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NOMENCLATURES

A	Alumina
A	Angstrom
A_c	Cross-sectional area of incident beam
A.M.	Alumina Modulus
C	Calcium oxide
c	Velocity of light
CA	Monocalcium Aluminate
C_3A	Tricalcium Aluminate
C_2AS	Gehlenite ($2CaO.Al_2O_3.SiO_2$)
$C_{12}A_7$	Calcium Aluminate, metastable compound ($12CaO.7Al_2O_3$)
C_3AH_6	Calcium Alumino Hydrate ($3CaO.Al_2O_3.6H_2O$)
C_4AF	Tetracalcium Alumino Ferrite
CF	$CaO.Fe_2O_3$, Calcium Ferrite
C_2F	Dicalcium Ferrite
CH	Calcium Hydroxide
CSH	Calcium Silicate Hydrate
C_2S	Dicalcium Silicate
C_3S	Tricalcium Silicate
d	Interplanar spacing
e	Charge of the electron
e^{-2M}	Temperature factor (a function of θ)

F : Ferric oxide
$|F|$: Structure factor
H : Water, H_2O
H.M. : Hydraulic Modulus
I : Integrated intensity per unit length of diffraction line
Io : Intensity of incident beam
$I\alpha$: Intensity of the selected line fo the α phase
$I\alpha p$: Intensity of the selected line of the α phase diffraction from the pure phase
K : Potassium oxide, K_2O
L.C.F. : Lime Concentration Factor
L.S.F. : Lime Saturation Factor
m : Mass of the electron
N : Sodium oxide, Na_2O
P : Multiplicity
r : Radius of diffractometer circle
S : Silica oxide, SiO_2
S.M. : Silica Modulus
v : Volume of unit cell
$W\alpha$: Weight fraction
θ : Bragg angle
Λ : Wavelength of incident radiation
μ : Linear absorption coefficient
μ_m : Micrometer

Appendix A. CHEMICAL ANALYSIS OF RAW MATERIALS AND CEMENT.

Preparation of Solution A

Solution A is used in the determinations of SiO_2 and Al_2O_3. Portions of the samples and standards are decomposed by fusion with NaOH at a comparatively low temperature for about 5 min in nickle crucibles. After cooling, the melts are leached with water, and the solutions are acidified with hydrochloric acid.

Occasionally, with some fine-grained materials such as clays, there is a tendency during fusion for some of the sample powder to float on the surface of the molten NaOH and remain unattacked. When this occurs, the crucible is swirled, allowed to cool, and then reheated so that complete decomposition will result.

For silicate rocks, 50-mg portions of samples and standard are used. This relatively small portion eliminates the necessity for double dilutions, which would be required with larger samples Furthermore, silicon is known to be unstable in solution above concentrations of 200 ppm: this procedure assures the stability of the silicon. For carbonate or phosphate rocks, a 200-mg portion of sample powder is used.

The high percentages of SiO_2 in silicate rocks (about 40 to 100 percent) require that the accuracy and precision of this determination be greater than those necessary for most of the other constituents. For this reason, particular care must be given to mixing and weighing the samples, to the fusions, and to the handling of the relatively concentrated solutions of the melts before dilution .

Reagents

NaOH solution, 30-%: Dissolve 450 g of NaOH pellets in 1,500 ml of water in a stainless steel beaker. Cool and store in a plastic bottle.

HCI, 1+1: Prepare 1 1.

National bureau of Standards standard sample No. 70a (feld-spar) or equivalent.

Procedure

1. Transfer 5-ml portions of 30-percent NaOH solution, measured with a plastic graduate, to a series of 75-ml nickel crucibles. One crucible will be needed each sample, two for standards, and one for a blank. The crucibles should be cleaned with dilute HCI before use.
2. Evaporate the solutions to dryness over gas burners or electric, heaters. Slight spattering can be ignored.
3. Accurately weigh (to the nearest 0.1 mg) 50 mg of each sample of silicate-rock powder (200 mg of samples of carbonate and phosphate rocks) and two 50-mg portions of National Bureau of Standards sample No. 70a (feldspar). As each portion is weighed, transfer to a crucible containing the fused NaOH.
4. Cover and heat the crucibles to dull redness for about 5 min. Remove each crucible from the heat and swirl the melt around

the sides. Allow the melts to cool.

5. Transfer the crucibles plus contents to a series of 1-l plastic beakers.
6. Add about 980 ml of water to each beaker using a rapid 1-l water dispenser (the exact amount is not important except that the same amount must go into each beaker). Allow to stand for at least 1 hr.
7. Stir the contents of each beaker using a plastic stirring rod.
8. Use a tilting pipet to add 20 ml of 1+1 HCl to each beaker, stirring each at the time of addition.
9. Use nichrome tongs to lift each nickel crucible out and examine it; there should be no adhering melt. If any melt remains, allow some more time for dissolution with frequent stirring. When all the melts are dissolved, remove the crucibles and set them aside for washing with HCl before storing them. When the crucibles are removed the solutions are ready to be used for SiO_2 and Al_2O_3 determinations.

A.1 Determination of SiO_2 in Silicate, Carbonate and Phosphate Rocks.

A molybdedum blue method is used for the determination of silica (SiO_2) (Bunting, 1944). As in the preparation of sol$^{\underline{n}}$ A, factors that govern precision should be given particular attention in order to avoid errors that might seriously affect accuracy. All of the aliquots of the A solution should be precisely measured with the same 8-ml pipet. Adequately precise measurements can be made with an ordinary transfer pipet, but better precision is assured if a stopcock pipet is used.

The procedures as written are based on the use of a pipetting machine to disperse reagents. Use of this pipet assures a high degree of reproducibility and eliminates the need for the use of volumetric flasks. The face of agitation obtained by its use eliminates the need for stirring and because the aliquots and all reagents are measured precisely the final volume is the same for blanks, standards and samples. Even when as few as six samples are being analyzed the mechanical pipet is preferred for the addition of reagents for the SiO_2 and Al_2O_3 determinations.

A.1.1 Reagents

- Ammonium Molybdale solution: Dissolve 6.0 gm of $(NH_4)_6$-$Mo_7O_{24}.4H_2O$ in 1 l of water.

- Tartaric acid sol$^{\underline{n}}$: Dissolve 16 gm of $H_2C_4H_4O_6$ in 1 l of water

- Reducing sol$^{\underline{n}}$: Place 0.28 gm of sodium sulphite, 3.6 gm of Sod. bisulphite, and 0.06 gm of 1-amino-2 napthol-4 sulphonic acid into 1 l bottle, add 1 l of water and stir to dissolve.

- Acidified water: Add 5 ml of 1+1 H_2SO_4 to 1 l of water.

Note: Highly consistent results are readily obtained when these sol$^{\underline{ns}}$ are prepared fresh, not more than 48 hr before use. To avoid the inconvenience of weighing reagents at the time an analysis is underway, a supply of 12 or more containers of each dry reagent is preweighed at same convenient time and stored for use as required

A.1.2 Procedure

A.1.2.1 With an 8-ml stopcock pipet, transfer 8 ml of sol$^{\underline{n}}$ A reagent blank sol$^{\underline{n}}$, 8 ml of each standard sol$^{\underline{n}}$, and 8 ml of each sample sol$^{\underline{n}}$ to 150-ml beakers. Add the following reagents.

A.1.2.2 Add 25 ml of acidified water to each beaker.

A.1.2.3 Add 25 ml of the molybdate reagent to each beaker.

A.1.2.4 Add 25 ml of the Tartaric acid sol$^{\underline{n}}$ to each beaker.

A.1.2.5 Add 25 ml of reducing sol$^{\underline{n}}$ to each beaker. Allow to stand for at least 45 min.

A.1.2.6 Determine the percent transmission for each sol$^{\underline{n}}$ at 640 nm using the reagent blank as the reference, or read percent SiO_2 using an automated spectrophotometer.

A.1.3 Results

limestone	1.78%	SiO_2
spent shale	41.77%	SiO_2
clay	66.54%	SiO_2

A.2 Determination of Al_2O_3 in Silicate and Carbonate Rocks

Alumina (Al_2O_3) is determined by measuring the absorption of light at 475 nm by a sol$^{\underline{n}}$ in which Alumina has been converted to a calcium aluminum-alizarin-red-S-complex (Parker and Goddard, 1950)

When 50-mg samples are used for the preparation of the A solutions and a 15-ml aliquot is used, the determination can be made on samples containing as much as 24 percent Al_2O_3. For samples containing from 24% to 36% Al_2O_3, a 10-ml aliquot of sol$^{\underline{n}}$ A should be used instead of the usual 15 ml. When a 10-ml aliquot is used, 5 ml of blank $sol^{\underline{n}}$ should also be added to the flask to maintain the same pH and concentration of inert salts as in the standard solutions. Carbonate-rock samples are run using a 200-mg sample.

Appreciable quantities of iron and titanium also form colored complexes that absorb at 475 nm. Interference from iron is eliminated by the use of potassium ferricyanide and thioglycolic acid as complexing agents, both of these reagents are required to completely eliminate this interference. The interference of titanium is not eliminated, but an empirically derived table is provided to correct for the effect of titanium.

A.2.1 Reagents

- Complexing $sol^{\underline{n}}$: To 880 ml of water add 0.3 gm potassium ferricyanide $K_3Fe(CN)_6$; 40 ml of hydroxylamine hydrochloride ($NH_2OH.HCl$) $sol^{\underline{n}}$, 10%, and 80 ml of $CaCl_2$ solution (Dissolve 14 gm $CaCO_3$ in 30 ml concentiated HCl and dilute to 1 l). Prepare this $sol^{\underline{n}}$ the same day it is to be used.

- Thioglycolic acid solution: Dilute 3 ml of the pure acid to 1 l with water.

- Buffer $sol^{\underline{n}}$: Dissolve 80 gm of sodium acetate $NaC_2H_3O_2.3H_2O$ in 975 ml H_2O and add 24 ml glacial acetic acid.

- Alizarin red-S stock $sol^{\underline{n}}$, 0.05%: Dissolve 0.5 gm of the pure dye in 1 l of water and filter.

- Alizarin red-S, 0.01%. Dilute 200 ml of the stock $sol^{\underline{n}}$ to 1 l with water.

A.2.2 Procedure

A.2.2.1 Transfer 15 ml each of the sol$^{\underline{n}}$ A reagent blank the std. sol$^{\underline{n}}$, and the sample solutions to a series of 150-ml beakers using a high precision piston pipet. Add the following reagents to each beaker.

A.2.2.2 Add 25 ml of the complexing sol$^{\underline{n}}$.

A.2.2.3 Add 25 ml of the thioglycolic acid sol$^{\underline{n}}$. Allow to stand for 5 min.

A.2.2.4 Add 25 ml of the buffer sol$^{\underline{n}}$. Allow to stand for 10 min.

A.2.2.5 Add 25 ml of the 0.01% alizarin red-S sol$^{\underline{n}}$. Allow to stand for 45 to 75 min.

A.2.2.6 Determine the % transmission at 475 nm for each sol$^{\underline{n}}$ using the blank sol$^{\underline{n}}$ as a reference, or read percent constituent with an automated spectrophotometer.

A.2.3 Results

limestone	0.65%	Al_2O_3
spent shale	14.67%	Al_2O_3
clay	15.67%	Al_2O_3

Preparation of Solution B

Solution B is used in the determination of total iron, MgO, CaO, TiO_2, P_2O_5, MnO, Na_2O, and K_2O.

The samples are digested on a steam bath overnight with a mixture of HF, H_2SO_4, and NHO_3. This procedure decomposes almost all the minerals that are normally present.

Organic matter is destroyed by the addition of a few drops of a mixture of perchloric and nitric acids after heating to sulfuric acid fumes. Any manganese dioxide formed by oxidation is then reduced and rendered soluble by the addition of hydrazine sulfate.

Rarely, appreciable residues do remain. If so, they should be removed, identified, and analyzed by suitable methods.

Reagents

Solution B acid mixture: Working under a fume hood, transfer the contents of a 454-g bottle of HF (48%) to a 1-l polyethylene bottle. Chill the HF in a bath of cold water. Keep the polyethylene bottle in the cold-water bath, add 165 ml of concentrated H_2SO_4, mix, and allow to cool. Add 40 ml of concentrated HNO_3 and mix.

$HCIO_4$-HNO_3 mixture: Mix 100 ml of $HCIO_4$ (72%) and 100 ml of concentrated HNO_3 Store in a 200-ml glass-stop-pered Pyrex bottle.

Hydrazine-sulfate solution, 0.2%: Dissolve 0.2 g $NH_2NH_2SO_4$ in 100 ml of water. Prepare fresh solution for each run.

Procedure

1. Transfer 0.500 g of each sample to special Teflon beakers.
2. Under a fume hood, add 15 ml of the solution B acid mixture to each Teflon beaker and swirl to wet the sample powder.
3. Cover the Teflon beakers with the Teflon covers, place the beakers in the steam bath so that most of each beaker is suspended in

steam, and heat overnight.

4. Remove the covers and continue to heat the beakers on the steam bath for about 1 hr or until acid fumes are no longer emitted.
5. Completely transfer the contents of the Teflon beakers to 400-ml Vycor beakers, using a minimum of water and a policeman to effect the transfer.
6. Place the Vycor beakers on a hot plate and heat until SO_3 fumes evolve, then remove the beakers.
7. When SO_3 fuming stops, add about 4 drops of the $HClO_4$-HNO_3 mixture to each beaker from a dropping bottle and replace the beakers on the hot plate. Heat each beaker until strong fumes are evolved and any color caused by organic matter had disappeared. Repeat this step if necessary.
8. Remove the beakers from the hot plate, allow them to cool for a few minutes, and then add about 225 ml of water, 5 ml of concentrated HNO_3, and 1 ml of the hydrazine sulfate solution.
9. Replace the beakers on the hot plate, and heat to boiling, if a brown precipitate of MnO_2 remains after the solutions have boiled for a few minutes, an additional milliliter of hydrazine sulfate should be added. If a residue remains after the solutions have been boiled for a few minutes, boiling should be continued for a additional half hours. A small residue can be ignored; an appreciable residue should be separated, identified, and analyzed by suitable means.
10. Cool the solutions to room temperature, then transfer them to 250-ml volumetric flasks.

11. Dilute to volume, mix, and transfer to polyethylene bottles.

A.3 Determination of Total Iron in Silicate, Carbonate and Phosphate Rocks.

The method for determining total iron (as Fe_2O_3) is based on the orange color developed with orthophenanthroline after the iron is reduced with hydroxylamine hydrochloride and the sol$^{\underline{n}}$ is buffered with sodium citrate (Bandemer and Schaible, 1944). When 5 ml of sol$^{\underline{n}}$ B in used, the method is designed to accommodate samples containing from a to 15% total iron (as Fe_2O_3). For the occasional sample in which Fe_2O_3 exceeds 15%, a smaller aliquot of sol$^{\underline{n}}$ B can be used.

A.3.1 Reagents

- Hydroxylamine hydrochloride, 10%: Dissolve 100 gm $NH_2OH.HCl$ in 1 l of water.

- Orthophenanthroline sol$^{\underline{n}}$ 0.1%: Dissolve 1 gm $C_{12}H_8N_2.H_2O$ in 1 l of water.

- Sodium citrate sol$^{\underline{n}}$, 10%: Dissolve 100 gm $Na_3C_6H_5O_7.2H_2O$ in 1 l of water.

- Standard iron solution: Add 0.491 gm $FeSO_4.(NH_4)_2SO_4.6H_2O$ to a 500 ml volumetric flask. Add 16 ml of 1+1 H_2SO_4, 10 ml of concentrated HNO_3 and about 300 ml of water.

- Stir until completely dissolved, then add water to the mark, and mix. The Fe_2O_3 concentration in this sol$^{\underline{n}}$ is 0.2 mg/ml, which is equivalent to that in a sol$^{\underline{n}}$ prepared from a sample containing 10% Fe_2O_3.

A.3.2 Procedure

A.3.2.1 With 5-ml pipet, transfer 5 ml of standard iron $sol^{\underline{n}}$ and 5 ml of each sample $sol^{\underline{n}}$ B to a series of 100 ml volumetric flasks. Use an additional flask for the preparation of a reagent blank $sol^{\underline{n}}$.

A.3.2.2 With a tilting pipet add 5 ml of the hydroxylamine hydrochloride $sol^{\underline{n}}$ to each flask and allow to stand for 10 min.

A.3.2.3 Add 20 ml of the orthophenanthroline $sol^{\underline{n}}$ to each flask, preferably with a pipetting machine.

A.3.2.4 Add 10 ml of the sodium citrate to each flask with a tilting pipet and dilute to 100 ml with water.

A.3.2.5 After 1 hr determine the % transmission at 555 nm for each $sol^{\underline{n}}$ by using the blank $sol^{\underline{n}}$ as the 100% reference.

A.3.3 Results

limestone	0.40%	Fe_2O_3
spent shale	3.90%	Fe_2O_3
clay	5.08%	Fe_2O_3

A.4 Determination of MgO, Na_2O and K_2O in Silicate, Carbonate and Phosphate Rocks and CaO in Silicate Rocks.

The alkaline earths, which were determined by automatic photometric titrations with EDTA in Shapin and Brannock (1962) and the alkalies previously determined by flame-emission techniques are now being determined by atomic absorption spectrometry. Lanthanum is added to all blanks, standards, and samples as a releasing agent (Dinnin, 1960) and the four elements are determined directly from the same $sol^{\underline{n}}$. In general, results can be expected to be ±1-2% of the amount present, which is adequate accuracy for these constituents at the levels normally encountered. At levels above 15%, the maximum concentration readable without furthur dilution, a better value for CaO or MgO can be obtained by visual titration with EDTA. For Na_2O and K_2O, a dilution is made, and the sol$^{\underline{ns}}$ are reread more carefully in duplicate and triplicate by AAS.

A.4.1 Reagents

- Rock standard solution.
- Lanthanum solution: Carefully dissolve 58 gm La_2O_3 (99.99% pure or better) in 250 ml of concentrated HCl in a 2-l beaker. When the reaction has subsided, dilute to 1 l with water.

A.4.2 Procedure

A.4.2.1 Transfer 4 ml of the std. sol$^{\underline{n}}$ and the sol$^{\underline{n}}$ B of each cample, using a transfer pipet, to a series of 200-ml plastic bottles or beakers. Plastic bottles are preferable if the solutions are to be stored for sometime before analysis.

A.4.2.2 Add 16 ml of Lanthanum sol$^{\underline{n}}$ to each bottle, preferably with a mechanical pipet.

A.4.2.3 Add 50 ml of water to each bottle, preferably with a mechanical pipet. These sol$^{\underline{ns}}$ are ready to be analyzed, or if they are tightly stoppered, they may be stired as long as a month.

A.4.2.4 Atomic absorption measurements are made following the proceding described by the manufacturer for the equipment in use. The std. solutions cover the workable range for each constituent and encompass concentrations derived from rocks. Where concentrations exceed 15% of the rock, the sol$^{\underline{n}}$ B can be diluted appropriately, but accuracy will be diminished unless duplicates or triplicates are run to improve the results.

A.4.2.5 Results are calculated from std. curves made from the std. solution. As the concentration of these solutions are already stated in terms of % constituent, no calculations are required once the curve is drawn.

A.4.3 Results from atomic absorption measurements:

	%K_2O	%Na_2O	%MgO	%CaO
limestone	0.10	trace	0.91	n.d.
spent shale	2.32	1.21	7.78	17.16
clay	1.95	0.30	2.05	1.46

%CaO of limestone is more than 20%, Hence, titration is used.

A.5 Total CaO + MgO in Carbonate and Phosphate Rocks:

Because CaO, P_2O_5 and CO_2 are the major constituents of carbonate or phosphate rocks, a higher accuracy is desirable for them than for the other components of these rocks. The comparatively low level of Al_2O_3 and Fe_2O_3 in these rocks, and the use of cyanide and triethanolamine (Biedermann and Schwarzenback, 1948; Pribil, 1953) to complex them makes a visual EDTA titration for CaO plus MgO feasible. Experience has shown that at the levels found in these rocks the visual titration for CaO + MgO is more accurate than the AAS techniques and is therefore to be preferred.

Calcium carbonate of American Chemical Society (A.C.S.) grade is used as std. A measured small quantity of Magnesium is added to all sol$^{\underline{ns}}$ prior to titration to ensure the presence of sufficient magnesium to convert the dye to its red complex so that the end point change will be sharp. The titration, always a large one with this type of rock, can be speeded up by decanting a small portion of the sol$^{\underline{n}}$ to be titrated into a small beaker and then titrating the main portion rapidly going slightly past the end point. This allows a good view of the appearance of the end point. When the decanted portion is restored to the main portion, the end point can then be relocated with more care. A mixed indicater is used to provide a small amount of yellow to serve as an internal filter which sharpens the appearance of the end point. Without the added yellow, the color changes from red to purple and then to blue, making it difficult to determine the end point. If the yellow is added, the end point is the complete disappearance of red, a distinctly discernible point.

Results obtained for CaO + MgO can be used, with results obtained for MgO by AAS to calculate CaO.

A.5.1 Reagents

- Standard sol$^{\underline{n}}$ for CaO + MgO: Transfer 0.5 gm of calcium carbonate (low in alkalies), A.C.S. grade, to a 150-ml beaker, add about 25 ml of water and 5 ml of HNO_3, cover with a watch glass, and bring to a boil on a hot plate. Cool to room temp, add 8 ml of 1+1 H_2SO_4, dilute with water to 250 ml in a volumetric flask, and mix.

- EDTA sol$^{\underline{n}}$, 0.3%: Dissolve 12 gm of disodium ethylenediamine tetraacetate in 4 l of water.

- Eriochrome black T sol$^{\underline{n}}$, 0.05%: Dissolve 50 mg of the dye in 100 ml of water. Do not store for more than 3 days.

- Hydroxylamine hydrochloride sol$^{\underline{n}}$, 10%: Dissolve 100 gm $NH_2OH.HCl$ in 1 l of water.

- Buffer sol$^{\underline{n}}$: Dissolve 66 gm of NH_4Cl in 500 ml of water, add 500 ml of concentrated NH_4OH and mix.

- Complexing sol$^{\underline{n}}$: Dissolve 64 gm of KCN in 600 ml of water, add 400 ml of triethanolamine, and mix.

- Methyl red sol$^{\underline{n}}$, 0.02%: Dissolve 200 mg of the pure dye in 1 l of water.

- Magnesium sulfate sol$^{\underline{n}}$: Dissolve 6 gm of $MgSO_4.7H_2O$ in 1 l of water.

