Designation: D4337 – 89 (Reapproved 2017)

Standard Test Methods for Analysis of Linear Detergent Alkylates[1]

This standard is issued under the fixed designation D4337; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover chemical and physical tests applicable to linear detergent alkylates, evaluating those properties which characterize linear detergent alkylates with respect to its suitability for desired uses.

NOTE 1—Linear detergent alkylates comprises linear alkylbenzenes prepared by varying processes of varying linear alkyl chain length. The alkylate is sulfonated for surfactant use, the largest application being in detergent products. Careful control of linear detergent alkylate characteristics is desired; during sulfonation, variations of the sulfonate can occur that may result in either desirable or undesirable end-use properties.

1.2 The test methods appear as follows:

	Section
Composition by Gas Chromatography	7 – 15
Chlorides	16 – 27
Color	28 – 30
Refractive Index	31 – 33
Specific Gravity	34 – 36
Water	37 – 39

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage. See also Note 1 *and Section 21*.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D270 Methods of Sampling Petroleum and Petroleum Products (Withdrawn 1984)[3]

D1122 Test Method for Density or Relative Density of Engine Coolant Concentrates and Engine Coolants By The Hydrometer

D1193 Specification for Reagent Water

D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

D1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids

D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

3. Significance and Use

3.1 These test methods are suitable for setting specifications on linear detergent alkylates and for quality control where these alkylates are manufactured or are used in a manufacturing process.

3.2 The gas chromatography test is useful in identifying linear detergent alkylates produced by the various manufacturing processes and for determining the applicability of a linear detergent alkylate to a particular end use. Test conditions have been optimized for resolution of the C-9 to C-15 linear detergent alkylates and the presence of higher or lower chain lengths will be readily apparent but may produce erroneous results. The tetralin structures have not been sufficiently identified to allow determination of tetralins, and small unidentified components are reported as tetralins and unidentifieds.

3.3 Some linear detergent alkylates use a manufacturing process with an organic chloride as a precursor. This test may be used to ensure that the chloride content of the alkylate is not excessive for a particular end use.

3.4 The platinum-cobalt color test is useful in determining that the color of the linear detergent alkylate will not contribute to the color of the end use product.

3.5 The refractive index and specific gravity tests are possible aids in the identification of linear detergent alkylates and in evaluating alkylates for gross contaminants.

3.6 The water test is suitable for determining that linear detergent alkylates do not contain amounts of water deleterious to further processing.

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[1] These test methods are under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and are the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

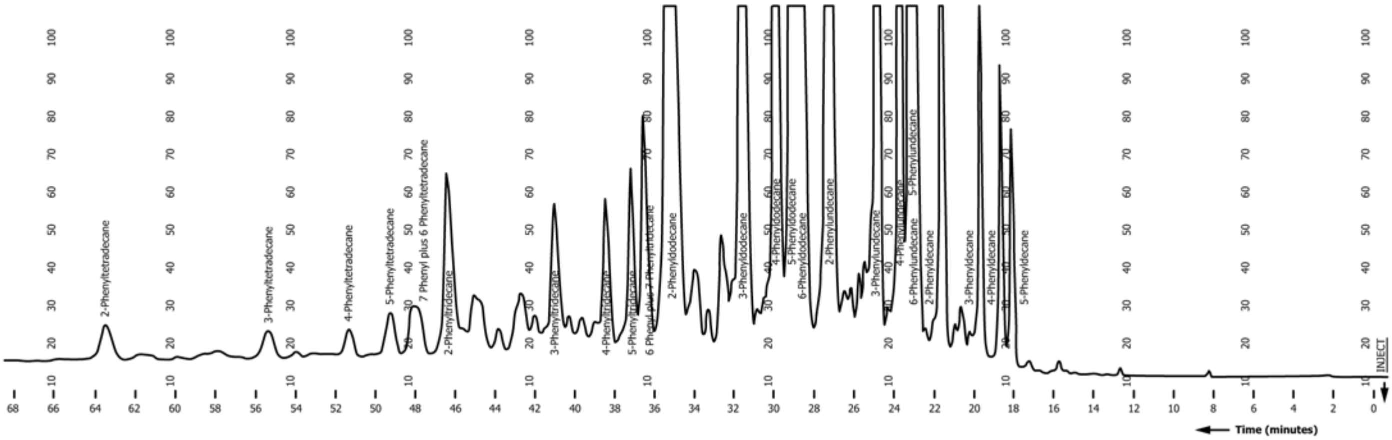

FIG. 1 Typical Gas Chromatic Scan of an Alkylate

4. Purity of Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[4] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Unless otherwise indicated, references to water shall be understood to mean Type III water conforming to Specification D1193.

