NBS MONOGRAPH 28

Causes of Variation in Chemical Analyses and Physical Tests of Portland Cement

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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B. Leonard Bean and John R. Dise

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Preface

It is believed that reference to this Monograph will enable a cement-testing laboratory to identify and eliminate many of the more frequently encountered causes of random or constant errors in test results.

In consideration of the fact that some laboratories exhibit a tendency to obtain high values in comparison with computed averages for certain tests, while other laboratories are always low; the reader is cautioned to bear in mind that the attainment of reproducibility through elimination of one particular source of variation does not, necessarily, indicate that accuracy has also been attained. It therefore seems advisable for each laboratory to conduct general inspections of equipment and procedures at regular intervals, and it is suggested that this compilation would be a helpful reference in these operations.

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Causes of Variation in Chemical Analyses and Physical Tests of Portland Cement

B. Leonard Bean and John R. Dise

(June 7, 1960)

Variations in testing that could lead to the rejection of a material fully conforming to specification requirements, or the acceptance of a material with undesirable chemical or physical properties, are apparent in the results reported by laboratories participating in comparative tests of portland cements. Many of the causes for variation in chemical analyses and physical test results are listed in this discussion, and remedies for some of the more frequently encountered deficiencies in apparatus and methods are suggested. Particular consideration is given to problems which do not seem to have been covered in sufficient detail in previous discussions of cement testing procedures. Literature references are given for additional information.

1. Introduction

Many of the organizations engaged in the testing of portland cements have found it advantageous to use frequent interlaboratory tests on carefully prepared samples of the material to evaluate the performance of the laboratories involved. A report on a recent comprehensive effort of this kind was submitted to the American Society for Testing Materials at its 1959 Annual Meeting in Atlantic City, New Jersey [1].*

All too frequently, variations in testing that could lead to the rejection of a portland cement fully conforming to specification requirements, or the acceptance of one with undesirable properties, are apparent in the results reported by the participants in these programs. This is due in large measure to the fact that many steps are involved in making the standard chemical analyses and physical tests, and that the results are sometimes greatly influenced by seemingly minor variations in equipment and procedures. Many of these influences have been taken into account in the preparation of the standard methods of test. However, it is not uncommon to find that inadequate attention has been given to certain important specification requirements because their significance is not fully understood, or that essential considerations are overlooked simply because they are not readily apparent. For these reasons it has been suggested that a discussion of the causes of variation, in which particular attention is given to matters not already covered in detail in current publications, might prove helpful to laboratories participating in comparative test programs such as the recently established Cement Reference Sample Program of NBS.

Part I. Possible Causes of Error in Cement Analysis

B. Leonard Bean

The suggestion was made that this author compile information on analytical techniques, both from personal experience and from the literature, which might aid analysts in securing uniform and reliable results. The suggestions which follow, it is hoped, will benefit one who is seeking some means to bring his own results into line with the average values reported by all chemists taking part in a co-operative study.

2. General Precautions

2.1. The Weighing Operation

The weights used in interlaboratory analyses should conform to Class S–1 requirements of NBS Circ. 547. The weights of 1 g and larger units should preferably be made of stainless steel or other corrosion and abrasion-resistant alloy not requiring protective coating. These weights which are used for regular work should frequently be checked against a reference set of standard weights which conform to Class S (or Class M) requirements. This reference set should contain weights from 50 g to 10 mg. The material used to adjust the weights should have properties of corrosion and abrasion resistance comparable to the material of the weights. Nickel-chromium resistance wire is recommended as adjusting material as it is highly constant and generally available. If weights have been variable, the cavities should be cleaned carefully and all old adjusting material should be removed and drilled out when necessary. Guard against traces of materials such as oil, corrosion products, or miscellaneous unidentified adjusting material. Whenever a weight from a working set is known to have been dropped, it should be cleaned and

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*Figures in brackets indicate the literature references at the end of this Monograph.

its weight checked carefully against weights from a reference set before further use. In such instances and whenever the full accuracy of the weights is required the certified corrections to each individual standard weight should be made as part of the weighing procedure. When weights from a reference set which carries NBS certification are involved in an accident such as being dropped to the floor, the Bureau calibration of the weights involved in the accident must be considered to be in doubt. Such weights should be returned to the Bureau for recalibration.

Balances are now sold with many added features to speed the weighing process: damping devices, notched beams to accommodate poises, chains, projected direct-reading scales, and sets of built-in weights. These can save weighing time. It must be recognized, however, that each such added device may contribute to the error of the weighing. Tests must be made occasionally to assure freedom from significant errors.

Electrostatic effects must be guarded against in all weighing operations. Sometimes grounding the balance is helpful. The claim is made for certain radioactive materials on the market that they may be placed in the balance to ionize the air and permit the charge to flow away from insulating materials. The moisture that may condense on a dry crucible on a balance pan, due to humid atmospheres, can be compensated to some extent by placing a tare crucible on the other balance pan; this expedient cannot be used, of course, with a balance of the single-arm type with only one pan.

A balance or set of weights which is suspect may be returned to the manufacturer for repairs, adjustment, or modernization. There are several persons who travel through the country specializing in repairing and adjusting balances and weights. Although they probably cannot maintain all the equipment and facilities which a balance or weight manufacturer has at his disposal, use of their services does minimize the length of time the balance or set of weights is out of use in the laboratory. In addition, the balance or weights does not require transportation after adjustment on the spot. A balance put into excellent condition by a manufacturer may suffer a mishap while being returned to its home laboratory, which undoes all the careful adjustment made on it.

Excellent information on testing balances and the terminology used in describing the performance of balances may be found in Federal Specification AAA–B–92 [2]; and articles published in Analytical Chemistry by the Committee on Balances and Weights in the Division of Analytical Chemistry of the American Chemical Society [3], and by Lashoff and Macurdy [4].

2.2. Ignition Procedures

Where platinum crucibles are used, they should, in general, be made of pure platinum, that is, not intentionally alloyed with other platinum metals. At the temperatures of 1,100 and 1,200 °C used for certain ignitions in cement analysis, other alloying metals are liable to volatilize slowly and vitiate the results obtained. Such alloyed crucibles or lids are sold and used for some purposes because they are stiffer and do not deform as readily as pure platinum. Some manufacturers have equipped pure platinum crucibles with alloyed lids to obviate sticking of crucible and lid. Such practice may be permissible at lower temperatures than those used in cement analysis. Any empty crucible and lid which consistently loses more than 0.2 mg per hr at 1,200 °C should be suspected [5]. Use of an empty alloyed crucible and correction for the loss it sustains applied to the crucibles containing precipitates is liable to lead to erroneous results. The alloying metal covered with precipitate will probably not volatilize at the same rate as in the empty crucible.

The beginning of the ignition of a precipitate contained in a wet filter paper should be made at a rate slow enough so that no spattering, popping, or flaming occurs. After the moisture is expelled the paper should be "smoked off." Then, when all danger of flaming is past, the crucible may be placed in a muffle furnace which is at the proper temperature for final ignition.

Some check on the temperatures of the muffle furnaces is advisable from time to time. Contamination of thermocouples may occur resulting in erroneous temperature readings. A standard thermocouple and portable potentiometer may be used to check the accuracy of the thermocouples and temperature-indicating devices ordinarily used. The National Bureau of Standards issues a copper melting-point standard, No. 45c, which melts at 1,083.3 °C. Use of pyrometric cones may be helpful in checking temperatures [6].

E. T. Carlson [7] recommends a modification of Roberts' technique [8] utilizing the melting point of potassium sulfate (1,069.1 °C) as a check on muffle temperatures. Carlson breaks up the K_2SO_4 with a mortar and pestle and places about a gram in each of several covered platinum crucibles. These are placed at several points in the furnace with the indicated temperature somewhat below 1,069 °C. They are removed and inspected at about 15 min intervals. The indicated temperature of the muffle is raised by 10 °C intervals. In this manner the temperature of the various zones in the furnace can be estimated. Almost invariably the K_2SO_4 in the back of the furnace will melt first and that at the front last. One must not be confused by an inversion that takes place in K_2SO_4 at about 583 °C, but without melting. The K_2SO_4 was first crushed to avoid the spattering that takes place on inversion.

When igniting precipitates in a number of crucibles, six, for example, it is well to reverse the order of the crucibles when about half the ignition period has elapsed. Thus crucible No. 1 which may at first have been at the front of the furnace in the coolest position will spend the remainder of the time in the back of the furnace which is usually the hottest zone. (In some furnaces, the middle portion may be hottest.) It should be borne in mind also that the temperature shown by the indicating device is the temperature of the end of the thermocouple. If the thermocouple is covered by a protective tube, as is usually the case, the temperature of any one crucible on or near the floor of the furnace will probably differ somewhat from the thermocouple temperature. The position of the thermocouple is important. It should be near the middle of the heated zone. If it is accidentally bumped while placing crucibles in the furnace, it may be so misplaced that it no longer indicates the average furnace temperature. The thermocouple position should be checked every time the muffle furnace is used.

2.3. Desiccation

No absolute rules can be given for desiccation. The time and technique which will give satisfactory desiccation while the crucibles are cooling to room temperature so that they can be weighed, may be a function of many factors: 1. Size of the desiccator, 2. the number of crucibles, their weight, and the weight of the contents, 3. the character or composition of the contents, 4. the material of which the desiccator is constructed, 5. the temperature and relative humidity in the room, 6. the desiccant used, and 7. the degree to which the lid fits the desiccator. An excellent discussion of the subject is given by Hillebrand, Lundell, Bright, and Hoffman [9]. In general, sulfuric acid or anhydrous magnesium perchlorate are the preferred desiccants for cement analysis. Either is satisfactory in glass desiccators; obviously, sulfuric acid should not be used in an aluminum desiccator. The lid should be ground to fit the desiccator on which it is used. Generally no difficulty is encountered in purchasing glass desiccators whose lids fit properly. A well-constructed aluminum desiccator has the advantage over glass of speeding the cooling of ignited precipitates, but, in the writer's experience, the lids rarely fit properly and require regrinding before use. It has been claimed that the cost of such grinding by the manufacturer would be prohibitive. Ordinary silicone stop cock grease (not High-Vac) is a good sealing and lubricating material for aluminum desiccators if the lids fit. Petrolatum is satisfactory for glass desiccators.

When the lid of a desiccator fits and is lubricated properly, air will be heard to rush in when the cooling period (usually 30 to 60 min) is over and the lid is opened slightly by cautiously sliding or by opening a valve, if the desiccator is so equipped. If this sound is not heard one is always uncertain as to how much moisture-laden room air may have entered the desiccator during the cooling period. Care must be exercised to avoid blowing precipitates out of the crucibles by this inrushing air. If a desiccator has been found not to have been kept tightly closed overnight, it is good practice to recharge with fresh desiccant.

Hot crucibles should never be placed in a desiccator at quitting time and then weighed in the morning. Such weights will seldom agree with those obtained after the usual cooling period. Weighings made just prior to quitting time should be viewed with suspicion if there has been any tendency to shorten the usual desiccating period in favor of leaving at a given hour. Crucibles and contents which appear to gain weight on the balance during weighing may have been left in the desiccator for too short a period. Slightly warm crucibles cause convection currents in the balance and affect it in other ways which generally result in a weight which is too low and somewhat unstable during the weighing period. This latter phenomenon may be superimposed on the uncertainty involved in weighing hygroscopic materials which have been properly cooled for a sufficient length of time in a good desiccator.

Ignited silica, ammonium hydroxide group (R_2O_3), and calcium oxide should be treated as hygroscopic materials, or potentially so, and kept tightly covered in the crucible during desiccation and weighing. Calcium oxide should be desiccated over magnesium perchlorate or preferably, phosphorus pentoxide and weighed as rapidly as possible.

It is not easy to know when a desiccator should be recharged with fresh desiccant. G. Boehm [9, p. 33, note 22] describes a technique [1] for checking the effectiveness of sulfuric acid as a drying agent, by using acid containing 18 g of $BaSO_4$ dissolved in each liter. Some analysts have found it useful to mix a few pieces of indicating Drierite with magnesium perchlorate in a desiccator; when the blue color changes to pink the desiccant should be discarded. In fact, it probably should have been discarded prior to that. Most laboratories do not have the vacuum equipment required to reactivate magnesium perchlorate and prefer to discard the used material and recharge the desiccator with fresh material.

