Designation: E 1090 – 96

Standard Test Method for Dicumyl Peroxide and Dicumyl Peroxide Decomposition Products in Resins[1]

This standard is issued under the fixed designation E 1090; 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 This test method is applicable to the determination of dicumyl peroxide[2] and the decomposition products dimethylbenzyl alcohol and acetophenone in cured and uncured polyethylene (PE) and ethylene vinyl acetate (EVA) resins. These uncured polymers normally contain from 1 to 2 % dicumyl peroxide, whereas the residual peroxide level in the cured polymers is usually less than 0.1 %.

1.2 *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.* Specific precautionary statements are given in Section 7.

1.3 Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[3]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]

E 300 Practice for Sampling Industrial Chemicals[4]

E 682 Practice for Liquid Chromatography Terms and Relationships[5]

E 685 Practice for Testing Fixed-Wavelength Photometric Detectors Used in Liquid Chromatography[5]

E 755 Test Method For Assay of Dicumyl Peroxide by Liquid Chromatography[4]

3. Summary of Test Method

3.1 Dicumyl peroxide and dimethylbenzyl alcohol are extracted from a cryogenically ground sample with methylene chloride. The extract is concentrated, redissolved in methanol, and analyzed by high performance liquid chromatography (HPLC). Acetophenone is extracted from a separate sample with methanol and analyzed directly by HPLC. The analyses are performed on a reversed phase octadecylsilane (ODS) column using acetonitrile/water as the mobile phase and an ultraviolet detector at 254 nm. The concentration of each component is determined by the internal standard technique, using peak height ratios of the sample and standard chromatograms.

4. Significance and Use

4.1 Knowledge of the peroxide content of uncured PE and EVA samples is required to regulate the degree of crosslinking in the cured product. As end use applications of the cured product can be affected by residual amounts of the peroxide or its decomposition products—dimethylbenzyl alcohol and acetophenone—knowledge of these levels is also important. This test method provides a procedure for determining the concentration of these compounds. A method for the HPLC assay of dicumyl peroxide is described in Test Method E 755.

5. Apparatus

5.1 *Liquid Chromatograph*, equipped with a 254-nm UV detector, injection valve, and an isocratic-solvent delivery system capable of operating to a gage pressure of 3000 psi. The detector should be equipped with an attenuator switch to change the sensitivity range as required. (See Practices E 682 and E 685.)

5.2 *Recorder*, 0 to 1 mv range, 1 s or less full-scale deflection, with a chart speed of 0.1 in./min or other convenient speed that will produce a satisfactory chromatogram. As an alternative, an electronic data system can be used.

5.3 *Chromatographic Column*, reversed phase C-18, from 250 to 300-mm by 3.9-mm inside diameter, containing octadecylsilane chemically bonded to microparticulate silica.[6]

NOTE 1—Commercial HPLC columns may vary in physical dimensions, degree of substrate loading, and size and type of support material. For these reasons, some modification in the operating parameters may be required to achieve optimum separation.

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[1] This test method is under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and is the direct responsibility of Subcommittee E15.22 on Functional Groups.

Current edition approved March 10, 1996. Published May 1996. Originally published as E 1090 – 86. Last previous edition E 1090 – 91.

[2] Dicumyl peroxide; peroxide, bis(1-methyl-1-phenylethyl) $C_{18}H_{22}O_2$; CAS Registry No. 80-43-3.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 14.01.

[6] Satisfactory results were obtained using Waters μ-Bondapak C-18 (Cat. No. 27324) and Waters Radial PAK C-18 (Cat. No. 84720) columns in a round-robin evaluation of the test method. Equivalent results should be obtained with other commercial C-18 reversed phase columns.

5.4 *Guard Column*, reversed phase C-18, containing octadecylsilane chemically bonded to microparticulate silica.

5.5 *Filter Funnel*, Buchner, 60-mL capacity, with medium porosity glass frit.

5.6 *Vials*, screw cap, 4-dram and 1-dram capacities, with PTFE-lined caps.