A.5.2 Procedure

A.5.2.1 With a 25-ml transfer pipet, transfer two aliquots of the std. sol$^{\underline{n}}$ for CaO + MgO, an aliquot of the magnesium sulfate sol$^{\underline{n}}$, and aliquots of each B sol$^{\underline{n}}$ to a series of 400-ml beakers.

A.5.2.2 Add 5 ml of the hydroxylamine hydrochloride sol$^{\underline{n}}$ to each of the beakers and allow them to stand for 5 min.

A.5.2.3 Add about 200 ml of water to each beaker.

A.5.2.4 Add 5 ml of complexing sol$^{\underline{n}}$ to each beaker. Use a graduate or pipet that does not require mouth suction.

A.5.2.5 Add 25 ml of the buffer sol$^{\underline{n}}$ to each beaker using a graduate.

A.5.2.6 Add with a transfer pipet 2 ml of the magnesium sulfate sol$^{\underline{n}}$ to each beaker, including the beaker already containing magnesium sulfate.

A.5.2.7 To one of the solutions containing the std. CaO + MgO, add 1 ml of the Eriochrome black T sol$^{\underline{n}}$ and 1 ml of the methyl red sol$^{\underline{n}}$.

A.5.2.8 Decant about 10 ml of the sol$^{\underline{n}}$ into a small beaker and set aside for the moment.

A.5.2.9 Adjust the level of EDTA in the buret to zero, then add the EDTA rapidly to the sol$^{\underline{n}}$ in the 400-ml beaker in slight excess of that needed to get complete color change from red to blue.

A.5.2.10 Pour the sol$^{\underline{n}}$ which had been set aside back into the 400-ml beaker. The color of the sol$^{\underline{n}}$ should change back to red.

A.5.2.11 Titrate carefully until the red color of the sol$^{\underline{n}}$ disappears and the sol$^{\underline{n}}$ is dull grey-green in color. This is the end point.

A.5.2.12 Repeat steps 6-11 with each of the sample solutions, the magnesium sulfate sol$^{\underline{n}}$, and finally with the remaining std. CaO + MgO sol$^{\underline{n}}$.

A.5.3 Calculations:

A.5.3.1 Divide the volume for the titration of the magnesium sulfate sol$^{\underline{n}}$ by 13.5 to obtain the volume of the EDTA equivalent to 2 ml of the magnesium sol$^{\underline{n}}$ 82/13.5 = 6.074.

A.5.3.2 Subtract the volume of EDTA equivalent to 2 ml of the magnesium sol$^{\underline{n}}$ from each titration volume to obtain the volume of EDTA required for the 25 ml of each sol$^{\underline{n}}$ 67.920-6.074 = 61.846.

A.5.3.3 The % CaO in pure $CaCO_3$ is 56.0, therefore to calculate a factor:

56.000/Ave. of titration volumes for 25 ml of standards

56.000/61.846 = 0.9055 = 0.906

A.5.3.4 Calculate the percent total CaO + MgO, as CaO in each sample:

(Factor) x (titration volume for 25 ml of the sample sol$^{\underline{n}}$)

= % CaO + MgO calculated as CaO

0.906 x 60.668 = 54.965

A.5.3.5 The result obtained for CaO + MgO as CaO is used in the following manner %MgO (by atomic absorption) x 1.39 = CaO equivalent of MgO = 0.91 x 1.39 then

CaO + MgO (from step A.5.3.4) - CaO equivalent of MgO = %CaO

54.965 - 1.265 = 53.70

%CaO of limestone = 53.70%

A.6 Determination of loss on ignition in raw materials and cement.

Loss on ignition signifies the weight loss in percent which the sample undergoes when heated to a certain temperature during certain time intervals-dependent on the nature of the sample until constancy of weight is obtained.

A.6.1 Procedure

1 gramme (weighed to 4 decimals) is used for the determination of the loss on ignition. The sample is placed in the bottom of a heated and weighed platinum crucible. Before the heating all raw materials are incinerated over a Meker-burner for 15 min. So that only the bottom of the crucible is brought to a red heat. The crucible and the sample are heated in an electric furnace under one of the conditions mentioned below and cooled for 10 min. before each weighing, whereafter heating and weighing are continued until constancy of weight is obtained. In case of slag heating is finished when maximum of weight loss is reached.

A.6.2 Choice of heating temperature and heating time:

Materials	Temperature (°C)	1st heating (min).	2nd and following heating (min.)
Gypsum	400	15	5
Cement Clinker Slag	950	15	5
Raw materials with <72% total carbonate	1200	30	15
limestone Raw materials with >72% total carbonate	1200	60	0

A.6.3 Calculation:

$$\% \text{ loss on ignition} = \frac{(G-A) \times 100}{G}$$

where

A = ignition residue in grammes

G = weight of sample in grammes

A.6.4 Results:

Raw Materials

$$\text{limestone} = \frac{(1.0000-0.5754)100}{1.0000} = 42.46\%$$

$$\text{S.S.} = \frac{(1.0000-0.881)100}{1.0000} = 11.19\%$$

$$\text{clay} = \frac{(1.0000-0.9305)100}{1.0000} = 6.95\%$$

Product

$$\text{clinker} = \frac{(1.0000-0.9854)100}{1.0000} = 1.46\%$$

A.7 Determination of SiO_2 in cement.

SiO_2 in acid decomposable silicates ($3CaO, SiO_2$ and $2CaO, SiO_2$) is precipitated by dissolution and evaporation of the sample in strong acid. Any precipitated R_2O_3 is determined by treatment of the SiO_2 precipitate with HF ($SiO_2 + 4HF = SiF_4 + 2H_2O$).

A.7.1 Reagents:

Hydrochloric acid	HCl concentrated p.a.
Silver nitrate-nitric acid sol$^{\underline{n}}$	10 grammes of $AgNO_3$ + 30 ml HNO_3 concentrated p.a. + distilled water up to 100 ml.
Sulphuric acid	H_2SO_4 concentrated p.a.
Hydrofluoric acid	HF concentrated p.a.

A.7.2 Procedure:

A.7.2.1 A 0.5 gramme sample (weighed to 4 decimals) is placed in an evaporation dish and stirred with 10 ml water and 50 drops of HCl (3.3 ml). When all the material is decomposed, the sol$^{\underline{n}}$ is evaperated to dryness on a hot plate and placed in a quartz oven at 120-130^{o}C for 1 hour, or until HCl can no longer be smelt. After cooling, the substance is moistened with 20 drops of HCl and is left for 10 min. covered with a watchglass. Boiling water from a wash bottle is added, the solution is heated to boiling and filtered through an OOR-filter into a 400 ml beaker. The filtrate is washed free from chloride with boiling water. The filtrate is used in A.9.2 analysis R_2O_3.

A.7.2.2 The wet filter is placed in an ignited and weighed platinum crucible (c), it is dried and incinerated in the incinerator, and thereafter ignited over a Meker-burner until all the eval has been burnt away. It is then placed in an electric oven at 1200^{o}C for half an hour, after which it is cooled in the desiccator for 10 min. and weighed (b).

A.7.2.3 The silica is moistened with a little water, and the crucible is filled three quarter full with HF, and one drop of H_2SO_4 is added. After evaporation to dryness in the incinerater, the material is ignited over a Meker-burner, first cautiously, then strongly for 5 min. Finally, the material is ignited in an electric oven at 1200^{o}C for half an hour followed by cooling and weighing (a). The analysis is continued as described under A.9.2.

A.7.3 Calculation:

See A.9.3 (R_2O_3) a,b,c and d refer to this item

A.8 Determination of Insoluble residue in cement.

A.8.1 Reagents:

Hydrochloric acid	HCl concentrated p.a.
Sodium hydroxide solution	10 grammes of NaOH per 1000 ml of water (1%).
Methyl red	0.2% methyl red in 60% alcohol.
Ammonium nitrate solution	20 grammes of NH_4NO_2 per 1000 ml of water (2%).

A.8.2 Procedure:

An exact determination of the insoluble residue is made as follows:

a 1 gramme sample (weighed to 4 decimals) is stirred in a glass dish with 10 ml water, and 5 ml HCl are added. The solution is heated cautiously, diluted to 50 ml, and left on a hot plate for 15 min. covered with a watchglass (if necessary, on a triangle so that boiling is avoided). The solution is filtered through an OOR-filter and washed about 6 times with hot water.

The filter and the precipitate are placed in a dish and treated with 100 ml hot NaOH solution, heated to ebullition, and placed on a hot plate for 15 min. After neutralization with HCl (methyl red) + 4 to 5 drops in excess, the material is filtered through an OOR-filter and washed 12 to 15 times with a hot NH_4NO_3 solution. The filter and the insoluble residue are placed in an ignited and weighed platinum crucible, incinerated in the incinerator, and heated at 950°C for half an hour, followed by cooling and weighing.

A.8.3 Calculation:

$$\% \text{ insoluble residue} = \frac{A \times 100}{G}$$

A = residue of ignition in grammes

G = weight of sample in grammes

A.8.4 Results:

$$\% \text{ insoluble residue} = \frac{0.0030 \times 100}{1.0000}$$

$$\therefore \text{insoluble residue} = 0.30\%$$

A.9 Determination of R_2O_3 (Al_2O_3 + Fe_2O_3) in cement.

R_2O_3 designates the sum of aluminium and iron calculated as oxides. These substances are precipitated as hydroxides with ammonia (or as phosphates in a phosphate containing sample). Fe^{++} being oxidized beforehand with Br_2 to Fe^{+++}

$$Al^{+++} + 3OH^- = Al(OH)_3$$
$$Fe^{+++} + 3OH^- = Fe(OH)_3$$

After ignition the hydroxides are weighed as oxides (or phosphates)

$$2Al(OH)_3 = Al_2O_3 + 3H_2O$$
$$2Fe(OH)_3 = Fe_2O_3 + 3H_2O$$

Aluminum oxide is generally calculated as the difference between R_2O_3 (after expulsion of silicon), and the Fe_2O_3 content determined as described under A.13 any content of titanium and phosphate being thereby included in the aluminum oxide value.

A.9.1 Reagents:

Ammonium chloride	NH_4Cl solid p.a.
Bromine water	excess of Br_2 in distilled water.
Ammonia	NH_3 concentrated p.a. (about 25%).
Methyl red	0.2% methyl red in 60% alcohol.
Ammonium nitrate	NH_4NO_3 solid p.a.
Nitric acid	HNO_3 concentrated p.a.
Hydrofluoric acid	HF concentrated p.a.
Wash solution	10 grammes of NH_4NO_3 + concentrated NH_3 water till basic reaction (methyl red) + distilled water up to 1000 ml.

A.9.2 Procedure:

A.9.2.1 The filtrate from the SiO_2 determination (A.7.2.1) is evaporated to about 150 ml, and 2 grammes of NH_4Cl and 5 drops of bromine water are added. Excess of bromine is boiled off. Then add NH_3 by drops till basic reaction (2 drops of methyl red) and furthur 2 drops of NH_3. The boiling is continued for a few minutes, the basic reaction must keep (NH_3 vapours). The hot solution is filtered through an OOR-filter into a 600 ml beaker and is washed a few times with the boiling wash solution.

A.9.2.2 The filter and the precipitate are placed in the beaker used for the precipitation. The filter is destroyed, and the funnel is rinsed. 2 grammes of NH_4NO_3 and 100 ml water are added, and after heating to boiling, drops of HNO_3 are added until the precipitate is dissolved. In the boiling solution R_2O_3 is precipitated again by addition of a slight excess of NH_3. After a few minutes boiling, the solution is filtered quickly, and the precipitate washed free from chloride with the boiling wash solution (The Filtrate is used in A.10.2, CaO).

A.9.2.3 The filter and the precipitate are placed in the ignited platinum crucible used for the SiO_2 determination, they are dried and incinerated in the incinerator and ignited over a Meker-burner for half an hour (the crucible being held in an inclined position), afterwards in an electric oven at 1200°C for half an hour. After 10 minutes' cooling in the desiccator, the substance is weighed (e). After moistening the ignition residue with a little water, any coprecipitated SiO_2 is determined by HF and H_2SO_4 treatment. The precipitate is covered completely with HF and after evaporation to dryness in the incinerator, HF is treated in the incinerator with 5 or 6 drops of H_2SO_4, and the residue ignited over a Meker-burner, first cautiously, then strongly for 30 min. Finally, the crucible is left in an electric oven at 1200°C for half an hour, followed by cooling and weighing (d).

A.9.3 Calculation of SiO_2 and R_2O_3:

$$\% \ SiO_2 = \frac{[(b-a) + (e-d)] \times 100}{G}$$

$$\% \ R_2O_3 = \frac{(d-c) \times 100}{G}$$

where a,b,c,d,e are

SiO_2	crucible after treatment with HF	a	30.4572
	crucible + SiO_2	b	30.5594
	crucible	c	30.4564
R_2O_3	crucible after treatment with H_2SO_4	d	30.4987
	crucible + R_2O_3	e	30.4987
	crucible	c	30.4564

and G = weight of sample in grammes.

A.9.4 Results:

$$\% \ SiO_2 = \frac{[(30.5594-30.4572) + (30.4987-30.4987)]\,100}{0.5000}$$

$$\% SiO_2 \text{ in cement} = 20.44\%$$

$$\% \ R_2O_3 = \frac{(30.4987-30.4564)100}{0.5000}$$

$$\% R_2O_3 \text{ in cement} = 8.46\%$$

A.10 Determination of CaO in cement.

In the filtrate from the R_2O_3 determination (A.9.2) calcium is precipitated by addition of ammonium oxalate as the insoluble white calcium oxalate.

$$Ca^{++} + (COO^-)_2 = Ca\ (COO)_2$$

The precipitate is ignited and weighed as calcium oxide:

$$Ca\ (COO)_2 = CaO + CO_2 + CO$$

A.10.1 Reagents:

Ammonia	NH_3 concentrated p.a. (about 25%).
Methyl red	0.2% methyl red in 40% alcohol.

Ammonium oxalate	$(COONH_4)_2\ H_2O$ solid p.a.
Silver nitrate-nitric acid	10 grammes of $AgNO_3$ + 30 ml HNO_3 concentrated p.a. + distilled water up to 100 ml.
Wash solution	10 grammes of $(COONH_4)_2\ H_2O$ + distilled water up to 1000 ml.

A.10.2 Procedure:

A.10.2.1 The total filtrate from the R_2O_3 determination (A.9.2.2) is evaporated to about 300 ml, and after heating to boiling, add NH_3 till basic reaction (methyl red) and 2 grammes of $(COONH_4)_2\ H_2O$ (4 times the weight of the sample). After two minutes boiling the beaker is left for two hours on a hot plate covered with a watch glass. The solution is filtered through an OOM-filter into a 600 ml beaker and washed free from chloride with the boiling wash solution.

A.10.2.2 The filter and the precipitate are transferred to an ignited and weighed platinum crucible and incinerated in the incinerater, ignited over a Meker-burner and thereafter in an electric oven at 1200°C for 3/4 hour. The material is cooled in the desiccater (only three crucibles at a time) for exactly 10 min and weighed, then ignited repeatedly for half an hour to constancy of weight.

A.10.3 Chloride test:

About 1 ml of the filtrate is placed in a test tube, by addition of 1 ml of silver nitrate-nitric acid solution this sample must not show sign of turbidity. The sample is discarded. In the chloride test an ample quantity of HNO_3 is added to secure complete dissolution of $(COOAg)_2$.

A.10.4 Notes:

Hot plate corresponds to water bath, i.e. a temperature of about 90°C on the surface of the plate.

It is important that the cooling in the desiccator lasts exactly 10 min., since this time limit permits the material to get cold at the same time as an absorption of CO_2 is avoided.

A.10.5 Calculation:

$$\% \text{ CaO} = \frac{A \times 100}{G}$$

where

A = ignition residue in grammes
G = weight of sample in grammes

A.10.6 Results:

$$\% \text{ CaO} = (0.31985 \times 100)/0.5000$$
$$\text{CaO} = 63.97\%$$

A.11 Determination of MgO in cement.

In the filtrate from the CaO precipitation (A.10.2) magnesium is precipitated as magnesium ammonium phosphate by addition of disodium hydrogene phosphate and ammonia.

$$Mg^{++} + NH_3 + HPO_4^{---} = MgNH_4PO_4$$

The precipitate is ignited and weighed as magnesium pyrophosphate:

$$2MgNH_4PO_4 \;=\; Mg_2P_2O_7 + 2NH_3 + H_2O$$

The result is converted into %MgO.

A.11.1 Reagents:

- Hydrochloric acid	HCl concentrated p.a.
- Ammonia	NH_3 concentrated p.a. (about 25%)
- Disodium hydrogene phosphate solution	100 gm of $Na_2HPO_4.12H_2O$ + distilled water up to 1000 ml (10%).
- Ammonia water	100 ml NH_3 concentrate p.a. + distilled water up to 1000 ml (2.5%).

A.11.2 Procedure:

A.11.2.1 The total filtrate from the CaO precipitation (A. 10.2.1), CaO is made slightly acid by addition of HCl and evaporated to about 200 ml. After cooling, 40 ml (NH_3) ammonia and 10 ml Na_2HPO_4 solution are added, the mixture is beated with a glass spatula till it becomes turbid and left for complete precipitation during at least 12 hours.

A.11.2.2 The cold solution is filtered through an OOH-filter, and the precipitate is washed with cold ammonia water. The filter with precipitate is transferred to an ignited and weighed porcelain crucible, incinerated in the incinerator and ignited for half an hour over a Meker-burner (but not at so high a temperature that the material is melted), then cooled and weighed.

A.11.3 Calculation:

$$\%MgO = \frac{0.3623 \times A \times 100}{G}$$

where

A = ignition residue in grammes

G = weight of sample in grammes

$$0.3623 = \frac{\text{molecular weight of MgO}}{0.5 \times \text{molecular weight of } Mg_2P_2O_7}$$

A.11.4 Notes:

A.11.4.1 During the evaporation of the filtrate from CaO the liquid should be slightly acid in order to prevent precipitation of magnesium salts.

A.11.4.2 $MgNH_4PO_4$ is somewhat soluble in water for which reason a wash solution containing NH_3 is used.

A.11.4.3 A percelain crucible must be used, since ignited $MgNH_4PO_4$ in combination with C (from the filter paper) may attack the platinum, possibly because of a reduction of the material to phosphorus or free hydrogene.

A.11.5 Results:

$$\%MgO = \frac{0.3623 \times 0.0319 \times 100}{0.5000}$$

$$MgO = 2.31\%$$

A.12 Determination of SO_3 in cement.

In this method, sulfate is precipitated from an acid solution of the cement with barium chloride ($BaCl_2$). The precipitate is ignited and weighed as barium sulfate ($BaSO_4$) and the SO_3 equivalent is calculated.

A.12.1 Procedure:

To 1 gm of the sample add 25 ml of cold water and, while the mixture is stirred vigorously, add 5 ml of HCl (note A.12.1.1). If necessary, heat the solution and grind the material with the flattened end of a glass rod until it is evident that that decomposition of the cement is complete (note A.12.1.2). Dilute the solution to 50 ml and digest for 15 min. at a temperature just below boiling. Filter through a medium textured paper and wash the residue thoroughly with hot water. Dilute the filtrate to 250 ml and heat to boiling. Add slowly, dropwise, 10 ml of hot $BaCl_2$ (100 g/liter) and continue the boiling until the precipitate is well formed. Digest the solution for 12 to 24 hour at a temperature just below boiling (note A.12.1.3). Take care to keep the volume of solution between 225 and 260 ml and add water for this purpose if necessary. Filter through a retentive paper, wash the precipitate with hot water, place the paper and contents in a weighed platinum crucible, and slowly char and consume the paper without inflaming. Ignite at 800 to 900°C, cool in a desiccator, and weigh.

A.12.1.1 Note:

The acid filtrate obtained in the determination of the insoluble residue (A.8) may be used for the determination of SO_3 instead of using a separate sample.

A.12.1.2 Note:

If a rapid determination is desired, the time of digestion may be reduced to as little as 3 hour. However, the cement may be rejected for failure to meet the specification requirement only on the basis of results obtained when using 12 to 24 hour digestion times.

A.12.2 Blank:

Make a blank determination following to same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

A.12.3 Calculation:

Calculate the percentage of SO_3 to the nearest 0.01 as follows:

$$SO_3\ (\%) = W \times 34.3$$

where:

W = grams of $BaSO_4$

34.3 = molecular ratio of SO_3 to $BaSO_4$ (0.343) multiplied by 100

A.12.4 Results:

$$SO_3\ (\%) = 0.0668 \times 3.43$$

$$SO_3 = 2.29\%$$

A.13 Determination of Fe_2O_3 in cement.

Divalent iron (ferrous) may be oxidized quantitatively to trivalent (ferric) with potassium dichromate in a cold, acid solution:

$$6Fe^{++} + Cr_2O_7^{--} + 14H^{+} = 6Fe^{+++} + 2Cr^{+++} + 7H_2O$$

The sodium salt of diphenyl-amino-p-sulpho-acid is used as an indicator. Any ferric ions present are reduced before the titration with stannous chloride:

$$2Fe^{+++} + Sn^{++} = 2Fe^{++} + Sn^{++++}$$

Excess of stannous chloride is removed with mercuric chloride:

$$Sn^{++} + 2Hg^{++} = Sn^{++++} + 2Hg^{+}$$

The mercurous ions formed are precipitated at once as mercurous chloride:

$$2Hg^{+} + 2Cl^{-} = Hg_2Cl_2$$

A.13.1 Reagents:

Hydrochloric acid	HCl concentrated p.a.
Stannous chloride solution	5 gm $SnCl_2.2H_2O$ are dissolved in 5 ml concentrated HCl and diluted with 95 ml distilled water, add a few metallic tin lumps.

Mercuric chloride solution	5 gm $HgCl_2$ + distilled water up to 100 ml.
Acid solution	350 ml distilled water + 150 ml 85% H_3PO_4 are mixed after cooling with a cooled mixture of 350 ml distilled water + 150 ml 96% H_2SO_4.
Indicator solution	0.2% aqueous solution of the sodium salt of diphenyl-amino-p-sulpho-acid.
Sodium chromate solution	0.98 gm $K_2Cr_2O_7$ p.a., dried in advance for 1 hour at 200°C + distilled water up to 1000 ml.

$$\text{normality} = \frac{\text{gm } K_2Cr_2O_7}{49.04}$$

(about 0.02)

A.13.2 Procedure:

The filtrate from the decomposition is parboiled, and drops of $SnCl_2$ solution are added until the liquid is colourless, and after complete cooling add 10 ml $HgCl_2$ solution, 15 ml acid mixture and 4 drops of the indicator solution. The liquid is titrated with $K_2Cr_2O_7$ solution until colour change from green over grey to purple.