2.4. Additional Precautions

Filter paper should be visually inspected before folding and, of course, discarded if evidence of a weak spot or a hole is detected.

A piece of glass broken from a wash bottle tip is an occasional cause of high results.

Failure to decompose the sample of cement completely in the initial hydrochloric acid treatment can lead to erroneous results for almost all subsequent determinations.

Silicone stopcock grease is not recommended for use in burettes. It may tend partially to water-

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[1] ". . . no precipitate will form in sulfuric acid containing 18 g of dissolved barium sulfate per liter until enough water has been taken up to reduce the concentration of acid to about 93 percent. Between 93 and 84 percent, needlelike crystals of $BaSO_4 \cdot 2H_2SO_4 \cdot H_2O$ separate freely. On further dilution by absorbed water, the needlelike crystals change to fine crystals of $BaSO_4$, at which point the acid is no longer effective as a drying agent."

proof the inner surface of the burette. Silicone grease can be removed from a burette by use of fuming sulfuric acid. Extreme caution must be exercised in using fuming sulfuric to avoid injury.

Individual Determinations

Many of the statements which follow are intended to apply to comparative analyses in which, presumably, the utmost in accuracy is sought. Less rigorous techniques may be satisfactory for routine analyses, where conformance to specification requirements is not in doubt.

3. Silicon Dioxide

High Results. For the determination of silica, ignition temperature of 1,200°C for at least one hour is recommended, with subsequent heating periods of 30 min at the same temperature until constant weight within 0.2 mg has been established [10, also 9, p. 679].

Recovery of residual silica from the ignited ammonium hydroxide precipitate together with a correction for the silica recovered from a reagent blank can lead to high results. This is discussed on page 682 of [9], where the importance is stressed of adding as much iron and alumina to the reagent blank as is contained in the sample being analyzed. This is time-consuming and makes necessary the use of two blanks, if silica is to be recovered: one containing nothing but reagents in order to correct the R_2O_3 for filter paper ash; the other with added iron and alumina to carry down silica from the reagents and glassware. The present ASTM method [11] does not take this into account. The present Federal method [10, Method 1101, pars. 3.1.1 and 3.1.2] does not require the recovery of silica from the R_2O_3 because the amount involved is likely to be almost insignificant, the probable errors of recovery high, and the time required excessive.

Low Results. In careful work, the HF residue can generally be kept below 1 mg in cement analysis. If the referee (double dehydration) method is used this residue should seldom be more than half a milligram. Whenever the HF residue is much higher than these values, uncertainty in results for silica exists, usually tending to give low silica values. (Subsequent determinations of other constituents may also be affected.) Impurities may be weighed as chlorides during the ignition of the crude silica and as sulfates or oxides after treatment with HF and H_2SO_4. The error can be minimized by following the usual dilute HCl (1:99) and water washings of the precipitated silica with one or two washings with dilute H_2SO_4 (1:4) or by moistening the combined papers containing the silica with a few drops of dilute sulfuric acid before drying and igniting [12, also 9, p. 863]. Thorough washing of silica to remove contaminants is the best insurance against high HF residues. The higher the HF residue the more error can be introduced if the contaminants are not present in the same form before and after treatment with HF and H_2SO_4 [9, p. 681]. Special precautions need to be taken in washing when the optional ammonium-chloride method of silica separation is used. The bulk of the gelatinous silicic acid should be transferred to the funnel without dilution with water and the small volume of concentrated acid solution allowed to drain through. Thorough washing with hot HCl (1:99) and then hot water follow. It is useful to tear off small pieces of filter paper and use them together with a rubber policeman to scrub the inside of the beaker thoroughly in order to remove all adhering silica. This use of filter paper should be delayed until no more ferric chloride color can be seen. If the scraps of filter paper are added to the funnel too soon, their presence impedes the washing procedure and causes high HF residue.

Low results for silica can be caused by baking the second dehydration at temperatures over 110 °C or for longer than 1 hr. Such treatment can cause recombination of silica and magnesia, resulting in formation of an acid soluble silicate [9, p. 678].

4. Ammonium Hydroxide Group (R_2O_3)

High Results. In the determination of the ammonium hydroxide group ignition of R_2O_3 at 1,100 °C for one hour is recommended, followed by subsequent 30-min ignition periods until constancy of weight within 0.2 mg is indicated.

If 10 to 15 ml of HCl is not present before neutralization with NH_4OH, coprecipitation of some magnesium hydroxide with the R_2O_3 may occur. The presence of that quantity of HCl insures the formation of a sufficient amount of NH_4Cl so that the ionization of NH_4OH is depressed to a point where insufficient hydroxyl ion is present to exceed the solubility product of $Mg(OH)_2$. Ammonium chloride also tends to keep magnesium in solution by formation of an ammonia complex, assists in buffering the solution, and aids in coagulation of the precipitate.

Use of ammonium hydroxide which contains carbon dioxide can cause coprecipitation of calcium and magnesium carbonates. Reagent ammonium hydroxide bottles should be kept stoppered to prevent contamination. Digestion of the ammonical solution and precipitate should not be prolonged beyond 10 min. Otherwise CO_2 may be absorbed from the air.

The results for R_2O_3 may be affected by the completeness of recovery of silica in the previous step. Any silica which has escaped the double dehydration or ammonium-chloride separation will cause high results for R_2O_3 unless a careful recovery of residual silica is made with due regard for a properly determined blank. In addition, a reagent blank correction must be made to correct for the impurities in the reagents and for the filter paper ash. This blank will generally amount to about 0.5 mg.

Low Results. Failure to neutralize with ammonia to the exact end point will cause low results.

Stopping short of the methyl-red end point will leave some hydroxides unprecipitated. Going past the end point of methyl red by more than the one drop of 1:1 NH_4OH prescribed, will redissolve alumina and cause low results. The presence of any iron in the ferrous condition will cause results to be low because ferrous hydroxide is not quantitatively precipitated at this *p*H. Whenever ferrous iron is suspected, such as by a blackened precipitate, redissolve with hydrochloric acid, add four or five drops of concentrated nitric acid, and boil for a few minutes to oxidize all the iron. Then precipitate with NH_4OH as before. It may be necessary to add more methyl-red indicator from time to time because of the effect of the nitric acid on the indicator.

In the event the first precipitation of R_2O_3 has been completed at the end of the working day, the filter paper together with its precipitate should not be treated with acid, pulped up, and allowed to stand overnight in this condition. It may even be better not to do this over a lunch hour. In a strongly acidic environment organic matter from the filter paper pulp tends to complex some of the aluminum and prevent completeness of precipitation on subsequent treatment with NH_4OH [9, pp. 66 and 98]. In this respect filter paper may act a little like tartaric or citric acid. Use of filter paper for this separation which is less retentive than Whatman #40 is not recommended because some $Fe(OH)_3$ might pass through.

5. Total Iron as Ferric Oxide

In the determination of iron, the reduction process with stannous chloride is critical. If less than the required amount is added, results will be low. An excess of one drop after the yellow color of ferric ions disappears, will give satisfactory results provided the specified concentration of stannous chloride is used. This solution has a tendency to become more concentrated on standing in the presence of metallic tin. After cooling to room temperature and adding the mercuric chloride solution, a *small amount* of white silky precipitate should be in evidence. If none appears, insufficient stannous chloride was used and the determination should be repeated. The presence of much mercurous chloride is also objectionable because it may become oxidized. If the precipitate is gray, it indicates the presence of finely divided elemental mercury because too much stannous chloride had been used. In the latter event, also repeat the determination because the results will be high [12, pp. 573 and 580].

After reduction with stannous chloride, the solution should be cooled in a cool water bath and the remainder of the determination completed immediately thereafter. The reduced solution should not be allowed to stand overnight before titrating; air oxidation could cause low results.

6. Aluminum Oxide

Alumina is not actually determined in cement but is calculated by difference. The ammonium hydroxide precipitate in cement is assumed to consist of Al_2O_3, Fe_2O_3, TiO_2, P_2O_5, and residual SiO_2, together with possible traces of ZrO_2, V_2O_3, and Cr_2O_3. If, as is usually done, only the percentage of Fe_2O_3 is subtracted in the calculation, the results for Al_2O_3 will always be expected to be high by the amount of the other undetermined oxides. Any error in the R_2O_3 or Fe_2O_3 determinations will cause corresponding errors in the Al_2O_3 percentage reported. Note the suggestions given in the two preceding sections.

7. Calcium Oxide

High Results. In the determination of calcium oxide high results can be caused by failure to remove manganese, or to separate iron or alumina completely. When the optional method is used, the results obtained may be either high or low depending on the degree of washing with hot water. The suggestion in the specification method to use about 75 ml of hot water in washing will generally be satisfactory. However, variations in size of wash bottle tips, lung power of individual chemists, and the degree to which the precipitated calcium oxalate is churned up during washing, will cause variations in the result. It is suggested that each analyst occasionally analyze a sample of NBS Standard No. 177 and vary his operations throughout the analysis, particularly his washing technique, until he is able to get CaO values close to the certificate value for that cement. If the final titration in the presence of the added filter paper is prolonged unduly some of the paper will be oxidized. The one-hour standing period for precipitation of the oxalate is critical and should not be prolonged lest coprecipitation of magnesium result.

It is difficult to ignite CaO to constant weight for the referee determination. Ignition at 1,200 °C for at least one hour is recommended, followed by heating at the same temperature for 30-min periods until constancy of weight within 0.2 mg is indicated. The crucibles should have well fitting covers. Magnesium perchlorate is preferable to sulfuric acid for desiccating CaO. Phosphorus pentoxide is definitely superior to either. The second weighing of CaO should be made in reverse order from the first weighing to detect any errors due to standing in a desiccator already opened. It is especially necessary to rotate the crucibles in the furnace for the CaO ignition.

Low results. The presence of CO_2 during the precipitation of the ammonium hydroxide group can cause some of the calcium to precipitate there instead of later as the oxalate. This was discussed more fully under the R_2O_3 precipitation. Failure to wash the R_2O_3 precipitate adequately may leave calcium absorbed on the gelatinous precipitate.

8. Magnesium Oxide

There are many precautions to be heeded in order to obtain correct results for the gravimetric determination of magnesium. The subject is covered thoroughly in authoritative discussions by Hoffman and Lundell [13], Kolthoff and Sandell [12, p. 352–62], and Hillebrand, Lundell, Bright, and Hoffman [9, p. 636–40]. It is impossible in a paper of this type to list more than a few precautions to be observed in the determination of magnesium by either the phosphate or oxyquinolate method.

High Results. If manganese has not been removed—and it is not mandatory—high values for magnesium may be obtained by the oxyquinolate (optional) method.

Use of filter paper that is too coarse for filtration of R_2O_3 may allow iron to run through and increase the subsequent MgO values. A green precipitate of what is supposed to be magnesium oxyquinolate is evidence of contamination by iron. This may also be caused by failure to keep all the iron in the ferric state during its precipitation and washing. The magnesium oxyquinolate precipitate should be yellow.

Berman found [14] that this method gave high results even after manganese removal in the case of cements containing over 2 percent MgO.

When the phosphate (referee) method is used, any mechanical loss of calcium by creeping over or passing through the filter paper, will produce high results for magnesium because of coprecipitation of calcium phosphate.

If the ignition of magnesium ammonium phosphate in the presence of filter paper is carried out too rapidly, incorrect results, either high or low, may be obtained. The carbon may become partially fireproofed, rendering it impossible to burn it out [9, p. 637]. Or, the pyrophosphate may be slowly reduced and phosphorus volatilized. This latter action can ruin a platinum crucible. It is probably safer to ignite such precipitates in porcelain. The final ignition temperature should be 1,100 °C. At 1,200 °C, as permitted in some specification methods, the pyrophosphate slowly loses weight [12, p. 359].

Low Results. Absence of sufficient NH_4Cl during the R_2O_3 precipitation will cause some of the magnesium to precipitate there and subsequent values for MgO will be low.

During the calcium precipitation, if the oxalate is added to an ammoniacal solution, or if the solution and precipitate are boiled, some coprecipitation of magnesium will result [12, pp. 347–8]. Older specification methods recommended such procedure and some laboratories may still be using this technique, causing low magnesium results.

Berman [14] reported low results for MgO by the optional oxyquinolate method at levels less than 1 percent MgO.

