STATE OF CALIFORNIA—BUSINESS, TRANSPORTATION AND HOUSING AGENCY

DEPARTMENT OF TRANSPORTATION
DIVISION OF ENGINEERING SERVICES
Transportation Laboratory
5900 Folsom Blvd.
Sacramento, California 95819-4612

METHOD OF TEST FOR CEMENT OR LIME CONTENT IN TREATED AGGREGATE BY THE TITRATION METHOD

A. SCOPE

This test method describes the procedures for determining the percentage of portland cement or lime in freshly mixed, treated aggregate. These determinations are based upon chemical titration methods that relate the cement or lime contents of field-treated samples to known contents in laboratory samples.

Two procedures for determining cement content are included in this test method. The acid-base method is preferred, but, because the acid will attack certain minerals such as calcite and dolomite that are present in some aggregates, the constant-neutralization method is provided as an alternate when needed. A procedure for determining the appropriate titration method to be used is also included.

This test method is divided into the following parts:

1. Field Sampling of Cement or Lime Treated Materials.
2. Preparation of Test Samples.
3. Determination of Appropriate Titration Method.
4. Acid-Base Method for Determining Cement Content.
5. Constant-Neutralization Method for Determining Cement Content.
6. Constant-Neutralization Method for Determining Lime Content.

B. REFERENCES

AASHTO M 92 - Wire Cloth Sieves for Testing Purposes
California Test 226 - Determination of Moisture Content by Oven Drying

C. APPARATUS

A complete list of necessary equipment is included in each part of this test procedure. Detailed descriptions of some of these items are included below:

1. Sieves: U.S. Standard Sieves conforming to AASHTO M 92 and of convenient size and dimensions for separating the material into the required fractions. Sieves in 12 in. diameter frames are satisfactory, but other sizes may be used.

2. Titrating burettes: 100 mL automatic zeroing type with stopcock and necessary stand, clamp, etc. Burettes for use with screw-cap bottles to provide self-contained units are preferred, but stand-mounted units with side tubes for filling are acceptable.

3. Pipet: 100 mL pipet with hand-operated bulb filler.

4. Carboy: Plastic, 5 gal with neoprene or Tygon™ tubing, clamps, and fittings necessary to transfer small quantities of the solution to the burette or the measuring flask. Do not substitute glass containers for the plastic carboys.

5. Fluorescent light (optional), 36 in. long: A portable light which can be placed directly behind the samples during titration testing may be of help in detecting color changes of the solution.

6. Emergency washing facility: An ample water supply which is immediately available in the event acids or acid solutions are splashed or spilled on the skin and/or in the eyes.

D. MATERIALS

The materials required to perform the different tests are listed in the individual parts of this test procedure. Instructions for preparing and handling these materials are given below:

1. Hydrochloric acid (approximately 3 N) prepared as follows:

   a. Pour approximately 3 gal of water into a 5 gal plastic carboy.

   b. Pour the contents of two full standard 6 lb bottles of concentrated reagent grade hydrochloric acid into the carboy.

   c. Stir or mix the solution vigorously for about 1 min.

   d. Add water as necessary to bring the total volume of water and acid to 5 gal; then stir or mix the solution vigorously for another minute.

   e. Allow the acid solution to stand overnight then remix it once again prior to using.

   f. Remix the acid solution once a week during use and before using after storage for more than a week.

2. Sodium hydroxide (approximately 1 N) prepared as follows:

   a. Pour approximately one gal of tap water at a temperature of approximately 110˚F into a 5 gal plastic carboy. (This temperature is about as hot as can be tolerated on a person's hand.)

   b. Add 800 g of sodium hydroxide reagent grade pellets to the water, stopper the carboy, and shake it vigorously for about 1 min.

   c. Add tap water in approximately one gal increments until 5 gal of solution are attained. Stopper and vigorously shake the carboy after each addition. Successively reduce the temperature of each subsequent increment of water until the last portion is added at the temperature of the cold tap.

   d. After the solution has cooled to room temperature, examine the solution for suspended matter by looking down through the neck of the carboy while shining a strong light through the sides near the bottom. The presence of suspended matter is an indication of incompletely dissolved sodium hydroxide due to insufficient heat or insufficient agitation during the initial

mixing. Since reheating is normally required to dissolve the suspended material, it is usually more practical to discard the solution and mix a new batch.

3. Phenolphthalein indicator solution, 1 %:

   a. If the 1 % solution is not available, it can be prepared by dissolving 5 g of phenolphthalein powder U.S.P. in 250 mL of ethanol and then diluting this solution with 250 mL of deionized water.

4. Prepare Separan NP-10 solution as follows:

   a. Mix 0.5 g of NP-10 powder with 1000 mL of water in a glass beaker.

   b. Stir at frequent intervals until no suspended material is visible; normally about an hour.

   c. Pour the solution into a container suitable for storage and dispensing.

   d. Prepare a fresh solution weekly.

5. Water:

   a. Tap water is satisfactory for performing the titration test and preparing solutions.

   b. Calibration-curve samples must be prepared using the same source of water being used in the field.

E. CONTROLS

1. Standard Curing Time

Hydration of cement and lime begins with the introduction of water. Because the hydration time prior to starting the titration test can affect the test results, it is important that the field samples and calibration curve samples be cured for equal periods of time. A standard curing time should be established for control of titration testing on each project based on the following criteria:

   a. The curing time is the time lapse between the introduction of mixing water and beginning the test.

   b. The standard curing time shall be established on the basis of time elapsed between initial mixing and preparation of the field test specimens.

   c. The curing time for individual field samples shall be within ± 15 min of the standard curing time.

   d. If it becomes necessary for the curing time of the field samples to deviate from the standard curing time, a calibration curve shall be established using a corresponding curing time.