5. Precautions

5.1 Consult the latest OSHA regulations regarding all reagents and materials used in these test methods.[5]

6. Sampling

6.1 Unless otherwise indicated in a specific test method, sample the material in accordance with Method D270, with special application of Sections 41 and 42 pertaining to industrial aromatic hydrocarbons, Section 12 pertaining to Bottle or Beaker Sampling, and Section 15 pertaining to Tap Sampling. Samples must be taken and stored in amber, screw-cap, glass bottles to protect them from light.

COMPOSITION BY GAS CHROMATOGRAPHY

7. Scope

7.1 The gas chromatography test was developed to allow determination of C-9 through C-15 linear detergent alkylate isomer distribution before sulfonation. The analyses of stocks having a chain length distribution above or below the interval covered in this test method will require a different set of operating conditions. If water, extremely high boiling components, or unusual impurities are present in the linear detergent alkylate, this test would not necessarily detect them and may produce erroneous results.

8. Summary of Test Method

8.1 A sample is injected by means of a microsyringe into a vaporization chamber of a gas chromatograph. A stream splitter allows the majority of the sample to be vented into the atmosphere through a valve that is incorporated in the vaporization chamber. A small portion of the sample is then swept by the carrier gas into a capillary column that has been coated with a thin layer of the stationary phase. A hydrogen flame ionization detector provides a very sensitive means of detecting the extremely small concentrations of separated vapor components as they elute from the column. An electrometer-amplifier converts the detector signal into a suitable signal that can be recorded on a standard strip-chart recorder and integrated by a suitable integration device. The concentrations of the components are obtained by normalizing the integrated areas, based on the assumption that all components of the mixture are eluted under the conditions used.

9. Apparatus

9.1 *Gas Chromatograph,* with the following characteristics:

9.1.1 *Sample Injection Port,* operable at 250°C and containing a stream splitter capable of linearly splitting sample injections up to 1000 to 1.

9.1.2 *Column Oven,* capable of temperature programming from 120 to 165°C at a rate of 1 or 2°C per min.

9.1.3 *Flame Ionization Detector,* capable of operating at 300°C.

9.2 *Recorder,* 0 to 1 mv range with 0.1 second full scale deflection.

9.3 *Integration Device,* capable of accurately measuring areas of up to 250 components.

9.4 *Column,* 150 ft (45.7 m) by 0.01 in. (0.25 mm) inside diameter stainless steel wall; open tubular capillary column

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] Available from Occupational Safety and Health Review Commission, 1825 K Street, Washington, DC 20006.

coated with phenyl methyl silicone DC-550[6] or other column that will perform required separation as indicated in Fig. 1.

9.5 *Syringe,* 10-μL capacity with fixed 2-in. (5.1 cm) needle.[7]

10. Reagents

10.1 *Carrier Gas,* high-purity nitrogen.

10.2 *Hydrogen,* 99.996 mol % pure.

10.3 *Compressed Air.*

11. Calibration

11.1 No calibration or correction factors are necessary as the response of the flame ionization detector is essentially constant for aromatic hydrocarbons. The individual peak area percentages may be regarded as the actual weight percent in the sample.

12. Procedure

12.1 Adjust the chromatograph to the following conditions:

Column temperature, °C (Note 2)	120 programmed to 160 at 1 to 2°C per min
Injection port temperature, °C	250
Detector temperature, °C	300
Carrier gas pressure, lbf	60
Sample size (Note 3), μL	0.5 to 1
Chart speed, cm/min	1
Hydrogen flow (Note 4), mL/min	30
Air flow (Note 4), mL/min	240
Range	X1
Attenuator setting	X4
Splitter (Note 3)	1000/1

NOTE 2—These conditions should give adequate separation. It may be necessary to adjust conditions slightly to meet the separation criteria of the sample chromatogram (Fig. 1), depending upon the performance of the capillary column.

