which they were to be used in distillation flasks by making use of the corrections obtained in this paper. However, owing to the confusion which might arise in the use of such instruments and the difficulty which would be encountered in attempting to graduate or test them, such a procedure is hardly thought justified at the present time.

A method suggested for finding the total correction to thermometers used in distillation flasks by reading the thermometer first in the vapor of boiling naphthalene and then in the vapor of boiling anthracene was investigated and found to be practicable and sufficiently accurate (about 1° or 2°) for distillation practice.

A significant disagreement in the fractions will result if corrections are made in one case for emergent stem and not in another.

As a final conclusion, it might be said that even if the methods suggested here are not adopted the investigation will at least call attention to the great possibility of wide disagreement in the results obtained in different commercial practices where mercurial thermometers are used with utter disregard to the emergent stem correction errors. To repeat what has already been said,

how can the results in different laboratories be expected to check when in one case a correction is made for the emergent stem, in another it is not, and in another perhaps a thermometer scaled for partial immersion may be used under conditions totally different from those for which it was intended? It is to be hoped that this paper will assist in bringing about greater uniformity in the making of distillation tests, especially from the standpoint of temperature measurements.

WASHINGTON, March 29, 1915.