2. Calibration Curves

   a. The calibration curve must be established by the same technician who will be testing the field samples. Testing technique and ability to see changes in

color may affect the quantities of solutions used; however, since the test results are relative, accuracy will be maintained as long as the technician is consistent.

b. A calibration curve shall be established each day that cement/lime content tests are performed.

c. The average amount of acid (or sodium hydroxide) required to neutralize either pair of duplicate samples in the current calibration curve should not differ by more than ± 2 mL from the average amount required in the preceding calibration-curve test. When this tolerance is exceeded, the calibration-curve testing shall be repeated. If the retest confirms the curve in question, use these two curves to establish the new curve. If the retest corresponds with the previous curve, discard the curve in question, and use the retest.

d. When it is determined, on the basis of 5 or more tests, that the calibration curves are consistent, it is permissible to reduce the frequency of calibration curves to not less than one per 7 calendar days, or one for each 10 control tests. At any time a calibration curve does not meet the tolerances for consistency, the frequency of calibration curves shall be increased to not less than one per day until consistency is reestablished.

e. Changes in the acid solution or in the source of aggregate, cement, or water, may affect the test results. Any time the source of any of these items is changed, or a new reagent solution is mixed, it will be necessary to establish a new calibration curve.

3. Operator Qualifications

It is imperative that the technician performing the titration test establish a consistent technique for stirring the test samples and adding the titrating solutions. The purpose of stirring the test sample is to break up all agglomerations and expose all portions of the cement to the solution. It is of utmost importance in the constant-neutralization test that the addition of acid be carefully controlled. An excess of acid will attack reactive aggregates and will also alter the cement, thus causing inconsistencies in test results.

F. PROCEDURE

PART 1. FIELD SAMPLING OF CEMENT OR LIME-TREATED MATERIALS

1A. SCOPE

Since the methods of adding and mixing cement or lime may vary from one project to another, it is important that proper consideration be given to the sampling program to ensure meaningful test results. This Part describes acceptable sampling procedures for use under normal construction conditions. Other sampling schemes may be employed as necessary to accomplish the desired objectives.

1B. EQUIPMENT

1. Shovel

2. Hand scoop

3. Covered containers with a minimum capacity of 3000 g.

1C. CONTROL

1. Field samples should always be taken in sets of four or more. When the acid-base method is being used, up to eight samples can be tested simultaneously.

2. Field samples should normally be taken from the in place mixture as soon as possible after mixing and spreading, and before initial compaction.

3. Samples of in place material must be representative of the full depth of the layer. This can be accomplished best by digging a straight-sided hole through the layer and then removing material from the side of the hole until a properly sized sample is obtained.

4. Each field sample should weigh approximately 3000 g.

5. Retain field samples in individual covered containers until ready to prepare test samples.

6. The time differential between mixing the aggregate, cement/lime, and water of any two samples of a set shall not exceed 30 min.

1D. PROCEDURE

1. Sampling to Determine the Efficiency of Batch or Continuous-Mix Plants.

    a. Samples shall be taken from random locations after the mixed material has been spread on the roadbed.

    b. All of the samples shall be taken from a single batch or truckload of material.

2. Sampling to Determine the Efficiency of Road-Mixing Equipment.

    a. Samples shall be taken at equal intervals across the material mixed by a single pass of the mixer.

    b. If the normal mixing operation requires remixing the materials, sampling shall be done after the final mixing.

    c. When lime-treated materials are being mixed and placed in layers greater than 0.50 ft, samples shall be taken from the top and bottom halves of the layer at each sampling location.

3. Sampling to Determine Conformance to Overall Quantity Requirements.

    a. Samples may be taken from random locations within the test area or at predetermined intervals across the roadbed.

    b. The random selection of sampling locations should be done only after the efficiency of the mixing equipment has been determined to be satisfactory.

PART 2. PREPARATION OF TEST SAMPLES

2A. SCOPE

Properly prepared, representative test samples are critical to the accuracy of the titration tests. This Part describes procedures to be followed in preparing test samples from material placed in the field and laboratory-mixed samples for use in establishing calibration curves.

It is also important that the moisture content of the test samples be considered. This can be done either by preparing the calibration samples at the same moisture content as the field samples (Alternate 1) or by preparing the calibration samples on a dry weight basis and applying a correction factor (Alternate 2). Both alternatives are described.

2B. EQUIPMENT

1. A ⅜ in. sieve.

2. A 1½ in. sieve.

3. A balance or scale with a capacity of 5 kg and accuracy of 1 g.

4. A balance or scale with a capacity of 1 kg and accuracy of 0.1 g.

5. Two quart capacity, wide mouth polyethylene containers with covers (one required for each test sample).

6. Stainless steel stirring rods (one for each sample).

7. A timer.

8. Drying ovens, 230°F ± 9°F and 100°F ± 4°F.

2C. PROCEDURE

1. Preparation of samples for determining appropriate titration method.

   a. Follow the procedures for preparing calibration-curve samples, except that no cement will be added, and the dry mass of the aggregate shall be 300 g.

   b. Prepare two 300 g aggregate samples.

   c. Prepare two additional samples weighing 300 g each from dry Ottawa sand. Glass beads may be substituted for the Ottawa sand.

2. Preparation of calibration-curve samples (Alternate 1).

   This procedure provides for adjustment in batch masses to correlate with the assumed moisture content of field samples. No correction of titration results will be required.

   a. Obtain a representative sample of the aggregate and cement/lime being used on the project.

   b. Dry the aggregate to constant mass at 230°F ± 9°F. Drying may be waived provided the moisture content is determined and the batch masses are adjusted to provide the specified dry-aggregate masses. When testing

reclaimed aggregates containing traces of asphalt or asphalt concrete, the drying temperature shall not exceed 100°F.

c. Separate the aggregate on the 1½ in. and ⅜ in. sieves. Discard any aggregate retained on the 1½ in. sieve.

d. Using the project records, determine the moisture content representative of the completed mixture being placed or anticipated on the project.

e. Prepare four 300 g test samples conforming to the grading and moisture content of the field samples. Two samples shall contain 1 % more cement/lime than specified for the project, and two samples shall contain 1 % less than specified. The appropriate mass of aggregate, water, and cement/lime can be determined from Table 1 (See page 19) when whole percentages are required.

(1) Place the dry aggregate in a polyethylene container, then thoroughly blend in the cement/lime using a stainless steel stirring rod.

(2) Add the appropriate amount of water, and thoroughly mix again.

(3) Cover the container and allow the sample to cure for a period of time conforming to the requirements in the testing procedures.