A.13.3 Notes:

A.13.3.1 The Hg_2Cl_2 precipitated should be white. If it is gray it indicates that too much $SnCl_2$ solution was used for the reduction, and that free Hg has therefore been precipitated which might react with $K_2Cr_2O_7$ and thus make the determination inaccurate.

A.13.3.2 H_3PO_4 is added in order to elucidate the change of colour, since the yellowish coloured ferric ions form the complex ferric phosphate which is colourless.

A.13.4 Calculation:

$$\% \ Fe_2O_3 = \frac{b.n.\ 7.985}{G}$$

where

b = ml $K_2Cr_2O_7$ solution used

n = normality of $K_2Cr_2O_7$ solution

G = weight of sample in grammes

$$7.985 = \frac{159.7 \times 100}{1000 \times 2} \quad \left(\frac{159.7}{2} = \text{equivalent weight for } Fe_2O_3\right)$$

A.13.5 Results:

$$\% \ Fe_2O_3 = \frac{8.08 \times 0.02 \times 7.985}{0.5000}$$

$$= 2.581$$

% Fe_2O_3 in cement = 2.58%

Find % Al_2O_3 = % R_2O_3 - % Fe_2O_3

= 8.46-2.58

% Al_2O_3 in cement = 5.88%

A.14 Determination of Na_2O and K_2O in cement.

A.14.1 Reagents:

Hydrochloric acid	HCl, conc. p.a.
Cement Standard	cement which contain Na_2O, K_2O = 0.2, 0.4, 0.5%

A.14.2 Procedure:

A 0.2 gramme sample (weighed to 4 decimals) is placed in a 100 ml beaker. 30 ml distilled water and 3-4 ml HCl are added. To dissolved the suspended solution, warm if necessary. Filtered through into the volumetric flask, wash the filter and beaker with distilled water. Then make up the filtrate to 250 ml.

Run the solution compare to the standard cement by AAS. Record the result.

A.14.3 Results:

% Na_2O in cement = 0.40

% K_2O in cement = 0.35

A.15 Determination of Free lime (CaO) in cement.

Glycol Titration

The free lime in cements in determined by extraction with glycol:

$$CaO + (CH_2OH)_2 = (CH_2O)_2\,Ca + H_2O$$

and the calcium glycolate formed is titrated with hydrochloric acid:

$$(CH_2O)_2\,Ca + 2HCl = (CH_2OH)_2 + CaCl_2$$

This process does not run completely to the end, but stops at an equilibrium. In the calculation the percentage of free lime is, therefore, determined from a standard curve, where ml acid is plotted against a known percentage of free lime.

A.15.1 Reagents:

Glycol	$(CH_2OH)_2$; pure and waterfree.
Bromocresolgreen	1% bromocresolgreen solution in alcohol (96%).
Hydrochloric acid	HCl, 0.1 normal solution (standardized against 0.35 g $Na_2B_4O_7.10H_2O$ with bromocresolgreen as an indicator)

$$N = \frac{(\text{grammes of } Na_2B_4O_7.10H_2O)\,1000}{190.74 \times (\text{ml HCl})}$$

A.15.2 Procedure:

In a dry 100 ml conical flask weigh out 1,000 g of sample; in the case of samples containing more than 3% free lime, only about 0.3 gramme (weighed to 4 dec.) is used.

Add 50 ml dry, warm (65-70°C) glycol to the flask, and close it with a cork stopper. Place the flask in a water bath at 65-70°C, with a magnetic stirrer, for 30 min. The solution is then filtered under suction through a 5 cm Buchner funnel (with two filter papers) into a 250 ml suction flask. The precipitate is washed with 10 ml warm (65-70°C) glycol three times (total 30 ml). The filtrate, which must be quite clear, is titrated after addition of 2 drops of bromocresolgreen indicator, with the standardized 0.1 normal HCl solution to a blue-green colour change.

A.15.3 Calculation:

A.15.3.1 The percentage of free lime is found from the standard curve (Fig. A.15.1) as the value corresponding to the amount of ml 0.1 n HCl used.

If the weight of the sample has not been 1.000 gramme, the calculated value must be divided by the weight used.

A.15.3.2 If the titration has used more HCl than the highest value on the standard curve, the free lime percentage can be calculated like this:

$$\% \text{ CaO} = \frac{n \times A - \{k \times (n-1)\}}{G}$$

where

n = the factor with which the ml 0.1 n HCl used is divided to get a usable value for the curve = $\frac{56}{60}$ = 0.93,

A = % CaO calculated from the curve with the value referred to above.

k = the % CaO which indicates the intersection of the curve and the abscissa = 0.05.

G = weight of sample in grammes.

A.15.4 The standard curve:

In a platinum crucible analytically pure $CaCo_3$ is ignited in an electric oven at 1200°C for 30 min.

From the ignition residue (pure CaO) weigh out 10 samples of:

5 mg-10 mg-20 mg-30 mg-40 mg

5 mg-10 mg-20 mg-30 mg-40 mg

The samples are transferred to 300 ml conical flasks and dissolved in 50 ml warm (65-70°C) glycol, and placed in a water bath at 65-70°C with magnetic stirrer for 30 min. As pure CaO is soluble in glycol, filtration is not necessary. To each of the solutions add 30 ml warm (65-70°C) glycol and carry out the titration as described under A.15.2.

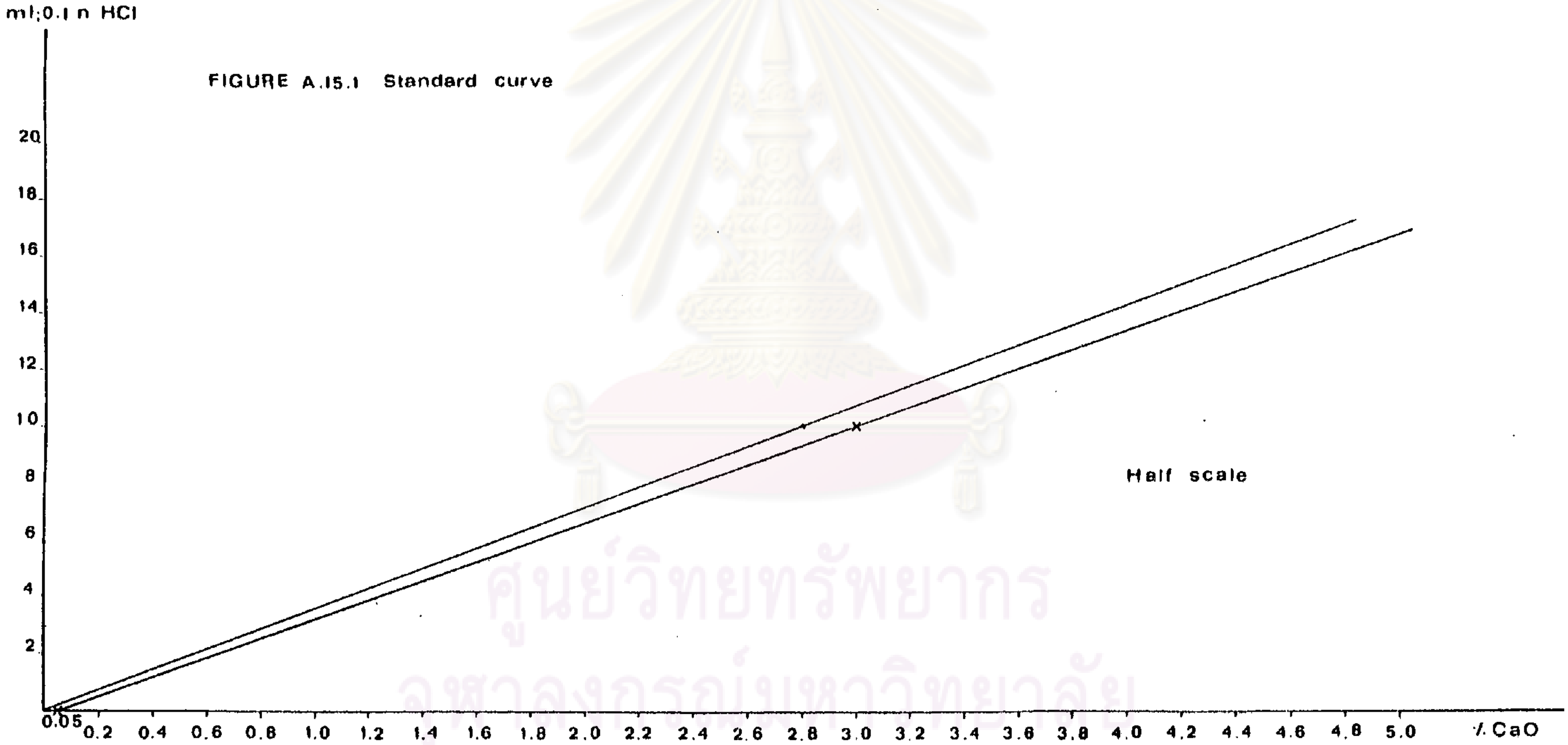
The standard curve is now drawn by plotting the amount in ml of 0.1 normal HCl used on the axis of the ordinates (1 ml∿10 mm) and the corresponding % CaO (grammes of CaO x 100 = % CaO) on the abscissa (0.1% = 10 mm).

A.15.5 Notes:

A.15.5.1 The small amounts of acid or base which might be present in the glycol, are neutralized in the following way: 50 ml glycol are titrated with 0.1 N HCl (possibly, with 0.1 N NaOH) with bromocresolgreen as an indicator. It should be calculated how many ml acid or base are required to neutralize 1000 ml glycol. After neutralization, dry the 1000 ml glycol, see A.15.5.2.

A.15.5.2 The maximum content of water in the glycol is 1%: 50 ml of glycol are shaken for 1 hour with 0.5 gm waterfree $CuSO_4$,

FIGURE A.15.1 Standard curve



and after shaking, the mixture must only have a faint green colour, if the colour is stronger, the water must be removed by heating the glycol to 120°C for 3 hours.

A.15.5.3 It is important to place the filter papers carefully on the Buchner funnel (A.15.2). The paper are sucked firmly to the funnel after moistening with glycol.

A.15.5.4 During the filtration (A.15.2) it is necessary to place a big filter flask between the sample flask and the pump to prevent water from being sucked into the sample flask.

A.15.5.5 The weighing out of the ignited CaO (A.9.4) must be carried out very fast to prevent water absorption.

A.15.5.6 As an indicator a mixed indicator bromocresolgreen methyl red, might as well be used:

Dissolve 0.050 gramme of bromocresolgreen in 40 ml absolute ethanol. Dissolve 0.050 gramme of methyl red in 40 ml absolute ethanol. Mix the solutions and dilute to 100 ml with absolute ethanol in a volumetric flask. Colour change: green over colourless to pink on addition of 2 drops of acid.

A.15.6 Results:

3-component raw mixture at various temp and time

Temp , Time	ml 0.1 n HCl used	% free lime
1300°C, 45 min	31.0	26.95
1300°C, 1 hr	18.6	16.14
1300°C, 2 hr	7.4	2.30
1350°C, 45 min	32.6	9.72
1350°C, 1 hr	17.3	5.20
1350°C, 2 hr	0.4	0.22
1400°C, 45 min	0.5	0.25
1400°C, 1 hr	0.3	0.17
clinker	1.6	0.45

Appendix B: PHYSICAL PROPERTIES TEST

B.1 Standard Test Method for Fineness of Portland Cement By Air Permeability Apparatus (ASTM:C 204-55)

B.1.1 Scope

This method covers determination of the fineness of portland cement, using the Blaine air permeability apparatus, in terms of the specific surface expressed as total surface area in cm^2/gm, or m^2/kg, of cement.

B.1.2 Apparatus

B.1.2.1 Nature of apparatus - The Blaine air permeability apparatus consists essentially of a means of drawing a definite quantity of air through a prepared bed of cement of definite porosity. The number and size of the pores in a prepared bed of definite porosity is a function of the size of the particles and determines the rate of airflow through the bed. The apparatus, illustrated in Figure B.1.1, shall consist specifically of the parts described in B.1.2.2 to B.1.2.8.

B.1.2.2 Permeability cell - The permeability cell shall consist of a rigid cylinder 12.7±1 mm in inside diameter, constructed of glass or noncorroding metal. The interior of the cell shall have a finish of 32μ in (0.81μm). The top of the cell shall be a right angles to the principal axis of the cell. The lower portion of the cell must be able to form an airtight fit with the upper end of the manometer, so that there is no air leakage between the contacting surfaces. A ledge 0.5 to 1.0 mm in width shall be an integral part of the cell or be firmly fixed in the cell 50±15 mm from the top of the cell for support of the perforated metal disk.

B.1.2.3 Disk - The disk shall be constructed of noncorroding metal and shall be 0.9±0.1 mm in the thickness, perforated with 30 to 40 holes 1 mm in diameter equally distributed over its area. The disk shall fit the inside of the cell snugly. The center portion of one side of the disk shall be marked or inscribed in a legible manner so as to permit the operator always to place that side downwards when inserting it into the cell. The marking or inscription shall not extend into any of the holes, nor touch their peripheries, nor extend into that area of the disk that rests on the cell ledge.

B.1.2.4 Plunger - The plunger shall be constructed of noncorroding metal and shall fit into the cell with a clearance of not more than 0.1 mm. The bottom of the plunger shall sharply meet the lateral surfaces and shall be at right angles to the principal axis. An airvent shall be provided by means of a flat 3±0.3 mm. wide on one side of the plunger. The top of the plunger shall be provided with a collar such that when the plunger is placed in the cell and the collar brought in contact with the top of the cell, the distance between the bottom of the plunger and the top of the perforated disk shall be 15±1 mm.

B.1.2.5 Filter paper - The filter paper shall be medium retentive, corresponding to Type 1, Grade B, as prescribed in Federal Specification for Paper; Filtering (UU-P-236). The filter paper disks shall be circular, with smooth edges, and shall have the same diameter as the inside of the cell.

B.1.2.6 Manometer - The U-tube manometer shall be constructed according to the design indicated in Fig. B.1.1, using nominal 9 mm outside diameter, standard-wall, glass tubing. The top of one arm of the manometer shall form an airtight connection with the permeability cell. The manometer arm connected to the permeability cell shall have a line etched around the tube at 125 to 145 mm below the top side outlet and also others at distances of 15±1 mm, 70±1 mm and 110±1 mm above that line. A side outlet shall be provided at 250 to 305 mm above the bottom of the manometer for use in the evacuation of the manometer arm connected to the permeability cell. A positive airtight value or clamp shall be provided on the side outlet not more than 50 mm from the manometer arm. The manometer shall be mounted firmly and in such a manner that the arms are vertical.

B.1.2.7 Manometer liquid - The manometer shall be filled to the midpoint with a nonvolatile, nonhydroscopic liquid of low viscosity and density, such as dibutyl phthalate (dibutyl 1, 2-benzene-dicarboxylate) or a light grade of mineral oil.

B.1.2.8 Timer - The timer shall have a positive starting and stopping mechanism and shall be capable of being read to the nearest 0.5 second or less.

B.1.3 Calibration of Apparatus

B.1.3.1 Sample - The calibration of the air permeability apparatus shall be made using the current lot of National Bureau of Standards. Standard Reference Material No. 114. The sample shall be at room temperature when tested.

B.1.3.2 Bulk volume of compacted bed of powder - Determine the bulk volume of the compacted bed of powder by the mercury displacement method as follows:

B.1.3.2.1 Place two filter paper disks in the permeability cell, pressing down the edges, using a rod having a diameter slightly smaller than that of the cell, until the filter disks are flat on the perforated metal disk; then fill the cell with mercury, ACS reagent grade or better, removing any air bubbles adhering to the wall of the cell. Use tongs when handling the cell. If the cell is made of material that will amalgamate with mercury, the interior of the cell shall be protected by a very thin film of oil just prior to adding the mercury. Level the mercury with the top of the cell by lightly pressing a small glass against the mercury surface until the glass is flush to the surface of the mercury and rim of the cell, being sure that no bubble or void exists between the mercury surface and the glass plate. Remove the mercury from the cell, weigh, and record the weight of the mercury. Remove one of the filter disks from the cell. Using a trial quantity of 2.80 gm of cement (Note B.1.1) compress the cement (Note B.1.2) in accordance with 4.5 with one filter disk above and one below the sample. Into the unfilled space at the top of the cell, add mercury, remove entrapped air, and level off the top as before. Remove the mercury from the cell, weigh, and record the weight of the mercury.

B.1.3.2.2 Calculate the bulk volume occupied by the cement to the nearest 0.005 cm^3 as follows:

$$V = (W_A - W_B)/D \ldots\ldots\ldots\ldots\ldots\ldots \qquad 1$$

where

V = bulk volume of cement, cm^3

W_A = grams of mercury required to fill the cell, no cement being in the cell.

W_B = grams of mercury required to fill the portion of the cell not occupied by the prepared bed of cement in the cell.

D = density of mercury at the temperature of test, g/cm^3 (see Table B.1.1)

B.1.3.2.3 Make at least two determinations of bulk volume of cement, using separate compactions for each determination. The bulk volume value used for subsequent calculations shall be the average of two values agreeing within ±0.005 cm^3. Note the temperature in the vicinity of the cell and record at the beginning and end of the determination.

Note B.1.1 It is not necessary to use the standard sample for the bulk volume determination.

Note B.1.2 The prepared bed of cement shall be firm. If too loose or if the cement cannot be compressed to the desired volume, adjust the trial quantity of cement used.

B.1.3.3 Preparation of Sample - Enclose the contents of a vial of the standard cement sample in a 4-oz jar (approximately 120 cm^3) and shake vigorously for 2 min to fluff the cement and break up lumps or agglomerates. Allow the jar to stand unopened for a further 2 min, then remove the lid and stir gently to distribute throughout the sample the fine fraction that has settled on the surface after fluffing.

B.1.3.4 Weight of Sample - The weight of the standard sample used for the calibration test shall be that required to produce a bed of cement having a porosity of 0.500±0.005, and shall be calculated as follows:

$$W = \rho V(1-\varepsilon) \quad \ldots\ldots\ldots\ldots 2$$

where:

W = grams of sample required,

ρ = density of test sample (for portland cement a value of 3.15 Mg/m^3 or 3.15 g/cm^3 shall be used),

V = bulk volume of bed of cement, cm^3, as determined in accordance with B.1.3.2.

ε = desired porosity of bed of cement (0.500± 0.005).

B.1.3.5 Preparation of Bed of Cement - Seat the perforated disk on the ledge in the permeability cell, inscribed or marked face down. Place a filter paper disk on the metal disk and press the edges down with a rod having a diameter slightly smaller than that of the cell. Weigh to the nearest 0.001 g the quantity of cement determined in accordance with B.1.3.4 and place in the cell. Tap the side of the cell lightly in order to level the bed of cement. Place a filter paper disk on top of the cement and compress the cement with the plunger until the plunger collar is in contact with the top of the cell. Slowly withdraw the plunger a short distance, rotate about 90 deg, repress, and then slowly withdraw. Use of fresh disks is required for each determination.

B.1.3.6 Permeability Test:

B.1.3.6.1 Attach the permeability cell to the manometer tube, making certain that an airtight connection is obtained and taking care not to jar or disturb the prepared bed of cement.

B.1.3.6.2 Slowly evacuate the air in the one arm of the manometer U-tube until the liquid reaches the top mark, and then close the valve tightly. Start the timer when the bottom of the meniscus of the manometer liquid reaches the second (next to the top) mark and stop when the bottom of the meniscus of liquid reaches the third (next to the bottom) mark. Note the time interval measured and record in seconds. Note the temperature of test and record in degrees Celsius.

B.1.3.6.3 In the calibration of the instrument, make at least three determinations of the time of flow on each of three separately prepared beds of the standard sample (Note B.1.3). The calibration shall be made by the same operator who makes the fineness determination.

Note B.1.3 The sample may be refluffed and reused for preparation of the test bed, provided that it is kept dry and all tests are made within 4 hr of the opening of the sample.

B.1.3.7 Recalibration - The apparatus shall be recalibrated (Note B.1.4)

B.1.3.7.1 At periodic intervals to correct for possible wear on the plunger or permeability cell,

B.1.3.7.2 If any loss in the manometer fluid occurs, or

B.1.3.7.3 If a change is made in the type of quality of the filter paper used for the tests.

Note B.1.4 It is suggested that a secondary sample be prepared and used as a fineness standard for the check determinations of the instrument between regular calibrations with the standard cement sample.

B.1.4 Procedure:

B.1.4.1 Temperature of Cement - The cement sample shall be at room temperature when tested.

B.1.4.2 Size of Test Sample - The weight of sample used for the test shall be the same as that used in the calibration test on the standard sample, except that when determining the fineness of Type III cements, or other types of fine-ground cements whose bulk for this weight is so great that ordinary thumb pressure will not cause the plunger collar to contact the top of the cell, the weight of sample shall be that required to produce a test bed having a porosity of 0.530±0.005.

B.1.4.3 Preparation of Bed of Cement - Prepare the test bed of cement in accordance with the method described in B.1.3.5.

B.1.4.4 Permeability Tests - Make the permeability tests in accordance with the method described in B.1.3.6, except that only one time-of-flow determination need be made on each bed.

B.1.5 Calculation

B.1.5.1 Calculate the specific surface values in accordance with the following equations:

$$S = \frac{S_s\sqrt{T}}{\sqrt{T_s}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3$$

$$S = \frac{S_s\sqrt{\eta_s}\sqrt{T}}{\sqrt{T_s}\sqrt{\eta}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 4$$

$$S = \frac{S_s(1-\varepsilon_s)\sqrt{\varepsilon^3}\sqrt{T}}{\sqrt{\varepsilon_s^3}\sqrt{T_s}(1-\varepsilon)} \quad \cdots\cdots\cdots\cdots 5$$

$$S = \frac{S_s(1-\varepsilon_s)\sqrt{\varepsilon^3}\sqrt{\eta_s}\sqrt{T}}{\sqrt{\varepsilon_s^3}\sqrt{T_s}\sqrt{\eta}(1-\varepsilon)} \quad \cdots\cdots\cdots\cdots 6$$

$$S = \frac{S_s\rho_s(1-\varepsilon_s)\sqrt{\varepsilon^3}\sqrt{T}}{\rho(1-\varepsilon)\sqrt{\varepsilon_s^3}\sqrt{T_s}} \quad \cdots\cdots\cdots\cdots 7$$

$$S = \frac{S_s\rho_s(1-\varepsilon_s)\sqrt{\eta_s}\sqrt{\varepsilon^3}\sqrt{T}}{\rho(1-\varepsilon)\sqrt{\varepsilon_s^3}\sqrt{T_s}\sqrt{\eta}} \quad \cdots\cdots\cdots\cdots 8$$

where:

S = specific surface of the test sample, cm^2/g,

S_s = specific surface of the standard sample used in calibration of the apparatus, cm^2/g,

T = measured time interval, S, of manometer drop for test sample (Note B.1.5)

T_s = measured time interval, S, of manometer drop for standard sample used in calibration of the apparatus (Note B.1.5)

η = viscosity of air, poises, at the temperature of test of the test sample (Note B.1.5)

η_s = viscosity of air, poises, at the temperature of test of the standard sample used in calibration of the apparatus (Note B.1.5)

ε = porosity of prepared bed of test sample (Note B.1.5)

ε_s = porosity of prepared bed of standard sample used in calibration of apparatus (Note B.1.5)

ρ = density of test sample (for portland cement a value of 3.15 Mg/M^3 or 3.15 g/cm^3 shall be used), and

ρ_s = density of standard sample used in calibration of apparatus (assumed to be 3.15 Mg/m^3 or 3.15 g/cm^3).