NOTE 3—Sample size and splitter ratio should be adjusted to obtain a peak height of 10 to 15 mV for the largest components.

NOTE 4—Hydrogen and air flows should be adjusted to give maximum detector sensitivity. See instructions in instrument instruction manual.

12.2 When a new column is installed, leak-test the plumbing connections by applying the operating carrier gas pressure. The air and hydrogen connections to the detector should be leak tested periodically. In particular, the hydrogen supply should be leak-tested at the cylinder each time the cylinder is changed, due to the hazardous nature of hydrogen gas.

12.3 Wet and rinse the syringe with the sample and draw an excess of liquid into the syringe barrel. Exercise caution that all air bubbles are excluded from the liquid in the syringe. Carefully depress the plunger until the exact amount of liquid desired is contained in the syringe.

12.4 Open the split valve and quickly pierce the septum, thrusting the needle of the syringe completely into the inlet. In the same motion quickly and completely depress the plunger. Immediately withdraw the syringe and start the column oven temperature program sequence. The split valve may be closed 1 min after the injection of the sample, but it must always be closed at exactly the same time after injection.

12.5 Compare the sample chromatogram with the standard chromatogram (Fig. 1) and from the retention times of the components identify all straight chain alkyl benzene isomers. Label all peaks whose identity has been established. Many unidentified minor peaks will appear as minor constituents of the sample and may be attributed to tetralin structures. Include these peaks in the calculations and report in a pooled percentage as tetralins and unknowns.

13. Calculations

13.1 Calculate the percentage for each component by dividing the area of each component by the total area of all components and multiplying by 100.

13.2 Calculate the total 2-phenyl isomer by summing together the 2-phenyl isomers for all chain lengths.

13.3 Calculate the chain length distribution by summing together all percentages for the identified linear alkyl benzene isomers of each chain length.

13.4 Calculate lights (components eluting before C_{10} alkyl benzene) by summing together the percentages of all the components eluting before 5-phenyldecane.

13.5 Calculate heavies (components eluting after C_{14} alkyl benzene) by summing together all the percentages of the components eluting after 2-phenyltetradecane.

13.6 Calculate tetralins and unknowns by summing together the percentages of all unidentified peaks eluting between the 5-phenyldecane peak and the 2-phenyltetradecane peak.

13.7 Determine the average molecular weight by assuming the tetralins and unknowns to have a molecular weight of 244. The average molecular weight is calculated to the third decimal place using the following equation:

$$\begin{aligned}\text{Average molecular weight} = \big[100 - \big(\text{lights} \\ + \text{heavies}\big)/\big[\big(\textstyle\sum C_{10}\ \text{isomers}/218\big) \\ + \big(\textstyle\sum C_{11}\ \text{isomers}/232\big) + \big(\textstyle\sum C_{12}\ \text{isomers}/246\big) \\ + \big(\textstyle\sum C_{13}\ \text{isomers}/260\big) + \big(\textstyle\sum C_{14}\ \text{isomers}/274\big) \\ + \ \big(\textstyle\sum \text{tetralins} + \text{unknowns}/244\big)\big]\end{aligned} \tag{1}$$

14. Report

14.1 Report the components and average molecular weight as specified in Table 1. Duplicate runs that agree within the checking limits shown in Table 1 are acceptable for averaging (95 % confidence level).

15. Precision and Bias

15.1 The following criteria should be used for judging the acceptability of results:

15.1.1 *Repeatability (Single Analyst)*— The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the value shown in Table 1 with the indicated degrees of freedom.

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[6] Capillary column, Part 009-0354, available from Perkin-Elmer Corporation, Norwalk, CT, has been found satisfactory.

[7] Microsyringe No. 701, available from Hamilton Co., P. O. Box 307, Whittier, CA, has been found satisfactory.