3. Preparation of calibration-curve samples (Alternate 2).

This procedure does not require adjustments to batch masses because of field moisture contents. A correction factor is applied to the test results of the field samples based on their actual moisture contents.

a. Obtain a representative sample of the aggregate and cement/lime being used on the project.

b. Dry the aggregate to constant mass at 230°F ± 9°F. Drying may be waived provided the moisture content is determined and the batch masses are adjusted to provide the specified dry aggregate masses.

c. Separate the aggregate on the 1½ in. and ⅜ in. sieves. Discard any aggregate retained on the 1½ in. sieve.

d. Prepare four 300 g test samples conforming to the grading of the field samples. Two samples shall contain 1 % more cement/lime than specified for the project, and two samples shall contain 1 % less than specified. The appropriate masses of aggregate and cement/lime can be determined from Table 2 (See page 8).

(1) Place the aggregate in a polyethylene container; then thoroughly blend in the cement/lime using a stainless steel stirring rod.

(2) Add water to provide a moisture content approximately equal to that being used in the field.

(3) Cover the container, and allow the sample to cure for a period of time conforming to the requirements in the testing procedures.

e. Proceed with testing as described in the appropriate section.

4. Preparation of Field Samples.

   a. Obtain 3000 g field samples according to the instructions in PART 1.

   b. Keep the material in covered containers until ready to prepare the test specimens.

   c. Separate the material on the 1½ in. and ⅜ in. sieves. Sieving is to be done while the material is in a moist condition. It is not intended that all of the fines coating the coarse aggregate be removed. Separating the material into coarse and fine fractions in this manner makes it possible to obtain representative test samples without drying and splitting.

TABLE 2
Batch Masses for Calibration-Curve Specimens
(Alternate 2)

% Cement/ Lime	Mass g	
	Dry Aggregate	Cement/ Lime
1.0	297	3.0
1.5	296	4.4
2.0	294	5.9
2.5	293	7.3
3.0	291	8.7
3.5	290	10.2
4.0	288	11.5
4.5	287	12.9
5.0	286	14.3
5.5	284	15.6
6.0	283	17.0
6.5	282	18.3
7.0	280	19.6
7.5	279	20.9
8.0	278	22.2
8.5	276	23.5
9.0	275	24.8
9.5	274	26.0
10.0	273	27.3

   d. Determine the amount of material retained on the 1½ in. and ⅜ in. sieves and the amount passing the ⅜ in. sieve. Calculate the percentage of each fraction based on the total mass of the sample.

   e. Discard the material retained on the 1½ in. sieve.

   f. Reconstitute a 300 g test sample using the original proportions of retained and passing ⅜ in. sieve material. 1½ in. by ⅜ in. material shall be used in place of the discarded plus 1½ in. material.

   g. Place the test sample in a polyethylene container, cover it, and allow it to stand for the period of time conforming to the standard curing time for the project.

   h. If the calibration-curve samples are prepared according to the instructions of Alternate 2, it will be necessary to prepare a second specimen from each field sample for moisture content determination.

(1) Place the second sample in a container suitable for drying; then determine the moisture content in accordance with California Test 226.

(2) It is permissible to use an accelerated drying method provided the resulting moisture content is within ± 1 % of that determined in accordance with California Test 226.

PART 3. DETERMINATION OF APPROPRIATE TITRATION METHOD

3A. SCOPE

The hydrochloric acid, which is used to digest the cement in the acid-base method, also will attack certain minerals present in some aggregates. This part of the test procedure is used to detect the presence of these minerals and thus indicate when the acid-base method should not be used.

3B. APPARATUS

See PART 4.

3C. MATERIALS

See PART 4.

3D. PROCEDURES

1. Prepare two 300 g aggregate samples and two 300 g Ottawa sand samples following the instructions in Section 2C. of this method.

2. Perform the acid-base test on each test sample following the instructions in Part 4 of this test method.

3. Determine the difference between the amounts of sodium hydroxide required to achieve the end point for each aggregate sample and the average required for the Ottawa sand samples.

4. If the difference is less than 5 mL, the acid-base method should be used.

5. If the difference is 6 mL or more for either aggregate sample, the aggregate is reactive to acid and the constant-neutralization method must be used.

PART 4. ACID-BASE METHOD FOR DETERMINING CEMENT CONTENT

4A. SCOPE

This part describes a procedure for cement content determination based upon the neutralization of the cement with an excess of hydrochloric acid. The residual excess acid, as measured by back titration with an alkali, is inversely proportional to the cement content of the treated base sample. This test procedure cannot be used if the aggregate is reactive to acid as determined by PART 3 of this test method.

4B. APPARATUS

1. 100 mL titrating burette.

2. 100 mL pipet with filler.

3. Volumetric flask or graduated cylinder suitable for accurately measuring 200 mL.

4. Volumetric flask or graduated cylinder suitable for accurately measuring 400 mL.

5. 250 mL Erlenmeyer flask – one for each test sample.

6. Plastic beakers with a capacity of approximately 500 mL – one for each test sample.

7. Medicine dropper.

8. Two quart capacity, wide-mouth polyethylene containers with covers – one for each test sample.

9. Stainless steel stirring rods – one for each test sample.

10. Two 5 gal plastic carboys. A third carboy may be required if tap water is not available.

4C. MATERIALS

1. Hydrochloric acid (approximately 3 N).

2. Sodium hydroxide (approximately 1 N).

3. Phenolphthalein indicator solution, 1 %.

4D. PROCEDURE

1. Prepare test specimens according to the instructions in PART 2, "Preparation of Test Samples". Up to eight samples can be tested simultaneously by this procedure; therefore, a calibration set and four field samples can be tested at one time if desired.

2. Allow the prepared test samples to stand in the covered containers to complete the curing time established as a standard for the project.

3. Accurately measure 200 mL of 3 N HCL for each test sample. Pour the acid into the Erlenmeyer flasks and set one flask behind each sample.

4. Accurately measure 400 mL of water for each test sample. Pour the water into beakers, and set one beaker behind each sample.

5. When the standard curing time has elapsed, start the timer, and pour the pre-measured acid into the test samples.

    a. Add the acid to one sample at a time at 45 s intervals.

    b. Immediately after adding acid, use the stainless steel rod to stir the sample for the remainder of the 45 s period. Use a combination of circular and zigzag motions with the stirring rod across the bottom of the container to break up all agglomerations of material and expose all portions of the cement to the acid.

6. At 6 min after adding the acid, stir each sample in sequence for 45 s.

7. At 12 min after adding the acid, stir each sample in sequence for 45 s.

8. At 18 min after adding the acid, pour the pre-measured water into the test samples.

    a. Add the water to one sample at a time at 45 s intervals.

    b. Immediately after adding water, stir the sample for the remainder of the 45 s period.