5.7 *Freezer Mill*, for pulverizing samples at liquid nitrogen temperature.[7]

5.8 *Bottles*, screw cap, wide-mouth, 2-oz capacity, with PTFE-lined caps.

5.9 *Sample Filter*, consisting of a syringe and 0.45-µm filter assembly to remove microparticulate matter from the prepared sample solution.[8]

5.10 *Tube*, borosilicate glass, approximately 8-in. long by 1-in. diameter with tapered end, for warming cryogenically ground resin samples to ambient temperature (see Fig. 1).

5.11 *Solvent Evaporation Assembly*—See Fig. 2.

5.12 *Silica Gel Purification Column.*[9]

6. Reagents

6.1 *Methanol*, chromatographic grade, distilled in glass.

6.2 *Acetonitrile*, chromatographic grade, distilled in glass.

6.3 *Water*, prepare Type II reagent water in accordance with Specification D 1193, or distill deionized water. Filter through a 0.45-µm filter[10] and store in a glass container.

6.4 *Acetonitrile:Water, 70:30*—Mix 7 volumes of acetonitrile with 3 volumes of water.

6.5 *Acetonitrile:Water, 30:70*—Mix 3 volumes of acetonitrile with 7 volumes of water.

6.6 *Acetonitrile:Water, 95:5*—Mix 9.5 volumes of acetonitrile with 0.5 volumes of water.

6.7 *Methylene Chloride*, chromatographic grade, distilled in glass.

6.8 *Dibutyl Phthalate*, purified.[11]

6.9 *Dibutyl Phthalate Internal Standard* (approximately 7.0 mg/mL)—Weigh 7.0 ± 0.1 g of dibutyl phthalate to the nearest 0.1 mg. Dissolve in methanol and quantitatively transfer to a 1-L volumetric flask. Dilute to volume with methanol and mix thoroughly. Calculate the exact concentration of dibutyl phthalate.

6.9.1 Long-term storage of a methanolic solution of dibutyl phthalate should be avoided. Dibutyl phthalate in the presence of traces of acidic or basic impurities may transesterify. If

[7] A Spex Freezer/Mill, Catalog No. 6700, has been found to be satisfactory for this purpose. This mill is available from Spex Industries, Inc., Metuchen, NJ.

[8] Waters Associations Sample Clarification Kit, Catalog No. 26870, was found to be satisfactory for this purpose.

[9] SEP-PAK silica gel cartridges, Waters No. 51-900, have been found to be suitable for this purpose.

[10] A0.45-µm Millipore type HA filter was found to be satisfactory for this purpose.

[11] Dibutyl phthalate, Aldrich Chemical Co. No. 15243-9, was found to be satisfactory for this use.