In the phosphate (referee) method, supersaturated solutions of magnesium ammonium phosphate sometimes prevent complete precipitation in the case of low magnesia contents. This can be remedied by longer standing before filtration. It is generally advisable not to discard the filtrate from the phosphate separation for at least one day so that in the event postprecipitation does occur, the additional precipitate may be filtered off and added to the main precipitate. The blank should be treated in the same manner.

The amounts of ammonium oxalate called for in the specification methods for precipitation of calcium, generally do not leave enough excess to interfere with the magnesium determination according to Kolthoff and Sandell [12, p. 360]. In a few instances, in the author's experience, removal of oxalate and ammonium salts by evaporation with 75 ml of nitric acid [9, p. 133] and [12, p. 370] has increased the amount of magnesium ammonium phosphate that could be precipitated from certain cements. For very careful work, the additional time consumed may be warranted.

9. Sulfur Trioxide

9.1. Turbidimetric Method

When the optional, turbidimetric, method is used for the determination of sulfur trioxide, it is recommended that the operator study Rudy's paper [15] upon which the specification method was based. It contains many excellent suggestions and precautions to be observed: necessity for close control of temperature, changes of variables which require preparation of a new calibration curve, and necessity for maintaining fairly uniform $BaCl_2 \cdot 2H_2O$ crystal size. In connection with the latter point, a further precaution might be in order. It is usual to separate a rather large supply of the $BaCl_2 \cdot 2H_2O$ that will pass a No. 40 sieve and be retained on a No. 80 sieve, because a new calibration curve must be prepared every time a new batch is sieved. If, however, the operator should fall into the habit of shaking the bottle before taking out each scoop of crystals in the mistaken idea that mixing is desirable, he will actually change the crystal size by such shaking and obtain incorrect results. If four or five months have elapsed since the supply of crystals was sieved, it is advisable to resieve the $BaCl_2 \cdot 2H_2O$ and prepare a new calibration curve anyway. The crystal size may have changed just by standing.

Molded glass sedimentation tanks should be used for SO_3 determinations because hydrochloric acid solutions are liable to dissolve the material used to hold the glass together in forming the other type of tank.

Many of the suggestions given under 17.2 in Part II of this Monograph will be helpful in maintaining the turbidimeter in proper operating condition.

9.2. Gravimetric Method

From a study of the discussions on the determination of sulfur as barium sulfate in well-known textbooks such as those by Kolthoff and Sandell [12, pp. 322–335] and Hillebrand, Lundell, Bright, and Hoffman [9, pp. 711–722], one almost reaches the conclusion that it should be impossible to obtain the correct result by precipitating $BaSO_4$ in the presence of all the constituents of portland cement. Evidently, the reasonably good results generally obtained are because compensating errors just about balance each other. It will be noted in the certificate of analysis of NBS Standard Sample 177 that J. R. Spann, of our laboratory, reported a value of 1.59 percent SO_3 found in that cement. His method of determination included reduction of iron with hydroxylamine, addition of $BaCl_2$ solution rapidly and treatment of the ignited $BaSO_4$ with HF to remove silica [12, pp. 329, 330, 332]. In comparison with this value of 1.59 percent by a refined method, Spann obtained values of 1.56 percent and 1.63 percent SO_3 for the same cement by the ASTM and Federal referee specification method.

Failure to wash out all the occluded $BaCl_2$, or to burn off all the carbon of the filter paper, are probably the two most common causes of high results.

A digestion time much shorter than 12 hr generally leads to low results. After filtration, the precipitated $BaSO_4$ should be washed thoroughly with hot water, as stated in the specification method. The temptation to use dilute HCl solution for washing in order to remove iron stains must be avoided. Any $Fe_2(SO_4)_3$ present already has a lower molecular weight than its equivalent amount of $BaSO_4$ and will probably lose its SO_3 during ignition. Washing out the $Fe_2(SO_4)_3$ with acid will make the results still lower. The silica occluded possibly compensates for the lower weight caused by some of the sulfate coming down as $Fe_2(SO_4)_3$.

Slow ignition with good access of air will avoid reduction of $BaSO_4$.

10. Sulfide Sulfur

The evolution method for sulfide sulfur [16] is not regarded as being the most accurate available. However, it is fairly rapid, and even though all the assumptions upon which it is based may not hold rigidly, it is widely used throughout industry.

11. Loss on Ignition

High Results for Portland Cement. If the ignition temperature is too high, approaching 1,200 °C, SO_3 will be lost and some alkali volatilized. This will not only cause high results but will make it difficult to attain constant weight. A muffle furnace with an accurate temperature control is much to be preferred to ignition over a gas burner where the temperature can only be estimated. A 1 g sample of hot cement takes a long time to cool in a desiccator. If it is weighed slightly warm, low weights can be obtained which cause high results. Generally, at least 1 hr in the desiccator is required.

Sulfide-Containing Cements. Recent work of several investigators has shown that the regular ignition loss procedure used in the analysis of portland cement is not applicable to cements containing sulfide constituents. ASTM and Federal methods have been recently revised to include suitable corrections, based on an additional sulfate determination performed on the ignited material together with suitable calculations.

Chaiken [17] indicated that a direct ignition method utilizing a helium atmosphere gave satisfactory results for blast-furnace slag cements.

12. Sodium Oxide and Potassium Oxide

No attempt will be made to discuss the gravimetric determination of sodium oxide and potassium oxide in cement because it has been rather extensively covered in standard texts [9, pp. 925–930] and is now seldom used because of the greater ease of using flame photometry for the purpose.

In general, the flame photometric determination of potassium is rather straighforward and not subject to many sources of error within the accuracy generally required in cement analysis. The same cannot be said for the sodium determination. Errors, if present, generally tend to give slightly low results. The calcium present emits light in the same wave length region, 589 mμ, at which sodium is measured. Silica, present in varying amounts in cement, inhibits the emission of calcium. Because the standard solutions used do not normally contain silica, this leads to somewhat low results for sodium (by a few hundredths of a percent) unless the silica is removed [18]. In fact, one or two cements manufactured domestically contain such a low content of Na_2O that some flame photometers indicate a slight negative Na_2O content if silica is not removed. It is rather embarrassing to try to report a negative Na_2O content for a sample of cement. Flame photometers equipped with adjustable-width slits can be made to minimize this effect by using a rather narrow slit. Some older flame photometers of the motor-driven chopper type tend to give low results if the motor mountings become loose.

If the standard solutions are stored in glass bottles for several months, a small amount of sodium may be leached from the glass. This makes the actual sodium content of the solution higher than its nominal value. This can cause results reported for sodium oxide to be low by a few hundredths of a percent. An exchange between constitutents of the glass and the solution may occur under some conditions. The best remedy is to store the standard solutions in polyethylene bottles. These have the added advantage of not supplying any chips of glass to plug up the atomizer.

13. Phosphorus Pentoxide

The only referee specification method described for the determination of phosphorus pentoxide in cement is the ASTM method [11, pp. 77–78] in which the phosphorus is first isolated as ammonium phosphomolybdate and finally precipitated as magnesium ammonium phosphate, ignited, and weighed as magnesium pyrophosphate. This is a classical method. The majority of portland cements in this country probably contain not over one tenth of a percent P_2O_5. Results obtained are liable to be low because of the tendency of magnesium ammonium phosphate to form supersaturated solutions when the phosphorus content is low. It is not unusual for one to find that the blank weighs slightly more than the pyrophosphate obtained from one of the samples. Filtrates from the magnesium ammonium phosphate precipitation should not be discarded for several days. If any evidence of postprecipitation occurs, it should be filtered off, ignited, and the weight so obtained added to that already found. The blank should be treated in the same manner. The addition of the same measured volume of a standard solution of a phosphate to each sample as well as to the blank is one means of dealing with this difficulty. By this procedure there is less tendency for the formation of supersaturated solutions. The author would like to suggest that the respective ASTM and Federal committees give consideration to incorporation of a spectrophotometric method for determining the small amounts of phosphorus pentoxide in portland cements.

14. Manganic Oxide

The bismuthate method for determining manganic oxide [11, pp. 78–79 and 9, pp. 443–446] is fairly straightforward. A few precautions might be emphasized. It is important that the 1:33 HNO_3 added after oxidation with $NaBiO_3$, be completely freed of HNO_2 by boiling. In the titration it may take practice to recognize the end point. This is the point at which no further color change can be noted on further addition of sodium arsenite. The end point of the solution should be observed in a strong light and against a white background. Here, again, it is suggested that a spectrophotometric method for determining manganic oxide in portland cement would be a welcome addition to the specification methods.

15. Insoluble Residue

Halstead and Chaiken made a thorough study of the various factors affecting results obtained for the insoluble residue determination. Their published [19] recommendations have been used as the basis for recent revisions in ASTM and Federal specification methods. These modifications became necessary largely because the previous specification methods were not giving reproducible results for slag cements. These investigators recommended more specific requirements for heating equipment, dilution and digestion techniques, and thorough washing.

Concluding Remarks

It is generally recognized that analytical chemistry still is a mixture of science and art. As long as this situation prevails there will be differences in results obtained by different analysts for the same sample of material. The suggestions given in this paper are presented with the hope that they may be of some assistance in minimizing these differences in the analysis of cement.

The author acknowledges advice and assistance from the Mass and Scale Section of NBS on material pertaining to weighing and weighing equipment.

II. Possible Causes of Variation in Physical Tests of Portland Cements

(John R. Dise)

The physical properties of portland cement for which specification requirements and standard methods of test have been developed are Fineness, Soundness, Time of Setting, Heat of Hydration, Air Content of Mortar, Compressive Strength, and Tensile Strength. Fineness tests are made on the cement itself; soundness, time of setting, and heat of hydration tests are made on pastes consisting of cement and water; and the air content, compressive strength, and tensile strength tests are made on mortars consisting of cement, water, and a natural silica sand. In the following discussion of the possible causes of variation in these tests, particular attention is given to matters not already covered in detail in current publications. A limited amount of background information, including the year in which each method was first published by the American Society for Testing Materials, has been incorpoiated in the belief that it will foster greater appreciation for the efforts that have been expended in the development of the details of the tests. A brief description of each method has also been included in order that the discussion might have more meaning for a casual reader.

16. General Considerations in the Physical Testing of Cements

Important considerations which are generally applicable to several or all test procedures are discussed herein. It is intended that the pertinent parts of this section be considered an integral part of all subsequent sections.

16.1. Laboratory

(a) Temperature and relative humidity. The atmosphere of a laboratory can have a significant effect upon the behavior of mixes and test specimens, and for this reason the standard methods of test for cements require that the relative humidity be maintained at not less than 50 percent, and the temperature between 20.0 and 27.5 °C. In nearly all instances, automatically controlled conditioning equipment must be used in order to accomplish this. Drafts of air as well as low relative humidity can cause rapid evaporation of water from both mixes and specimens, and therefore mixing slabs and testing apparatus should be shielded from excessive drafts from air conditioner duct outlets, and also from direct sunlight and local sources of heat such as radiators, steam pipes, hot plates, autoclaves, and ovens.

(b) Lighting. The laboratory should be well illuminated with both natural and artificial light so that operators will have no difficulty in reading instruments and gages or dials on test apparatus.

(c) Arrangement of equipment. Equipment should be so located that all items are readily accessible to laboratory personnel. Piling and jamming of apparatus is to be avoided because these conditions promote carelessness in testing and create safety problems.

16.2. Treatment of Samples

Aeration of cement markedly affects its physical characteristics. For this reason, samples should be kept in air-tight, moisture-proof containers from the time they are taken until all tests are completed. Before testing, each sample should be passed through a No. 20 sieve in order to mix the sample, break up lumps, and remove foreign materials. Foreign materials and hardened lumps that do not break up on sieving or brushing should be discarded. An unusual amount of lumpy material may be an indication that moisture is present in the sample.

16.3. Mixing Water

In general, water fit for drinking is considered to be satisfactory for use in the testing of cement if it does not contain abnormal concentrations of a particular mineral or minerals. In instances where the suitability of the mixing water is questioned, it may be helpful to compare results obtained with that which is customarily used to results obtained with distilled water. Water from specimen storage tanks should not be used in the preparation of mixes.

It is required that the temperature of mixing water be maintained between 21.3 and 24.7 °C. Since the temperature in outside pipes will vary over a broad range in any 12-month period, it is usually necessary to make special provisions for bringing the temperature of tap water within the specified range prior to use.