9. Rinse out the Erlenmeyer flasks.

10. Allow the samples to stand undisturbed until a total of 30 min has elapsed from the time the acid was added; then use the pipet to draw off a 100 mL portion of the residual acid solution from each specimen, and deposit it in clean Erlenmeyer flasks.

11. Add two full droppers (approximately 40 drops) of phenolphthalein solution to the solution in each Erlenmeyer flask.

12. Titrate the solution in each flask by adding one normal sodium hydroxide from the burette until the solution becomes a deep red color, which does not fade when the flask is shaken for 1 min.

13. Read and record to the nearest 0.5 mL the amount of sodium hydroxide added to each flask.

14. Use the titration values to establish a calibration curve or to determine the cement content of field samples by the procedures in Sections 4E. or 4F. below.

4E. ESTABLISH THE CALIBRATION CURVE

1. Plot the amount of sodium hydroxide, determined in Section 4D. above, against the known cement content of each sample as illustrated in Figure 1 on Page 19.

2. Draw a straight line between the average amounts of sodium hydroxide added to duplicate test samples.

3. This is the calibration curve for determining the cement content of field samples.

4F. DETERMINING CEMENT CONTENT OF FIELD SAMPLES

1. Locate the amount of sodium hydroxide added to the test sample on the ordinate of the curve plotted for calibration samples.

2. Extend a horizontal line from this point to its intersection with the calibration curve.

3. From this point extend a vertical line until it intersects the abscissa of the curve plotted for calibration samples.

4. This intersection indicates the amount of cement in the sample.

5. If the calibration samples were prepared according to Alternate 1 of the preparation procedures in PART 2, the cement content indicated in paragraph 4F.4. above is correct and requires no adjustment.

6. If the calibration samples were prepared according to Alternate 2 of the preparation procedures in PART 2, the cement content indicated in paragraph 4F.4. above must be corrected. To determine the actual cement content, multiply the indicated cement content by the correction factor in Table 3, which corresponds to the moisture content of the field sample.

TABLE 3
Correction Factor for Moisture Content

Moisture Content (%)	Correction Factor	Moisture Content (%)	Correction Factor	Moisture Content (%)	Correction Factor
1	1.01	11	1.11	21	1.21
2	1.02	12	1.12	22	1.22
3	1.03	13	1.13	23	1.23
4	1.04	14	1.14	24	1.24
5	1.05	15	1.15	25	1.25
6	1.06	16	1.16	26	1.26
7	1.07	17	1.17	27	1.27
8	1.08	18	1.18	28	1.28
9	1.09	19	1.19	29	1.29
10	1.10	20	1.20	30	1.30

PART 5. CONSTANT-NEUTRALIZATION METHOD FOR DETERMINING CEMENT CONTENT

5A. SCOPE

This part is an alternate to the acid-base test procedure, PART 4, and is used for determining cement content when the aggregate is found to react with acid. The determinations are based upon the continuous neutralization of an aqueous solution of the cement treated aggregate sample for a specified time. This is accomplished by adding small amounts of acid to neutralize the OH ion as it is liberated during the hydration of the cement. The amount of acid used is directly proportional to the cement content of the treated-base sample.

5B. APPARATUS

1. 100 mL titrating burettes – one for each sample.

2. Volumetric flask or graduated cylinder suitable for accurately measuring 1000 mL.

3. Medicine dropper.

4. Two-quart capacity, wide-mouth polyethylene containers with covers – one for each test sample.

5. Stainless steel stirring rods – one for each test sample.

6. Five gal plastic carboy. A second carboy may be required if tap water is not available.

7. A timer.

8. Fluorescent light (optional).

5C. MATERIALS

1. Hydrochloric acid (approximately 3 N).

2. Phenolphthalein indicator solution, 1 %.

5D. PROCEDURE

1. Prepare test samples according to the instructions in PART 2, "Preparation of Test Samples."

2. Allow the prepared test samples to stand in the covered containers to complete the curing time established as a standard for the project.

3. Fill the titrating burettes with 3 N hydrochloric acid.

4. At the end of the curing period, add 1000 mL of tap water to each test sample.

   a. Add the water to one sample at a time at 2 min intervals.

   b. Immediately after adding the water, use the stainless steel stirring rod to stir the sample thoroughly.

5. Start the timer when the water is added.

6. Add two full droppers (approximately 40 drops) of phenolphthalein solution to each sample. The water will turn red due to the presence of cement.

7. Place one test sample in front of each titrating burette.

8. At 10 min after adding the water, begin stirring the first test sample and adding acid from the burette.

   a. Stir the sample using a combination of circular and zigzag motions with the stirring rod across the bottom of the container. The objective of stirring the test specimen is to break up all agglomerations of cement and aggregate, and to expose the cement to water so that the OH ion will be released.

   b. Add the acid carefully and slowly. The objective of adding the acid is to neutralize the OH ions as they are released into the solution. When all of the free OH ions in the solution have been neutralized, the red color will disappear. Any excess acid (beyond the amount required to eliminate the red color) will attack the cement, as well as any acid-reactive aggregates, and can affect the test results.

   c. It is imperative that the tester establishes a consistent technique for stirring the test samples and adding acid. Variations in the stirring effort, and particularly differences in the rate of introducing the acid, can affect the test results.

   d. The instructions above apply to each subsequent step that requires stirring or adding acid.

9. During the first minute of stirring and adding acid, stir the test sample continuously, and add acid only as necessary to cause the color to disappear.

10. During the second, fourth, sixth, and eighth minutes and for the remainder of the testing period thereafter, add acid only as necessary to eliminate any red color. The sample shall be stirred as acid is added and for a minimum of 5 s of each minute of the testing period.

11. During the third, fifth, and seventh minutes do not stir or add acid to the samples already in progress. This time will be devoted to the first minute stirring of subsequent samples in a series.

12. Continue stirring and adding acid to each sample for a period of 50 min. One hour will have elapsed from the time the testing water was added.

13. Read and record to the nearest 0.5 mL the amount of acid added to each test sample.

    a. Determine the amount of acid used by direct reading of the graduated burette.

    b. If individual burettes are not available for each test sample, the acid for all of the samples can be added through a single burette, and the amount added to individual samples can be determined by weighing the samples at the start and completion of the test. When using this method, care must be exercised to avoid non-uniform mass losses due to evaporation. Mass determinations can be simplified, with less chance of weighing error, if the total mass including sample, water, container, and stirring rod is used.