Note B.1.5 Values for $\sqrt{\eta}$, $\sqrt{\varepsilon^3}$, and $\sqrt{T}$ may be taken from Tables B.1.1, B.1.2, and B.1.3.

Equations 3 and 4 shall be used in calculations of fineness of portland cements compacted to the same porosity as the standard fineness sample. Equation 3 is used if the temperature of test of the test sample is within ±3°C of the temperature of calibration test, and Eq 4 is used if the temperature of test of the test sample is outside of this range.

Equations 5 and 6 shall be used in calculation of fineness of portland cements compacted to some porosity other than that of the standard fineness sample used in the calibration test. Equation 5 is used if the temperature of test of the test sample is within ±3°C of the temperature of calibration test of the standard fineness sample, and Eq 6 is used if the temperature of test of the test sample is outside of this range.

Equations 7 and 8 shall be used in calculation of fineness of materials other than portland cement. Equation 7 shall be used when the temperature of test of the test sample is within ±3°C of the temperature of calibration test, and Eq 8 is used if the temperature of test of the test sample is outside of this range.

B.1.5.2 To calculate the specific surface values in square metres per kilogram, multiply the surface area in cm^2/g by the factor of 0.1.

B.1.5.3 Round values in cm^2/g to the nearest 10 units (in m^2/kg to the nearest unit). Example: 3447 cm^2/g is rounded to 3450 cm^2/g or 345 m^2/kg.

Table B.1.1 Density of Mercury, Viscosity of Air (η), and $\sqrt{\eta}$ at Given Temperatures

Room Temperature, °C	Density of Mercury, g/cm^3	Viscosity of Air, η	$\sqrt{\eta}$
16	13.56	0.0001788	0.01337
18	13.55	0.0001798	0.01341
20	13.55	0.0001808	0.01345
22	13.54	0.0001818	0.01348
24	13.54	0.0001828	0.01352
26	13.53	0.0001837	0.01355
28	13.53	0.0001847	0.01359
30	13.52	0.0001857	0.01363
32	13.52	0.0001867	0.01366
34	13.51	0.0001876	0.01370

Table B.1.2 Values for Porosity of Cement Bed

Porosity of Bed, ε	$\sqrt{\varepsilon^3}$
0.495	0.348
0.496	0.349
0.497	0.350
0.498	0.351
0.499	0.352
0.500	0.354
0.501	0.355
0.502	0.356
0.503	0.357
0.504	0.358
0.505	0.359
0.506	0.360
0.507	0.361
0.508	0.362
0.509	0.363
0.510	0.364
0.525	0.380
0.526	0.381
0.527	0.383
0.528	0.384
0.529	0.385
0.530	0.386
0.531	0.387
0.532	0.388
0.533	0.389
0.534	0.390
0.535	0.391

Table B.1.3 Time of Airflow

T = time of airflow in seconds; √T = the factor for use in the equations

T	√T	T	√T	T	√T	T	√T	T	√T	T	√T
26	5.10	51	7.14	76	8.72	101	10.05	151	12.29	201	14.18
26½	5.15	51½	7.18	76½	8.75	102	10.10	152	12.33	202	14.21
27	5.20	52	7.21	77	8.77	103	10.15	153	12.37	203	14.25
27½	5.24	52½	7.25	77½	8.80	104	10.20	154	12.41	204	14.28
28	5.29	53	7.28	78	8.83	105	10.25	155	12.45	205	14.32
28½	5.34	53½	7.31	78½	8.86	106	10.30	156	12.49	206	14.35
29	5.39	54	7.35	79	8.89	107	10.34	157	12.53	207	14.39
29½	5.43	54½	7.38	79½	8.92	108	10.39	158	12.57	208	14.42
30	5.48	55	7.42	80	8.94	109	10.44	159	12.61	209	14.46
30½	5.52	55½	7.45	80½	8.97	110	10.49	160	12.65	210	14.49
31	5.57	56	7.48	81	9.00	111	10.54	161	12.69	211	14.53
31½	5.61	56½	7.52	81½	9.03	112	10.58	162	12.73	212	14.56
32	5.66	57	7.55	82	9.06	113	10.63	163	12.77	213	14.59
32½	5.70	57½	7.58	82½	9.08	114	10.68	164	12.81	214	14.63
33	5.74	58	7.62	83	9.11	115	10.72	165	12.85	215	14.66
33½	5.79	58½	7.65	83½	9.14	116	10.77	166	12.88	216	14.70
34	5.83	59	7.68	84	9.17	117	10.82	167	12.92	217	14.73
34½	5.87	59½	7.71	84½	9.19	118	10.86	168	12.96	218	14.76
35	5.92	60	7.75	85	9.22	119	10.91	169	13.00	219	14.80
35½	5.96	60½	7.78	85½	9.25	120	10.95	170	13.04	220	14.83
36	6.00	61	7.81	86	9.27	121	11.00	171	13.08	222	14.90
36½	6.04	61½	7.84	86½	9.30	122	11.05	172	13.11	224	14.97
37	6.08	62	7.87	87	9.33	123	11.09	173	13.15	226	15.03
37½	6.12	62½	7.91	87½	9.35	124	11.14	174	13.19	228	15.10
38	6.16	63	7.94	88	9.38	125	11.18	175	13.23	230	15.17
38½	6.20	63½	7.97	88½	9.41	126	11.22	176	13.27	232	15.23
39	6.24	64	8.00	89	9.43	127	11.27	177	13.30	234	15.30
39½	6.28	64½	8.03	89½	9.46	128	11.31	178	13.34	236	15.36
40	6.32	65	8.06	90	9.49	129	11.36	179	13.38	238	15.43
40½	6.36	65½	8.09	90½	9.51	130	11.40	180	13.42	240	15.49
41	6.40	66	8.12	91	9.54	131	11.45	181	13.45	242	15.56
41½	6.44	66½	8.15	91½	9.57	132	11.49	182	13.49	244	15.62
42	6.48	67	8.19	92	9.59	133	11.53	183	13.53	246	15.68
42½	6.52	67½	8.22	92½	9.62	134	11.58	184	13.56	248	15.75
43	6.56	68	8.25	93	9.64	135	11.62	185	13.60	250	15.81
43½	6.60	68½	8.28	93½	9.67	136	11.66	186	13.64	252	15.87
44	6.63	69	8.31	94	9.70	137	11.70	187	13.67	254	15.94
44½	6.67	69½	8.34	94½	9.72	138	11.75	188	13.71	256	16.00
45	6.71	70	8.37	95	9.75	139	11.79	189	13.75	258	16.06
45½	6.75	70½	8.40	95½	9.77	140	11.83	190	13.78	260	16.12

Table B.1.3 (Cont.)

T	√T	T	√T	T	√T	T	√T	T	√T	T	√T
46	6.78	71	8.43	96	9.80	141	11.87	191	13.82	262	16.19
46½	6.82	71½	8.46	96½	9.82	142	11.92	192	13.86	264	16.25
47	6.86	72	8.49	97	9.85	143	11.96	193	13.89	266	16.31
47½	6.89	72½	8.51	97½	9.87	144	12.00	194	13.93	268	16.37
48	6.93	73	8.54	98	9.90	145	12.04	195	13.96	270	16.43
48½	6.96	73½	8.57	98½	9.92	146	12.08	196	14.00	272	16.49
49	7.00	74	8.60	99	9.95	147	12.12	197	14.04	274	16.55
49½	7.04	74½	8.63	99½	9.97	148	12.17	198	14.07	276	16.61
50	7.07	75	8.66	100	10.00	149	12.21	199	14.11	278	16.67
50½	7.11	75½	8.69	100½	10.02	150	12.25	200	14.14	280	16.73

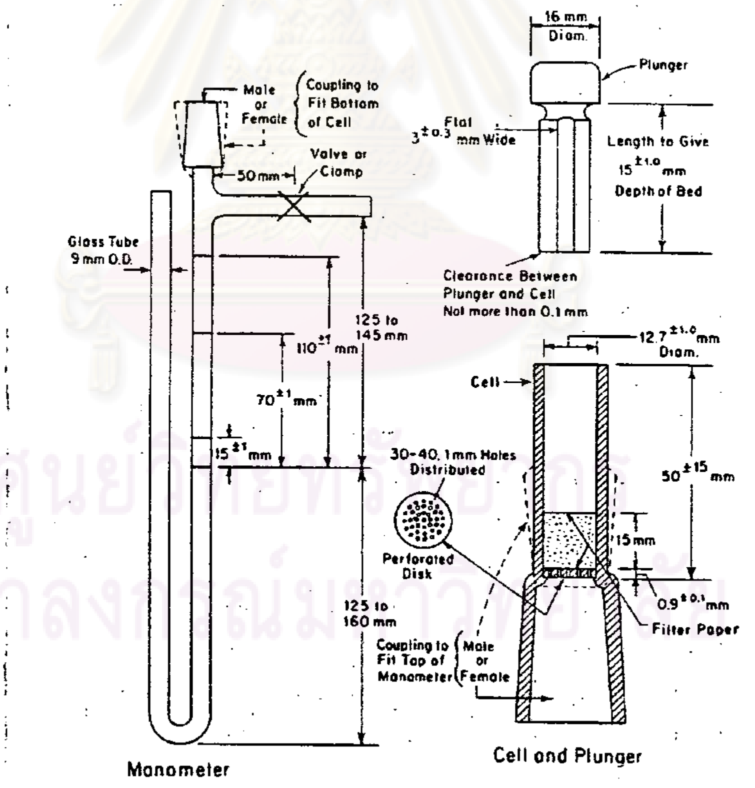


Fig. B.1. Blaine Air Permeability Apparatus

B.1.6 Results:

Temperature 26°C

Standard NBS 114L:

S_s = 3380 cm^2/gm

T_s = 66.95 sec.

ε_s = 0.5

From $\omega = \rho V(1-\varepsilon)$

ω = grams of sample required

ρ = density of test sample (for portland cement use 3.15 g/cm^3)

V = bulk volume of bed of cement, cm^3

ε = desired porosity of bed of cement (0.5)

$\therefore \omega = 3.15 \times 1.8578\ (1-0.5) = 2.926$ gm

From $$S = \frac{S_s \rho_s (1-\varepsilon_s)\sqrt{\eta_s}\sqrt{\varepsilon^3}\sqrt{T}}{\rho(1-\varepsilon)\sqrt{\varepsilon_s^3}\sqrt{T_s}\sqrt{\eta}}$$

$\varepsilon, \varepsilon_s = 0.5$ and $\eta = \eta_s = 0.01355$

$S_s = 3380\ cm^2/gm \quad \rho = \rho_s = 3.15$

From experiment $T = 29.2$ sec $\quad \sqrt{T} = 5.406$

$$S = \frac{3380 \times 5.406}{8.186} = 2232.14\ cm^2/gm$$

B.2 Standard Test Method for Normal Consistency of Hydraulic Cement (ASTM:C 187-79)

B.2.1 Scope

This method covers determination of the normal consistency of hydraulic cement.

B.2.2 Apparatus

B.2.2.1 Scales - On scales in use the permissible variation at a load of 9.8 N shall be ±0.01 N.

B.2.2.2 Weights - The permissible variations on weights in use in weighing the cement shall be as prescribed in Table B.2.1. The permissible variations on new weights shall be one half of the values in Table B.2.1.

B.2.2.3 Glass graduates; 200 or 250-ml capacity, and conforming to the requirements of Specification C490.

B.2.2.4 Vicat apparatus - The vicat apparatus shall consist of a frame A (Fig. B.2.1) bearing a movable rod B, weighing 300 g, one end C, the plunger end, being 10 mm in diameter for a distance of at least 50 mm, and the other end having a removable needle D, 1 mm in diameter and 50 mm in length. The rod B is reversible, and can be held in any desired position by a set screw E, and has an adjustable indicator F which moves over a scale (graduated in millimetres) attached to the frame A. The paste is held in a rigid conical ring G, resting on a glass plate H about 100 mm square rod B shall be made of stainless steel having a hardness of not less than HRC 35 (Note) and shall be straight with the plunger end which is perpendicular to the rod axis. The ring shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 70 mm at the base and 60 mm at the top, and a height of 40 mm. In addition to the above, the vicat apparatus shall conform to the following requirements:

weight of movable rod	300±0.5	g
Diameter of plunger end of rod	10±0.05	mm
Diameter of needle	1±0.05	m
Inside diameter of ring at bottom	70±3	mm
Inside diameter of ring at top	60±3	mm
Height of ring	40±1	mm (1.57±0.04 in).
Graduated scale	The graduated scale, when compared with a standard scale accurate to within 0.1 mm at all points, shall not show a deviation at any point greater than 0.25 mm.	

Note - The requirement that the rod be made of stainless steel shall apply only to new Vicat apparatus or replacement rods and not to equipment in use which meets the other requirements of C 187.

B.2.3 Temperature and Humidity

B.2.3.1 The temperature of the air in the vicinity of the mixing slab, the dry cement, molds, and base plates shall be maintained between 20 and 27.5°C (68 and 81.5°F). The temperature of the mixing water shall not vary from 23°C (73.4°F) by more than 1.7°C (±3°F).

B.2.3.2 The relative humidity of the laboratory shall be not less than 50%.

B.2.4 Procedure

B.2.4.1 Preparation of Cement Paste - Mix 650 g of cement with a measured quantity of clean water following the procedure prescribed in Section 5 of Method C 305.

B.2.4.2 Molding Test Specimen - Quickly form the cement paste, prepared as described in B.2.4.1, into the approximate shape of a ball with gloved hands. Then toss six times through a free path of about 6 in. (150 mm) from one hand to another so as to produce a nearly spherical mass that may be easily inserted into the Vicat ring with a minimum amount of additional manipulation. Press the ball, resting in the palm of one hand, into the larger end of the conical ring G, Fig. B.2.1 held in the other hand, completely filling the ring with paste. Remove the excess at the larger end by a single movement of the palm of the hand. Place the ring on its larger end on a plane, nonabsorptive plate H, and slice off the excess paste at the smaller end at the top of the ring by a single oblique stroke of a sharp-edged trowel held at a slight angle with the top of the ring, and smooth the top, if necessary, with a few light touches of the pointed end of the trowel. During these operations of cutting and smoothing, take care not to compress the paste.

B.2.4.3 Consistency Determination - Center the plate confined in the ring, resting on the plate, under the rod B, Fig. B.2.1, the plunger end C of which shall be brought in contact with the surface of the paste, and tighten the set-screw E. Then set the movable indicator F to the upper zero mark of the scale, or take an initial reading, and release the rod immediately. This must not exceed 30 s after completion of mixing. The apparatus shall be free of all vibrations during the test. The paste shall be of normal consistency when the rod settles to a point 10±1 mm below the original surface in 30 s after being released. Make trial pastes with varying percentages of water until the normal consistency is obtained. Make each trial with fresh cement.

B.2.5 Calculation

B.2.5.1 Calculate the amount of water required for normal consistency to the nearest 0.1% and report it to the nearest 0.5% of the weight of the dry cement.

Table B.2.1 Permissible Variations on Weights

Weight, g	Permissible Variations on Weights in Use, plus or minus, g
500	0.35
300	0.30
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

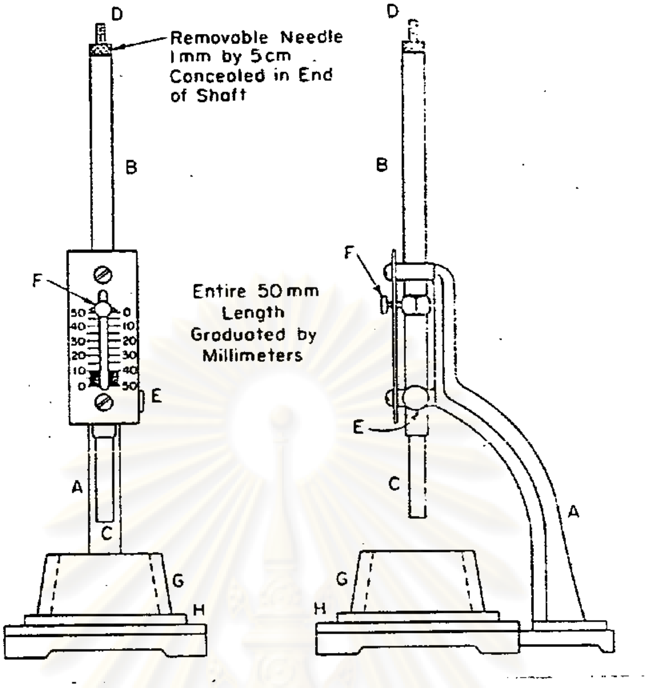


Fig. B.2.1 Vicat Apparatus.

B.2.6 Results:

Temp 74°F ∿ 23°C

normal consistency = 26.6%

B.3 Standard Test Method for Time of Setting of Hydraulic Cement by Vicat needle (ASTM:C 191-79)

B.3.1 Scope

This method covers determination of the time of setting of hydraulic cement by means of the Vicat needle.

B.3.2 Apparatus

All are the same as standard test method for normal consistency (ASTM C: 187-79). But for Vicat apparatus the needle end is used instead of plunger end.

B.3.3 Temperature and Humidity

B.3.3.1 The same as B.2.3.1

B.3.3.2 The relative humidity of the laboratory shall be not less than 50%. The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90%.

B.3.4 Preparation of Cement Paste

B.3.4.1 Mix 650 g of cement with the percentage of mixing water required for normal consistency following the procedure described in Method C 305. Distilled water is preferable and shall be used for all referee or cooperative tests.

B.3.5 Procedure

B.3.5.1 Molding Test Specimen - Quickly form the cement paste, prepared as described in Section B.3.4, into a ball with the gloved hands and toss six times from one hand to the other, maintaining the hands about 6 in. (152 mm) apart. Press the ball, resting in the palm of the hand, into the larger end of the conical ring, G, Fig. B.3.1, held in the other hand, completely filling the ring with paste. Remove the excess at the larger end by a single movement of the palm of the hand. Place the ring on its larger end on a glass plate, H, and slice off the excess paste at the smaller end at the top of the ring by a single oblique stroke of a sharpedged trowel held at a slight angle with the top of the ring. Smooth the top of the specimen, if necessary, with one or two light touches of the pointed end of the trowel. During the operation of cutting and smoothing, take care not to compress the paste. Immediately after molding, place the test specimen in the moist closet or moist room and allow it to remain there except when determinations of time of setting are being made. The specimen shall remain in the conical mold, supported by the glass plate, H, throughout the test period. A time of set specimen and an autoclave bar may be made from the same batch.

B.3.5.2 Time of Setting Determination - Allow the time of setting specimen to remain in the moist cabinet for 30 min after molding without being disturbed. Determine the penetration of the 1-mm needle at this time and every 15 min thereafter (every 10 min for Type III cements) until a penetration of 25 mm or less is obtained. For the penetration test, lower the needle D of the rod B until it rests on the surface of the cement paste. Tighten the set screw, E, and set the indicator, F, at the upper end of the scale, or take an initial reading. Release the rod quickly by releasing the set screw, E, and allow the needle to settle for 30 s; then take the reading to determine the penetration. (If the paste is obviously quite soft on the early readings, the fall of the rod may be retarded to avoid bending the 1-mm needle, but the rod shall be released only by the set screw when actual determinations for the setting time are made.) No penetration test shall be made closer than ¼ in. (6.4 mm) from any previous penetration and no penetration test shall be made closer than 3/8 in. (9.5 mm) from the inside of the mold. Record the results of all penetration tests and, by interpolation, determine the time when a penetration of 25 mm is obtained. This is the initial setting time. The final setting time is when the needle does not sink visibly into the paste.

B.3.5.3 Precautions - All the apparatus shall be free from vibration during the penetration test. Take care to keep the 1-mm needle straight, and the needle must be kept clean as the collection of cement on the sides of the needle may retard the penetration, while cement on the point may increase the penetration. The time of setting is affected not only by the percentage and the temperature of the water used and the amount of kneading the paste received, but also by the temperature and humidity of the air, and its determination is therefore only approximate.

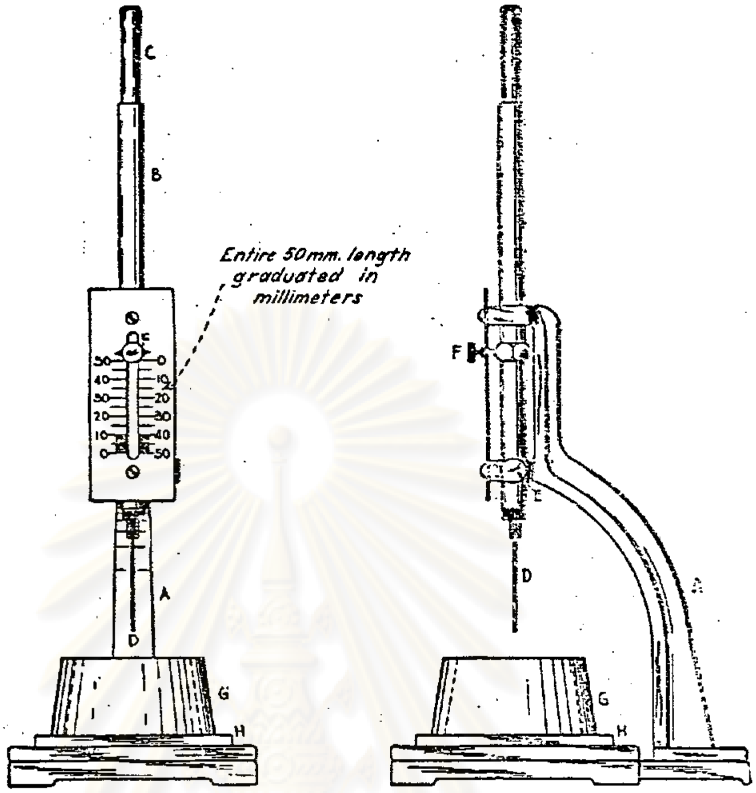


Fig. B.3.1 Vicat Apparatus

B.3.6 Results:

Temp 74^{o}F 23^{o}C

Initial setting time 1 hr 38 min

Final setting time 2 hr 45 min

B.4 Standard Test Method for Autoclave Expansion of Portland Cement (ASTM:C 151-77)

B.4.1 Scope

B.4.1.1 This method covers determination of the autoclave expansion of portland cement by means of a test on a neat cement specimen.