With respect to time of setting and strength tests, it may be helpful to bear in mind that warm water hastens the hardening of cement and cold water retards it.

16.4. Measurement of Mixing Water

Mixing water should be measured in a clean glass graduate of the capacity and type specified in the applicable test method. In no case should the capacity be more than twice the volume required in the mix. A graduate which is not clean will not deliver the indicated amount of water, and for that reason frequent cleaning with dilute HCl, or a suitable glassware cleaning solution, is recommended.

Significant errors can sometimes be attributed to bad practices in reading graduates. A technique which avoids a number of possible variations consists of permitting the filled graduate to come to rest in a vertical position while it is held lightly at the top between the tips of two fingers; after which the elevation of the hand is adjusted to bring the bottom of the meniscus into a horizontal plane with the line of sight. The reading is made using the bottom of the meniscus.

The degree of uniformity being attained in these measurements should be checked periodically by weighing a series of measured volumes of the same magnitude on a mix balance. In this check, the graduate may be used as both the weighing container and the measuring device. It may be of interest to compare the weights thus obtained with the theoretical weight of a given volume of water, at the temperature involved, as derived from a standard volumetric table.

16.5. Standard Sands

The two gradations of natural silica sand from Ottawa, Illinois, used in making mortars for test purposes are customarily referred to as Standard Sand and Graded Standard Sand. Because a number of other gradations are also prepared by the processing plants, it is advisable for a laboratory to check each new shipment to ascertain that the proper sand has been received.

Contamination of the sand with an agent capable of entraining air in mortars sometimes occurs during processing, shipment, or storage. In many instances, the first evidence of an accident is a substantial increase in the air content, or a noticeable decrease in the strength, of test mortars. The test method for air-entrainment of standard Ottawa sand developed by DeFore and Corah [20] provides a convenient way in which to determine whether or not a given lot of sand is contaminated. Another is to run parallel check tests with sand which is considered to be uncontaminated.

16.6. Weighings

(a) Mix balance. The balance should be mounted on a plane, nonskid surface, on a vibration-free table with a rigid top. It should be shielded from direct sunlight and from stray currents of air. The permissible variation on balances at loads from 1 to 2 kg should be ±1 g.

(b) Mix weights. A common mistake in weighings is the use of mix weights which do not conform to the accuracy requirements of the various cement test methods. The difficulty can usually be attributed to the loss of slugs from a "two-piece" weight, or surface wear on the bottom of a solid weight. Because of the wear problem, it is most desirable that weights be lifted rather than skidded from one location to another, and that they be stored in a suitable wooden block when not in use. Every laboratory should be equipped with a spare set of new mix weights which can be used for periodic checking of the weights in daily use.

16.7. Timing Device

One or more timing devices, preferably those equipped with sweep second hands, should be so located that operators can conveniently observe the time from any point in the laboratory. In general, stop watches and pocket watches are less suitable than devices such as wall clocks and bench-type timers with large faces.

16.8. Oiling of Molds

Several of the test procedures require that the surfaces of molds be *thinly covered* with mineral oil. Nondetergent oil is preferable. Animal fats or vegetable oils should not be used in lieu of mineral oils.

16.9. Specimen Storage Facilities

Well made specimen storage facilities are essential. Some of the important considerations are as follows:

(a) Temperature. It is required that the temperature of the air in a moist cabinet or moist room, and the temperature of the water in a storage tank, be maintained between 21.3 and 24.7 °C. Many cement testing laboratories have found it necessary to install automatic controls in order to maintain storage temperatures within these limits.

(b) Relative humidity. It is required that the moist closet or moist room be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 percent. Specimens in storage should both look moist and feel moist.

(c) Storage of filled molds. It is intended that molds containing specimens be so stored that the upper surfaces are freely and uniformly exposed to the atmosphere of the moist storage unit. Molds should not be piled one upon the other in such a way that the plates of the upper molds exclude air from the specimens beneath. When sprays are used to humidify the moist storage unit, precautions should be taken to prevent water from falling upon the surfaces of freshly molded specimens.

16.10. Storage Period for Molded Specimens

Molded specimens are to be stored in a moist atmosphere until they have attained the age of 24 hr, at which time they are either tested or placed in water storage. In no case should specimens be removed from the molds until they are at least 20 hr old. If removed before 24 hr have elapsed, they should be placed on the shelves of the moist storage unit until the expiration of the 24-hr period.

16.11. Removal of Specimens from Molds

Specimens which are damaged during mold stripping operations should be discarded even though the damage may be considered to be slight.

Damage to test specimens, and also to molds, can be minimized if a rawhide hammer is used to apply the gentle taps usually needed to break the bond between mold and specimen. When stripping is completed, care should be taken to insure that mold parts are not inadvertently interchanged. In many instances, the dimensions of molds will not conform to specification requirements after an interchange of parts.

16.12. Marking Specimens

Reliable identification marks, preferably numerals, must be made on each test specimen if errors due to mistaken identity are to be avoided. The marks should be made on paste specimens, such as the Gillmore pat and the autoclave bar, immediately after they are formed. Identification tags should be used for freshly molded mortar specimens such as briquets and cubes. Under no circumstances should identification of a specimen depend upon its position on a shelf or table, or its proximity to a marked specimen. When mortar specimens, such as briquets and cubes, are removed from moist storage, the identification should be marked on the bottom surfaces with a soft crayon after the plates have been removed, but while the specimens are still in the molds. A graphite crayon will make very distinct and durable markings. Colored crayons should not be used because their marks may fade very quickly after specimens are placed in a water storage tank. The practice of using a sharp tool to scratch marks in the surfaces of mortar specimens is objectionable.

16.13. Trowels

The specifications require that the trowels used in making specimens have steel blades, 4 to 6 in. in length, with straight edges. Many operators have found that a slightly curved blade is of great assistance in making briquets. Such trowels are not to be used for other purposes. Damaged, blunt, or badly worn implements should not be used in any operation.

Excessive troweling of the paste in a Vicat ring or an autoclave bar mold often occurs after the surface of the material is torn in the process of slicing off that which projects above the top edge of the mold. Torn and uneven surfaces will be encountered frequently when an implement with blunt edges is employed, and therefore, it is advantageous to reserve a trowel with specially sharpened edges for use in the preparation of paste specimens.

16.14. Care and Cleaning of Apparatus

It is usually difficult, if not impossible, to obtain satisfactory test results with testing apparatus which is not clean, and for this reason much helpful information on cleaning has been incorporated in the various cement testing standards. Particular attention is invited to the fact that power-driven wire brushes and brushes with steel bristles are not suitable for use in cleaning molds.

Numerous situations arise wherein the laboratory finds it necessary to adjust the weight of test apparatus. A common mistake on these occasions is the failure to clean the apparatus prior to starting the adjustment. Another is the use of an inaccurate balance or weight for checking the work. These mistakes may have a very significant effect in tests employing a Vicat apparatus, the Gillmore needles, or a 400-ml measure.

17. The Determination of Fineness

The literature advises that fineness has long been recognized as one of the important physical properties of a cement. Early tests were based on the use of sieves and it was not until 1933 that a satisfactory replacement, the Turbidimeter Fineness Method, was introduced. Another new test, the Air Permeability Fineness Method, was introduced in 1943. Currently, both of these procedures are used as standard tests of cement.

17.1. Common Causes of Variations

Although the two fineness tests are distinctly different in technique and theory, there are a number of considerations which are of importance in either procedure. Some of these are presented herewith.

(1) *Problems with the standard fineness sample.* Although great care is taken in the blending and packaging of standard fineness samples, an occasional vial is found to be unsuitable for calibration purposes. It therefore is advisable to check the calibration of an instrument with a freshly opened sample whenever an unexpected difficulty is encountered. The risk of contaminating fresh samples with glass chips when the containers are opened may be reduced if opening is accomplished by inverting the vial and pressing the sides in the empty section against a thin, curved, high-resistance wire mounted between the terminals of a small transformer.

(2) *Exposure of apparatus to a source of variable heat.* The location of a turbidimeter or an air permeability apparatus above, or near, a source of variable heat such as a radiator, hot plate, oven, or autoclave, may cause considerable variation in test results.

(3) *Errors in calculations.* Among the common errors in calculations are the following: (a) Mistakes in the use of arithmetic, algebra, or logarithms. (b) Errors in the transcription of figures. (c) Mistakes in the use of slide rules and other calculating devices. (d) Selection of the wrong equation for a basic calculation.

(4) *Conversion of one test value to another by means of a factor.* Since the air permeability value is frequently about 1.7 times greater than the turbidimeter value, this factor is sometimes used in converting from one test value to the other. The practice is unsound because the results in these tests may be affected by such unpredictable

influences as differences in the ignition loss of a cement from day to day, or differences in the particle size distribution caused by variations in grinding, and values obtained in this manner may be seriously in error.

17.2. The Turbidimeter Fineness Test

The method of test for fineness of portland cement by the turbidimeter was introduced in a paper presented by L. A. Wagner in 1933 [21], and adopted as a standard test for cement in 1934. The procedure provides for the determination of specific surfaces by measurement of the turbidities of cements in liquid suspensions. Essentially, the turbidimeter consists of a source of light of constant intensity adjusted so that approximately parallel rays of light pass through a suspension of cement in clear white kerosene and impinge upon the sensitive plate of a photoelectric cell. The current generated by the cell is measured with a microammeter and the indicated reading is a measure of the turbidity of the suspension. The turbidity is in turn a measure of the surface area of the suspended cement.

An attractive feature of the test is that it provides a means for determining the particle size distribution as well as the specific surface of portland cements. This advantage has been nullified to a certain extent, however, by the difficulties a laboratory encounters in maintaining all of the various elements of the apparatus in satisfactory operating condition, and in teaching new operators to make the determination. It is important to bear in mind that the test is not intended for use in determining the fineness of materials other than portland cement.

17.2.1. Causes of Variations

A. *Apparatus*

(1) *Apparatus improperly mounted.* Vibration of the wall, or other object, to which the shelf supporting the sedimentation tank has been fastened may disturb the suspension and thereby affect test results. The shelf should be so located that the indicator on the scale on the side of the case reads zero when the center of the slot in the photoelectric cell is at the same elevation as the surface of the liquid in the tank.

(2) *Water cell in poor condition.* When the cell is not completely filled with water, a difference in transmission of light may be encountered above and below the water line. Also, a wavering film of matter inside the cell, or dirt and sedimentation lines on the glass ends of the cell, may influence the transmission of light. Keeping the cell completely filled with distilled water helps to avoid these problems.

(3) *Sedimentation tank in poor condition.* Scratches on the faces of the sedimentation tank may affect the transmission of light.

(4) *Reflection of light from bright surfaces inside turbidimeter case.* It is required that the interior of the case, and the exterior surfaces of the shelf, parabolic reflector, water cell, shield, and photoelectric cell hood be painted with a dull, optical black paint. In addition, the cabinet should be free of light reflecting dust, light-colored wiring, and shiny pieces of metal, and the gaskets used to seal the end of the water cell should be black.

(5) *Trouble in the electrical system.* Among the possible causes of difficulty are the following:

(a) Corrosion of electrical contacts and of the wires in the rheostats.

(b) Broken or loose connecting wires.

(c) A weak storage battery.

(d) Deterioration of the photoelectric cell. Photoelectric cells tend to have different fatigue characteristics, and therefore the cell always constitutes a potential source of difficulty even though it may have been in use for only a relatively short time.

(e) Looseness of the light bulb in its socket.

(f) Looseness of the reflector in its holder.

(g) Improper focusing of the light beam.

(h) Changes in the color, or intensity, of light emitted by a bulb after prolonged usage.

(i) Malfunctioning of voltage regulator. In laboratories where storage batteries have been replaced with a voltage regulator, malfunctioning of the regulator may constitute a problem.

(6) *Trouble with the microammeter.* Among the possible difficulties with the microammeter are the following:

(a) Damage in usage. A bent tip on the indicating hand may be an indication that the instrument has been overloaded.

(b) Internal corrosion of parts while instrument is in storage. A microammeter that has not been used for some time may have to be "exercised" before it will give uniform readings.

(c) Static charges on the instrument, or on the person using it. In some instances, it has been found necessary to ground both the microammeter and the operator.