14. Use the titration values to establish a calibration curve or to determine the cement content of field samples by the procedures in Sections 5E. or 5F. below.

5E. ESTABLISHING THE CALIBRATION CURVE

1. Plot the amount of acid, determined in Section 5D above, against the known cement content of each calibration-curve sample as illustrated in Figure 2.

2. Draw a straight line between the average amounts of acid added to duplicate test samples.

3. This is the calibration curve for determining the cement content of field samples.

5F. DETERMINING CEMENT CONTENT OF FIELD SAMPLES

1. Locate the amount of acid added to the test sample on the ordinate of the plotted calibration curve.

2. Extend a horizontal line from this point to its intersection with the calibration curve.

3. From this point, extend a vertical line until it intersects the abscissa of the curve plotted for calibration samples.

4. This intersection indicates the amount of cement in the sample.

5. If the calibration samples were prepared according to Alternate 1 of the preparation procedures in PART 2, the cement content indicated in paragraph 5F.4. above is correct and requires no adjustment.

6. If the calibration samples were prepared according to Alternate 2 of the preparation procedures in PART 2, the cement content indicated in paragraph 5F.4. above must be corrected. To determine the actual cement content, multiply the indicated cement content by the correction factor in Table 3, which corresponds to the moisture content of the field sample.

PART 6. CONSTANT-NEUTRALIZATION METHOD FOR DETERMINING LIME CONTENT

6A. SCOPE

This part describes a procedure for determining the percentage of lime in soils or aggregates which have been treated with lime. The test is based upon the continuous neutralization of an aqueous solution of the lime treated sample for a specified time. This is accomplished by adding small amounts of acid to neutralize the OH ion as it is liberated during the hydration of the lime. The amount of acid used is proportional to the lime content of the treated sample.

6B. APPARATUS

1. 100 mL titrating burette – one for each sample.

2. Volumetric flask or graduated cylinder suitable for accurately measuring 1000 mL.

3. Volumetric flask or graduated cylinder suitable for accurately measuring 50 mL.

4. Medicine dropper.

5. Two quart capacity, wide mouth, polyethylene containers with covers – one for each test sample.

6. Stainless steel stirring rods – one for each test sample.

7. Five gal plastic carboy. A second carboy may be required if tap water is not available.

8. A timer.

6C. MATERIALS

1. Hydrochloric acid (approximately 3 N).

2. Phenolphthalein indicator solution, 1 %.

3. Separan NP-10 solution.

6D. PROCEDURE

1. Prepare test samples according to the instructions in PART 2, “Preparation of Test Samples.”

2. Allow the prepared test samples to stand in the covered containers to complete the curing time established as a standard for the project.

3. Fill the titrating burettes with 3 N hydrochloric acid.

4. At the end of the curing period add 1000 mL of tap water and 50 mL of Separan NP-10 to each test sample.

    a. Add the water and NP-10 to one sample at a time at 2 min intervals. The water and NP-10 may be pre-measured and stored in individual containers prior to starting the test if desired.

    b. Immediately after adding the water use the stainless steel stirring rod to stir the sample thoroughly.

5. Start the timer when the water is added.

6. Add two full droppers (approximately 40 drops) of phenolphthalein solution to each sample. The water will turn red due to the presence of lime.

7. Place one test sample in front of each titrating burette.

8. At 10 min after adding the water and NP-10, begin stirring the test sample and adding acid from the burette.

    a. Stir the sample using a combination of circular and zigzag motions with the stirring rod across the bottom of the container. The objective of stirring the test specimen is to break up all agglomerations of lime and aggregate or soil, and to expose the lime to water so that the OH ions will be released.

    b. Add the acid carefully and slowly. The objective of adding the acid is to neutralize the OH ions as they are released into solution. When all of the free OH ions in the solution are neutralized, the red color will disappear. Any excess acid (beyond the amount required to eliminate the red color) will attack the lime as well as any acid-reactive aggregates and can affect the test results.

    c. It is imperative that the tester establishes a consistent technique for stirring the test samples and adding acid. Variations in the stirring effort, and particularly differences in the rate of introducing the acid, can affect the test results.

    d. The instructions above apply to each subsequent step that requires stirring or adding acid.

9. During the first minute of stirring and adding acid, stir the test sample continuously, and add acid only as necessary to cause the color to disappear.

10. During the second, fourth, sixth and eighth minute, and for the remainder of the testing period thereafter, add acid only as necessary to eliminate any red color. The sample shall be stirred as acid is added and for a minimum of 5 s of each minute of the testing period.

11. During the third, fifth, and seventh minute, do not stir or add acid to the samples already in progress. This time will be devoted to the first minute stirring of subsequent samples in a series.

12. At 25 min after adding the initial water and NP-10, stir the sample vigorously for 30 s, and then add an additional 50 mL of NP-10.

13. Continue stirring and adding acid to each sample until a total of 50 min has elapsed after adding the initial water and NP-10.

14. Read and record, to the nearest 0.5 mL, the amount of acid added to each test sample.

    a. Determine the amount of acid used by direct reading of the graduated burette.

    b. If individual burettes are not available for each test sample, the acid for all of the samples can be added through a single burette, and the amount added to individual samples can be determined by weighing the samples at the start and completion of the test. The mass of the second addition of NP-10 must be determined also and a correction applied. When using this method, care must be exercised to avoid non-uniform mass losses due to evaporation. Mass determinations can be simplified, with less chance of weighing error, if the total mass, including sample, water, container, and stirring rod is used.

15. Use the titration values to establish a calibration curve or to determine the lime content of field samples by the procedures in Sections 6E. and 6F. below.

6E. ESTABLISHING THE CALIBRATION CURVE

1. Plot the amount of acid determined in Section 6D. above against the known lime content of each calibration-curve sample as illustrated in Figure 3.

2. Draw a straight line between the average amounts of acid added to duplicate test samples.

3. This is the calibration curve for determining the lime content of field samples that have been cured for the same period of time.

6F. DETERMINING LIME CONTENT OF FIELD SAMPLES

1. Locate the amount of acid added to the test sample on the ordinate of the plotted calibration curve.

2. Extend a horizontal line from this point to its intersection with the calibration curve.

3. From this point, extend a vertical line until it intersects the abscissa of the curve plotted for calibration samples.