TABLE 1 Composition by Gas Chromatography Precision Values

Component	Level, Area %	Report To, Area %	Checking Limits	Repeatability			Reproducibility		
				Standard Deviation	Degrees of Freedom	95 % Confidence Interval	Standard Deviation	Degrees of Freedom	95 % Confidence Interval
Lights	0–1	0.01	0.14	0.053	46	0.15	0.099	7	0.33
Total C-10 Isomers	0–1	0.01	0.08	0.021	13	0.06	0.138	6	0.44
	5–30	0.1	0.8	0.27	26	0.8	0.37	6	1.3
Total C-11 Isomers	0–3	0.1	0.3	0.07	13	0.2	0.26	6	0.9
	20–40	0.1	1.9	0.65	27	1.9	1.05	6	3.6
Total C-12 Isomers	10–45	0.1	1.3	0.34	40	1.0	0.84	6	2.9
Total C-13 Isomers	0–15	0.1	1.1	0.26	26	0.8	0.72	6	2.5
	40–60	0.1	2.3	0.40	14	1.2	1.60	6	5.5
Total C-14 Isomers	0–2	0.1	0.2	0.06	19	0.2	0.38	5	1.4
	4–7	0.1	0.5	0.20	6	0.7	0.50	5	1.8
	20–35	0.1	1.1	0.21	12	0.6	0.76	5	2.7
Eluting After C-14	0–3	0.01	0.44	0.16	34	0.46	0.52	5	1.88
Total 2-Phenyl Isomer	10–30	0.1	1.2	0.35	47	1.0	0.54	7	1.8
Average Molecular Weight	230–265[A]	0.1[A]	0.9[A]	0.25[A]	38	0.7[A]	0.45[A]	5	1.6[A]

[A] Units are molecular weight units, not area percent.

Two such results should be considered suspect (95 % confidence level) if they differ by more than the values in Table 1.

15.1.2 *Reproducibility (Multilaboratory)*— The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be the value shown in Table 1 with the indicated degrees of freedom. Two such results should be considered suspect (95 % confidence level) if they differ by more than the values shown in Table 1 (Note 5).

NOTE 5—The preceding precision estimates are based on an interlaboratory study of six samples of linear detergent alkylates. A total of eight laboratories cooperated in the studies in which duplicate determinations were performed on each of 2 days. Practice E180 was used in developing these precision estimates.

ORGANIC CHLORIDES

16. Scope

16.1 This test method is suitable for the rapid determination of organic chlorides in linear detergent alkylates before sulfonation. The test method is applicable to samples which include chloride-containing compounds that are volatile at 700°C. Compounds such as sodium chloride do not completely volatilize under the operating conditions specified and are not quantified. By using the proper sample size, this test method is applicable to the direct detection of chlorides at levels up to 700 ppm. Halogen compounds other than those containing chlorides tend to be nonquantitatively detected by this method.

17. Summary of Test Method

17.1 Liquid samples are injected into a flowing stream of gas containing about 80 % oxygen and 20 % inert gas such as nitrogen, helium or argon. The gas and sample flow through a combustion tube maintained at about 800°C. The chlorine is converted to chloride and oxychlorides which then flow into a titration cell where they react with silver ions in the titration cell. The silver ions thus consumed are coulometrically replaced. The total current required to replace the silver ions is a measure of the chlorine present in the injected samples.

18. Interferences

18.1 Sulfur in excess of 1 000 times the chlorine content.

18.2 Nitrogen in excess of 6.5×10^4 times the chlorine content.

19. Apparatus[8] (see Fig. 2)

19.1 *Electric Furnace,* having at least two separate and independently controlled heat zones. The first or inlet zone is held at a temperature sufficient to volatilize all of the sample. The second zone (and third zone, depending on system configuration) is held at a temperature sufficient to pyrolyze and oxidize the sample. For routine operation with syringe inlet sampling, the temperatures should be:

Inlet zone	700°C
Oxidation Zone(s)	800°C

19.2 *Combustion Tube,* fabricated from quartz. The inlet end of the tube holds a septum for syringe entry of the sample and has a side arm for introduction of O_2 and inert gas. The construction is such that the inert gas sweeps the inlet zone transporting all of the volatilized sample into the oxidation zone. The oxidation section should be large enough (approximately 300 mm long and 24 mm in diameter) to ensure complete oxidation of the sample. The outlet end of the pyrolysis tube should be constructed to hold a demountable quartz insert tube, which is packed with quartz wool. A conventional pyrolysis tube is depicted in Fig. 2. Other configurations are acceptable if precision is not degraded.