(d) Location of the microammeter on a table containing magnetic material, or near some strong external magnetic field.

(7) *Variations in the character of the suspension.* Among the causes of variations in the suspension are the following:

(a) Appreciable variations in room temperatures.

(b) Use of a suspending medium other than clear, white kerosene.

(c) Traces of water in the kerosene. Water may cause foaming inside the tank during agitation of the suspension.

(d) Changes which often occur in oleic acid or linseed oil with age or exposure to heat and light. When difficulties are encountered in calibrating the apparatus, one of the first corrective actions should be to obtain fresh supplies of dispersing agent and kerosene. In general, technical grade oleic acid does not make a suitable dispersing agent.

(8) *Trouble with the timing buret.* Lint in the capillary of the buret, even though practically invisible, can slow down the flow of kerosene and thereby cause an appreciable variation in test results. Accordingly, utmost precautions should be taken to keep lint out of the buret, and the kerosene. As a safeguard, a filter made of No. 325 sieve cloth should be placed across the top of the buret whenever kerosene is being added, and the top opening should always be covered when the buret is not in use. Use of an improperly made, or a broken timing buret is to be avoided.

(9) *Trouble with the wet sieving apparatus.* Variations may occur as the result of plugging of holes in the spray nozzle, fluctuations in the pressure of the water passing through the spray nozzle, or errors in setting the water pressure. The latter difficulty may be caused by the use of an inaccurate pressure gage, a badly marked gage, or a gage of unsuitable capacity. Fluctuations in water pressure may be controlled by installing a pressure regulator in the water line back of the gage. A gas gage is not suitable for use with water unless it has been modified for such usage.

B. *Test Procedure*

(1) *Improper cleaning of tank prior to starting test.* If an oily film has accumulated on the interior faces of a tank, it should be removed with a "lint-free" wiper before a test is started. A "squeegee" of synthetic rubber makes a good wiping device. Wiping of a dry tank with a dry cloth is to be avoided because this may create static charges on the tank.

(2) *Variations in the agitation of suspensions.* Variations in the agitation process include allowing the cement to slide along the inner faces of the tank during rotation, and loss of a part of the suspension during alternate inversions. A number of laboratories have found that the use of a rotatable tank holder helps to promote uniformity in agitation operations. Customarily, this holder is provided with an adjustable screw in the center of the top to hold the glass lid of the tank in place, and so designed that it can be mounted to a wall by a pivot arrangement at its center, and rotated through 180° by means of a handle located near one end.

(3) *Fouling of tank faces during handling of the tank.* Care must be taken, especially during the agitation process, to avoid getting liquid or finger prints on the faces of the tank which are to be placed in the path of the light. If it becomes necessary to clean the tank after agitation, the possibility that a static charge may be created on the faces of the tank as a result of wiping must be considered.

(4) *Variations in the wet seiving operation.* Precautions should be taken to avoid loss of cement due to splashing at the beginning of the wet sieving operation.

(5) *Errors in calculation.* A common source of error is the failure to use standard procedures in the calculation and application of correction factors for No. 325 sieves.

17.3. The Air Permeability Fineness Test

The method of test for fineness of portland cement by the air permeability apparatus was introduced in a paper presented by R. L. Blaine in 1943 [22], and adopted as a standard test for cement in 1946. The procedure provides for the determination of specific surfaces by measurement of the rates of air flow through prepared beds of cements of definite porosities. Essentially, the air permeability apparatus consists of a permeability cell and a U-tube manometer filled to about the midpoint with a suitable liquid. The test is accomplished by attaching the cell, containing a carefully measured amount of cement, to the manometer and evacuating the air in one arm thereof until the liquid rises to a point just beneath the cell, after which the liquid is released and the time required for the meniscus to fall through a distance of approximately 5.5 cm recorded. The specific surface is then calculated from this time-of-flow determination.

The procedure is favored by many investigators because it is economical to perform, and because it can be used to determine the fineness of materials other than portland cements. It is to be noted that the test is not suitable for determining the specific surface of materials finer than 7,000 cm^2 per g, or the specific surface of porous materials such as diatomaceous earth and fly ashes containing porous cinders. Also, that in testing materials other than portland cement, it is generally necessary to use a specific gravity other than that (3.15) which is customarily assigned to portland cement.

Air permeability fineness determinations are sometimes influenced by factors which are peculiar to tests in a single laboratory, or to those in a small group of laboratories, and it is not uncommon to find that a modification of the standard procedure has been made in an effort to compensate for such influences. It is very probable that some of the variations in comparative test results can be attributed to the fact that values reported by several laboratories were obtained by a modified procedure rather than the standard procedure because the operators concerned were not aware of the difference in techniques.

17.3.1. Causes of Variations

A. *Apparatus*

(1) *Nonconformance of permeability cell with specification requirements.* Variations in the volume of the test bed may result from wearing or damaging of the cell and plunger, or nonplaneness of the perforated disk. When a unit is being used regularly, the dimensions of the component parts should be checked at least once in every six months

with particular attention being given to the clearance between the plunger and cell, and the volume of the compacted bed should be checked at least once in every twenty-four months. If the disk is warped, it should be marked so that it can be placed in the apparatus in the same position for every test.

(2) *Uneven edges of disks of filter paper.* It is often difficult to seat disks of filter paper properly inside the cell if the edges are torn or uneven. The use of manufactured disks in preference to homemade disks will usually solve such problems.

(3) *Unsatisfactory stopcock lubricant.* Stopcock lubricants sometimes foul the upper part of the manometer to the extent that droplets of manometer fluid will cling to the inner surfaces after the liquid has receded. When this occurs, the inside of the manometer should be cleaned, and a check made to insure that petrolatum, or a petrolatum base stopcock lubricant, is available for future use. One way in which a manometer tube may be cleaned is, *under the supervision of an expert chemist,* to fill it with an organic solvent to remove all traces of manometer fluid, rinse out the solvent with water, allow the interior to dry, fill it with a 20-percent solution of fuming sulfuric acid which is then allowed to stand for a period of from 2 to 12 hours, and finally rinse with water to remove the acid.

(4) *Incorrect amount of fluid in the manometer.* The specifications require that the manometer be filled with fluid to the midpoint. Since the calculated midpoint often falls either above or below the lower mark, in the interest of uniformity, it is customary to consider that the lower mark is at the midpoint of the tube. When this feature is checked, it should be remembered that the level of the liquid will often be 2 to 3 mm below the normal level immediately after a test.

(5) *Changes in viscosity of manometer fluid.* Use of a liquid whose viscosity may vary appreciably with changes in temperature is undesirable.

B. *Test Procedure*

(1) *Errors in calibration of apparatus.* Among the common errors in calibration are the following:

(a) Incorrect porosity of bed. A loosely compacted test bed is highly sensitive to slight bumps whereas a tightly compacted bed may spring back when the plunger is released. Since the porosity of bed has an important relationship to these problems, it is essential that a suitable value be selected. In general, a value greater than 0.500 should be used for materials finer than Type I cement, and a value less than 0.500 should be used for coarser materials.

(b) Failure to fluff the standard fineness sample prior to use.

(2) *Cement not at room temperature at time of test.*

(3) *Variations in the volume of the test bed.* Variations in the volume of the bed may be caused by the following:

(a) Failure to seat filter papers properly. It is required that a rod slightly smaller than the cell diameter be used to press the edges of the bottom piece of filter paper against the metal disk.

(b) Failure to level the surface of the bed prior to compaction. It is required that the side of the cell be tapped lightly in order to level the bed of cement. Too much tapping is undesirable, particularly when coarse cements are being tested.

(c) Loss of cement around sides of rapidly descending plunger.

(d) Improper compaction techniques. Among these is rotation of the plunger after it comes in contact with the top piece of filter paper.

(4) *Errors in timing.* Such errors are frequently encountered where the operator fails to use the bottom of the meniscus of the manometer liquid in gaging when to start and stop his timer.

(5) *Leakage of air around outside of cell during test.*

(6) *Theoretical errors.* A common mistake is the failure of an individual operator to calibrate himself by determining his own T_s for the standard fineness sample, or a suitable reference sample. Another is the selection of the wrong equation for calculation of fineness. The latter problem may be avoided by using the following composite equation for all calculations:

$$S=\frac{S_s\sqrt{T}}{\sqrt{T_s}}\times\frac{p_s(1-e_s)}{p(1-e)}\times\frac{\sqrt{n_s}}{\sqrt{n}}\times\frac{\sqrt{e^3}}{\sqrt{e_s^3}}$$

where:

S=specific surface in sq cm per g of the test sample,
S_s=specific surface in sq cm per g of the standard sample used in calibration of the apparatus,
T=measured time interval in seconds of manometer drop for test sample,
T_s=measured time interval in seconds of manometer drop for standard sample used in calibration of the apparatus,
n=viscosity of air in poises at the temperature of test of the test sample,
n_s=viscosity of air in poises at the temperature of test of the standard sample used in calibration of the apparatus,
e=porosity of prepared bed of test sample,
e_s=porosity of prepared bed of standard sample used in calibration of apparatus,
p=specific gravity of test sample (for portland cement a value of 3.15 shall be used), and
p_s=specific gravity of standard sample used in calibration of apparatus (assumed to be 3.15).

18. Consistency

Both hand mixing and mechanical mixing procedures are used in the preparation of mortars and pastes of the consistency required for the standard tests of portland cement. Standard 1:3

mortars for tensile strength specimens are mixed by hand using a calculated amount of water. All other mortars are mixed with a mechanical mixer, and their consistencies are measured with the 10-in. flow table. With the exception of those used in heat of hydration tests, it is generally the practice to mix pastes by hand and to measure their consistencies with the Vicat apparatus. Pastes for heat of hydration tests are first mixed by hand with a spatula, and then with an electric stirrer, using the amount of water indicated in the test method.

18.1. Mixing of Pastes by Hand

The hand mixing procedure begins with the addition of water to the cement, and mixing begins at the point where normal portland cements will have absorbed an appreciable percentage of the water. The operation consists of continuous and vigorous mixing, squeezing, and kneading of the paste with the hands for 1½ min. It has long been recognized that the plasticity of a mass of cement paste is affected by the amount of work used in the mixing operation, and that it is often possible to attribute a large portion of the variations in results for tests employing pastes to this one factor.

A. *Causes of Variations*

Among the common sources of variations in hand mixing operations are the following:

(1) *Use of wrong amount of cement or mixing water.*

(2) *Use of equipment coated with oil or other contaminants.*

(3) *Unsatisfactory work gloves.* Rubber gloves must be pliable and snug-fitting so that they will not prevent uniform and vigorous movement of the fingers throughout the mixing period.

(4) *Use of gloves, trowels, or mixing slabs which have not been permitted to dry.*

(5) *Loss of mixing water due to evaporation.* Use of too large an area on the mixing slab during hand mixing promotes evaporation of water.

(6) *Inadequate mixing of paste.* Lack of vigor in mixing is a principal factor.

(7) *Retempering to obtain paste of the desired consistency.*

18.2. The Normal Consistency Determination

The amount of water to be used in preparing pastes for time of setting and autoclave expansion tests, and in preparing mortars for tensile strength tests, is established by the normal consistency determination. The apparatus for this determination was developed out of equipment used in about 1818 by L. J. Vicat, Chief Engineer of Bridges and Roads in France [23], and the test had been in use for many years in both the United States and Europe prior to its adoption as a standard cement testing procedure in 1904. The method provides for the determination of the consistency of hand-mixed paste by observation of the depth to which the 1-cm-diam plunger of a Vicat apparatus penetrates a ring-confined sample during a 30-sec period, and the paste is considered to be of normal consistency when the penetration is between 9 and 11 mm. Among the more important variables in this procedure are improper preparation of the paste, and failure to observe the specified time limits on the various operations.

18.2.1. Causes of Variations

A. *Apparatus*

(1) *Incorrect plunger weight.* Incorrect plunger weight may result from loss of shot from the barrel of the plunger, reduction of weight due to loss of metal in a machining operation, accumulation of dirt on the plunger, or replacement of the 1-mm needle. The weight may be adjusted by varying the amount of shot in the barrel (when construction of the apparatus permits); or by filing material away from, or adding solder to, the customary knurled knob on the needle assembly. When solder is added, it must be determined that the adjustment is permanent.