4. This intersection indicates the amount of lime in the sample.

5. If the calibration samples were prepared according to Alternate 1 of the preparation procedures in PART 2, the lime content indicated in paragraph 6F.4. above is correct and requires no adjustment.

6. If the calibration samples were prepared according to Alternate 2 of the preparation procedures in PART 2, the lime content indicated in paragraph 6F.4. above must be corrected. To determine the actual lime content, multiply the indicated lime content by the correction factor in Table 3, which corresponds to the moisture content of the field sample.

G. PRECAUTIONS

Glass carboys are not to be substituted for the plastic carboys for mixing or storing the hydrochloric acid solution. Glass containers having a maximum capacity of 1 gal may be used as reservoirs with automatic burettes. All containers of hydrochloric acid or sodium hydroxide solutions shall be clearly labeled and stored in separate areas.

Store the hydrochloric acid in closed containers in a cool, dry, well ventilated area away from sources of ignition, strong oxidizers, strong bases, out of direct sunlight and away from incompatible chemicals. Storage areas should have acid resistant floors and approved drainage facilities.

Store sodium hydroxide pellets in a tightly sealed non-metallic container, at a temperature below 140°F, in a well-ventilated area away from water, acids, metals, flammable liquids and organic halogens. Storage area should be equipped with trapped floor drains, curbs or gutters.

Do not use compressed air to agitate the solutions in the carboys. Do not use pressure to transfer the solution between containers if failure of the system would result in uncontrolled flow. Exposed spigots, valves, or other discharge points should be installed below eye level and be pointed downward.

H. HEALTH AND SAFETY

It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Prior to handling, testing or disposing of any materials, testers must be knowledgeable about safe laboratory practices, hazards and exposure, chemical procurement and storage, and personal protective apparel and equipment.

Caltrans Laboratory Safety Manual is available at:

http://www.dot.ca.gov/hq/esc/ctms/pdf/lab_safety_manual.pdf

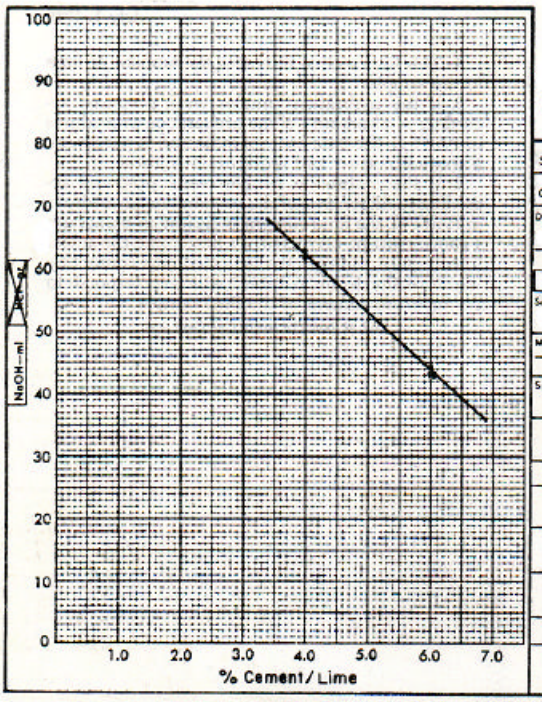
End of Text
(California Test 338 contains 22 pages)

Figure 1

Batch Weights for Calibration-Curve Samples (Alternate 1)

%Cement/Lime		1 %		2 %		3 %		4 %		5 %		6 %		7 %		8 %		9 %		10 %	
% moisture	mls H_2O	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm	Dry Wt. Agg. gm	Cem./Lime gm
5	14	283	2.8	280	5.6	278	8.3	275	11.0	272	13.6	270	16.2	267	18.7	265	21.2	262	23.6	260	26.0
6	17	280	2.8	277	5.5	275	8.2	272	10.9	270	13.5	267	16.0	264	18.5	262	21.0	260	23.4	257	25.7
7	20	277	2.8	275	5.5	272	8.2	269	10.8	267	13.4	265	15.9	262	18.3	260	20.8	257	23.2	255	25.5
8	22	275	2.8	272	5.5	270	8.1	267	10.7	265	13.2	262	15.7	260	18.2	257	20.6	255	22.9	253	25.3
9	25	272	2.7	270	5.4	267	8.0	264	10.6	262	13.1	259	15.6	257	18.0	255	20.4	253	22.7	250	25.0
10	27	270	2.7	268	5.4	265	8.0	262	10.5	260	13.0	258	15.5	255	17.9	253	20.2	250	22.5	248	24.8
11	30	267	2.7	265	5.3	262	7.9	260	10.4	257	12.9	255	15.3	252	17.7	250	20.0	248	22.3	246	24.6
12	32	265	2.7	263	5.3	260	7.8	258	10.3	255	12.8	253	15.2	250	17.5	248	19.8	246	22.1	244	24.4
13	34	262	2.6	260	5.2	257	7.7	255	10.2	252	12.6	250	15.0	248	17.3	246	19.7	244	21.9	241	24.1
14	37	260	2.6	258	5.2	255	7.7	253	10.1	250	12.5	248	14.9	246	17.2	244	19.5	241	21.7	239	23.9
15	39	258	2.6	256	5.1	253	7.6	251	10.0	249	12.4	246	14.8	244	17.1	242	19.3	239	21.5	237	23.7
16	41	256	2.6	254	5.1	251	7.5	249	10.0	247	12.3	244	14.6	242	16.9	239	19.2	237	21.4	235	23.5
17	44	253	2.5	251	5.0	249	7.5	246	9.8	244	12.2	242	14.5	239	16.7	237	19.0	235	21.2	233	23.3
18	46	252	2.5	249	5.0	247	7.4	244	9.8	242	12.1	240	14.4	238	16.6	235	18.8	233	21.0	231	23.1
19	48	250	2.5	247	4.9	245	7.3	242	9.7	240	12.0	238	14.3	236	16.5	233	18.7	231	20.8	229	22.9
20	50	248	2.5	245	4.9	243	7.3	240	9.6	238	11.9	236	14.2	234	16.4	231	18.5	229	20.6	227	22.7
21	52	246	2.5	243	4.9	241	7.2	238	9.5	236	11.8	234	14.0	232	16.2	230	18.4	227	20.5	225	22.5
22	54	244	2.4	241	4.8	239	7.2	236	9.4	234	11.7	232	13.9	230	16.1	228	18.2	226	20.3	224	22.4
23	56	242	2.4	239	4.8	237	7.1	235	9.4	232	11.6	230	13.8	228	16.0	226	18.1	224	20.1	222	22.2
24	58	240	2.4	237	4.7	235	7.1	233	9.3	230	11.5	228	13.7	226	15.8	224	17.9	222	20.0	220	22.0
25	60	238	2.4	235	4.7	234	7.0	231	9.2	229	11.4	226	13.6	224	15.7	222	17.8	220	19.8	218	21.8
26	62	236	2.4	233	4.7	231	6.9	229	9.2	227	11.4	225	13.5	222	15.6	220	17.6	218	19.7	216	21.6
27	64	234	2.3	231	4.6	229	6.9	229	9.1	225	11.3	223	13.4	221	15.5	219	17.5	217	19.5	215	21.5
28	66	232	2.3	229	4.6	227	6.8	225	9.0	223	11.2	221	13.3	219	15.3	217	17.4	215	19.4	213	21.3
29	68	231	2.3	228	4.6	226	6.8	224	9.0	222	11.1	220	13.2	218	15.3	215	17.2	213	19.2	211	21.1
30	69	229	2.3	226	4.5	224	6.7	222	8.9	220	11.0	218	13.1	216	15.1	214	17.1	212	19.1	210	21.0
31	71	227	2.3	225	4.5	222	6.7	220	8.8	218	10.9	216	13.0	214	15.0	212	17.0	210	18.9	208	20.8
32	73	225	2.3	223	4.5	220	6.6	218	8.7	216	10.8	214	12.8	212	14.8	210	16.8	209	18.8	207	20.7
33	75	224	2.2	222	4.4	219	6.6	217	8.7	215	10.8	213	12.8	211	14.8	209	16.7	207	18.6	205	20.5
34	76	222	2.2	220	4.4	217	6.5	215	8.6	213	10.7	211	12.7	209	14.6	207	16.6	205	18.5	204	20.4
35	78	220	2.2	218	4.4	216	6.5	213	8.5	211	10.6	209	12.5	207	14.5	206	16.5	204	18.3	202	20.2