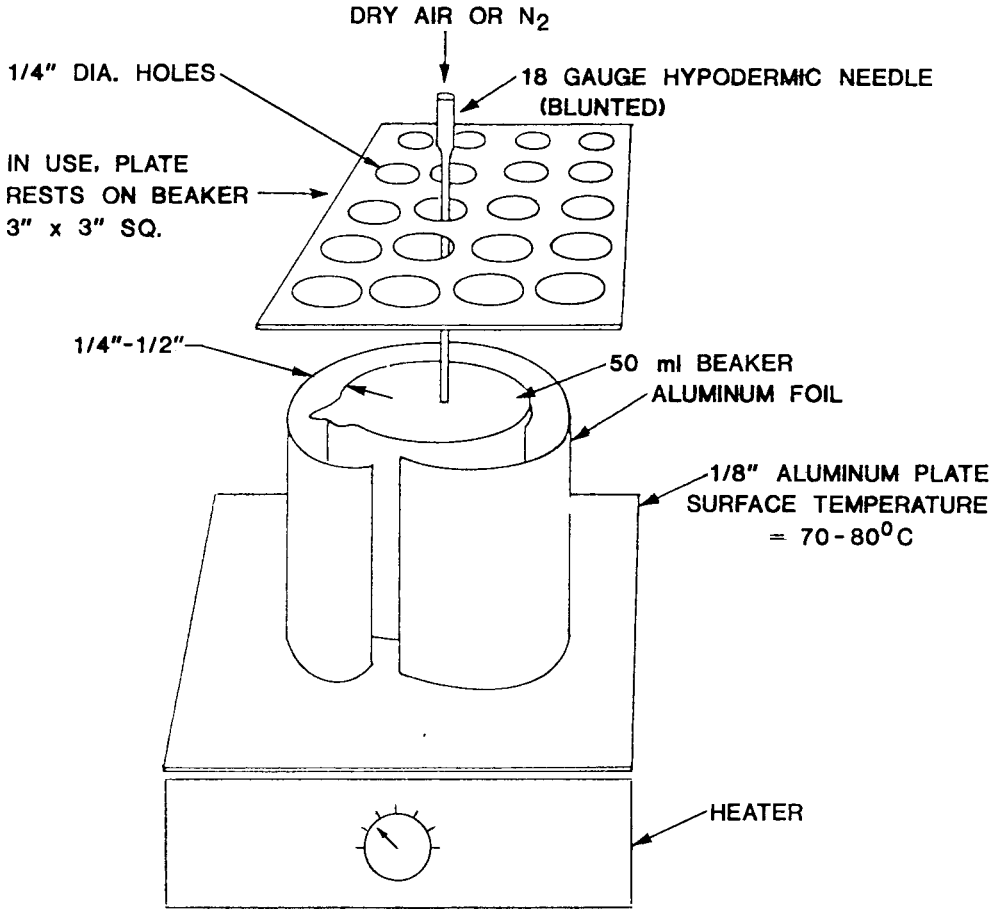

FIG. 2 Solvent Evaporation Assembly for Preventing Accumulation and Loss of Volatile Compounds

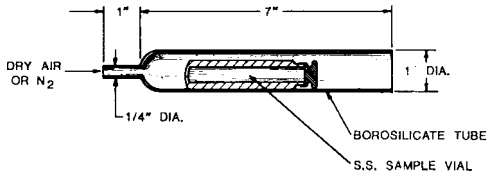

FIG. 1 Tube for Warming Cryogenically Ground Resin Samples to Ambient Temperature

transesterification occurs, the dibutyl phthalate peak will slowly decrease, and the appearance of the methylbutyl phthalate peak (k_1 value about 3.8) will be noted.

6.10 *Dibutyl Phthalate Internal Standard* (approximately 0.7 mg/mL)—Pipet 100 mL of dibutyl phthalate standard (6.9, approximately 7 mg/mL) into a 1-L volumetric flask. Dilute to volume with methanol and mix thoroughly. Calculate the exact concentration of dibutyl phthalate.

6.11 *Benzyl Alcohol*, purified.[12]

6.12 *Benzyl Alcohol Internal Standard* (approximately 15.0 mg/mL)—Weigh 15.0 ± 0.1 g of benzyl alcohol to the nearest 0.1 mg. Dissolve in methanol and quantitatively transfer to a 1-L volumetric flask. Dilute to volume with methanol and mix thoroughly. Calculate the exact concentration of benzyl alcohol.

6.13 *Benzyl Alcohol Internal Standard* (approximately 1.5 mg/mL)—Pipet 100 mL of benzyl alcohol standard (6.12, approximately 15 mg/mL) into a 1-L volumetric flask. Dilute to volume with methanol and mix thoroughly. Calculate the exact concentration of benzyl alcohol.

6.14 *Dicumyl Peroxide, Recrystallized*—Transfer 25.0 g of commercial refined dicumyl peroxide into a 100-mL Erlenmeyer flask. Add 8.0 mL of methanol and gently warm the solution in a water bath while swirling, to effect complete solution. Cool to 0°C in an ice bath. Transfer the contents to a medium-porosity sintered glass crucible and vacuum filter. Allow air to pass through the filter for 10 to 15 min, to dry the peroxide. Repeat the crystallization twice using approximately 1 mL of methanol for every 3 g of peroxide. Place the recrystallized dicumyl peroxide in a tightly capped bottle and store in the refrigerator. *Caution*—see Section 7.