(2) *Incorrect plunger diameter.* The diameter of the plunger may be incorrect due to errors in machining or damage in usage. Also, a substantial reduction in diameter of the end of the plunger may occur over a period of time from polishing the sides with emery cloth.

(3) *End of plunger in poor condition.* The condition of the end should always be checked after an uncontrolled drop, or after it has come in forcible contact with a hard object.

(4) *Frictional resistance to fall of plunger.* Among the conditions which tend to retard the fall of the plunger are the following:

(a) Use of viscous oil or grease on plunger. The plunger should be oiled at the contact point with the frame. Only light oils are suitable for this purpose.

(b) Hardened cement or rust on the sides of the plunger.

(c) Accumulation of dirt at point where plunger contacts frame. Dirt will accumulate rapidly on the portion of the plunger that slides through the frame of the Vicat apparatus unless the operator develops the habit of holding the lower end while making adjustments of the zero setting.

(5) *Vicat apparatus in poor condition.* The apparatus is in poor condition if the indicator is bent or twisted in such fashion that it does not point to comparable lines on both scales; or if the scale is tarnished and difficult to read.

B. *Test Procedure*

(1) *Failure to completely fill the Vicat mold with paste.* Such failures may be minimized if the ball of paste is gently shaken into an ellipsoidal shape

while resting in the palm of the hand prior to insertion of the mass into the larger end of the mold.

(2) *Failure to make the proper zero setting.* It is a common practice to make the zero setting by adjusting the indicator to read zero when the bottom of the plunger is resting on the top of the side of the mold, which in turn is resting on the glass plate used with the mold. It is to be noted that this is only an approximation of the place where the surface of the paste will actually be. Individuals wearing bifocal glasses sometimes find it difficult to make an accurate setting.

(3) *Improper release of the plunger.* The manipulation of the set-screw holding the plunger must be such that the plunger will be released gently, quickly, and completely.

(4) *Failure to release the plunger exactly 30 sec after mixing.* The time that elapses between completion of mixing and release of the plunger has an important bearing on test results, particularly when cement with false setting characteristics, or highly plastic cements such as Type III or Masonry Cements, are under test. In many instances, it will be found desirable to time the operation of filling the mold in such fashion that the plunger can be released as soon as the mold is placed in the testing position.

(5) *Vibration of work table while penetration tests are in progress.*

18.3. Mechanical Mixing of Mortars

Interest in the use of mechanical mixing in the preparation of pastes and mortars had existed for many years prior to 1953 when the current procedure for mixing certain kinds of mortars was adopted as a standard test method. A method for mechanical mixing of pastes has also been developed, but use of this newer procedure is being postponed pending the possible development of some new parts for the mixing apparatus. One of the more important difficulties with the mixer at present is inadequate mixing of mortar materials as a result of excessive clearance between the end of the paddle and the bottom of the bowl.

18.3.1. Causes of Variations

A. *Apparatus*

(1) *Nonconformance of mixer design with specification requirements.* It is required that the mixer be of the epicyclic type and have a mechanical speed control device, that the mixing bowl have a nominal capacity of 5 quarts, and that the paddle and bowl be made of stainless steel. The Model N–50 Mixer, made by the Hobart Manufacturing Co., is considered to conform with these requirements. However, it is to be noted that the bowl and paddle often supplied as original equipment are not made of stainless steel, and therefore must be replaced if the mixer is to be used in the testing of cement. Mixers which do not conform with specification requirements should not be used. Paddles equipped with wipers should not be used unless authorized.

(2) *Failure to provide correct current for mixer motor.* Mixer motors are customarily designed to operate on 115 v, 60 cycle, alternating current. Speed control problems will be encountered when efforts are made to operate the conventional motor with a different kind of current.

(3) *Failure to maintain correct paddle to bowl clearances.* Incorrect clearance between the end of the paddle and the bottom of the bowl is a frequent problem in mechanical mixing procedures. When the clearance is too great, materials accumulate around the bottom of the bowl with the result that the mixture obtained is not of the expected consistency. When the clearance is too small, the paddle grinds up a portion of the standard sand, thereby changing the consistency of the mix and often causing excessive wear of both the paddle and the bowl.

(a) Adjustment of clearances. Small adjustments can often be made simply by bending the lugs on the side of the bowl. (Note: In general, the use of washers beneath the lugs to raise the bowl is unsatisfactory.) Major adjustments can be made by raising or lowering the motor housing, or by having a competent machinist rework a given paddle to fit a given bowl. Recently, it has been found that a homemade adjustment bracket can be used to good advantage.

(b) Marking of bowls and paddles. Since they are seldom, if ever, identical in every small detail, paddles and bowls, in most instances, cannot be interchanged at random after clearances have been adjusted for a given mixer-paddle-bowl combination. Therefore, if equipment is carefully adjusted in such manner that two paddle-bowl sets can be used with a single mixer, each set must be match-marked so that the proper combination can be easily recognized. Where more than one mixer is in use, each mixer-paddle-bowl set must be match-marked.

(4) *Bowl damage.* Bowls may be accidentally damaged by dropping of the bowl, dropping of the paddle into the bowl, or by raising the bowl into the mixing position before it is properly seated in the frame. Careless or nonprofessional efforts to repair a dent may result in a change in the form of the bowl which renders it unsatisfactory for use.

(5) *Failure to provide a lid for the bowl.* A cover is needed to prevent evaporation losses during pauses in the mixing operation. This lid should be made of material not attacked by the cement, and should be sufficiently rigid that it will lie flat across the top of the bowl. Rags should not be used in lieu of a lid.

(6) *Leakage of oil from gear housing into mixing bowl.* A change in the consistency of the mix may occur if oil falls into the bowl from the gear housing

during a mixing operation. When a mixer has not been used for some time, the bowl should be checked for oil, and cleaned if necessary, prior to starting a test.

B. *Test Procedure*

(1) *Failure to remove all moisture from paddle and bowl before use.*

(2) *Failure to introduce materials into the bowl in the prescribed sequence.*

(3) *Loss of materials as they are being introduced into the bowl.* Accidental losses can be minimized by the use of a funnel-shaped hopper.

(4) *Failure to follow the prescribed mixing procedure.*

18.4. The Flow Table Test

A comprehensive specification for the 10-in. flow table, based on the knowledge gained during more than 30 years of experimentation with and use of this device for measuring the consistency of mortars, was adopted in 1949. The apparatus consists essentially of a 10-in. diam rigid table, with a perpendicular shaft, mounted on an integrally cast rigid iron frame fastened to the top of a 300-lb concrete pedestal. Accessory items are a mold in the shape of the frustrum of a cone, and a specially marked caliper. The test is accomplished by leaving the amount of mortar required to fill the mold on the table while it is raised and dropped a specified number of times by means of an electrically-driven cam fastened to the frame. The flow is the resulting increase in diameter of the mortar mass expressed as a percentage of the original base diameter.

18.4.1. Causes of Variations

All of the important requirements for the table are covered in detail in the specification, and a lengthy check list for use in trouble shooting is available [24]. Since so much information has already been printed, only a few causes of variations other than those taken into account in section 16 are presented herewith.

A. *Apparatus*

(1) *Nonconformance of apparatus with specification requirements.* This is a common problem with brand new and old apparatus alike. New apparatus which does not conform to specification requirements should not be accepted. Old apparatus should be repaired or replaced whenever the following signs of wear or damage are noted:

(a) Damage to contact surfaces.

(b) Excessive clearances between bore and shaft.

(c) Leakage of water from beneath flow mold due to damage to bottom edge.

(d) Inaccurate flow measurements due to wearing of caliper.

(e) Curvature in straightedge of trowel used in striking excess material from the flow mold.

(f) Improper alinement of cam with table shaft due to bending of the threaded end of the cam shaft.

(2) *Failure to provide correct current for flow table motor.*

B. *Test Procedure*

(1) *Failure to dry and clean the table top prior to use.*

(2) *Variations in filling the flow mold.* Variations in the amount of material contained within the mold may be caused by variations in compacting mortars into the mold, including the failure to use the prescribed 20 tamping pressures. The practice of throwing mortar into the mold also constitutes a variable. The mold should be held firmly against the table top during the filling operation in order to reduce seepage under the bottom edge.

(3) *Failure to dry and clean the table top after filling the mold.* Any water that may have seeped from beneath the mold, and all spilled mortar, should be wiped away prior to proceeding with the test. A circular shield approximately 10 in. in diam, with a center opening approximately 4 in. in diam, should be used while the mold is being filled to prevent spillages from reaching the table top.

(4) *Errors in reading the flow caliper.*

(5) *Adjustment of flows by application of correction factors.* A mixture of ground silica and mineral oil has been used for a number of years as a means of gaging flow table performance. Some laboratories have attempted to correct mortar flow values by adding or subtracting the difference between assigned flow values and actual values obtained with the silica-oil mixture. Test results are not reliable when adjusted in this manner.

19. The Autoclave Soundness Test

Soundness tests have been used for many years to detect those qualities of a cement which tend to destroy its strength and durability, and at least four procedures employing artificial conditions which would hasten the development of such defects were either being used or studied in the early 1900's. An autoclave test was among these, and ultimately, in 1940, it was adopted as a standard test for cement. The method of test for autoclave expansion of portland cement provides for the exposure of a prism (1 by 1 by 10 in.) of hardened cement paste to a steam pressure of 295 psi within a specially designed autoclave for a period of 3 hours. Changes in length of the prism are measured by means of a suitable length comparator.

19.1. Causes of Variations

A. *Apparatus*

(1) *Use of multicompartmented molds.* Single or double compartment molds are preferred because they make it possible to put bars in moist storage soon after molding is completed.

(2) *Thin end plates in bar molds.* Thin end plates may permit the gage studs to become so deeply imbedded in the ends of bars that an accurate measurement of length cannot be obtained.

(3) *Operational problems with the autoclave.* The rate at which an autoclave reaches test pressure is an important factor in some soundness tests. Since operational problems often affect the rate of heating, all of the following items should receive special consideration.

(a) Leakage of steam through joint between lid and chamber. In a number of instances this difficulty has been eliminated by replacing the old-style asbestos gaskets with lead, or metal and asbestos, gaskets.

(b) Leakage of steam through safety valve. Use of a pressure release device which employs a platinum rupture disk may help to solve problems associated with defective safety valves.

(c) Low line voltage.

(d) Deterioration of heating elements. The failure of an autoclave to heat at the specified rate, or its failure to maintain the specified test pressure, can often be corrected by replacing the heating elements.

(e) Errors in indication of pressure. In numerous instances, gages have been reset by laboratory personnel so that the indicated steam pressure would be in agreement with the temperature within the chamber as indicated by a total immersion thermometer. Such adjustments are usually in error because the thermometer well of an autoclave is designed for the use of a partial immersion thermometer. For this and other reasons, the accuracy of indication of the pressure gage should be verified periodically on a gage tester.

(f) Improper manipulation of heater controls. The initial heating rate is significantly affected by the number of bars in the chamber, and therefore it must be controlled by manipulating the heater controls in a sequence previously found to be satisfactory for the number of bars under test. Only one sequence is required if vacancies in the rack are filled with previously tested bars so that the number in the chamber will be the same for every run. Use of a voltage regulator may also prove helpful.

(g) Use of too much water in the chamber. A measured quantity of water, which is 7 to 10 percent the volume of the chamber, should be added prior to the start of each test. Use of an amount greater than 10 percent may cause an appreciable change in the heating characteristics of an autoclave. Any water remaining in the chamber at the end of a test should be removed with a siphon and the walls wiped dry with a sponge to prevent rusting.

(h) Improper adjustment of automatic pressure controls.

(i) Plugging of connecting pipes. The gradual accumulation of material in the connecting pipes may prevent the pressure regulator, the pressure gage, or the safety device from performing in a satisfactory manner.

(4) *Errors in measurements due to comparator.* The graduations on some dials are very difficult to read, and in some instances the physical condition of the dial face is such that accurate readings may be difficult to obtain. Dials in these categories should be replaced. The use of a small cup on the end of the dial stem to receive the gage stud, and another on the comparator base, helps to prevent mistakes in measurement.

B. *Test Procedure*

(1) *Use of an incorrect amount of cement or mixing water.* The practice of using a fixed water content for all bars made from a certain type of cement from a given source is undesirable.

