



Technical Information

DI-CUP[®] Dicumyl Peroxide

High-Performance Liquid Chromatographic Assay

This method is applicable for assaying the active dicumyl peroxide (*a,a'*-dimethylbenzyl peroxide) content of DI-CUP[®] R, DI-CUP[®] T, DI-CUP[®] 40C, and DI-CUP[®] 40KE.

The procedure consists of chromatographing an aliquot of a sample solution containing an internal standard on a μ Bondapak-C18 column (reversed-phase) with an isocratic mobile-phase ratio of 70:30 acetonitrile:water. UV detection at 254 nm is utilized. The amount of dicumyl peroxide in the sample is calculated through the use of appropriate peak area ratios.

Apparatus

1. Liquid chromatograph. Any liquid chromatograph equipped with a UV detector capable of monitoring at 254 nm.
2. Column, μ Bondapak-C18, Waters Associates, Part No. 27324.
3. 15 ml vial, screw cap, with Teflon (Dupont) cap liner.
4. Pipette, transfer, 10-mL.
5. Disposable filters, nylon, 0.25 μ m.
6. Flask, volumetric, 1,000-mL.
7. Flask, volumetric, 100-mL.

Reagents

1. Methanol, HPLC grade.
2. Water, HPLC grade.
3. Acetonitrile, HPLC grade.
4. Stock solution containing the internal standard. Weigh to the nearest 0.0001 g, about 7.0 g dibutyl phthalate (reference standard). Quantitatively transfer to a 1,000-mL volumetric flask, fill to the mark with methanol, and thoroughly mix. Calculate the exact concentration in mg/mL.

Reference Standards

1. Dicumyl peroxide, 99+% pure.
2. Dibutyl phthalate, 99+% pure.

Column Operating Conditions

Column	ambient
Flow rate	1 mL/min.

Calibration

1. Weigh, to the nearest 0.0001 g, 0.45 ± 0.05 g dicumyl peroxide standard into a 100-mL volumetric flask. Add 50 mL of methanol and swirl until the sample has completely dissolved. With a pipette, transfer 10.00 mL of dibutyl phthalate internal standard (7.0 mg/mL) into the flask, dilute to volume with methanol, and mix thoroughly. Calculate in mg/mL the exact concentration of dicumyl peroxide. Filter a portion of standard solution through a 0.25- μ m disposable nylon filter into a clean, dust free 15-mL vial.
2. When the column is in equilibrated with the mobile phase (acetonitrile: water, 70:30), inject 10 μ L of filtered standard solution.



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Reagents

- a Melt, at a maximum temperature of 55°C, and weigh, to the nearest 0.0001 g, 0.45 ± 0.05 g of Di-Cup[®] R into a 100-mL volumetric flask. Add 50 mL methanol and swirl until the sample is completely dissolved. Add 10.00 mL stock solution containing internal standard, dilute to volume with methanol, mix thoroughly, filter using the 0.25 µm disposable nylon filters, and chromatograph according to the conditions specified.
 - b For Di-Cup[®] 40C and Di-Cup[®] 40KE, weigh to the nearest 0.0001 g, 1.1 ± 0.05 g into a 100-mL volumetric flask. Add 50 mL of methanol and 10.00 mL of dibutyl phthalate internal standard stock solution; mix well. Dilute to volume with methanol and mix thoroughly. Let stand approximately 30 min. to ensure complete extraction. Filter a small portion using the 0.25 µm disposable nylon filters, and chromatograph according to the conditions specified.
2. Measure the peak areas of the internal standard and dicumyl peroxide peaks.

Calculations

1. Measure the areas of the dibutyl phthalate and dicumyl peroxide peaks in the chromatogram of the standard solution.
2. Calculate the response factor, F, for the dicumyl peroxide as follows:

$$F = \frac{A \times C}{B \times D}$$

where
A = Concentration of dicumyl peroxide in prepared standard solution, mg/mL.
B = Concentration of dibutyl phthalate in prepared standard solution, mg/mL.
C = Peak area of dibutyl phthalate in chromatogram of standard, mAU.
D = Peak area of dicumyl peroxide in chromatogram of standard, mAU.