6.15 *Acetophenone*, purified.[13]

6.16 *α,α-Dimethylbenzyl Alcohol (DMBA)*—Dissolve 0.2 g of α,α-dimethylbenzyl alcohol[14] in 2 mL of 98:2 *n*-hexane:chloroform. Transfer the solution into a 5-mL syringe and carefully pass the solution through a SEP-PAK silica gel cartridge. Discard the eluate. Wash the column with an additional 2 mL of 98:2 *n*-hexane:chloroform and again discard the eluate. Then, elute the DMBA with 5 mL of chloroform, collecting the eluate in a 50-mL filtering flask. Stopper the flask and attach the side arm to a water aspirator. Immerse the flask in a water bath maintained at 35 to 40°C until the chloroform has completely volatilized. Store the purified DMBA in a sealed vial.

[12] Benzyl alcohol, Aldrich Chemical Co. No. B1620-8, was found to be satisfactory for this use.

[13] Acetophenone, 99 %, Aldrich Chemical Co. No. A1,070-1, was found to be satisfactory for this use.

[14] α,α-Dimethylbenzyl alcohol, 99 %, Fluka Chemical Corp. No. 78940, was found to be satisfactory for this use.

7. Precautions

7.1 Organic peroxides are strong oxidizing agents and present potential fire and explosion hazards. Reactivity varies widely and some compounds may explode when shocked. While dicumyl peroxide is one of the more stable peroxides, contact with reducing agents and sources of heat, sparks, or open flame must be avoided. Organic peroxides in general are irritating to the skin, eyes, and mucous membranes. Avoid bodily contact and handle only in a well-ventilated area.

7.2 Small quantities of solid or molten dicumyl peroxide can be safely handled at temperatures up to 55°C. Dicumyl peroxide should not be heated above 55°C as the rate of peroxide decomposition rapidly increases with increasing temperatures above this point.

7.3 Only a water bath that has been preheated to the desired temperature and removed from the heat source should be used for warming vessels containing dicumyl peroxide. Electrically heated water baths should not be used as they may cause localized hot spots. Other sources of heat considered unsafe for warming containers of dicumyl peroxide include ovens, hot plates, and direct steam.

8. Sample Preparation

8.1 Obtain at least 3 g of a representative sample and reduce the particle size, if required, to approximately ⅛ in. or less using stainless-steel shears. (See Practice E 300.)

8.2 Charge the stainless-steel sample vial with approximately 1.5 g of sample, add the stainless-steel impactor rod, and cap the vial with the stainless-steel cover head.

8.3 Carefully position the vial in the freezer/mill which has been precooled and filled with liquid nitrogen to the proper level.

8.4 Cool for 4 to 5 min, then activate the impactor and allow to pulverize for 3 to 4 min at optimum impactor rate. Consult manufacturer's instructions for detailed operating procedure.[7]

8.5 Remove the sample vial and immediately place in a borosilicate tube through which a flow of dry air or nitrogen is maintained. See Fig. 1. Allow to warm to ambient temperature under the dry air or nitrogen flow in order to exclude atmospheric moisture.

8.6 Remove the pulverized sample and store in a clean, capped vial.

8.7 Repeat 8.2-8.5 and combine with the pulverized product obtained in 8.6. Reserve for extraction and HPLC analysis.

9. Procedure

9.1 *Preparation of Sample for Determination of Dicumyl Peroxide and Dimethylbenzyl Alcohol*:

9.1.1 Weigh a 2.0 ± 0.1-g sample of cryogenically ground resin to the nearest 0.1 mg and transfer to a 2-oz bottle equipped with a PTFE-lined screw cap.

9.1.2 Add approximately 30 mL of methylene chloride, cap, and shake. Allow to stand at ambient temperature for 18 h with occasional shaking to complete the extraction.