B.4.2 Significance

B.4.2.1 The autoclave expansion test provides an index of potential delayed expansion caused by the hydration of CaO or MgO or both.

B.4.3 Apparatus

B.4.3.1 Scales and Weights, for weighing materials conforming to the requirements of ASTM Specification C 490.

B.4.3.2 Glass Graduates, 200 or 250-ml capacity, and conforming to the requirements of Specification C 490.

B.4.3.3 Mold, 1 by 1-in. (25.4 by 25.4-mm) cross section, conforming to the requirements of Specification C 490.

B.4.3.4 Trowel, having a straight-edged steel blade 4 to 6 in. (100 to 150 mm) in length.

B.4.3.5 Autoclave, consisting of a high-pressure steam vessel provided with a thermometer well. The autoclave shall be equipped with an automatic pressure control and a rupture disk with a bursting pressure of 350 psi (2.4 MPa) ±5%. In locations where the use of a rupture disk is not permitted, the autoclave shall be equipped with a safety valve. In addition, the autoclave shall be equipped with a vent valve to allow the escape of air during the early part of the heating period and to release any steam pressure remaining at the end of the cooling period. The pressure gage shall have a dial with a nominal diameter of 4½ in. (114 mm) and shall be graduated from 0 to 600 psi (0 to 4.1 MPa) with scale divisions of not more than 5 psi (0.03 MPa). The error in the gage shall not exceed ±3 psi (±0.02 MPa) at the operating pressure of 295 psi (2 MPa). The capacity of the heating unit shall be such that with maximum load (water plus specimens) the pressure of the saturated steam in the autoclave may be raised to a gage pressure of 295 psi in 45 to 75 min from the time the heat is turned on. The automatic pressure control shall be capable of maintaining the gage pressure at 295±10 psi (2±0.07 MPa) for at least 3 h. A gage pressure of 295±10 psi corresponds to a temperature of 420±3 F (216±2 C). The autoclave shall be designed to permit the gage pressure to drop from 295 psi to less than 10 psi in 1½ h after the heat supply has been shut off.

B.4.3.5.1 Rupture Disk - The rupture disk shall be made of a material having a tensile strength that is relatively insensitive to temperature in the range 68 to 420 F (20 to 216 C) and that is electrochemically compatible with the pipe leading to it and to its holder.

B.4.3.6 Length Comparator - The comparator used for measuring length change of specimens shall conform to the requirements of Specification C 490.

B.4.4 Temperature and Humidity

B.4.4.1 Molding Room - Maintain the temperature of the molding room, dry materials and mixing water, and the relative humidity of the molding room within the limits of Specification C 490.

B.4.4.2 Moist Storage Facilities - Maintain the temperature and humudity of the moist storage facilities to the requirements of 7 ASTM Specification C 511.

B.4.5 Number of Test Specimens

B.4.5.1 Make at least one test specimen.

B.4.6 Preparation of Specimen Molds

B.4.6.1 Prepare the specimen molds in accordance with the requirements of Specification C 490.

B.4.7 Preparation of Test Specimens

B.4.7.1 Mixing Cement Paste - Prepare the standard batch consisting of 650 g of cement and sufficient water to give a paste of normal consistency. Mix this batch in accordance with the procedure described in ASTM Method C 305, for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency. Both time of set specimens and an autoclave bar may be made from the same batch.

B.4.7.2 Molding Specimens - Immediately following preparation of time of set specimens or completion of mixing, mold the test specimen in two approximately equal layers, each layer being compacted with the thumbs or forefingers by pressing the paste into the corners, around the gage studs, and along the surface of the mold until a homogeneous specimen is obtained. Compact the top layer, cut off the paste flush with the top of the mold with a thin-edged trowel, and smooth the surface with a few strokes of the trowel. During the operations of mixing and molding, protect the hands with rubber gloves.

B.4.7.3 Storage of Test Specimens - After filling the mold, place it in the moist closet or moist room. Store the specimens in the molds in the moist enclosure for at least 20 h; if removed from the molds before 24 h, they shall be kept in the moist closer or moist room until time of test.

B.4.8 Procedure

B.4.8.1 At 24 h ±30 min after molding, remove the specimens from the moist atmosphere, immediately measure for length and place in the autoclave at room temperature in a rack so that all sides of the specimen will be exposed to saturated steam. The autoclave shall contain enough water, at an initial temperature of 68 to 82 F (20 to 28 C), to maintain an atmosphere of saturated steam vapor during the entire test. Ordinarily 7 to 10 percent of the volume of the autoclave should be occupied by the water.

B.4.8.2 To permit air to escape from the autoclave during the early portion of the heating period, leave the vent valve open until steam begins to escape. Then close the valve and raise the temperature of the autoclave at a rate that will bring the gage pressure of the steam to 295 psi (2 MPa) in 45 to 75 min from the time the heat it turned on. Maintain the 295±10-psi (2±0.07-MPa) pressure for 3 h. At the end of the 3-h period, shut off the heat supply and cool the autoclave at such a rate that the pressure will be less than 10 psi at the end of 1½ h. At the end of the 1½-h period, slowly release any remaining pressure by partially opening the vent valve until atmospheric pressure if attained. Then open the autoclave and place the test specimen in water at a temperature above 194 F (90 C). Cool the water surrounding the bars at a uniform rate by adding cold water so that the temperature of the water will be lowered to 74 F (23 C) in 15 min. Maintain the water surrounding the specimens at 74 F for an additional 15 min; then, surface-dry the specimens and measure their lengths again.

Note B.4.1 - If it is preferred to make all measurements at 80 F (27 C), it is recommended that the specimens, after removal from the moist closet or moist room, be placed in water maintained at 80 F for at least 15 min, removed and measured for length, and then placed in the autoclave. Upon removal from the autoclave, cool the specimens and water to 80 F in 15 min. Keep the specimens in water at this temperature for an additional 15 min, then measure them for length.

B.4.9 Calculation

B.4.9.1 Calculate the difference in length of the test specimen before and after autoclaving as percent of the effective gage length and report to the nearest 0.01 percent. Report the percentage of increase in length as the autoclave expansion. Indicate a decrease in length by a minus sign prefixed to the percent value.

B.4.10 Results:

l_o = length before place into autoclave

l_i = length after place into autoclave

$$\text{Autoclave expansion \%} = \frac{(l_i - l_o)100}{lo} = \frac{(10.0143-9.9800)100}{9.9800}$$

$\therefore$ autoclave expansion = 0.34%

B.5 Standard Test Method for Air Content of Hydraulic Cement Mortar (ASTM:C 185-80)

B.5.1 Scope

B.5.1.1 This method covers determination of the air content of hydraulic cement mortar under the conditions hereinafter specified.

The values states in inch-pound units are to be regarded as the standard.

B.5.2 Summary of Method

B.5.2.1 A mortar is prepared with standard sand and the cement to be tested, using a water content sufficient to give a required flow. This mortar is compacted into a measure of known volume and weighed. The air content is calculated from the measured density of the mortar, the known densities of the constituents, and the mixture proportions.

B.5.3 Significance

B.5.3.1 The purpose of this method is to determine whether or not the hydraulic cement under test meets the air-entraining or non-air-entraining requirements of the applicable hydraulic cement specification for which the test is being made. The air content or concrete is influenced by many factors other than the potential of the cement for air entrainment.

B.5.4 Apparatus

B.5.4.1 Flow Table, Flow Mold, and Caliper conforming to the requirements of Specification C 230.

B.5.4.2 Measure - A cylindrical measure having an inside diameter of 3±1/16 in. (76.2±1.6 mm) and a depth (approximately $3\frac{15}{32}$ in. (88.1 mm). The measure shall be made of a metal not attacked by the cement mortar.

B.5.4.3 Mixer, Bowl, and Paddle conforming to Section 2 of Method C 305.

B.5.4.4 Straightedge - A steel straightedge not less than 4 in. (102 mm) long and not less than 1/16 in. (1.6 mm) nor more than 1/8 in. (3.2 mm) in thickness.

B.5.4.5 Spatula - A spatula with a metal blade 6 in. (152 mm) in length and ½ in. (13 mm) in idth, with straight edges and a wooden handle.

B.5.4.6 Scales - Scales having a capacity sufficient to weigh the mortar and the container (approximately 2 kg). The permissible variation at 2 kg shall be ±1 g.

B.5.4.7 Glass Graduates - Glass graduates of 250-ml capacity, with graduations at 2-ml intervals, for measuring the mixing water, made to deliver the indicated volume at 68°F (20°C). The permissible variation shall be ±2 mL.

B.5.4.8 Tamper, made of a nonabsorptive, non-abrasive, nonbrittle material such as a rubber compound having a Shore A durometer hardness of 80±10, or seasoned oak wood rendered nonabsorptive by immersion for 15 min in paraffin at approximately 392°F (200°C), and having a cross section of ½ by 1 in. (13 by 25 mm) and a convenient length (5 to 6 in. (127 to 152 mm)). The tamping face of the tamper shall be flat and at right angles to the length of the tamper.

B.5.4.9 Spoon - A metal restaurant serving spoon at least 9 in. (approximately 230 mm) in length and with a bowl approximately 4 in. (100 mm) in length.

B.5.5 Temperature and Humidity

B.5.5.1 The temperature of the room and dry materials shall be maintained between 20 and 27.5°C (68 and 81.5°F).

B.5.5.2 The mixing water and the measure, if it is being calibrated, shall be conditioned to the temperature required for moist cabinets or rooms in Specification C 511, 23±1.7°C (73.4±3°F).

B.5.5.3 The relative humidity of the laboratory shall not be less than 50%.

B.5.6 Standard Sand

B.5.6.1 The sand used for making the standard mortar (see

B.5.7.3) shall be natural silica sand, conforming to the requirements for 20-30 standard sand as described in Specification C 778.

B.5.7 Procedure

B.5.7.1 Batch - The proportions of the standard mortar shall be 350 g of cement to 1400 g of 20-30 standard sand and sufficient water to give a flow of 87½±7½% when determined in accordance with B.5.7.3.

B.5.7.2 Mixing of Mortar - Mix the mortar in accordance with Method C 305.

B.5.7.3 Flow Determination - Carefully wipe dry the flow-table top and place the flow mold at the center of it. Using the spoon, place a layer of mortar about 1 in. (25 mm) in thickness in the mold and tamp 20 times with the tamper. The tamping pressure shall be just sufficient to ensure uniform filling of the mold. Then fill the mold with mortar and tamp as specified for the first layer. Then cut off the mortar to a plane surface, flush with the top of the mold, by drawing the straightedge with a sawing motion across the top of the mold. Wipe the flow table top clean and dry, being especially careful to remove any water from around the edge of the mold. Lift the mold away from the mortar 1 min after completing the mixing operation. Immediately drop the table through a height of ½ in. (12.7 mm) 10 times in 6 s. The flow is the resulting increase in average diameter of the mortar mass, as determined with the calipers, measured on at least four diameters at approximately equispaced intervals, expressed as a percentage of the original diameter. Make trial mortars with varying percentages of water until the specified flow is obtained. Make each trial with fresh mortar.

B.5.7.4 Weight per 400 mL of Mortar - When the quantity of mixing water has been found that produces a flow of 87½±7½%, immediately determine the weight per 40 mL of mortar, using the mortar remaining in the mixing bowl after the flow has been determined. In the determination of the weight per 400 mL, do not use the portion of the mortar used in the flow determination. Using the spoon, place the mortar gently into the 400-mL measure in three equal layers, spading each layer 20 times with the spatula in one complete revolution around the inner surface of the measure. One complete up and down motion of the spatula held in a vertical position shall be considered one spading. In spading the first layer do not strike the spatula forcibly against the bottom of the measure. In spading the second and final layers, use only enough force to cause the spatula to penetrate the surface of the previous layer. After the measure has been filled and spaded in the above prescribed manner, tap the sides of the measure lightly with the side of the tapping stick once each at five different points at approximately equal spacing around the outside of the measure in order to preclude entrapment of extraneous air. No obvious space shall be left between the mortar and the inner surface of the measure as a result of the spading operation. Then cut the mortar off to a plane surface, flush with the top of the measure, by drawing the straightedge with a sawing motion across the top of the measure, making two passes over the entire surface, the second pass being made at right angles to the first. If, in the striking-off operation, loose sand grains cause the straightedge to ride above the top surface of the measure, these grains shall be removed, and the operation repeated. Complete the entire operation of filling and striking off the measure within 1½ min. Wipe off all mortar and water adhering to the outside of the measure. Weigh the measure and its contents. Subtract the weight of the container, and record the weight of the mortar in grams.

B.5.8 Calculation

B.5.8.1 Calculate the air content of the mortar from the following formula, which is based on use of the measure specified in B.5.4.2 and on the batch proportions given in B.5.7.1 taking the specific gravity of portland cement as 3.15 and of 20-30 standard sand as 2.65. When the hydraulic cement is other than portland, the appropriate value for its specific gravity shall be substituted for the value of 3.15 and the formula rederived accordingly:

Air content, volume % = $100 - W\left[(182.7 + P)/(2000 + 4P)\right]$

where:

W = weight of 400 mL of mortar, g, and

P = percentage of mixing water, based on weight of cement used.

Note B.5.1 This formula can be derived as follows:

Air content, volume % = $100\left[1-(W_a/W_c)\right]$

where:

W_a = actual weight per unit of volume as determined by Method C 185.

= W/400 g/mL, where W is the weight in grams of the specified 400 mL of mortar (B.5.7.4),

W_c = theoretical weight per unit of volume, calculated on an air-free basis as follows and using the values for quantities of materials and specific gravities as given in B.5.7.1 and B.5.8.1

$$= \frac{350 + 1400 + 350 \times P \times 0.01}{\frac{350}{3.15} + \frac{1400}{2.65} + \frac{350 \times P \times 0.01}{1}}$$

$$= \frac{(5 + 0.01P)}{(1.827 + 0.01P)}\text{ , and}$$

P = percentage of mixing water, based on weight of cement

Substituting for W_a and W_c we have:

$$\text{Air content, volume \%} = 100\left(1 - \frac{W}{400} \times \frac{1.827 + 0.01P}{5 + 0.01P}\right)$$

$$= 100 - \frac{W}{4} \times \frac{(1.827 + 0.01P)}{(5+ 0.01P)}$$

$$= 100 - 2.5W\frac{(182.7 + P)}{(5000 + 10P)}$$

$$= 100 - W\frac{(182.7 + P)}{(2000 + 4P)}$$

B.5.8.2 Make only one determination of air content on a batch.

Note B.5.2 - Difficulty has occasionally been experienced with Method C 185 by some persons. These difficulties usually have taken the form of values for air content that are abnormally high and that may be greater than the specified maxima in specifications for hydraulic cement. The air-entraining potential of the sand may be reduced by washing in the laboratory with potable water by decantation until a clear, colorless supernatant liquid is obtained. In case of dispute, such freshly washed sand shall be used.

B.5.9 Results:

Temperature = 73°F or = 22.78°C

Weight of container = 659.7 gm

Weight of container + mortar = 1481.65 gm

$\therefore$ Weight of mortar = 821.95 gm

From

$$\text{Air content, volume \%} = 100\text{-}W\left[(182.7 + P)/(2000 + 4P)\right]$$

where W = weight of 400 ml of mortar, gm

P = percentage of mixing water, based on weight of cement used

= 72

$$\text{Air content, volume \%} = 100 - \frac{821.95\ (182.7 + 72)}{2000 + 4(72)}$$

$$\therefore \text{air content of mortar} = 8.5\%$$

B.6 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens) (ASTM:C 109-80)

B.6.1 Scope

B.6.1.1 This method covers determination of the compressive strength of hydraulic cement mortars, using 2-in, or 50 mm cube specimens.

B.6.2 Apparatus

B.6.2.1 Scales, for weighing materials for mortar mixes,

B.6.2.2 Weights, the same as B.2.2.2 $0 + \frac{1400}{2.65} + \frac{350 \times P \times 1}{1}$

B.6.2.3 Sieves, square-hole, woven wire-cloth No. 100 (150-µm), No. 50 (300-µm), No. 40 (425-µm), No. 30 (600-µm) and No. 16 (1.18-mm) sieves conforming to Specification E 11 shall be used.

B.6.2.4 Glass Graduates,

B.6.2.5 Specimen Molds, for the 2-in or 50-mm cube specimens shall be tight fitting. The molds shall have not more than three cube compartments and shall be separable into not more than two parts. The parts of the molds when assembled shall be positively held together. The molds shall be made of hard metal not attacked by the cement mortar. For new molds the Rockwell hardness number of the metal shall be not less than HRB 55. The sides of the molds shall be sufficiently rigid to prevent spreading or warping. The interior faces of the molds shall be plane surfaces with a permissible variation of 0.001 in. (0.025 mm) for new molds and 0.002 in. (0.05 mm) for molds in use. The distances between opposite faces shall be 2±0.005 in. or 50±0.13 mm for new molds, and 2±0.02 in. or 50±0.50 mm for molds in use. The height of the molds, measured separately for each cube compartment, shall be 2 in. or 50 mm with permissible variations of +0.01 in. (0.25 mm) and -0.005 in. (0.13 mm) for new molds, and +0.01 in. and -0.015 in. (0.38 mm) for molds in use. The angle between adjacent interior faces, and between interior faces and top and bottom planes of the mold, shall be 90 $\pm 0.5^{\circ}$ measured at points slightly removed from the intersection of the faces.

B.6.2.6 Mixer, Bowl and Paddle, an electrically driven mechanical mixer of the type equipped with paddle and mixing bowl, as specified in C 305.

B.6.2.7 Flow Table and Flow Mold, conforming to the requirements of Specification C 230.

B.6.2.8 Tamper, a nonabsorptive, nonabrasive, nonbrittle material such as a rubber compound having a Shore A durometer hardness of 80±10 or seasoned oak wood rendered nonabsorptive by immersion for 15 min in paraffin at approximately $392^{\circ}F$ $(200^{\circ}C)$, and shall have a cross section of ½ by 1 in. (13 by 25 mm) and a convenient length of about 5 to 6 in. (120 to 150 mm). The tamping face shall be flat and at right angles to the length of the tamper.

B.6.2.9 Trowel, having a steel blade 4 to 6 in. (100 to 150 mm) in length, with straight edges.

B.6.2.10 Testing Machine, either the hydraulic or the screw type, with sufficient opening between the upper bearing surface and the lower bearing surface of the machine to permit the use of verifying apparatus. The load applied to the test specimen shall be indicated with an accuracy of ±1.0%. The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to tilt in any direction. The diagonal or diameter (Note B.6.1) of the bearing surface shall be only slightly greater than the diagonal of the face of the 2-in. or 50-mm cube in order to facilitate accurate centering of the specimen. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen shall have a Rockwell hardness number not less than HRC 60. These surfaces shall not depart from plane surfaces by more than 0.0005 in. (0.013 mm) when the blocks are new and shall be maintained within a permissible variation of 0.001 in. (0.025 mm).

Note B.6.1 - A diameter of 3 1/8 in. (79.4 mm), which is large enough for testing 3 by 6-in. (75 by 150-mm) cylinders, is satisfactory, provided that the lower bearing block has a diameter slightly greater than the diagonal of the face of the 2-in. or 50-mm cube but not more than

2.9 in. (75 mm), and is centered with respect to the upper bearing block and held in position by suitable means.

B.6.3 Materials

B.6.3.1 Graded Standard Sand:

B.6.3.1.1 The sand used for making test specimens shall be natural silica sand conforming to the requirements for graded standard sand in Specification C 778.

B.6.4 Temperature and Humidity

B.6.4.1 Temperature - The same as B.2.3.1.

B.6.4.2 Humidity - The relative humidity of the laboratory shall be not less than 50%. The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 95%.

B.6.5 Test Specimens

B.6.5.1 Three or more specimens shall be made for each period of test specified.

B.6.6 Preparation of Specimen Molds

B.6.6.1 Thinly cover the interior faces of the specimen molds with mineral oil or light cup grease. Thinly cover the contact surfaces of the halves of each mold with a heavy mineral oil or light cup grease such as petrolatum. After assembling the molds, remove excess oil or grease from the interior faces and the top and bottom surfaces of each mold. Set the molds on plane, nonabsorptive base plates that have been thinly coated with mineral oil, petrolatum, or light cup grease. Apply a mixture of 3 parts of paraffin to 5 parts of rosin by weight, heated between 230 and 248°F (110 and 120°C), at the outside contact lines of the molds and base plates so that watertight joints are effected between the molds and the base plates.

Note B.6.2 Watertight Molds - The mixture of paraffin and rosin specified for sealing the joints between molds and base plates may be found difficult to remove when molds are being cleaned. Use of straight paraffin in permissible if a watertight joint is secured, but due to the low strength of paraffin it should be used only when the mold is not held to the base plate by the paraffin alone. A watertight joint may be secured with paraffin alone by slightly warming the mold and base plate before brushing the joint. Molds so treated should be allowed to return to the specified temperature before use.

B.6.7 Procedure

B.6.7.1 Composition of Mortars:

B.6.7.1.1 The proportions of materials for the standard mortar shall be one part of cement to 2.75 parts of graded standard sand by weight. Use a water-cement ratio of 0.485 for all portland cements and 0.460 for all air-entraining portland cements. The amount of mixing water for other than portland and air-entraining portland cements shall be such as to produce a flow of 110±5 as determined in accordance with B.6.7.3 and shall be expressed as weight percent of cement.

B.6.7.1.2 The quantities of materials to be mixed at one time in the batch of mortar for making six and nine test specimens shall be as follows:

	Number of Specimens	
	6	9
Cement, gm	500	740
Sand, gm	1375	2035
Water, mL		
Portland (0.485)	242	359
Air-entraining portland (0.460)	230	340
Other (to flow of 110±5)	...	...

B.6.7.2 Preparation of Mortar

B.6.7.2.1 Mechanically mix in accordance with the procedure given in Section 6 of Method C 305.