(2) *Allowing gage studs to shift during molding of bars.*

(3) *Excessive troweling of the surface of bars.* Excessive troweling can be avoided if the operator uses a trowel with sharpened edges, and performs the smoothing operation by drawing the flat side of this trowel (with the leading edge slightly raised), over three-fourths of the length of the mold, in such fashion that the surface of the paste behind the trowel will be smoothed while any excess material will be "crowded" toward the end of the mold. Toward the end of the stroke, the leading edge of the trowel should be lowered to ride along the top edge of the mold, and the extra material then sliced away by the sharp edge of the trowel. The entire operation of smoothing and finishing can be completed in this manner in the course of a few passes along the mold.

(4) *Allowing bars to dry after molding is completed.* Bars should be put into moist storage immediately after molding is completed, and should not be disturbed until it is time to strip the mold. The procedure of smoothing the surface an hour or two after the mold has been filled does not conform with standard practice.

(5) *Autoclaving bars which are less than 24 hours old.*

(6) *Failure to measure bars immediately after removal from moist storage.*

(7) *Improper cooling of bars after autoclaving.* It is important that bars be cooled to room temperature prior to measurement. A cylindrical container approximately 8 in. in diam and 16 in. in height, equipped with a bail, makes a convenient cooling chamber. In use, this container is filled with water and placed on a hot plate at the end

of the 3-hr autoclaving period. Later, when the autoclave is opened, the rack and bars as a single unit may be lifted from the autoclave by means of a suitable metal hook and immersed in the near-boiling water in the container. The container may then be transferred to a sink, and cold water introduced through a rubber tube connected to a cold water spigot. The rate of cooling may be controlled by regulation of the flow of cold water through the tube.

(8) *Errors in calculations.* Such errors are usually mistakes in arithmetic.

20. Time of Setting

Setting properties have been of interest since the very earliest days of cement testing and numerous procedures for gaging this important quality have been developed. Presently, there are two standard methods of test, namely the Vicat Time of Setting Test, and the Gillmore Time of Setting Test.

20.1. Common Causes of Variations

The two tests are similar in many respects, and therefore, there are a number of considerations which are of importance in either procedure. Some of the common causes of variations other than those taken into account in previous sections of this discussion are presented herewith.

(1) *Error in the determination of the normal consistency.* It is generally understood that the more mixing water used, the slower the set. The amount of mixing water may be incorrect due to an error in the determination of the normal consistency.

(2) *Inadequate mixing of paste.* It has been illustrated by many investigators that the time of set varies with the vigor of the mixing used in the preparation of the paste [25]. For this reason, it is suggested that when difficulty is encountered in obtaining suitable reproducibility of test results the experimental procedure for mechanical mixing of pastes might be used in evaluating the effectiveness of an operator's hand mixing technique. When making such comparisons, it must be remembered that machine mixing will give unsatisfactory results if the mixer does not conform to *all* requirements of the applicable specifications.

(3) *Regaging of paste.* Regaging usually tends to make paste slower setting.

(4) *Irregularities in testing.* A Vicat apparatus, or a set of Gillmore needles, should be shielded from drafts of air and from direct sunlight which might cause evaporation of moisture from specimens while readings are being taken. Also, the length of time specimens are kept out of moist storage should be held to a minimum.

(*f*) *Errors in determining time intervals.* Where errors in reading times or in calculating time intervals are encountered, the use of a "military-type" clock with a face showing the hours from 0000 to 2400 may prove helpful.

20.2. The Vicat Time of Setting Test

The current method of test for time of setting of hydraulic cement by the Vicat needle was developed out of equipment and procedures used in about 1818 by L. J. Vicat [23]. The evolutionary process began in Europe and had been under way there and in the United States for many years before a crude version of the present procedure was adopted as a standard test for cement in 1904. A number of changes in apparatus and procedures have been made since the adoption, with a recent change of particular note being the replacement of the tests for "initial time of setting" and "final time of setting" with a single test for the "Vicat time of setting".

The test is accomplished by observing the time required for hand-mixed paste to harden to the extent that the 1-mm needle of a Vicat apparatus will penetrate a ring-confined sample to a depth of only 25 mm during a 30-sec period. This test is generally found to be more reproducible than the Gillmore test because there is greater uniformity among test specimens, and because the personal judgment factor as to when set occurs is eliminated.

20.2.1. Causes of Variations

A. *Apparatus*

(1) *Vicat apparatus in poor condition.* In general an apparatus that would not be suitable for use in normal consistency determinations would not be suitable for use in Vicat time of setting tests. Otherwise, particular attention should be given to the straightness of the needle, the diameter and the condition of its end, and the possible presence of hardened cement or rust on its sides. The condition of the needle should always be checked following a free fall through a mass of paste in a plastic state.

(2) *Use of oil on Vicat ring.* Use of oil on the ring to facilitate the removal of hardened paste is not particularly helpful, and introduces the possibility that the paste might be contaminated with oil during the test.

B. *Test Procedure*

(1) *Improper procedure.* The paste may not be suitable for test purposes if it has been used previously in a normal consistency determination.

(2) *Failure to completely fill the Vicat ring with paste.* Small holes in the paste may have a significant effect on test results.

20.3. The Gillmore Time of Setting Test

The use of weighted wires (needles) was originally proposed as a test for time of setting of cement by a Committee of the American Society of Civil Engineers in 1885. The wires were called

"Gillmore wires" as a courtesy to General Gillmore, the chairman of the committee, although they had not been proposed by him, but had been suggested by others [25]. A test based on that early method was adopted as a standard test for cement in 1916.

A set of Gillmore needles consists of an initial needle, weighing ¼ lb and equipped with a $\frac{1}{12}$-in. diam cylindrical tip; and a final needle, weighing 1 lb and equipped with a $\frac{1}{24}$-in. diam cylindrical tip. Initial and final setting times are determined by the application of these needles to the flattened top of a specimen of paste (a pat) 3 in. in diam at the bottom and ½ in. thick at the center. The Gillmore test is preferred by many testing agencies because it is simplier, and therefore more economical to perform, than the Vicat test. Its chief weaknesses are considered to be the failure to obtain a homogenous mass upon which to make the determination, the difficulty of applying the needles at right angles to the surface of the specimen, and the personal equation in determining when the needles cease to make an appreciable indentation.

20.3.1. Causes of Variations

A. *Apparatus*

(1) *Incorrect diameters of needle tips.* The diameter of a needle tip may be incorrect due to errors in machining, wear, dropping, or encrustations of hardened cement.

(2) *Incorrect needle weights.* The weight of a needle may be incorrect due to the inadvertent loss of shot from the ball, or the addition of excess shot.

(3) *Failure to provide required frame for needles.* The ends of needles stored in wooden blocks, or in drawers, are frequently found to be damaged.

(4) *Bent needle shafts.*

B. *Test Procedure*

(1) *Use of an incorrect amount of cement or water.* Since the specified batch size makes more paste than is required to make a pat, smaller samples are sometimes mixed by hand or with a trowel. These methods usually require more mixing water than that indicated for normal consistency, and therefore the rate of hardening of the paste is altered. Results obtained in this manner are only approximations of the time of setting.

(2) *Specimens improperly formed.* This is frequently due to the failure to follow specified procedures. Among the other contributing factors are the following:

(a) Excessive troweling. If operators seem to be over-troweling specimens, an effort should be made to form pats with less than 40 troweling strokes. It is preferred that only one application of pressure be used in flattening the top of the pat.

(b) Slanting or uneven surface on top of pat. The surface of the pat should be smooth, and it should be parallel to the surface of the glass plate on which the specimen is formed. A template may be helpful in preparing specimens which have smooth, level surfaces, and which are of a uniform height. This template should not be confused with the so-called "pat mold," a device which is not suitable for use in the preparation of specimens.

(3) *Variation in technique of applying needles.* The needles must always be applied at right angles to the surface of the pat.

(4) *The personal equation in judging indentations.* Variations in determining the point at which the pat will bear, without appreciable indentation, the Gillmore needle, may be due to inadequate lighting of the test area, and therefore the installation of special lights to insure that surfaces of pats are adequately illuminated may prove helpful. Use of fluorescent lights is recommended to avoid exposing pats to the "drying heat" of incandescent lights.

(5) *Too frequent checking of specimens.*

21. The Heat of Hydration Test

The Heat of Hydration Test is customarily listed among the physical tests of cement, and therefore it has been necessary to make several references to it. However, because the procedure has much in common with procedures for chemical analysis of cement, comments about the related methods and apparatus have not been included in this discussion. For those who wish to pursue it, considerable information about the test can be obtained by reference to a published bibliography [26].

22. The Determination of the Air Content of Mortar

The need for a rapid physical test whereby the air-entrainment characteristics of portland cements treated with air-entraining agents could be regulated led to the adoption, in 1944, of the air content of mortar determination as a standard test for cement. A number of inadequacies in the original equipment and procedures were soon recognized, and these were corrected in extensive revisions of the method in subsequent years. With these changes, the method of test for air content of hydraulic cement mortar has become one of the most satisfactory of all the physical tests from the standpoint of reproducibility.

The current procedure provides for the determination of air content of machine-mixed mortar; consisting of 1 part cement, 4 parts standard sand, and the amount of water required to obtain a flow of 80 to 95 percent after 10 drops of the flow table; by comparing the weight of a 400-ml sample thereof to the theoretical air-free weight of a similar volume as calculated by the absolute volume method. In general, the uniformity in results is found to be better in tests of air-entraining cements than in tests of non-air-entraining cements.

22.1. Causes of Variations

A. *Apparatus*

(1) *Variations in volume of 400-ml measure.* It is required that the volume of the measure be between 399 and 401 ml. Many of the containers presently in use do not conform to this requirement, and therefore, as a precaution, the volume of every new measure should be checked, and adjusted if necessary, prior to acceptance by the laboratory.

(2) *Balance not suitable for test.* It is difficult to obtain the desired accuracy in test results if the capacity of the balance is inadequate.

B. *Test Procedure.*

(1) *Use of an incorrect amount of cement, sand, or water.* If water requirements for all cements seem to be consistently in error, the flow table and mechanical mixer should be thoroughly checked for conformance with the requirements of applicable specifications.

(2) *Failure to place mortar gently in the measure.* The technique of throwing mortar into the measure does not conform with standard practice.

(3) *Failure to observe specified time limits on test operations.* The desired accuracy cannot be obtained if the time limits on various test operations are disregarded.

(4) *Improper handling of the 400-ml measure.* Shaking or jolting of the measure during the filling operation must be avoided.

(5) *Failure to use specified equipment in performance of test.* Particular attention is invited to the advantages of using the specified spoon, spatula, tapping stick, and straightedge in the filling of the 400-ml measure.

(6) *Variations in weighings.* In many instances, bench weights will give more uniform results than the beam and poise with which many mix balances are now equipped.

23. Strength of Mortars

The use of mortar strength tests as a criterion of the quality of cements was recorded in the literature as early as 1772 [23], but it was not until about 1848 that any real efforts were made to devise standard mortar strength tests for portland cements [27]. Much improved versions of the compressive strength and tensile strength tests conducted at that time are presently included among the standard physical tests for cement.

23.1. Common Causes of Variations

Although there are many actual or theoretical differences between the two strength tests, the common use of a mortar specimen ensures that there will be a number of considerations which would be of importance in either procedure. Some of these are presented herewith.

(1) *Nonconformance of mold design with specification requirements.*

(2) *Leakage of water from molds.* The loss of water through the joints of a mold, or the joint between a mold and its base plate, may permit specimens to develop strengths which are higher than average. In this connection, it is important that mold base plates be of sufficient thickness to preclude bending during molding and handling of specimens.

(3) *Failure to provide suitable water storage facilities.*

(4) *Improper treatment of specimens after removal from storage.* It is required that all strength specimens be placed in moist air storage immediately after molding. Specimens to be tested at 24 hours are taken directly from the moist air storage and tested at once. Specimens to be tested at later ages are transferred to water storage for additional curing. Air drying of any of these specimens just before testing can cause an increase in compressive strengths or a reduction in tensile strengths. For this reason, when several 24-hr specimens are to be tested at one time, it is recommended that they be placed in a pan immediately upon removal from moist storage, and kept covered with a moist cloth until ready for test. When two or more specimens, other than 24-hr specimens, are to be tested at one time, they should be removed from the water storage tanks, placed immediately in a pan of water, and kept there until actually tested. As immersion in water of an appreciably different temperature than the tank water just prior to testing can have a significant influence on results, it is recommended that the water in the pan be obtained from the storage tank.