19.3 *Quartz Insert Tube (Fig. 2),* packed with quartz wool and inserted into the exit end of the pyrolysis tube.

19.4 *Coulometric Titration Cell.* In operation these cells are light sensitive. All enclosures must be kept closed during measurements.

19.5 *Microcoulometer.*

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[8] The apparatus (19.1 – 19.8) manufactured by Dohrmann, Santa Clara, CA has been found to meet all requirements.

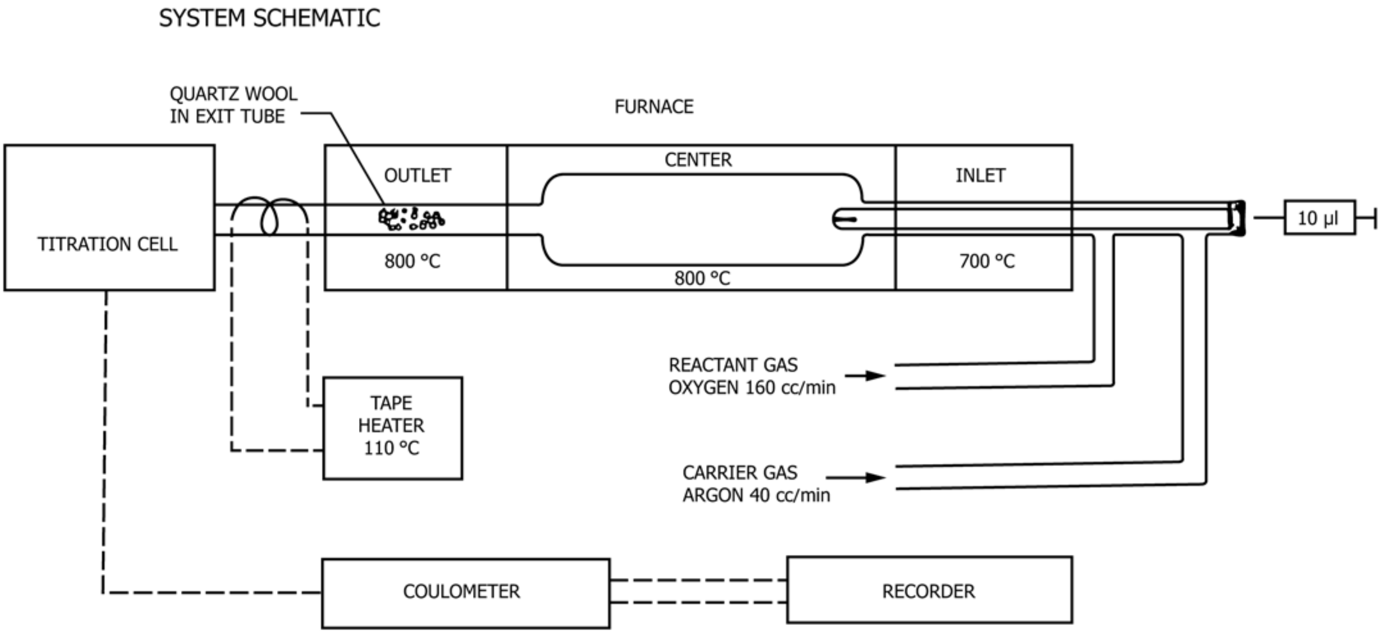

FIG. 2 Oxidative Chlorine System

19.6 *Integrator.*

19.7 *Recorder.*

19.8 *Microlitre Syringes* of 10, 25 or 50-µL capacity, capable of accurately delivering microlitre quantities, are required. The needle should be long enough to reach the hottest portion of the inlet furnace when injecting the sample. At or below 2 ppm chlorine, sample injection volumes of 30–40 µ L are suggested.