B.6.7.3 Determination of Flow:

B.6.7.3.1 Carefully wipe the flow-table top clean and dry, and place the flow mold at the center. Place a layer of mortar about 1 in. (25 mm) in thickness in the mold and tamp 20 times with the tamper. The tamping pressure shall be just sufficient to ensure uniform filling of the mold. Then fill the mold with mortar and tamp as specified for the first layer. Cut off the mortar to a plane surface, flush with the top of the mold, by drawing the straight edge of a trowel (held nearly perpendicular to the mold) with a sawing motion across the top of the mold. Wipe the table top clean and dry, being especially careful to remove any water from around the edge of the flow mold. Lift the mold away from the mortar 1 min after completing the mixing operation. Immediately drop the table through a height of ½ in. (12.7 mm) 25 times in 15 s. The flow is the resulting increase in average base diameter of the mortar mass, measured on at least four diameters at approximately equispaced intervals, expressed as a percentage of the original base diameter. Make trial mortars with varying percentages of water until the specified flow is obtained. Make each trial with fresh mortar.

B.6.7.4 Molding Test Specimens:

B.6.7.4.1 For portland and air-entraining portland cements, allow the mortar to stand in the mixing bowl 90 s without covering. During the last 15 s of this interval, quickly scrape down into the batch any mortar that may have collected on the side of the bowl. Then remix for 15 s at medium speed. Upon completion of mixing, the mixing paddle shall be shaken to remove excess mortar into the mixing bowl.

B.6.7.4.2 Immediately following completion of the flow test, in the case of other than portland and air-entraining portland cements (Note B.6.3), return the mortar from the flow table to the mixing bowl. Quickly scrape down into the batch the mortar that may have collected on the side of the bowl and then remix the entire batch 15 s at medium speed. Upon completion of mixing, the mixing paddle shall be shaken to remove excess mortar into the mixing bowl.

B.6.7.4.3 Start molding the specimens within a total elapsed time of not more than 2 min and 30 s after completion of the original mixing of the mortar batch. Place a layer of motar about 1 in. (25 mm) (approximately one half of the depth of the mold) in all of the cube compartments. Tamp the mortar in each cube compartment (B.6.2.8) 32 times in about 10 s in 4 rounds, each round to be at right angles to the other and consisting of eight adjoining strokes over the surface of the specimen, as illustrated in Fig. B.6.1 The tamping pressure shall be just sufficient to ensure uniform filling of the molds. The 4 rounds of tamping (32 strokes) of the mortar shall be completed in one cube before going to the next. When the tamping of the first layer in all of the cube compartments is completed, fill the compartments with the remaining mortar and then tamp as specified for the first layer. During tamping of the second layer bring in the mortar forced out onto the tops of the molds after each round of tamping by means of the gloved fingers and the tamper upon completion of each round and before starting the next round of tamping. On completion of the tamping, the tops of all cubes should extend slightly above the tops of the molds. Bring in the mortar that has been forced out onto the tops of the molds with a trowel and smooth off the cubes by drawing the flat side of the trowel (with the leading edge slightly raised) once across the top of each cube at right angles to the length of the mold. Then, for the purpose of leveling the mortar and making the mortar that protrudes above the top of the mold of more uniform thickness, draw the flat side of the trowel (with the leading edge slightly raised) lightly once along the length of the mold. Cut off the mortar to a plane surface flush with the top of the mold by drawing the straight edge of the trowel (held nearly perpendicular to the mold) with a sawing motion over the length of the mold.

Note B.6.3 - When a duplicate batch is to be made immediately for additional specimens, the flow test may be omitted and the mortar allowed to stand in the mixing bowl for 90 s and then remixed for 15 s at medium speed before starting the molding of the specimens.

B.6.7.5 Storage of Test Specimens - Immediately upon completion of molding, place the test specimens in the moist closet or moist room. Keep all test specimens, immediately after molding, in the molds on the base plates in the moist closet or moist room from 20 to 24 h with their upper surfaces exposed to the moist air but protected from dripping water. If the specimens are removed from the molds before 24 h, keep them on the shelves of the moist closet or moist room until they are 24-h old, and then immerse the specimens except those for the 24-h test, in saturated lime water in storage tanks constructed of noncorroding materials. Keep the storage water clean by changing as required.

B.6.7.6 Determination of Compressive Strength:

B.6.7.6.1 Test the specimens immediately after their removal from the moist closet in the case of 24-h specimens, and from storage water in the case of all other specimens. All test specimens for a given test age shall be broken within the permissible tolerance prescribed as follows:

Test Age	Permissible Tolerance
24 h	±½ h
3 days	±1 h
7 days	±3 h
28 days	±12 h

If more than one specimen at a time is removed from the moist closet for the 24-h tests, keep these specimens covered with a damp cloth until time of testing. If more than one specimen at a time is removed from the storage water for testing, keep these specimens in water at a temperature of 73.4±3°F (23±1.7°C) and of sufficient depth to completely immerse each specimen until time of testing.

B.6.7.6.2 Wipe each specimen to a surface-dry condition, and remove any loose sand grains or incrustations from the faces that will be in contact with the bearing blocks of the testing maching. Check these faces by applying a straightedge (Note B.6.4). If there is appreciable curvature, grind the face or faces to plane surfaces or discard the specimen. A periodic check of the cross-sectional area of the specimens should be made.

Note B.6.4 Specimen Faces - Results much lower than the true strength will be obtained by loading faces of the cube specimen that are not truly plane surfaces. Therefore, it is essential that specimen molds be kept scrupulously clean, as otherwise, large irregularities in the surfaces will occur. Instruments for cleaning molds should always be softer than the metal in the molds to prevent wear. In case grinding specimen faces is necessary, it can be accomplished best by rubbing the specimen on a sheet of fine emery paper or cloth glued to a plane surface, using only a moderate pressure. Such grinding is tedious for more than a few thousandths of an inch (hundredths of a millimetre); where more than this is found necessary, it is recommended that the specimen be discarded.

B.6.7.6.3 Apply the load to specimen faces that were in contact with the true plane surfaces of the mold. Carefully place the specimen in the testing machine below the center of the upper bearing block. Prior to the testing of each cube, it shall be ascertained that the sperically seated block is free to tilt. Use no cushioning or bedding materials. An initial loading up to one half of the expected maximum loads for specimens having expected maximum loads of more than 3000 lbf (13.3 kN) may be applied at any convenient rate. Apply no initial loading to specimens having expected maximum loads of less than 3000 lbf (13.3 kN). Adjust the rate of load application so that the remainder of the load (or the entire load in the case of expected maximum loads of less than 3000 lbf (13.3 kN)) is applied, without interruption, to failure at such a rate that the maximum load will be reached in not less than 20 nor more than 80 s from start of loading. Make no adjustment in the controls of the testing machine while a specimen is yielding before failure.

Note B.6.5 - It is advisable to apply only a very light coating of a good quality, light mineral oil to the spherical seat of the upper platen.

B.6.8 Calculation

B.6.8.1 Record the total maximum load indicated by the testing machine, and calculate the compressive strength in pounds per square inch (or pascals). If the cross-sectional area of a specimen varies more than 1.5% from the nominal, use the actual area for the calculation of the compressive strength. The compressive strength of all acceptable test specimens (see Section B.6.9) made from the same sample and tested at the same period shall be averaged and reported to the nearest 10 psi (70 kPa).

B.6.9 Faulty Specimens and Retests

B.6.9.1 In determining the compressive strength, do not consider specimens that are manifestly faulty, or that give strengths differing by more than 10% from the average value of all test specimens made from the same sample and tested at the same period (Note B.6.6). After discarding specimens of strength values, if less than two strength values are left for determining the compressive strength at any given period, make a retest.

Note B.6.6 - Reliable strength results depend upon careful observance of all of the specified requirements and procedures. Erratic results at a given test period indicate that some of the requirements and procedures have not been carefully observed; for example, those covering the testing of the specimens as prescribed in B.6.7.6.2 and B.6.7.6.3. Improper centering of specimens resulting in oblique fractures or lateral movement of one of the heads of the testing machine during loading will cause lower strength results.

Rounds 1 and 3 Rounds 2 and 4

Fig. B.6.1 Order of Tamping in Molding of Test Specimens

B.6.10 Results:

Temp 73°F or 22.78°C

Specimen mark	age (day)	Dimension (cm)	Weight (gm)	Ultimate strength		ave. stress (kg_f/cm^2)
				Load (kg_f)	Stress (kg_f/cm^2)	
1	3	5.06 x 5.06	284.3	3,980	155.45	159.97
2	3	5.07 x 5.10	287.5	4,160	160.89	
3	3	5.05 x 5.06	283.8	4,180	163.58	
4	7	5.10 x 5.10	296.8	5,650	217.22	216.63
5	7	5.05 x 5.06	283.7	5,400	211.33	
6	7	5.02 x 5.04	281.3	5,600	221.34	
7	28	5.08 x 5.09	291.4	7,450	288.12	293.86
8	28	5.07 x 5.08	289.5	7,700	298.96	
9	28	5.08 x 5.06	289.1	7.570	294.50	

B.7 Standard Specification for Standard Sand (ASTM:C 778-80)

B.7.1 Scope

B.7.1.1 This specification covers standard sand and graded standard sand for use in the testing of hydraulic cements.

The values stated in inch-pound units are to be regarded as the standard.

B.7.2 Definitions

B.7.2.1 Standard sand - Silica sand, composed almost entirely of naturally rounded grains of nearly pure quartz, used for preparing mortars in the testing of hydraulic cements.

B.7.2.2 20-30 sand - Standard sand, predominantly graded to pass a No. 20 (850-μm) sieve and be retained on a No. 30 (600-μm) sieve.

B.7.2.3 Graded sand - Standard sand, predominantly graded between the No. 30 (600-μm) sieve and the No. 100 (150-μm) sieve.

B.7.3 Requirements

B.7.3.1 Standard Sand - Natural silica sand from Ottawa, Ill., or Le Sueur, Minn., graded as follows:

Sieve	Accumulative Percentage Retained
No. 16 (1.18 mm)	none
No. 20 (850 μm)	15 (max)
No. 30 (600 μm)	95 to 100

B.7.3.2 Graded Standard Sand - Natural silica sand from Ottawa, Ill., graded as follows:

Sieve	Accumulative Percentage Retained
No. 16 (1.18 mm)	None
No. 30 (600 μm)	2±2
No. 40 (425 μm)	30±5
No. 50 (300 μm)	75±5
No. 100 (150 μm)	98±2

Methods of Sampling and Testing Standard Sands.

B.7.4 Apparatus

B.7.4.1 Sieves - The sieves shall be standard 8-in. (203-mm) diameter, full-height, wire-cloth sieves, conforming to the requirements of Specification E 11, and of the following sizes:

No. 16 (1.18 mm)
No. 20 (850 μm)
No. 30 (600 μm)
No. 40 (425 μm)
No. 50 (300 μm)
No. 100 (150 μm)

B.7.4.2 Sample Splitter - The sample splitter shall be of the riffle type and shall have an even number of equal-width chutes which alternately discharge in opposite directions. The device shall have no less than eight chutes having a maximum opening no greater than ½ in. (12.7 mm), and a minimum opening at least three times the diameter of the largest particle of sand in a sample to be split. It shall be equipped with a hopper or straightedged pan by which a sample may be fed to the chutes at a controlled rate, and two receptacles to hold the two halves of the sample following splitting. The length of the hopper or pan shall be approximately equal to the overall width of the assembly of chutes.

B.7.4.3 Scales - The scales shall conform to the requirements of Specification C 490.

B.4.7.4 Weights - The weights shall conform to the requirements of Specification C 490.

B.7.4.5 Sampling

B.7.4.5.1 Procedure - Pour an approximate quantity of sand from the bag or other container into the hopper or pan, leveling the surface from end to end and from edge to edge so that approximately equal amounts will flow through each chute when the sand is introduced to the chutes. The rate at which the sand is introduced shall be such as to allow free flow through the chutes into the receptacles below. When all of the original material has been divided, the portion in one of the receptacles shall be reintroduced to the hopper. Repeat as many times as necessary to obtain a sample of approximately 700 g from the contents of a full bag. As appropriate, the portion of the sample collected in the second receptacle each time may either be reserved for testing, reserved for reduction in size for other tests, or discarded.

B.7.6 Sieve Analysis

B.7.6.1 General - Conduct the sieve analysis by either hand sieving, or mechanical sieving.

B.7.6.2 Hand Sieving - Attach a snug-fitting pan to each individual clean, dry sieve. Place thereon a test sample of 80 to 90 g of sand split from the previously procured 700-g sample by continuing the reduction procedure described in B.7.5.1 and attach a snug-fitting cover. Hold the assembly in a slightly inclined position in one hand. Strike the side of the sieve sharply and with an upward motion against the heel of the other hand at the rate of about 150 times per minute, turning the sieve about 1/6 of a revolution at intervals of approximately 25 strokes. Continue sieving until not more than 0.5 g passes through the sieve in 1 min. Express the weight of the residue on each sieve as a percentage of the test sample.

B.7.6.3 Mechanical Sieving - Using snug-fitting sieves, pan and cover, nest the required number of sieves, attach the pan to the bottom sieve, place a test sample of 80 to 90 g of sand split from the previously procured sample of 700 g on the top sieve, attach the cover, and agitate the nest for 15 min. Express the weight of the residue on each sieve as a percentage of the weight of the test sample.

B.7.7. Rejection

B.7.7.1 Standard Sand - A bag of standard sand shall be rejected if the contents fail to meet one or more of the requirements of this specification. A shipment of standard sand may be accepted if the contents of a number of randomly selected bags, at least equivalent to the cube root of the total number of bags in the shipment, meet the requirements of this specification. If any of these randomly selected bags fail to meet one or more of the requirements of this specification, a second random selection of bags, equal in number to the first lot, shall be tested. If the total accumulative number of bags in the shipment that fail to meet one or more of the requirements of this specification equals or exceeds two bags, based on both samplings, the shipment shall be rejected.

B.7.7.2 The following table shows the number of randomly selected bags to be tested referenced to the cube root of the number of bags in the shipment. The number of bags in Column 2 is the number to be selected for the test and retest.

Total No. of Bags in Shipment	No. of Randomly Selected Bags to test($\sqrt[3]{}$)	No. of Failures to Reject Shipment on Accumulation of Repeat Tests
2 to 8	2	2
9 to 27	3	2
28 to 64	4	2
65 to 125	5	2

B.7.7.3 Graded Standard Sand - A bag of graded standard sand shall be rejected if the contents fail to meet one or more of the requirements of this specification. A shipment of graded standard sand shall be sampled by selecting every 15th bag by consecutive number beginning with the 5th bag of the shipment. Bags missing from the sequence shall be counted as if they were there. A shipment of graded standard sand may be accepted if the contents of the selected bags meet the requirements of this specification. If any of the selected bags fail to meet one or more of the requirements of this specification, the shipment shall be resampled by selecting every 15 th bag by consecutive number beginning with the 10th bag of the shipment. A shipment of graded standard sand may be accepted if the contents of the selected bags on the resampling meet the requirements of this specification. If any of the selected bags on the resampling fail to meet one or more of the requirements of this specification, and the rejected bag is within a 15-bag sequence of a bag rejected in the first selection of bags, the entire sequence of bags between accepted tests shall be rejected. The entire shipment shall be rejected if half of the number of bags tested fail to meet the requirements of this specification, or if the shipment consists of five bags or less if one randomly selected bag fails the specification.

B.7.7.4 Table B.7.1 shows for both sampling and resampling in Column 2 the number of bags to be tested based on the total number of consecutive bags in the shipment. Column 3 shows the identification of the bag to be selected for test based on the consecutive number of the bags.

Note B.7.1 - If, in a shipment of 30 bags, on first sampling, the 20th bag fails the specification, and on resampling, the 25th bag fails the specification, the entire shipment is rejected. If the shipment were 100 bags and on first sampling only the 20th bag fails, and on resampling only the 25th bag fails, only bags No. 11 to 34 would be rejected.

Table B.7.1 Sampling and Resampling of Graded Standard Sand

Total No. of Consecutive Bags in Shipment	No. of Bags to Test	Test in Consecutive No., the Following Bags
Sampling:		
less than 5	1	random
5 to 19	1	5th
20 to 34	2	5th and 20th
35 to 49	3	5th, 20th, and 35th
50 to 64	4	5th, 20th, 35th, and 50th
Resampling:		
less than 5	none	reject shipment
5 to 19	1	10th
20 to 34	2	10th and 25th
35 to 49	3	10th, 25th, and 40th
50 to 64	4	10th, 25th, 40th, and 55th

endix C. CHEMICAL COMPOSITION AND CHEMICAL PARAMETER OF CLINKER

Two-Component

ample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
1	19.79	6.98	2.11	65.20	4.31	1.10	0.51	2.26	2.18	3.31	100.00	31.61	65.07	7.66	14.93	6.42
2	20.20	7.12	2.13	64.52	4.38	1.12	0.53	2.19	2.18	3.34	97.25	32.18	58.21	14.01	15.27	6.48
3	20.60	7.25	2.17	63.85	4.44	1.15	0.54	2.13	2.19	3.34	94.39	32.76	51.51	20.21	15.54	6.60
4	20.98	7.40	2.20	63.20	4.51	1.16	0.55	2.07	2.19	3.36	91.72	33.37	44.93	26.27	15.89	6.70
5	21.39	7.55	2.22	62.52	4.57	1.19	0.56	2.01	2.19	3.40	89.00	33.97	38.00	32.67	16.25	6.76
6	21.78	7.68	2.26	61.86	4.63	1.21	0.58	1.95	2.19	3.40	86.50	34.55	31.42	38.75	16.53	6.88
7	22.18	7.80	2.31	61.17	4.71	1.24	0.59	1.89	2.19	3.38	84.01	35.14	24.69	44.97	16.76	7.03
8	22.57	7.96	2.33	60.51	4.77	1.26	0.60	1.84	2.19	3.42	81.66	35.21	17.94	51.19	17.15	7.09
9	22.98	8.09	2.37	59.84	4.82	1.29	0.61	1.79	2.20	3.41	79.33	36.32	11.16	57.48	17.43	7.21
10	23.37	8.24	2.40	59.16	4.90	1.30	0.63	1.74	2.20	3.43	77.11	36.95	4.38	63.71	17.78	7.30

Three-Component

ample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
1	23.72	8.26	2.42	58.85	4.83	1.30	0.62	1.71	2.22	3.42	75.70	36.97	0.26	67.83	17.80	7.36
2	22.36	7.88	2.32	60.84	4.73	1.24	0.60	1.87	2.19	3.40	82.79	35.44	21.21	48.19	16.98	7.05
3	23.47	8.08	2.39	59.53	4.68	1.26	0.59	1.75	2.24	3.39	77.51	36.15	6.21	62.62	17.38	7.27
4	22.12	7.70	2.29	61.53	4.58	1.21	0.57	1.92	2.22	3.37	84.84	34.61	27.26	42.87	16.56	6.95
5	22.67	7.81	2.32	60.86	4.55	1.22	0.57	1.86	2.24	3.36	82.02	34.98	19.65	50.19	16.77	7.06
6	23.21	7.91	2.36	60.21	4.52	1.23	0.56	1.80	2.26	3.36	79.37	35.34	12.15	57.39	16.97	7.17
7	23.75	8.01	2.39	59.55	4.50	1.24	0.56	1.74	2.29	3.35	76.83	35.70	4.63	64.62	17.18	7.27
8	21.87	7.66	2.27	61.84	4.59	1.20	0.57	1.95	2.20	3.38	86.18	34.45	30.77	39.51	16.46	6.91
9	21.33	7.43	2.22	62.86	4.45	1.16	0.55	2.03	2.21	3.35	89.89	33.44	40.68	30.48	15.94	6.75
10	21.87	7.53	2.25	62.20	4.43	1.18	0.54	1.97	2.23	3.34	86.90	33.81	33.16	37.69	16.15	6.86
11	22.41	7.63	2.29	61.54	4.40	1.19	0.54	1.90	2.26	3.34	84.04	34.17	25.65	44.91	16.35	6.96
12	22.94	7.73	2.32	60.89	4.38	1.20	0.54	1.85	2.28	3.33	81.32	34.53	18.19	52.08	16.56	7.07
13	23.49	7.83	2.36	60.23	4.35	1.20	0.54	1.79	2.31	3.32	78.69	34.90	10.65	59.32	16.77	7.18
14	24.03	7.93	2.39	59.57	4.33	1.22	0.53	1.73	2.33	3.32	76.19	35.26	3.15	66.51	16.98	7.28
15	20.54	7.15	2.15	64.20	4.32	1.12	0.52	2.15	2.21	3.32	95.35	32.26	54.12	18.05	15.31	6.55
16	21.07	7.25	2.19	63.54	4.30	1.13	0.52	2.08	2.23	3.32	92.14	32.63	46.67	25.20	15.52	6.65
17	21.61	7.35	2.22	62.88	4.28	1.14	0.52	2.02	2.26	3.31	89.04	33.00	39.14	32.43	15.74	6.76
18	22.14	7.46	2.26	62.22	4.25	1.15	0.52	1.95	2.28	3.31	86.11	33.37	31.66	39.61	15.95	6.87
19	22.67	7.56	2.29	61.57	4.23	1.16	0.51	1.89	2.30	3.30	83.32	33.74	24.22	46.75	16.16	6.97
20	23.21	7.66	2.33	60.91	4.21	1.17	0.51	1.84	2.33	3.29	80.63	34.11	16.71	53.96	16.37	7.08
21	23.75	7.76	2.36	60.25	4.19	1.18	0.51	1.78	2.35	3.28	78.06	34.48	9.24	61.12	16.58	7.18
22	24.29	7.86	2.40	59.59	4.16	1.19	0.51	1.72	2.37	3.28	75.57	34.85	1.65	68.40	16.79	7.29
23	19.74	6.87	2.08	65.54	4.19	1.08	0.50	2.28	2.20	3.30	101.25	31.08	67.59	5.60	14.69	6.35
24	20.27	6.98	2.12	64.88	4.17	1.09	0.49	2.21	2.23	3.29	97.76	31.45	60.13	12.76	14.90	6.45
25	20.80	7.08	2.15	64.22	4.15	1.10	0.50	2.14	2.25	3.29	94.47	31.83	52.71	19.88	15.12	6.56
26	21.33	7.18	2.19	63.57	4.13	1.11	0.49	2.07	2.28	3.28	91.29	32.20	45.21	27.07	15.33	6.66
27	21.87	7.28	2.23	62.90	4.11	1.12	0.49	2.01	2.30	3.28	88.26	32.58	37.71	34.26	15.54	6.77
28	22.40	7.38	2.26	62.25	4.09	1.13	0.49	1.94	2.32	3.27	85.40	32.95	30.30	41.37	15.75	6.87