(5) *Failure to clean specimens prior to testing.* Loose sand grains or incrustations must be removed from the bearing surfaces of specimens before they are inserted in the testing machine. If specimens seem to be excessively coated with a soapy substance, the use of a different mold lubricant should be considered.

(6) *Failure to observe suitable time tolerances on testing.* Every effort should be made to maintain equal intervals between the preparation and testing of strength specimens. Since it may not always be possible to do this, some tolerances on time should be observed. The following are suggested: 1-day, within ½ hour; 3-day, within 2 hours; 7-day, within 4 hours; and 28-day, within 8 hours.

(7) *Inaccuracy of testing machines.* The accuracy of indication of a testing machine should be verified following its installation in a laboratory, and at least once in every three years thereafter. When the accuracy of a machine is questioned, and it is not convenient to make an immediate verification, it may prove helpful to compare

results obtained with the machine in question against results obtained with machines in nearby laboratories. In these instances the comparative tests should be made on specimens from a single batch, and checks should be made in two properly equipped laboratories, rather than in just one. It may be helpful to remember that the failure to maintain a testing machine in proper operating condition is one of the common causes of errors in strength testing.

(8) *Failure to load test specimens at specified rate.* Appropriate rates for application of loads to strength specimens have been determined by careful experimentation. The failure to use the specified rate may have a significant effect on test results, particularly in compressive strength determinations.

23.2. The Compressive Strength Test

The procedure whereby the strength characteristics of a cement are evaluated by the compressive strength of 2-in. mortar cubes; consisting of 1 part cement, 2.75 parts Graded Standard Sand, and the amount of water required to obtain a flow of 100 to 115 percent after 25 drops of the flow table; was adopted as a standard test for cement in 1934. It is favored by many investigators because the progressive increase in the strength of cubes, with age, parallels the increase in strength of concrete, with age, and because the agreement between laboratories using correct techniques and equipment is generally satisfactory. Numerous refinements in apparatus and procedures have been made since 1934, i.e., machine mixing has replaced hand mixing and the instructions for molding of specimens have been expanded, with the result that the number of laboratories reporting unreasonable compressive strength values has been substantially reduced.

23.2.1. Causes of Variations

A. *Apparatus*

(1) *Nonplaneness of interior faces of molds.*

(2) *Failure to keep corners of molds clean.* The accumulation of hardened cement in the corners of the pockets in a cube mold reduces the overall size of test specimens, and thereby causes reductions in compressive strengths. Prompt cleaning of the molds after stripping will help to avoid this difficulty, and normally most of the residue can be removed with a damp cloth if cleaning is undertaken before the material has had an opportunity to harden in air.

(3) *Excessive use of oil on molds.*

(4) *Inadequate sealing of molds.* Operators frequently fail to follow the instructions for sealing of cube molds. In numerous instances, the departure is attributed to difficulty in applying the specified sealing material. One effective technique is to melt the material in a conventional oil can with a "cut-down" spout, and to apply the liquid thus obtained to the mold, as one might apply oil, while holding the hot can in a hand protected by an asbestos glove.

(5) *Problems with spherical blocks in compression machines.* Among the common problems are the following:

(a) Block frozen in place and therefore unable to adjust to specimen.

(b) Improper design of block.

(c) Damage to spherical surfaces which prevents normal movement of the block.

(6) *Nonplaneness of faces of compression machine bearing blocks.* All blocks tend to lose their planeness with usage, and this change may occur rapidly if the bearing faces have not been properly hardened. Planeness may be checked with a flashlight and a toolmaker's straightedge. If the planeness is within 0.001 in. as required, very little light, if any, will show between the straightedge and the surface of the block when the flashlight is held behind the straightedge.

(7) *Inadequate maintenance of compression testing machine.* Every laboratory should be provided with maintenance literature for the compression machine it uses, and all special maintenance equipment should be available, i.e., the special wrench and oil gun needed for machines equipped with a fluid support. The indicating hands should be checked periodically for freedom of movement, with particular attention being given to maximum hands. The level of oil in all oil reservoirs should be checked frequently. In the event of mechanical failure, the services of a competent repairman should be obtained promptly.

B. *Test Procedure*

(1) *Use of an incorrect amount of cement, sand, or water.* If the water requirements for all cements seem to be consistently in error, the flow table and mechanical mixer should be checked thoroughly for conformance with the requirements of applicable specifications.

(2) *Mixing of mortars by hand.* The mixing of mortars by hand, rather than with the mechanical mixer as specified, will usually produce lower test results.

(3) *Variations in tamping pressures.* Nonuniformity in the tamping pressures used in compacting mortar in the mold pockets is a variable in compressive strength determinations.

(4) *Improper positioning of specimens on compression machine bearing blocks.* It is important that cubes be centered carefully on the bearing blocks of the testing machine. The use of a special centering template may be helpful.

(5) *Failure to zero compression machine properly at start of test.*

(6) *Errors in reporting test results.* A confusing error, particularly where low strengths are concerned, is the failure to divide the load in pounds by four to obtain pounds per square inch.

23.3. The Tensile Strength Test

The procedure whereby the strength characteristics of a cement are evaluated by the tensile strength of 1-in. thick, figure-8 mortar specimens (briquets); consisting of 1 part cement, 3 parts Standard Sand, and the amount of water indicated by the normal consistency determination; was introduced about 1880 [27], and was adopted as a standard test for cement in 1904. The test has been favored by many investigators because it is simple, and therefore economical to perform. The retrogression of strength with age, which is sometimes encountered, is frequently mentioned as a disadvantage.

23.3.1. Causes of Variations

A. *Apparatus*

(1) *Use of worn test apparatus.* When strength values seem to be consistently in error, all apparatus should be carefully checked for conformance with the requirements of applicable specifications. A particular problem in recent years has been the low strength of specimens produced in molds which have worn so thin as to no longer comply with minimum thickness requirements. Another problem has been the leakage of water from molds which are worn in such fashion that the halves no longer meet at all points.

(2) *Variations in mold design.* The molds presently in use vary from the single pocket to the six pocket type, and fabrication techniques tend to vary with the type of mold. It would be advantageous for all laboratories to use the customary 3-pocket type.

(3) *Inadequate maintenance of testing machine.* From time to time, grips are observed wherein the rollers are no longer free to turn. This and other important maintenance problems are discussed in detail in existing publications [24].

B. *Test Procedure*

(1) *Use of an incorrect amount of cement, sand, or water.* Consistent errors in the amount of mixing water used may indicate an error in the normal consistency determination, or an error in calculating the amount of water from the normal consistency. In this latter calculation, the given percentage of water is expressed in terms of the weight of cement and sand, and not in terms of the weight of the cement alone.

(2) *Variations in techniques for filling molds.* Where two or more molds are being filled from the same batch, each phase of the fabrication process should be completed on each mold before the next phase is started, i.e., compaction of the mortar into each pocket in each mold should be completed before the heaping and smoothing operation is started. The mortar in each pocket should be flush with the top of the mold when smoothing is completed.

(3) *Improper compaction of mortar in the mold pockets.* It is recommended that the thumb pressures be applied in groups of two per briquet on each of six trips along the mold. Care should be taken that the thumb pressures are applied entirely to the surface of the mortar, and not to the surface of the mold. The specified thumb pressure of between 15 and 20 lb may be measured by molding the briquets on a platform scale or on other suitable apparatus that will indicate the pressure exerted.

(4) *Variations in techniques of finishing specimens.* In standard practice the excess material is removed from the molds with a smoothing motion rather than a sawing motion. Mastery of the smoothing operation requires a certain amount of practice, and therefore many laboratories permit inexperienced operators to use the simplier sawing procedure while learning the test. The prescribed smoothing technique should be adopted at the end of the apprenticeship period.

(5) *Failure to remove all sand grains from face of mold.* Sand grains between the mold and its base plate promote the loss of mixing water from finished specimens as well as an increase in thickness. Either of these conditions is capable of causing an apparent increase in strength.

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24. References

[1] W. J. Youden, Statistical aspects of the cement testing program, ASTM Proc., **59,** 1120, (1959).
J. R. Crandall and R. L. Blaine, Statistical evaluation of interlaboratory cement tests, Ibid., p. 1129.

[2] Federal Standard Stock Catalog, Section IV (pt. 5), Federal Specification for Balances; Analytical, AAA-B-92, (May 1949). For sale by the Superintendent of Documents, Washington 25, D.C. Price, 5 cents.

[3] L. B. Macurdy, H. K. Alber, A. A. Benedetti-Pichler, Hugh Carmichael, A. H. Corwin, Robert M. Fowler, E. W. D. Huffman, P. L. Kirk, and T. W. Lashof, Terminology for describing the performance of analytical and other precise balances, 1954 Report and Recommendations of the Committee on Balances and Weights, Anal. Chem., **26,** 1190, (July 1954).

[4] T. W. Lashof and L. B. Macurdy, Testing a quick-weighing balance, Anal. Chem., **26,** 707, (April 1954).

[5] Leonard Bean and Ethel J. Hackney, A study of some operations involved in cement analysis, ASTM Bull. **197,** 44 (April 1954).

[6] Information concerning pyrometric cones may be obtained from The Edward Orton Jr., Ceramic Foundation, 1445 Summit St., Columbus 1, Ohio.

[7] Private communication.
[8] H. S. Roberts, Phys. Rev. **23,** 386 (1924).
[9] W. F. Hillebrand, G. E. F. Lundell, H. A. Bright, and J. I. Hoffman, Applied inorganic analysis, pp. 32–33, 46–51, 503. (Published by John Wiley and Sons, New York, N.Y., 2d ed., 1953.)
[10] Fed. Test Method Std. No. 158, May 1957: Cements hydraulic; sampling, inspection, and testing; method 1001, par. 4.2.
[11] ASTM Standards, pt. 4, pp. 68 and 69, (1958).
[12] I. M. Kolthoff and E. B. Sandell, Textbook of quantitative inorganic analysis, p. 391, (Published by the Macmillan Co., New York, N.Y., 3d ed., 1952).
[13] J. I. Hoffman and G. E. F. Lundell, The precipitation and ignition of magnesium ammonium phosphate, BS J. Research **5,** 279–93 (1930).
[14] H. A. Berman, An improved 8-hydroxyquinoline method for the determination of magnesium oxide in portland cement, ASTM Bull., **237,** 53 (April 1959).
[15] R. B. Rudy, Determination of sulfuric anhydride in portland cement by means of the Wagner Turbidimeter, J. Research NBS, **16,** 555 No. 6, (June 1936).
[16] Harry A. Bright, Determination of sulphur occurring as sulphide in portland cement, J. Research NBS, **18,** 137 No. 2, (Feb. 1937) RP968.
[17] Bernard Chaiken, Determination of ignition loss in portland blast-furnace slag cements, ASTM Bull., No. 238, p. 53, (May 1959).
[18] J. J. Diamond and Leonard Bean, Improvements in flame photometric determination of sodium in portland cement, Anal. Chem., **25,** 1825, (Dec. 1953).
[19] W. J. Halstead and Bernard Chaiken, Insoluble residue determination in portland and portland-slag cements, ASTM Bull., No. 229, pp. 60–65, (April 1958).
[20] M. R. DeFore and H. J. Corah, A test method for air-entrainment of standard Ottawa sand, ASTM Bull. No. 248, (Sept. 1960).
[21] L. A. Wagner, A rapid method for the determination of the specific surface of portland cement, ASTM Proc., **33,** 553 pt. II, (1933).
[22] R. L. Blaine, A simplified air permeability fineness apparatus, ASTM Bull., No. 123, p. 51, (Aug. 1943).
[23] H. F. Gonnerman, Development of cement performance tests and requirements, P. C. A. Research Bull., No. 93, (March 1958).
[24] ASTM standards on cement (with related information). Appendix I. Manual of cement testing.
[25] Data Considered by Committee C–1 of the American Society for Testing Materials in Preparing the Standard Specifications and Tests for Portland Cement (Serial Designation C 9–17).
[26] R. F. Blanks and H. L. Kennedy, The technology of cement and concrete, p. 51, (John Wiley & Sons, Inc., New York, N.Y.).
[27] M. A. Swayze, The testing of cements for inherent strength, ASTM Bull., No. 209, (Oct. 1955).

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