9.1.3 Filter through a medium porosity sintered-glass filter collecting the filtrate in a 125-mL filter flask. Rinse the resin with several small portions of methylene chloride, collecting the washings in the flask.

9.1.4 Quantitatively transfer the filtrate to a 50-mL beaker

using a minimum amount of methylene chloride to aid in the transfer.

9.1.5 Place the beaker on the solvent-evaporation assembly and direct a gentle stream of N_2 or dry air against the surface of the extract. The surface temperature of the aluminum plate should be maintained from 70 to 80°C and a ¼ to ½-in. space maintained between the beaker and the aluminum foil as shown in Fig. 2.

9.1.6 Evaporate the filtrate until the volume is reduced from 1 to 2 mL. Do not evaporate to dryness.

9.1.7 Quantitatively transfer the solution to a 1-dram PTFE-lined screw cap vial using a minimum amount of methylene chloride to aid in the transfer.

9.1.8 Place the vial on the evaporation assembly and continue the evaporation until the solvent has almost completely evaporated.

9.1.9 Pipet 0.50 mL of dibutyl phthalate internal standard (0.7 mg/mL) and 0.50 mL of benzyl alcohol internal standard (1.5 mg/mL) into the vial, then cap and mix thoroughly.

9.1.10 Transfer the solution into the syringe of the sample clarification kit[8] and filter the solution through a 0.45-µm filter collecting the clear filtrate in a clean 1-dram vial fitted with a PTFE-lined screw cap. Cap and reserve for analysis.

9.2 *Preparation of Sample for Determination of Acetophenone*:

9.2.1 Weigh a 2.0 ± 0.1-g sample of cryogenically ground resin to the nearest 0.1 mg and transfer to a 25-mL volumetric flask.

9.2.2 Pipet 10.00 mL of benzyl alcohol internal standard (1.5 mg/mL) into the flask and dilute to volume with methanol. Stopper and mix. Allow to stand for 18 h at ambient temperature with occasional shaking to complete the extraction.

9.2.3 Transfer a portion of the supernatant extract into the syringe of the sample clarification kit[8] and filter through a 0.45-µm filter collecting the clear filtrate in a clean 1-dram vial fitted with a PTFE-lined screw cap. Cap and reserve for analysis.

9.3 *Preparation of Calibration Standards*:

9.3.1 *Dicumyl Peroxide Standard* (4.5 mg/mL)—Weigh 0.45 ± 0.05 g of recrystallized dicumyl peroxide to the nearest 0.1 mg. Dissolve in methanol and quantitatively transfer to a 100-mL volumetric flask. Pipet 10.00 mL of dibutyl phthalate internal standard (7.0 mg/mL) into the flask, dilute to volume with methanol, and mix. This solution contains 0.70 mg of dibutyl phthalate/mL. Calculate the exact concentration of dicumyl peroxide.

9.3.1.1 Filter a portion of this solution through a 0.45-µm syringe filter collecting the filtrate in a 4-dram vial. Cap tightly and store in a cool, dark location. The standard is stable for approximately one week.

9.3.2 *Dimethylbenzyl Alcohol Standard* (8 mg/mL)—Weigh 0.80 ± 0.05 g of dimethylbenzyl alcohol to the nearest 0.1 mg. Dissolve in methanol and quantitatively transfer to a 100-mL volumetric flask. Pipet 10.00 mL of benzyl alcohol internal standard (15 mg/mL) into the flask, dilute to volume with methanol, and mix. This solution contains 1.50 mg of benzyl alcohol/mL. Calculate the exact concentration of dimethylbenzyl alcohol.

9.3.2.1 Filter a portion of this solution through a 0.45-µm syringe filter, collecting the filtrate in a 4-dram vial. Reserve for calibration.

9.3.3 *Acetophenone Standard* (0.05 mg/mL)—Weigh 0.05 ± 0.005 g of acetophenone to the nearest 0.1 mg. Dissolve in methanol and quantitatively transfer to a 100-mL volumetric flask. Dilute to volume with methanol and mix thoroughly. This solution contains approximately 0.50 mg of acetophenone/mL. Calculate the exact concentration.