2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
29	22.93	7.49	2.29	61.59	4.07	1.14	0.49	1.88	2.34	3.27	82.62	33.32	22.77	48.59	15.97	6.98
30	23.47	7.59	2.33	60.93	4.05	1.15	0.49	1.83	2.37	3.26	79.99	33.70	15.33	55.72	16.18	7.09
31	24.00	7.70	2.36	60.27	4.03	1.16	0.48	1.77	2.39	3.26	77.43	34.08	7.77	62.97	16.40	7.19
32	24.54	7.80	2.40	59.60	4.01	1.17	0.48	1.72	2.41	3.25	75.00	34.45	0.26	70.17	16.61	7.30
33	19.46	6.70	2.05	66.23	4.04	1.05	0.47	2.35	2.22	3.26	103.91	30.27	73.70	0.22	14.28	6.25
34	19.99	6.80	2.09	65.57	4.03	1.05	0.47	2.27	2.25	3.26	100.32	30.65	66.26	7.34	14.49	6.35
35	20.52	6.90	2.12	64.91	4.01	1.07	0.47	2.20	2.27	3.25	96.93	31.03	58.87	14.43	14.71	6.46
36	21.06	7.01	2.15	64.24	3.99	1.08	0.47	2.13	2.30	3.25	93.59	31.41	51.23	21.74	14.23	6.56
37	21.59	7.11	2.19	63.58	3.97	1.09	0.47	2.06	2.32	3.25	90.48	31.79	43.77	28.90	15.14	6.67
38	22.12	7.22	2.23	62.92	3.95	1.10	0.46	1.99	2.34	3.24	87.52	32.17	36.34	36.01	15.36	6.77
39	22.66	7.32	2.26	62.26	3.93	1.11	0.46	1.93	2.37	3.24	84.67	32.55	28.84	43.20	15.57	6.88
40	23.18	7.42	2.30	61.61	3.91	1.12	0.46	1.87	2.39	3.23	81.96	32.93	21.39	50.35	15.79	6.99
41	23.71	7.53	2.33	60.95	3.89	1.13	0.46	1.82	2.41	3.23	79.38	33.30	13.98	57.45	16.00	7.09
42	24.24	7.63	2.36	60.29	3.88	1.14	0.46	1.76	2.43	3.23	76.88	33.69	6.49	65.62	16.22	7.20
43	19.72	6.63	2.01	66.24	3.88	1.02	0.45	2.33	2.27	3.23	102.90	29.86	72.27	2.03	14.09	6.25
44	20.25	6.74	2.09	65.58	3.87	1.03	0.44	2.26	2.30	3.22	99.35	30.25	64.78	9.20	14.31	6.36
45	20.78	6.84	2.12	64.93	3.85	1.05	0.44	2.18	2.32	3.22	96.01	30.63	57.38	16.29	14.53	6.47
46	21.30	6.94	2.16	64.27	3.83	1.06	0.44	2.11	2.34	3.22	92.81	31.01	49.95	23.41	14.75	6.57
47	21.84	7.05	2.19	63.60	3.81	1.07	0.44	2.05	2.36	3.21	89.73	31.40	42.43	30.61	14.96	6.68
48	22.36	7.15	2.23	62.94	3.80	1.08	0.44	1.98	2.38	3.21	86.83	31.78	35.04	37.69	15.18	6.78
49	22.89	7.26	2.26	62.28	3.78	1.09	0.44	1.92	2.41	3.21	84.00	32.17	27.49	44.92	15.40	6.89
50	23.42	7.36	2.30	61.62	3.76	1.10	0.44	1.86	2.43	3.20	81.34	32.55	20.07	52.03	15.62	6.99
51	23.95	7.46	2.33	60.97	3.75	1.11	0.44	1.81	2.45	3.20	78.80	32.93	12.69	59.09	15.83	7.10
52	24.47	7.57	2.37	60.31	3.73	1.12	0.43	1.75	2.46	3.20	76.35	33.31	5.24	66.23	16.05	7.21
53	19.70	6.51	2.04	66.59	3.74	1.00	0.42	2.36	2.30	3.19	103.77	29.28	74.67	0.17	13.80	6.21
54	20.23	6.62	2.07	65.93	3.72	1.01	0.42	2.28	2.33	3.19	100.18	29.67	67.15	7.37	14.03	6.32
55	20.75	6.72	2.11	65.27	3.17	1.02	0.42	2.21	2.35	3.19	96.84	30.05	59.80	14.40	14.24	6.42
56	21.29	6.83	2.14	64.60	3.69	1.03	0.42	2.14	2.37	3.18	93.56	30.44	52.25	21.62	14.46	6.53
57	21.81	6.93	2.18	63.95	3.67	1.04	0.42	2.07	2.39	3.18	90.51	30.83	44.87	28.69	14.68	6.63

2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
58	22.34	7.04	2.21	63.29	3.66	1.05	0.41	2.00	2.42	3.18	87.56	31.21	37.41	35.83	14.90	6.74
59	22.86	7.14	2.25	62.63	3.64	1.06	0.42	1.94	2.44	3.18	84.76	31.60	29.99	42.93	15.12	6.84
60	23.39	7.25	2.28	61.97	3.63	1.07	0.41	1.88	2.46	3.17	82.05	31.99	22.50	50.10	15.34	6.95
61	23.91	7.35	2.32	61.31	3.61	1.09	0.41	1.83	2.47	3.17	79.49	32.37	15.13	57.15	15.56	7.05
62	24.44	7.46	2.35	60.64	3.60	1.10	0.41	1.77	2.49	3.17	76.99	32.77	7.61	64.35	15.78	7.16
63	24.96	7.56	2.39	59.99	3.58	1.11	0.41	1.72	2.51	3.17	74.64	33.15	0.21	71.43	16.00	7.27
64	20.20	6.50	2.06	66.28	3.58	0.98	0.40	2.31	2.36	3.16	101.08	29.09	69.64	5.39	13.74	6.27
65	20.73	66.	2.09	65.62	3.57	0.99	0.39	2.23	2.38	3.15	97.68	29.48	62.23	12.49	13.96	6.37
66	21.25	6.71	2.13	64.96	3.55	1.01	0.39	2.16	2.40	3.15	94.43	29.87	54.81	19.58	14.18	6.48
67	21.78	6.82	2.16	64.30	3.54	1.01	0.39	2.09	2.43	3.15	91.30	30.26	47.32	26.75	14.40	6.59
68	22.85	7.03	2.24	62.94	3.51	1.04	0.39	1.96	2.47	3.15	85.37	31.07	32.08	41.32	14.86	6.80
69	22.82	7.03	2.23	62.98	3.51	1.04	0.39	1.96	2.46	3.15	85.53	31.05	32.50	40.92	14.85	6.80
70	23.35	7.13	2.27	62.31	3.50	1.05	0.39	1.90	2.48	3.15	82.80	31.44	25.02	48.08	15.07	6.90
71	23.87	7.24	2.30	61.60	3.48	1.06	0.39	1.85	2.50	3.14	80.20	31.83	17.58	55.19	15.29	7.01
72	23.49	7.34	2.34	61.00	3.47	1.07	0.39	1.79	2.52	3.14	77.72	32.22	10.19	62.26	15.51	7.11
73	24.91	7.45	2.37	60.34	3.45	1.08	0.39	1.74	2.54	3.14	75.34	32.61	2.80	69.33	15.73	7.22
74	20.44	6.44	2.06	66.29	3.44	0.96	0.37	2.29	2.40	3.12	100.21	28.73	68.32	7.06	13.58	6.28
75	20.96	6.55	2.10	65.63	3.42	0.97	0.37	2.22	2.43	3.12	96.85	29.12	60.90	14.15	13.80	6.38
76	21.47	6.65	2.13	64.98	3.41	0.99	0.37	2.15	2.45	3.12	93.69	29.51	53.56	21.17	14.02	6.49
77	22.00	6.76	2.16	64.31	3.40	1.00	0.37	2.08	2.47	3.12	90.62	29.91	46.10	28.30	14.25	6.59
78	22.52	6.86	2.20	63.65	3.39	1.01	0.37	2.02	2.49	3.12	87.70	30.30	38.68	35.39	14.47	6.70
79	23.04	6.97	2.24	62.99	3.37	1.02	0.37	1.95	2.50	3.12	84.90	30.70	31.23	42.51	14.69	6.80
80	23.56	7.07	2.27	62.34	3.36	1.03	0.37	1.90	2.52	3.12	82.26	31.09	23.91	49.52	14.91	6.91
81	24.08	7.18	2.30	61.68	3.35	1.04	0.37	1.84	2.54	3.12	79.70	31.49	16.50	56.60	15.13	7.01
82	24.60	7.29	2.34	61.01	3.34	1.05	0.37	1.78	2.56	3.12	77.22	31.89	9.02	63.75	15.36	7.12
83	25.13	7.39	2.37	60.35	3.32	1.07	0.37	1.73	2.57	3.11	74.86	32.28	1.59	70.85	15.58	7.23
84	20.14	6.27	2.03	66.97	3.31	0.93	0.35	2.35	2.43	3.09	102.85	27.98	74.46	1.58	13.19	6.18
85	20.66	6.38	2.06	66.31	3.30	0.94	0.35	2.28	2.45	3.09	99.39	28.38	67.07	8.64	13.42	6.28

C.2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
86	21.17	6.49	2.10	65.65	3.29	0.95	0.35	2.21	2.47	3.09	96.12	28.77	59.72	15.66	13.64	6.39
87	21.69	6.59	2.13	64.99	3.28	0.97	0.35	2.14	2.49	3.09	93.00	29.17	52.38	22.68	13.86	6.49
88	22.21	6.70	2.17	64.33	3.26	0.98	0.35	2.07	2.51	3.09	89.95	29.57	44.87	29.85	14.09	6.60
89	22.73	6.81	2.20	63.67	3.25	0.99	0.35	2.01	2.52	3.09	87.08	29.97	37.49	36.90	14.31	6.70
90	23.25	6.92	2.24	63.00	3.24	1.00	0.35	1.94	2.54	3.09	84.32	30.37	30.07	43.99	14.54	6.81
91	23.76	7.02	2.27	62.35	3.23	1.01	0.35	1.89	2.56	3.09	81.73	30.76	22.78	50.96	14.76	6.91
92	24.29	7.13	2.31	61.68	3.22	1.02	0.35	1.83	2.58	3.09	79.17	31.16	15.29	58.12	14.99	7.02
93	24.81	7.23	2.34	61.02	3.21	1.04	0.35	1.78	2.59	3.09	76.74	31.57	7.88	65.19	15.21	7.13
94	25.33	7.34	2.38	60.36	3.20	1.05	0.35	1.72	2.61	3.09	74.41	31.96	0.48	72.26	15.44	7.23
95	20.35	6.22	2.03	66.98	3.18	0.91	0.33	2.34	2.47	3.06	102.04	27.64	73.29	3.08	13.04	6.18
96	20.87	6.32	2.07	66.32	3.17	0.92	0.33	2.27	2.49	3.06	98.65	28.04	65.93	10.10	13.26	6.29
97	21.38	6.43	2.10	65.67	3.15	0.94	0.33	2.20	2.51	3.06	95.42	28.44	58.58	17.13	13.49	6.40
98	21.90	6.54	2.13	65.01	3.14	0.95	0.33	2.13	2.53	3.06	92.31	28.84	51.18	24.19	13.71	6.50
99	22.41	6.64	2.17	64.35	3.14	0.96	0.33	2.06	2.54	3.06	89.37	29.24	43.83	31.21	13.94	6.60
100	22.93	6.75	2.20	63.69	3.13	0.97	0.33	2.00	2.56	3.06	86.52	29.64	36.40	38.30	14.17	6.71
101	23.45	6.86	2.24	63.02	3.12	0.98	0.33	1.94	2.58	3.06	83.79	30.05	28.98	45.39	14.39	6.82
102	23.96	6.97	2.27	62.36	3.11	1.00	0.33	1.88	2.59	3.06	81.20	30.45	21.64	52.40	14.62	6.92
103	24.49	7.07	2.31	61.70	3.10	1.01	0.32	1.82	2.61	3.06	78.69	30.85	14.18	59.52	14.84	7.03
104	25.00	7.18	2.34	61.04	3.09	1.02	0.33	1.77	2.63	3.07	76.30	31.25	6.81	66.55	15.07	7.13
105	20.56	6.16	2.03	67.00	3.04	0.90	0.31	2.33	2.51	3.03	101.28	27.30	72.18	4.50	12.89	6.19
106	21.07	6.27	2.07	66.34	3.04	0.91	0.30	2.26	2.53	3.03	97.95	27.71	64.86	11.49	13.11	6.29
107	21.59	6.38	2.10	65.68	3.03	0.92	0.30	2.19	2.55	3.03	94.74	28.11	57.47	18.55	13.34	6.40
108	22.10	6.48	2.14	65.02	3.02	0.93	0.31	2.12	2.56	3.03	91.69	28.52	50.10	25.58	13.57	6.51
109	22.62	6.59	2.17	64.36	3.01	0.94	0.31	2.05	2.58	3.04	88.75	28.93	42.66	32.68	13.80	6.61
110	23.13	6.70	2.21	63.69	3.00	0.96	0.31	1.99	2.60	3.04	85.94	29.33	35.26	38.73	14.02	6.72
111	23.64	6.81	2.24	63.04	2.99	0.97	0.31	1.93	2.61	3.04	83.30	29.73	28.00	46.67	14.25	6.82
112	24.16	6.91	2.27	62.38	2.99	0.98	0.31	1.87	2.63	3.04	80.72	30.14	20.57	53.76	14.48	6.93
113	24.68	7.02	2.31	61.71	2.98	0.99	0.31	1.82	2.64	3.04	78.24	30.55	13.15	60.84	14.71	7.03

C.2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
114	25.19	7.13	2.34	61.06	2.97	1.00	0.31	1.76	2.66	3.04	75.88	30.95	5.81	67.84	14.93	7.14
115	20.76	6.11	2.04	67.01	2.92	0.88	0.28	2.32	2.55	3.00	100.54	26.99	71.06	5.93	12.74	6.20
116	21.67	6.21	2.07	66.36	2.91	0.89	0.28	2.25	2.57	3.00	97.27	27.39	63.77	12.87	12.97	6.30
117	21.78	6.32	2.10	65.70	2.91	0.90	0.28	2.18	2.58	3.00	94.11	27.80	56.41	19.90	13.20	6.41
118	22.30	6.43	2.14	65.03	2.90	0.92	0.29	2.11	2.60	3.01	91.06	28.21	48.96	27.01	13.43	6.51
119	22.80	6.54	2.17	64.38	2.89	0.93	0.29	2.04	2.62	3.01	88.21	28.62	41.68	33.95	13.65	6.62
120	23.32	6.65	2.21	63.71	2.88	0.94	0.29	1.98	2.63	3.01	85.44	29.02	34.28	41.01	13.88	6.72
121	23.83	6.76	2.24	63.06	2.88	0.95	0.29	1.92	2.65	3.01	82.81	29.43	26.97	47.98	14.11	6.83
122	24.34	6.86	2.28	62.39	2.87	0.97	0.29	1.86	2.66	3.01	80.26	29.84	19.57	55.04	14.34	6.93
123	24.86	6.97	2.31	61.73	2.86	0.98	0.29	1.81	2.68	3.02	77.82	30.25	12.19	62.08	14.57	7.04
124	25.37	7.08	2.35	61.07	2.86	0.99	0.29	1.75	2.69	3.02	75.46	30.66	4.76	69.16	14.80	7.14
125	20.44	5.95	2.00	67.70	2.80	0.85	0.26	2.39	2.57	2.97	103.30	26.26	77.73	0.19	12.37	6.10
126	20.95	6.06	2.04	67.03	2.80	0.86	0.26	2.31	2.59	2.97	99.85	26.68	70.01	7.27	12.60	6.20
127	21.46	6.16	2.07	66.37	2.79	0.88	0.27	2.23	2.61	2.97	96.58	27.09	62.63	14.30	12.83	6.31
128	21.97	6.27	2.11	65.71	2.79	0.89	0.26	2.17	2.62	2.98	93.48	27.50	55.30	21.29	13.06	6.41
129	22.48	6.38	2.14	65.05	2.78	0.90	0.27	2.10	2.64	2.98	90.53	27.91	48.01	28.24	13.29	6.52
130	22.99	6.49	2.18	64.39	2.77	0.91	0.27	2.03	2.65	2.98	87.66	28.32	40.61	35.30	13.50	6.62
131	23.50	6.60	2.21	63.73	2.77	0.92	0.27	1.97	2.67	2.99	84.94	28.73	33.28	42.28	13.75	6.73
132	24.01	6.71	2.25	63.06	2.76	0.94	0.27	1.91	2.68	2.99	82.31	29.15	25.88	49.34	13.98	6.83
133	24.52	6.82	2.28	62.41	2.76	0.95	0.27	1.86	2.70	2.99	79.83	29.55	18.61	56.27	14.21	6.94
134	25.04	6.93	2.31	61.74	2.75	0.96	0.27	1.80	2.71	2.99	77.39	29.97	11.17	63.36	14.44	7.04
135	25.54	7.03	2.35	61.09	2.74	0.98	0.27	1.75	2.72	3.00	75.10	30.38	3.87	70.33	14.67	7.15
136	20.62	5.90	2.01	67.71	2.69	0.83	0.24	2.37	2.61	2.94	102.59	25.96	76.41	1.50	12.23	6.10
137	21.13	6.00	2.04	67.05	2.68	0.85	0.25	2.30	2.63	2.94	99.20	26.38	69.03	8.53	12.46	6.21
138	21.64	6.11	2.07	66.39	2.68	0.86	0.25	2.23	2.64	2.95	95.99	26.79	61.69	15.53	12.70	6.31
139	22.15	6.22	2.11	65.73	2.67	0.87	0.25	2.16	2.66	2.95	92.92	27.20	54.35	22.52	12.93	6.42
140	22.66	6.33	2.14	65.06	2.67	0.88	0.25	2.09	2.67	2.96	89.97	27.62	46.98	29.55	13.16	6.52
141	23.17	6.44	2.18	64.40	2.66	0.90	0.25	2.03	2.69	2.96	87.16	28.03	39.64	36.53	13.39	6.63

C.2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
142	23.68	6.55	2.21	63.74	2.66	0.91	0.25	1.97	2.70	2.96	84.47	28.45	32.32	43.52	13.62	6.73
143	24.19	6.66	2.25	63.08	2.65	0.92	0.25	1.91	2.72	2.97	81.87	28.86	24.95	50.54	13.85	6.84
144	24.70	6.77	2.28	62.42	2.65	0.93	0.25	1.85	2.73	2.97	79.39	29.28	17.59	57.55	14.08	6.94
145	25.21	6.88	2.32	61.75	2.64	0.95	0.25	1.79	2.74	2.97	76.98	29.70	10.19	64.60	14.32	7.05
146	25.71	6.99	2.35	61.10	2.64	0.96	0.25	1.74	2.75	2.97	74.72	30.11	2.92	71.53	14.55	7.15
147	20.81	5.85	2.01	67.72	2.57	0.82	0.22	2.36	2.65	2.91	101.86	25.67	75.30	2.88	12.10	6.11
148	21.32	5.96	2.04	67.05	2.57	0.83	0.23	2.29	2.67	2.92	98.53	26.09	67.95	9.88	12.34	6.21
149	21.82	6.07	2.08	66.40	2.56	0.84	0.23	2.22	2.68	2.92	95.40	26.50	60.71	16.78	12.56	6.32
150	22.33	6.17	2.11	65.74	2.56	0.86	0.23	2.15	2.69	2.93	92.35	26.92	53.35	23.79	12.80	6.42
151	22.84	6.29	2.15	65.08	2.56	0.87	0.23	2.08	2.71	2.93	89.45	27.34	46.01	30.78	13.03	6.53
152	23.34	6.40	2.18	64.42	2.55	0.88	0.23	2.02	2.72	2.94	86.66	27.76	38.68	37.77	13.26	6.63
153	23.85	6.51	2.21	63.75	2.55	0.90	0.23	1.96	2.74	2.94	83.98	28.18	31.30	44.79	13.50	6.74
154	24.35	6.61	2.25	63.10	2.55	0.91	0.23	1.90	2.75	2.94	81.46	28.59	24.05	51.69	13.73	6.84
155	24.87	6.73	2.28	62.43	2.54	0.92	0.23	1.84	2.76	2.95	78.98	29.01	16.65	58.75	13.96	6.95
156	25.37	6.84	2.32	61.77	2.54	0.93	0.23	1.79	2.77	2.95	76.63	29.43	9.37	65.68	14.19	7.05
157	25.88	6.94	2.35	61.11	2.54	0.95	0.23	1.74	2.78	2.95	74.37	29.84	2.05	72.65	14.43	7.16
158	20.99	5.80	2.01	67.73	2.46	0.80	0.21	2.35	2.69	2.89	101.22	25.39	74.35	4.10	11.97	6.11
159	21.49	5.91	2.04	67.07	2.46	0.82	0.21	2.28	2.70	2.89	97.95	25.81	67.04	11.06	12.21	6.22
160	22.00	5.91	2.04	67.07	2.46	0.82	0.21	2.21	2.72	2.90	94.82	26.23	59.73	18.02	12.44	6.32
161	22.50	6.13	2.11	65.75	2.46	0.84	0.21	2.14	2.73	2.90	91.81	26.65	52.38	25.01	12.68	6.43
162	23.01	6.24	2.15	65.09	2.45	0.85	0.21	2.07	2.74	2.91	88.95	27.07	45.08	31.97	12.91	6.53
163	23.51	6.35	2.18	64.43	2.45	0.87	0.21	2.01	2.76	2.91	86.21	27.49	37.78	38.92	13.14	6.64
164	24.01	6.46	2.22	63.77	2.45	0.88	0.21	1.95	2.77	2.92	83.57	27.91	30.44	45.90	13.38	6.74
165	24.52	6.57	2.25	63.11	2.44	0.89	0.22	1.89	2.78	2.92	81.03	28.33	23.11	52.89	13.61	6.85
166	25.02	6.68	2.28	62.45	2.44	0.91	0.22	1.84	2.79	2.93	78.61	28.75	15.82	59.83	13.84	6.95
167	25.53	6.79	2.32	61.78	2.44	0.92	0.22	1.78	2.80	2.93	76.27	29.17	8.49	66.80	14.08	7.06
168	26.04	6.90	2.35	61.12	2.44	0.93	0.22	1.73	2.81	2.93	74.03	29.59	1.17	73.77	14.31	7.16
169	21.15	5.75	2.01	67.75	2.36	0.79	0.19	2.34	2.73	2.86	100.63	25.11	73.46	5.24	11.85	6.12