19.8.1 An automatic dispensing adapter which allows a slow, controlled sample injection rate is also appropriate. This makes an ideal injection rate of 0.2 to 0.5 µL/s easier.

20. Reagents

20.1 *Inert gas,* argon or helium only, ultra-high purity grade (UHP).

20.2 *Oxygen Ultra High Purity* (UHP).

20.3 *Cell Electrolyte,* as specified by the microcoulometer manufacturer. Typically, 70 % V/V acetic acid in water.

20.4 *Quartz Wool.*

20.5 *Calibration Standard,* chlorobenzene in high purity benzene or toluene.

21. Safety Precautions

21.1 High temperatures and flammable, potentially toxic solvents are used in this test method. Use proper care to minimize personnel exposure to such chemicals and to minimize accidental contact of the solvents with high temperature employed in the pyrolysis furnace.

22. Assembly of Apparatus

22.1 Assemble apparatus in accordance with the manufacturer's instructions.

23. Calibration and Standardization

23.1 Prepare a series of calibration standards using a stock solution covering the range of operation and consisting of chlorobenzene and a matrix similar to samples to be analyzed.

23.2 Adjust the gas flows and the pyrolysis temperature to the desired operating conditions.

23.3 Analyze the calibration standards as described in Section 24.

24. Procedure

24.1 Sample sizes ranging from 3 to 30 µL are acceptable. The size of the injected sample should conform to the size of the injected standard.

24.2 Flush the microlitre syringe several times with the standard or sample to be analyzed.

24.3 Obtain volumetric measurement by filling the syringe to the 80 % level, retracting the plunger so that the lower liquid meniscus falls on the 10 % scale mark, and recording the volume of liquid in the syringe. After the sample has been injected, again retract the plunger so that the lower liquid meniscus falls on the 10 % scale mark and record the volume of liquid in the syringe. The difference between the two volume readings is the volume of sample injected.

24.4 Alternatively, the sample injection device may be weighed before and after injection to determine the amount of sample injected. This test method provides greater precision than the volume delivery method, provided a balance with a precision of ± 0.01 mg is used.

24.5 Inject the sample into the pyrolysis tube at nominal rate of 0.5µ L per s, *rate not to exceed 1.0 µL per s.* If a 50-µL syringe is used with an automatic injection adaptor, the injection pulses should be spaced about 2 s apart.

24.6 Record the area of the peak generated with a suitable integrator.

25. Calculations

25.1 Calculate the chloride level using the following equation:

$$A \times C \times D \times E/B \times G \times H = \text{chlorides, ppm by weight} \quad (2)$$

where:

A = peak area of sample,
B = peak area of standard,
C = ppm chloride in standard,
D = weight of standard injected (µL standard × density, g/mL),
E = range in Ohms used for recording area of standard peak,
G = weight of sample injected (µL sample × density, g/mL), and
H = range in Ohms used for recording area of sample peak.

26. Report

26.1 Report the chloride content to the nearest part per million by weight. Duplicate runs that agree within the checking limits shown in Table 2 are acceptable for averaging (95 % confidence level).

27. Precision and Bias

27.1 The following criteria should be used for judging the acceptability of results.

27.1.1 *Repeatability (Single Analyst)*— The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the value shown in Table 2 with the indicated degrees of freedom. Two such results should be considered suspect if they differ by more than the values in Table 2.

27.1.2 *Reproducibility (Multilaboratory)*— The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be the value shown in Table 2 with the indicated degrees of freedom shown. Two such results should be considered suspect (95 % confidence level) if they differ by more than the values shown in Table 2 (Note 6).

NOTE 6—The above precision estimates are based on an interlaboratory study of six samples of linear detergent alkylates. A total of three laboratories cooperated in the studies in which duplicate determinations were performed on each of 2 days. Practice E180 was used in developing these precision estimates.[12]

PLATINUM-COBALT COLOR

28. Procedure

28.1 Determine the color of the linear detergent alkylate in accordance with Test Method D1209.

29. Report

29.1 Report the color of the sample to the nearest 5 platinum-cobalt units. Duplicate runs that agree within 5 units are acceptable for averaging.