3. Measure the areas of the dibutyl phthalate and dicumyl peroxide peaks in the chromatogram of the sample solution.
4. Calculate the percent of dicumyl peroxide present in the original sample as follows:

$$\% \text{ dicumylperoxide} = \frac{B' \times D' \times F}{A' \times C'} \times 100\%$$

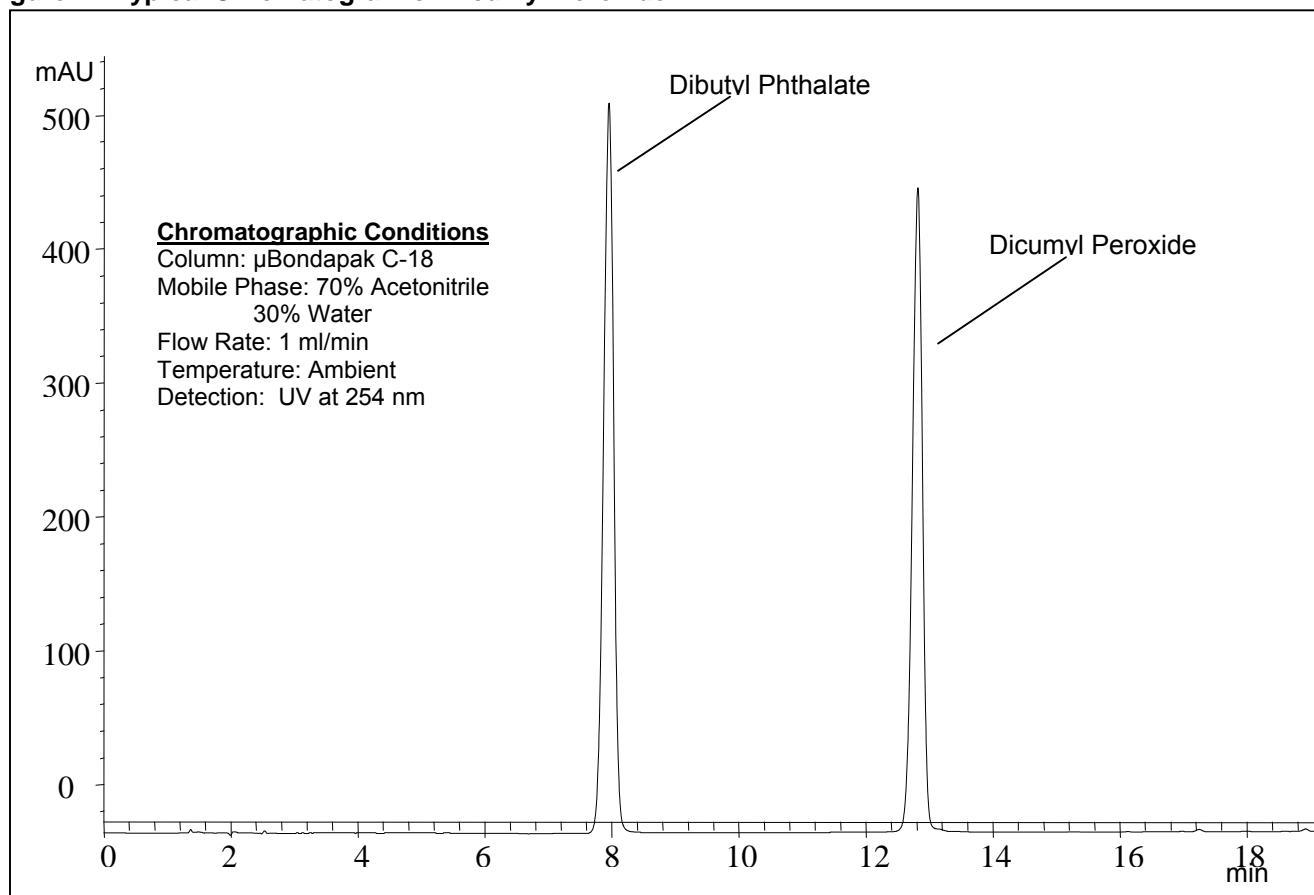
where
F = Response factor for the dicumyl peroxide standard.
A' = Concentration of sample in prepared sample solution, mg/mL.
B' = Concentration of dibutyl phthalate in prepared sample solution, mg/mL.
C' = Peak area of dibutyl phthalate in chromatogram of sample, mAU.
D' = Peak area of dicumyl peroxide in chromatogram of sample, mAU.

Precision

Preliminary data indicate that the standard deviation of the method based on 10 replicates is approximately 0.2.

NOTE: The foregoing procedure has been approved by ASTM Committee E-15 and appears under Method E 755 in the 1981 Annual Book of ASTM Standards, Part 30.

Figure 1 - Typical Chromatogram of Dicumyl Peroxide



For additional information, or to place an order or sample request, call 1.800.331.7654.

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