Figure 2



STATE OF CALIFORNIA
DEPARTMENT OF TRANSPORTATION
TRANSPORTATION LABORATORY
Field-Laboratory Record of
TITRATION TESTS
For Cement or Lime Content Determination

Sheet of Sheets			Date
Operator			
Dist.	Co.	Rte.	P. M.
SOURCE	CHARGE	EXPENDITURE AUTHORIZATION	SPECIAL DESIGNATION (USE WHEN APPLICABLE) / OBJECT
Source of Aggregate		Cement/Lime Brand	
Mixer		Road Mix ☐	Batch ☐ Cont. ☐
Spreader			
PLANNED	Thickness	Cement/Lime 5.0 %	Water 7.0 %

DETERMINATION OF METHOD OF TEST

Item	Specimen Type	Jar No.	NaOH ml	Control Values
A	Ottawa Sand	1	102.6	Av. Mls. 102.7
		2	102.8	
B	Aggregate Blank	3	100.0	Lowest Value
		4	100.5	100.0
C	Av. of A—Lowest Value of B			2.7

If C<6 mls use Acid-Base Proc. (Part I) . . . . ☒
If C≧6 mls use Const. Neut. Proc. (Part II) . . . . ☐

CALIBRATION CURVE TEST SAMPLES

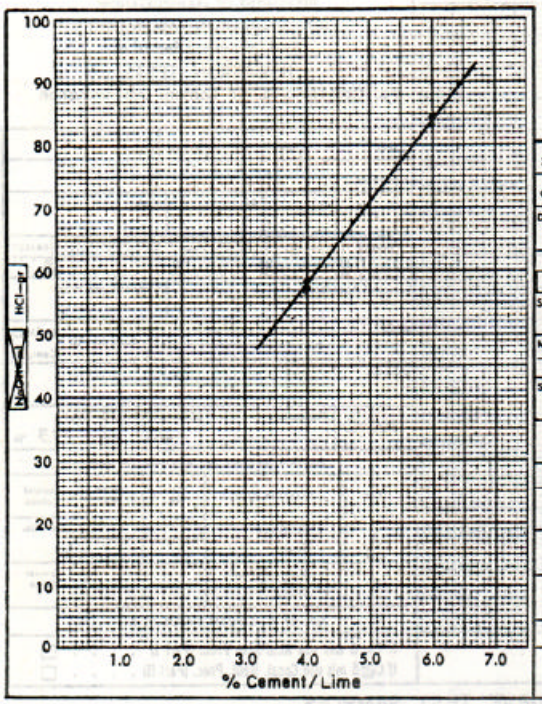
SAMPLE I.D.	Test Sample Proportions: Retained 9.5 mm	Pass 9.5 mm	Cement Lime	Water	Total Weight ☒ Wet ☐ Dry	Gross Jar Wt Grams: Start	Final	NaOH ml / HCl gr	% Cement Lime	Remarks
1	67	202	10.8	20	300	—	—	62.0	4	
2	67	202	10.8	20	300	—	—	63.0	4	
3	66	198	15.9	20	300	—	—	43.5	6	
4	66	198	15.9	20	300	—	—	44.0	6	

FIELD TEST SAMPLES

SAMPLE I.D. / Location	Field Sample: Total Weight	Pass 9.5 mm Wt	Pass 19 mm (%)	Test Sample: Total Weight	Pass 9.5 mm Wt	Gross Jar Wt Grams: Start	Final	NaOH ml / HCl gr	Indicated Cement Lime %	Moisture Content %	Correction Factor	Corrected Cement Lime %	
	3080	2395	78	300	234	—	—	66.5	3.6	—	—	3.6	
	3155	2445	77	300	231	—	—	55.0	4.8	—	—	4.8	
	3265	2440	75	300	225	—	—	50.0	5.3	—	—	5.3	
	3395	2580	76	300	228	—	—	55.5	4.7	—	—	4.7	

TL-3040 (Rev. 2/80)

Figure 3



STATE OF CALIFORNIA
DEPARTMENT OF TRANSPORTATION
TRANSPORTATION LABORATORY
Field-Laboratory Record of
TITRATION TESTS
For Cement or Lime Content Determination