9.3.3.1 Pipet 10.00 mL of the above solution (0.50 mg/mL) into another 100-mL volumetric flask. Pipet 10.00 mL of benzyl alcohol internal standard (15 mg/mL) into the flask, dilute to volume with methanol, and mix. This solution contains 1.50 mg of benzyl alcohol/mL and approximately 0.05 mg of acetophenone/mL. Calculate the exact concentration of acetophenone.

9.3.3.2 Filter a portion of the above solution through a 0.45-µm syringe filter, collecting the filtrate in a 4-dram vial. Reserve for calibration.

9.4 *Determination of Dicumyl Peroxide*:

9.4.1 Adjust the liquid chromatograph in accordance with the following parameters and allow the instrument to equilibrate until a stable base line is obtained on the recorder chart at the sensitivity setting to be used:

Column temperature	Ambient
Mobile phase	70:30 acetonitrile:water
Flow rate	1.0 mL/min
Chart speed	0.1 in./min
Detector	254 nm
Pump pressure	Normally 800 to 1200 psig
Sample injection	10 µL

NOTE 2—The parameters shown apply to a liquid chromatograph equipped with a Waters No. 27324 µ Bondapak C-18 reverse-phase column, 3.9 mm in inside diameter by 30 cm in length. Other columns may require some modification in the flow rate or mobile phase composition (see Note 1).

9.4.2 Determine the optimum sensitivity response by injecting 10 µL of the prepared standard solution (4.5 mg of dicumyl peroxide/mL, 9.3.1) and adjusting the detector attenuation to obtain approximately 85 % full-scale deflection for the larger of the dibutyl-phthalate and dicumyl-peroxide peaks. The capacity factor (k') for dicumyl peroxide should be within the approximate range of 5.0 to 6.6 in order to achieve optimum resolution of internal-standard and dicumyl-peroxide peaks. If necessary, adjust the composition of the mobile phase so that the capacity factor falls within this range. Typical retention times for dibutyl phthalate and dicumyl peroxide are approximately 10.5 and 15 min.

9.4.3 With conditions optimized, inject 10 µL of the prepared standard solution and record the chromatogram. A typical chromatogram of a dicumyl peroxide calibration standard obtained under the conditions outlined in 9.4.1 is shown in Fig. 3. When running actual samples, acetophenone, dimethylbenzyl alcohol, and benzyl alcohol coelute before the dibutyl phthalate at a retention time of approximately 3½ min.

9.4.4 Immediately after obtaining the chromatogram of the standard, inject 10 µL of the prepared sample solution for dicumyl peroxide (9.1) and record the chromatogram.

9.4.5 Calculate the dicumyl peroxide content of the original resin.

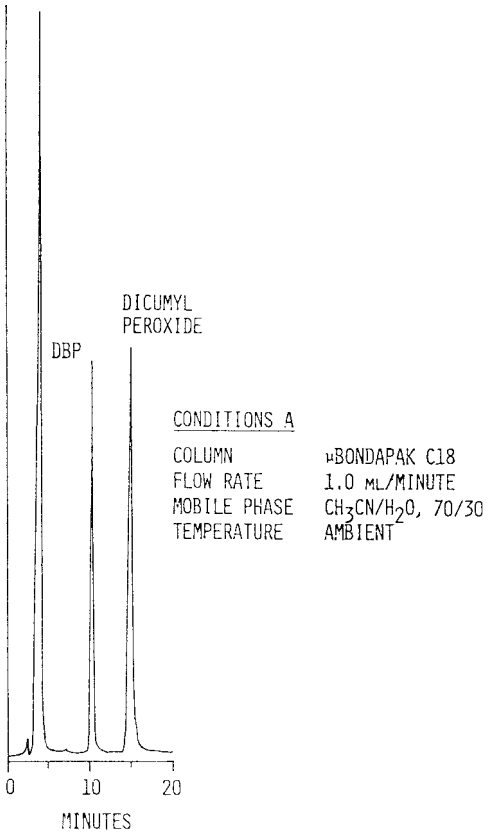

FIG. 3 Typical Chromatogram for Dicumyl Peroxide Calibration

9.5 *Determination of Dimethylbenzyl Alcohol*:

9.5.1 Adjust the liquid chromatograph in accordance with the following parameters and allow the instrument to equilibrate until a stable base line is obtained on the recorder at the sensitivity setting to be used (see Note 2):

Column temperature	Ambient
Mobile phase	30:70 acetonitrile:water
Flow rate	0.5 mL/min
Chart speed	0.1 in./min
Detector	254 nm
Pump pressure	Normally 800 to 2000 psig

9.5.2 Determine the optimum sensitivity response by injecting 10 µL of the prepared standard solution (8.0 mg of dimethylbenzyl alcohol/mL, 9.3.2) and adjusting the detector attenuation to obtain approximately 85 % full-scale deflection for the larger of the benzyl-alcohol and dimethylbenzyl-alcohol peaks. Typical retention times for benzyl alcohol and dimethylbenzyl alcohol are 13.5 and 23 min.

9.5.3 With conditions optimized, inject 10 µL of the prepared standard solution and record the chromatogram. A typical chromatogram of a dimethylbenzyl alcohol standard obtained under the conditions outlined in 9.5.1 is shown in Fig. 4.

9.5.4 Immediately after obtaining the chromatogram of the standard, inject 10 µL of the prepared sample solution for dimethylbenzyl alcohol (9.1) and record the chromatogram.

9.5.4.1 Following a sample injection under chromatographic conditions outlined under 9.5.1, dibutyl phthalate, dicumyl peroxide, and polymer components do not elute until several hours later. When multiple sample injections are made, the column should be purged with acetonitrile:water (95:5) after 2 h running time under 9.5.1 conditions, to remove later eluting compounds which might, in time, interfere with subsequent dimethylbenzyl alcohol determinations. When a steady base line is obtained continue as directed under 9.5.1.

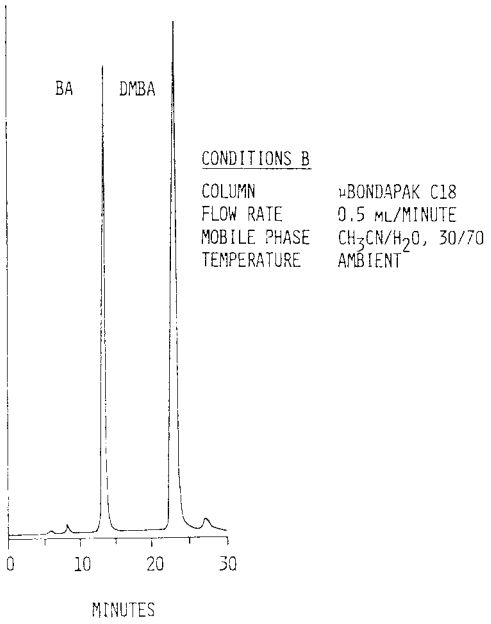

FIG. 4 Typical Chromatogram for Dimethylbenzyl Alcohol Calibration

9.5.5 Calculate the dimethylbenzyl alcohol content of the original resin.

9.6 *Determination of Acetophenone*:

9.6.1 Adjust the liquid chromatograph in accordance with the parameters outlined in 9.5.1 and allow the instrument to equilibrate until a stable base line is obtained on the recorder at the sensitivity setting to be used. See Note 2.

9.6.2 Determine the optimum sensitivity response by injecting 10 µL of the prepared standard solution (0.05 mg of acetophenone/mL, 9.3.3) and adjusting the detector attenuation to obtain approximately 85 % full-scale deflection for the larger of the benzyl-alcohol and acetophenone peaks. Typical retention times for benzyl alcohol and acetophenone are 13.5 and 27 min.