30. Precision and Bias

30.1 *Repeatability (Single Analyst)*— The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.5 platinum-cobalt units at 35 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 2 platinum-cobalt units.

30.2 *Reproducibility (Multilaboratory)*— The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 2.0 platinum-cobalt units at 8 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 7 platinum-cobalt units (Note 7).

NOTE 7—The above precision estimates are based on an interlaboratory study of six samples of linear detergent alkylates. A total of nine laboratories cooperated in the study in which duplicate determinations were performed on each of 2 days. Practice E180 was used in developing these precision estimates.

REFRACTIVE INDEX

31. Procedure

31.1 Determine the refractive index of the linear detergent alkylate at 20°C in accordance with Test Method D1218.

32. Report

32.1 Report the refractive index of the linear detergent alkylate to the nearest 0.0001 unit. Duplicate runs that agree within 0.0004 units are acceptable for averaging (95 % confidence level).

33. Precision and Bias

33.1 *Repeatability (Single Analyst)*— The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.00013 units at 29 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.0004 units.

33.2 *Reproducibility (Multilaboratory)*— The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.00043 units at 5 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.0016 units (Note 8).

TABLE 2 Organic Chlorides by Dohrmann Method

Chloride Level, ppm	Checking Limits[A]	Repeatability			Reproducibility		
		Coefficient Of Variation	Degrees of Freedom	95 % Confidence Interval[A]	Coefficient Of Variation	Degrees of Freedom	95 % Confidence Interval[A]
0–5	54.8 %	16.90	3	58.4 %	58.35 %	2	354.8 %
350–650	7.3 %	7.12	10	22.43 %	13.10 %	2	79.6 %

[A] Percentage of the determined amount.

NOTE 8—The above precision estimates are based on an interlaboratory study of five samples of linear detergent alkylates. A total of six laboratories cooperated in the study in which duplicate determinations were performed on each of 2 days. Practice E180 was used in developing these precision estimates.

SPECIFIC GRAVITY

34. Procedure

34.1 Determine the specific gravity of the linear detergent alkylate in accordance with Test Method D1122.

35. Report

35.1 Report the specific gravity of the sample to the nearest 0.001 unit. Duplicate runs that agree within 0.001 unit are acceptable for averaging (95 % confidence level).

36. Precision and Bias

36.1 *Repeatability (Single Analyst)*— The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.0004 unit at 30 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.001 units.

36.2 *Reproducibility (Multilaboratory)*— The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories, has been estimated to be 0.0008 units at 5 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.003 units (Note 9).

NOTE 9—The above precision estimates are based on an interlaboratory study of five samples of linear detergent alkylates. A total of six laboratories cooperated in the study in which duplicate determinations were performed on each of 2 days. Practice E180 was used in developing these precision estimates.

WATER OR MOISTURE

37. Procedure

37.1 Determine the water content of the linear detergent alkylate in accordance with Test Method D1364, with the following modifications:

37.1.1 Use 1 part pyridine to 4 parts anhydrous methanol as the sample solvent.

37.1.2 Use a 50-mL specimen (sample) size.

37.1.3 Use a weak Fischer reagent which is prepared by diluting one part of the strong reagent described in Test Method D1364 with 10 parts of anhydrous (less than 0.1 % water) 2-methoxyethanol.

38. Report

38.1 Report the water content of the sample to the nearest part per million (ppm). Duplicate runs that agree within 20 ppm are acceptable for averaging (95 % confidence level).

39. Precision and Bias

39.1 *Repeatability (Single Analyst)*— The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 5.0 ppm at 38 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 14 ppm.

39.2 *Reproducibility (Multilaboratory)*— The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 19.3 ppm at 5 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 70 ppm (Note 10).

NOTE 10—The preceding precision estimates are based on an interlaboratory study of six samples of linear detergent alkylates. A total of seven laboratories cooperated in the study in which duplicate determinations were performed on each of 2 days. Practice E180 was used in developing these precision estimates.

40. Keywords

40.1 linear alkylbenzenes; linear detergent alkylates