C.2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
170	21.66	5.86	2.05	67.09	2.35	0.80	0.19	2.27	2.74	2.87	97.39	25.54	66.15	12.20	12.08	6.22
171	22.16	5.98	2.08	66.42	2.35	0.82	0.19	2.20	2.75	2.87	94.27	25.96	58.79	19.20	12.32	6.33
172	22.67	6.09	2.11	65.76	2.35	0.83	0.19	2.13	2.77	2.88	91.29	26.39	51.44	26.20	12.56	6.43
173	23.17	6.20	2.15	65.10	2.35	0.84	0.19	2.07	2.78	2.88	88.47	26.81	44.18	33.12	12.79	6.54
174	23.67	6.31	2.18	64.44	2.35	0.85	0.20	2.00	2.79	2.89	85.77	27.23	36.91	40.03	13.02	6.64
175	24.17	6.42	2.22	63.78	2.34	0.87	0.20	1.94	2.80	2.90	83.15	27.65	29.57	47.02	13.26	6.75
176	24.68	6.53	2.25	63.12	2.34	0.88	0.20	1.89	2.81	2.90	80.65	28.07	22.27	53.96	13.49	6.85
177	25.18	6.64	2.29	62.46	2.34	0.89	0.20	1.83	2.82	2.91	78.24	28.50	14.94	60.94	13.73	6.96
178	25.69	6.75	2.32	61.79	2.34	0.91	0.20	1.78	2.83	2.91	75.92	28.92	7.61	67.92	13.97	7.06
179	26.19	6.86	2.36	61.13	2.34	0.92	0.20	1.73	2.84	2.91	73.70	29.34	0.32	74.85	14.20	7.17
180	21.32	5.71	2.01	67.77	2.25	0.77	0.17	2.33	2.76	2.84	100.05	24.85	72.58	6.38	11.73	6.12
181	21.82	5.82	2.05	67.10	2.25	0.79	0.17	2.26	2.77	2.84	96.82	25.28	65.21	13.39	11.97	6.23
182	22.32	5.93	2.08	66.44	2.25	0.80	0.18	2.19	2.79	2.85	93.77	25.70	57.94	20.31	12.20	6.33
183	22.82	6.04	2.12	65.78	2.25	0.81	0.18	2.12	2.80	2.86	90.85	26.12	50.66	27.23	12.44	6.44
184	23.33	6.15	2.15	65.11	2.25	0.83	0.18	2.06	2.81	2.86	88.00	26.56	43.27	34.26	12.67	6.54
185	23.83	6.26	2.18	64.46	2.25	0.84	0.18	2.00	2.82	2.87	85.34	26.98	36.04	41.14	12.91	6.65
186	24.33	6.38	2.22	63.79	2.25	0.85	0.18	1.94	2.83	2.87	82.76	27.40	28.74	48.09	13.15	6.75
187	24.84	6.49	2.25	63.12	2.25	0.87	0.18	1.88	2.84	2.88	80.25	27.83	21.35	55.11	13.38	6.86
188	25.34	6.60	2.29	62.46	2.25	0.88	0.18	1.83	2.85	2.89	77.87	28.26	14.05	62.06	13.62	6.96
189	25.84	6.71	2.32	61.80	2.25	0.89	0.19	1.77	2.86	2.89	75.58	28.68	6.76	68.99	13.86	7.07
190	21.48	5.66	2.01	67.78	2.15	0.76	0.16	2.33	2.80	2.81	99.49	24.59	71.72	7.48	11.61	6.13
191	21.98	5.78	2.05	67.11	2.15	0.77	0.16	2.25	2.81	2.82	96.29	25.02	64.35	14.49	11.85	6.23
192	22.48	5.89	2.08	66.45	2.15	0.79	0.16	2.18	2.82	2.83	93.25	25.45	57.03	21.45	12.09	6.34
193	22.98	6.00	2.12	65.79	2.15	0.80	0.16	2.12	2.83	2.84	90.36	25.88	49.75	28.37	12.32	6.44
194	23.49	6.11	2.15	65.12	2.15	0.82	0.16	2.05	2.84	2.84	87.55	26.31	42.39	35.37	12.56	6.55
195	23.98	6.22	2.19	64.47	2.15	0.83	0.16	1.99	2.85	2.85	84.93	26.73	35.20	42.21	12.80	6.65
196	24.49	6.34	2.22	63.80	2.15	0.84	0.16	1.93	2.86	2.85	82.34	27.17	27.81	49.24	13.04	6.76
197	24.98	6.45	2.25	63.14	2.15	0.86	0.17	1.88	2.87	2.86	79.90	27.59	20.58	56.11	13.27	6.86

C.2 Three-Component (Cont.)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	H.M.	S.M.	A.M.	L.S.F. (%)	liquid phase (%)	C_3S	C_2S	C_3A	C_4AF
198	25.48	6.56	2.29	62.48	2.15	0.87	0.17	1.82	2.88	2.87	77.54	28.02	13.28	63.05	13.51	6.97
199	25.99	6.67	2.32	61.81	2.16	0.88	0.17	1.77	2.89	2.87	75.24	28.45	5.90	70.07	13.75	7.07
200	21.13	5.51	1.98	68.45	2.05	0.74	0.14	2339	2.82	2.78	102.22	23.91	78.14	1.66	11.26	6.03
201	21.64	5.62	2.02	67.78	2.05	0.75	0.14	2.32	2.83	2.79	98.90	24.35	70.76	8.67	11.50	6.13
202	22.13	5.74	2.05	67.13	2.05	0.76	0.14	2.24	2.84	2.80	95.79	24.77	63.52	15.55	11.73	6.24
203	22.63	5.85	2.08	66.46	2.06	0.78	0.14	2.18	2.85	2.81	92.79	25.20	56.24	22.48	11.97	6.34
204	23.14	5.96	2.12	65.79	2.06	0.79	0.14	2.11	2.86	2.81	89.89	25.64	48.87	29.48	12.22	6.45
205	23.63	6.07	2.15	65.14	2.06	0.80	0.15	2.05	2.87	2.82	87.17	26.07	41.67	36.32	12.45	6.55
206	24.13	6.18	2.19	64.47	2.06	0.82	0.15	1.98	2.88	2.83	84.52	26.50	34.36	43.28	12.69	6.66
207	24.63	6.30	2.22	63.81	2.06	0.83	0.15	1.93	2.89	2.84	81.96	26.93	27.00	50.27	12.93	6.76
208	25.13	6.41	2.26	63.15	2.06	0.84	0.15	1.87	2.90	2.84	79.56	27.36	19.81	57.10	13.17	6.87
209	25.63	6.52	2.29	62.49	2.06	0.86	0.15	1.82	2.91	2.85	77.21	27.79	12.51	64.05	13.41	6.97
210	26.13	6.63	2.32	61.82	2.07	0.87	0.16	1.76	2.92	2.85	74.94	28.22	5.16	71.03	13.65	7.08
211	21.29	5.47	1.98	68.46	1.96	0.72	0.12	2.38	2.86	2.76	101.65	23.67	77.27	2.76	11.15	6.03
212	21.79	5.58	2.02	67.79	1.96	0.74	0.12	2.31	2.87	2.77	98.38	24.11	69.93	9.73	11.39	6.14
213	22.28	5.70	2.05	67.14	1.96	0.75	0.12	2.24	2.88	2.78	95.31	24.53	62.73	16.58	11.63	6.24
214	22.78	5.81	2.08	66.48	1.96	0.76	0.13	2.17	2.89	2.79	92.34	24.97	55.44	23.50	11.87	6.35
215	23.28	5.92	2.12	65.81	1.96	0.78	0.13	2.10	2.90	2.79	89.48	25.40	48.11	30.47	12.11	6.45
216	23.78	6.03	2.15	65.15	1.97	0.79	0.13	2.04	2.90	2.80	86.75	25.84	40.82	37.39	12.35	6.56
217	24.28	6.15	2.19	64.48	1.97	0.80	0.13	1.98	2.91	2.81	64.13	26.27	33.54	44.31	12.59	6.66
218	24.77	6.26	2.22	63.83	1.97	0.82	0.13	1.92	2.92	2.82	81.64	26.70	26.31	51.18	12.83	6.76
219	25.27	6.37	2.26	63.16	1.97	0.83	0.14	1.86	2.93	2.82	79.22	27.13	19.03	58.09	13.07	6.87
220	25.76	6.48	2.29	62.50	1.98	0.85	0.14	1.81	2.94	2.83	76.91	27.57	11.76	65.00	13.31	6.97
221	26.26	6.60	2.33	61.83	1.98	0.86	0.14	1.76	2.94	2.84	75.65	28.00	4.42	71.98	13.55	7.08
For spent shale = 0; the proportions between limestone: clay are 81.61:18.39 (A), 80.48:19.52 (B), 79.36:20.64 (C), 78.21:21.79 (D)																
A	21.37	5.32	1.96	68.83	1.75	0.69	0.08	2.40	2.94	2.71	102	22.53	79.25	1.48	10.78	5.96
B	22.37	5.56	2.03	67.49	1.75	0.71	0.09	2.25	2.95	2.74	96	23.42	64.49	15.48	11.30	6.18
C	23.34	5.80	2.11	66.16	1.76	0.74	0.09	2.12	2.95	2.75	90	24.34	49.97	29.22	11.80	6.42
D	24.33	6.00	2.18	64.83	1.78	0.77	0.11	1.99	2.97	2.75	85	25.16	35.59	42.91	12.21	6.63

Appendix D. X-RAY DIFFRACTION

D.1 Chemical Analysis by X-Ray Diffraction

A given substance always produces a characteristic diffraction pattern, whether that substance is present in the pure state or as one constituent of a mixture of substances. This fact is the basis for the diffraction method of chemical analysis. Qualitative analysis for a particular substance is accomplished by identification of the pattern of that substance. Quantitative analysis is also possible, because the intensities of the diffraction lines due to one constituent of a mixture depend on the proportion of that constituent in the specimen.

The particular advantage of diffraction analysis is that it discloses the presence of a substance as that substance actually exists in the sample and not in terms of its constituent chemical elements. For example, if a sample contains the compound A_xB_y, the diffraction method will disclose the presence of A_xB_y as such, whereas ordinary chemical analysis would show only the presence of elements A and B.

Diffraction analysis is therefore useful whenever it is necessary to know the state of chemical combination of the elements involved or the particular phases in which they are present. Compared with ordinary chemical analysis, the diffraction method has the additional advantages that it is usually much faster, requires only a very small sample, and is nondestructive. As a result, the diffraction method has been widely applied for the analysis of materials.

D.2 The Diffraction of X-Rays

In most methods of crystal analysis by X-rays, monochromatic radiation, is used. The monochromatic radiation is the radiation of a single wavelength. The x-rays are generated by the impact of an electron stream on a target and a characteristic spectrum of x-rays is produced, a single line of which is selected by means of filters. A frequently used radiation is the α_1 member of the doublet in the K series produced when copper is the target. This is known as CuKα_1, radiation of wavelength = 1.537395 kx units. The kx unit is a length equal to 1.00203 Angstrom units. Modern determinations have been converted from kx to ^{o}A

X-rays are scattered by the atoms in a crystal and since the atoms are in a regular arrangement the scattered rays will reinforce each other in certain directions and cancel each other out in others.

Consider a plane in a crystal defined by the Miller indices (hkl)[9]. It will be seperated from similar planes by a distance d, the interplanar spacing,written as d_{hkl}. If a beam of x-rays falls on a single plane at an angle of incidence it can easily be shown that a diffracted beam will be formed. (Fig D.2.1) in the plane of incidence emerging at an angle θ, exactly as if the beam were reflected from the plane. The location and nature of the diffracting points which lie in the plane affect the intensity of this reflected beam, but not its direction. When the beam is incident upon a three-dimensional array of points the diffracted rays from each layer must be in phase for an emergent beam to be formed and therefore the path difference AB-AC=2d sin θ (Fig D.2.2) must be an integral number of wavelengths. Therefore

$n\lambda = 2d\sin\theta$

where d= distance between planes

n= any integer

λ= wavelength of the x-rays used

θ= angle between plane and x-ray beam

In this case, therefore, a diffracted beam is formed only when the angle of incidence satisfies the Bragg equation written above.

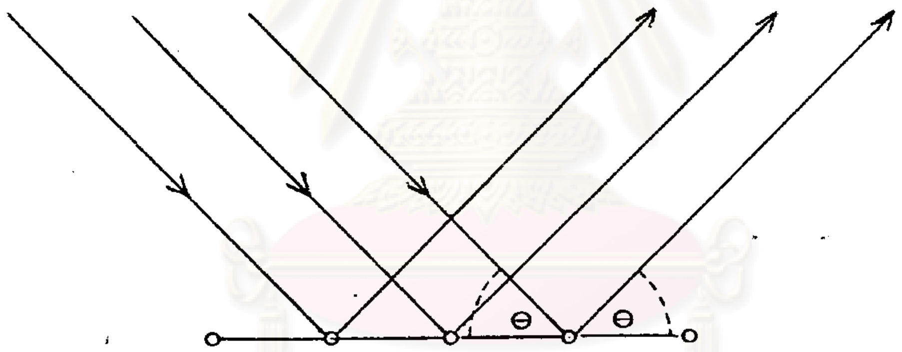


Fig D.2.1 Diffraction of x-rays by a single plane of atoms

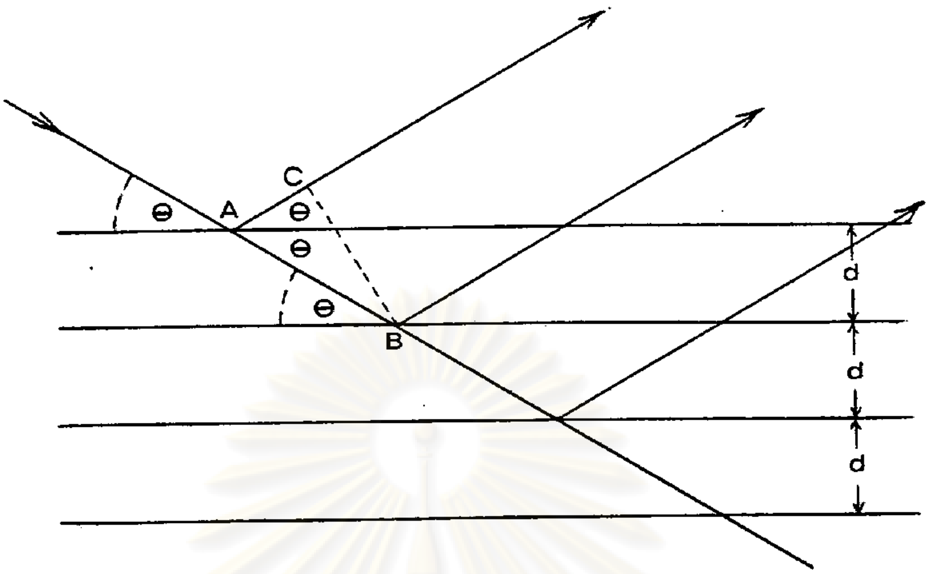


Fig D.2.2 Diffraction by a series of atomic planes of spacing d

Bragg's law nλ = 2d sinθ (CuKα)

CuKα$_1$, λ = 1,5405$_0$ Å

θ	0'	6' 1/10	12' 2/10	18' 3/10	24' 4/10	30' 5/10	36' 6/10	42' 7/10	48' 8/10	54' 9/10
0°	--	441,32	220.66	147,11	110,33	88,265	73.555	63.048	55,167	49.038
1	44,134	40.123	36,779	33.950	31,526	29.425	27.586	25.964	24.521	23,230
2	22,069	21.020	20,065	19,193	18,393	17,658	16,980	16,351	15,767	15.224
3	14.717	14,243	13.798	13,380	12.987	12.617	12,268	11.937	11.623	11.325
4	11.042	10,773	10.517	10,273	10.040	9,8172	9.6043	9.4004	9.2050	9,0176
5	8,8377	8,6648	8,4986	8,3387	8,1852	8,0363	7.8933	7,7553	7.6220	7.4932
6	7.3688	7,2485	7.1320	7,0192	6.9100	6.8042	6.7016	6.6021	6.5054	6,4115
7	6,3203	6,2317	6,1456	6,0619	5,9804	5.9011	5,8239	5.7487	5.6755	5,6041
8	5.5345	5,4666	5.4004	5,3358	5,2727	5,2111	5,1510	5,0922	5.0347	4,9785
9	4,9236	4,8700	4,8176	4,7663	4,7161	4,6669	4.6187	4.5715	4.5253	4.4800
10	4,4357	4,3923	4.3497	4,3079	4,2669	4,2267	4.1873	4.1486	4.1106	4,0733
11	4,0367	4,0008	3,9656	3.9310	3,8970	3.8635	3,8306	3.7983	3.7666	3.7354
12	3,7047	3,6745	3,6448	3.6156	3,5869	3,5587	3,5309	3.5036	3.4767	3.4502
13	3,4241	3,3984	3,3731	3,3482	3,3237	3,2995	3.2757	3.2522	3.2291	3.2063
14	3.1839	3,1618	3,1400	3,1185	3,0973	3,0764	3,0558	3,0354	3.0153	2 9955
15.	2,9700	2,9567	2,9377	2.9190	2,9005	2,8822	2.8642	2,8464	2,8289	2,8116
16	2,7945	2,7776	2,7609	2,7444	2,7281	2,7120	2,6961	2.6804	2.6659	2,6496
17	2,6345	2,6196	2,6048	2,5902	2.5758	2,5615	2.5474	2.5335	2,5196	2.5060
18	2,4926	2,4793	2,4661	2.4531	2,4402	2.4274	2.4148	2.4024	2.3901	2.3779
19	2.3659	2.3540	2,3422	2.3305	2,3189	2,3075	2.2962	2.2850	2.2739	2.2629
20	2.2520	2,2412	2.2306	2,2201	2,2097	2,1994	2,1892	2.1790	2.1690	2.1591
21	2,1493	2.1396	2,1299	2.1204	2.1110	2.1017	2.0924	2,0832	2.0741	2.0651
22	2,0562	2,0473	2.0385	2,0298	2,0213	2.0128	2,0044	1,9960	1,9877	1.9795
23	1,9713	1.9632	1,9552	1.9473	1.9394	1.9316	1.9239	1.9163	1.9087	1.9012
24	1,8938	1,8864	1,8790	1,8717	1,8645	1.8574	1,8503	1,8433	1,8363	1,8294
25	1,8226	1,8158	1,8091	1,8024	1,7958	1,7892	1,7826	1,7761	1,7697	1,7633
26	1,7571	1,7508	1.7446	1,7384	1,7323	1,7262	1,7202	1,7143	1,7084	1,7025
27	1,6967	1,6909	1,6851	1,6794	1,6737	1,6681	1.6625	1,6570	1,6515	1,6461
28	1,6407	1,6353	1,6300	1,6247	1,6195	1,6143	1,7091	1,6040	1,5989	1,5938
29	1,5887	1,5837	1,5788	1,5739	1,5690	1,5642	1,5594	1,5546	1,5499	1,5452
30	1,5405	1,5358	1,5312	1,5266	1,5221	1,5176	1,5131	1,5087	1,5043	1,4999

D.3 POWDER X-RAY DATA ON CEMENT MINERALS

The values of I represent relative intensity.

The d values for the spacings are given in Å, Angstrom units, the intensities are on an arbitrary numerical scale, 10 being the strongest and 1 the weakest. Where it is known that doublets would be shown by using a camera of very high resolving power (such as the Guinier camera), the lines are marked with an asterisk. The three strongest lines are underlined. Ths symbol B indicates a broad line

Calcium silicate hydrate. (I) [9]		Calcium silicate hydrate (II) [9]		Ca(OH)$_2$ [1]		3CaO, SiO$_2$ [2]		Alite [2]		β 2CaO, SiO$_2$ [5]	
d	I	d	I	d	I	d	I	d	I	d	I
9-14	10	9.80	9	4.900	7	5.901	4	3.861	3	4.920	1
3.06	10	4.90	2	3.112	2	3.862	3	3.517	1	4.645	1
2.81	8	3.07	10	2.628	10	3.510	2	3.334	2	3.790	3
1.83	8	2.85	5	2.447	1	3.346	2	3.144	2	3.380	1
1.67	4	2.80	9	1.927	4	3.227	1	3.022	8	3.335	1
1.53	2	2.40	4	1.796	4	3.022	8	2.959	6	3.090	1
1.40	4	2.20	1	1.687	2	2.957	6	2.880	2	3.040	2
1.17	1	2,10	1	1.634	1	2.891	3	2.804	1	2.874	2*
1.11	2	2.00	6	1.557	1	2.818	1	2.764	10	2.778	10*
1.07	1	1.83	9	1.434	2	2.776	10	2.739	9	2.740	10*
This is the data		1.72	1	1.449	2	2.730	8	2.682	3	2.714	1
for the poorly		1.62	1	1.314	1	2.670	1	2.592	9	2.607	10
crystalline		1.56	5	1.228	1	2.602	10	2.436	3	2.544	3
material. The		1.40	4	1.211	1	2.549	1	2.313	6	2.448	4*
long spacing		1.225	3	1.762	1	2.449	3	2.178	9	2.403	4
can vary consi-		1.165	3	1.1432	2	2.326	6	2.172	6	2.279	3
derably and		1.100	1	1.1275	1	2.304	5	2.089	1	2.189	6*
may also be		1.045	2	1.0599	2	2.277	2	2.060	1	1.163	4
undetected.		1.025	1	1.0366	1	2.234	1	2.028	1	2.128	1
		1.000	1	1.0143	2	2.185	10	1.973	5	2.088	1*
						2.159	1	1.928	6	2.044	2
						2.125	3	1.831	5	2.019	1
						2.083	4	1.819	5	1.982	7
						2.045	2	1.799	2	1.911	1
						2.011	1	1.761	9	1.892	4
						1.979	6/B	1.689	2	1.844	1
						1.940	7	1.640	2	1.806	2
						1.926	6	1.623	8	1.787	2
						1.900	2/B	1.537	6	1.763	1
						1.863	1	1.522	2	1.706	3
						1.825	6/B	1.485	9	1.632	7
						1.797	3			1.606	4
						1.771	9(1)			1.587	2
						1.752	8(1)			1.573	2
						1.642	2			1.550	1 B
						1.632	8			1.523	4
						1.623	6			1.483	3
						1.543	6			1.448	1
						1.526	2			1.427	1
						1.513	2			1.416	1
						1.497	6			1.406	1
						1.481	6			1.393	1

Pure 3CaO, SiO$_2$ can be distinguished from Alite since at (1) it gives doublets instead of single lines.

AUTOBIOGRAPHY

Miss Sunun Sakdivanichkul was born on January 1,1957 in Bangkok, Thailand. She graduated a Bachelor Degree of Science in Material Science from Chulalongkorn University in 1979 and continued her studies for Master Degree in Chemical Engineering at the same university. She was granted the Master Degree in April, 1982.