Sheet	of	Sheets	Date
Operator			
Dist.	Co.	Rte.	P. M.
SOURCE	CHARGE	EXPENDITURE AUTHORIZATION	SPECIAL DESIGNATION (USE WHEN APPLICABLE) / OBJECT
Source of Aggregate		Cement/Lime Brand	
Mixer		Road Mix ☐	Batch ☐ Cont. ☐
Spreader			
PLANNED	Thickness	Cement/Lime 5·0 %	Water 7·0 %

DETERMINATION OF METHOD OF TEST

Item	Specimen Type	Jar No.	NaOH ml	Control Values
A	Ottawa Sand	1	102·6	Av. Mls. 102·7
		2	102·8	
B	Aggregate Blank	3	78·3	Lowest Value 75·7
		4	75·7	
C	Av. of A—Lowest Value of B			27·0

If C<6 mls use Acid-Base Proc. (Part I) . . . . ☐
If C≥6 mls use Const. Neut. Proc. (Part II) . . . . ☒

CALIBRATION CURVE TEST SAMPLES

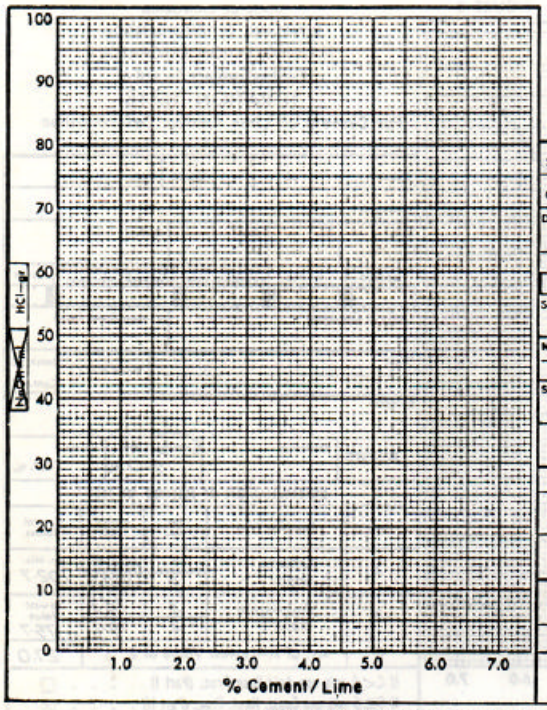
SAMPLE I.D.	Test Sample Proportions: Retained 9.5 mm	Pass 9.5 mm	Cement Lime	Water	Total Weight ☐ Wet ☒ Dry	Gross Jar Wt Grams: Start	Final	NaOH ml (HCl gr)	% Cement Lime	Remarks
1	72	216	11·5	21	300	—	—	56·5	4	
2	72	216	11·5	21	300	—	—	58·0	4	
3	71	212	17·0	21	300	—	—	83·0	6	
4	71	212	17·0	21	300	—	—	84·0	6	

FIELD TEST SAMPLES

SAMPLE I.D. / Location	Field Sample: Total Weight	Pass 9.5 mm Wt	Pass 19 mm (%)	Test Sample: Total Weight	Pass 9.5 mm Wt	Gross Jar Wt Grams: Start	Final	NaOH ml (HCl gr)	Indicated Cement Lime %	Moisture Content %	Correction Factor	Corrected Cement Lime %	
	3080	2395	78	300	234	—	—	48·0	3·3	8	1·08	3·6	
	3155	2445	77	300	231	—	—	63·5	4·5	7	1·07	4·8	
	3265	2440	75	300	225	—	—	70·5	5·0	6	1·06	5·3	
	3395	2580	76	300	228	—	—	62·5	4·4	7	1·07	4·7	

TL-3040 (Rev. 2/80)

Figure 4



STATE OF CALIFORNIA
DEPARTMENT OF TRANSPORTATION
TRANSPORTATION LABORATORY
Field-Laboratory Record of
TITRATION TESTS
For Cement or Lime Content Determination

Sheet of Sheets			Date
Operator			
Dist.	Co.	Rte.	P. M.
SOURCE	CHARGE	EXPENDITURE AUTHORIZATION	SPECIAL DESIGNATION (USE WHEN APPLICABLE) / OBJECT
Source of Aggregate		Cement/Lime Brand	
Mixer		Road Mix ☐	Batch ☐ Cont. ☐
Spreader			
PLANNED	Thickness	Cement/Lime 4.0 %	Water 13 %

DETERMINATION OF METHOD OF TEST

Item	Specimen Type	Jar No.	NaOH ml	Control Values
A	Ottawa Sand			Av. Mls.
B	Aggregate Blank			Lowest Value
C	Av. of A—Lowest Value of B			

If C<6 mls use Acid-Base Proc. (Part I) . . . . ☐
If C≧6 mls use Const. Neut. Proc. (Part II) . . . . ☐

CALIBRATION CURVE TEST SAMPLES

SAMPLE I.D.	Test Sample Proportions: Retained 9.5 mm	Pass 9.5 mm	Cement/Lime	Water	Total Weight ☐ Wet ☒ Dry	Gross Jar Wt Grams: Start	Final	NaOH ml (HCl gr)	% Cement/Lime	Remarks
1	0	286	14.3	39	300	—	—	77.0	5	
2	0	286	14.3	39	300	—	—	78.4	5	
3	0	291	8.7	39	300	—	—	41.8	3	
4	0	291	8.7	39	300	—	—	40.9	3	

FIELD TEST SAMPLES

SAMPLE I.D. / Location	Field Sample: Total Weight	Pass 9.5 mm Wt	Pass +19 mm (%)	Test Sample: Total Weight	Pass 9.5 mm Wt	Gross Jar Wt Grams: Start	Final	NaOH ml (HCl gr)	Indicated Cement/Lime %	Moisture Content %	Correction Factor	Corrected Cement/Lime %	
	3245	3245	100	300	300	—	—	63.0	4.2	15	1.15	4.8	
	3190	3190	100	300	300	—	—	61.5	4.1	11	1.11	4.6	
	3310	3310	100	300	300	—	—	61.2	4.1	9	1.09	4.5	
	3080	3080	100	300	300	—	—	59.5	4.0	12	1.12	4.5	

TL-3040 (Rev. 2/80)