9.6.3 With the conditions optimized, inject 10 µL of the prepared standard solution and record the chromatogram. A typical chromatogram of an acetophenone standard obtained under the conditions outlined in 9.5.1 is shown in Fig. 5.

9.6.4 Immediately after obtaining the chromatogram of the standard, inject 10 µL of the prepared sample solution for acetophenone (9.2) and record the chromatogram.

9.6.5 Calculate the acetophenone content of the original resin.

10. Calculation

10.1 Measure the peak heights of the compound and the internal standard of the standard solution.

10.2 Calculate the response factor, *F*, for the compound as follows:

$$F = \frac{A \times C}{B \times D} \quad (1)$$

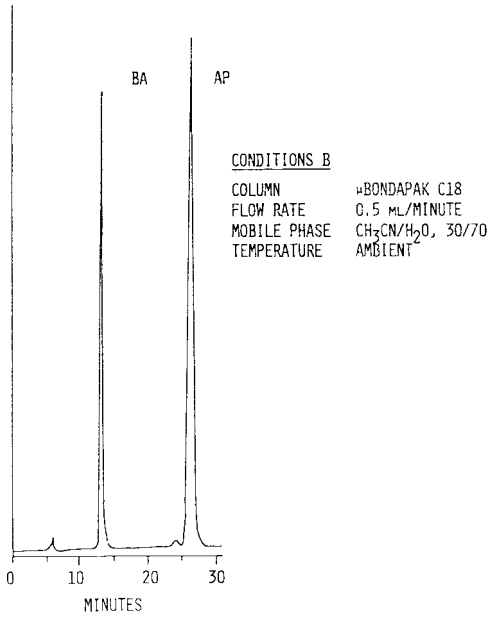

FIG. 5 Typical Chromatogram for Acetophenone Calibration

where:
A = concentration of the compound, mg/mL,
B = concentration of the internal standard solution, mg/mL,
C = peak height of the internal standard, mm, and
D = peak height of the compound, mm.

10.3 Measure the peak heights of the compound and the internal standard of the sample solution.

10.4 Calculate the percent of compound present in the sample as follows:

$$\text{Compound, \%} = \frac{B' \times D' \times F}{A' \times C'} \times 100 \quad (2)$$

where:
F = response factor of specific compound,
A' = concentration of sample, mg/mL,
B' = concentration of internal standard solution, mg/mL,
C' = peak height of internal standard peak, mm, and
D' = peak height of compound peak, mm.

11. Report

11.1 Report the percentage of each component to two significant figures.

12. Precision and Bias

12.1 The following criteria should be used for judging the acceptability of results (Note 3).

12.1.1 *Repeatability (Single Analyst)*—The coefficient of variation for a single determination has been estimated to be 12.3 %, relative, at 40 dF. The 95 % limit for the difference between two such runs is 34 %, relative.

12.1.2 *Laboratory Precision (Within-Laboratory, Between-Day Variation), formerly called repeatability*—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 7.8 %, relative, at 20 dF. The 95 % limit for the difference between two such averages is 22 %, relative.

12.1.3 *Reproducibility (Multi-Laboratory)*—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 39 %, relative, at 6 dF. The 95 % limit for the difference between two such averages is 108 %, relative.

NOTE 3—The above precision estimates are based upon an interlaboratory study on a sample of cured polyethylene electric cable containing about 0.005% dicumyl peroxide, 0.08 % dimethylbenzyl alcohol, and 0.06 % acetophenone. One analyst in each of 7 laboratories performed duplicate determinations and repeated on a second day, for a total of 84 determinations.[15] Practice E 180 – 90 was used in developing these precision estimates. The above precision estimates were recalculated from previous precision statements to conform with current precision definitions.

12.2 The bias of this test method has not been determined due to the unavailability of suitable reference materials.

13. Keywords

13.1 acetophenone; dicumyl peroxide; dimethylbenzyl alcohol; ethylene-vinyl acetate copolymers; high performance liquid chromatograph; HPLC; polyethylene; resins

[15] Supporting data are available from ASTM Headquarters. Request RR: E15–1034.