# High-Pressure Liquid Chromatographic Determination of Camphor and Parachlorophenol in Camphorated Parachlorophenol

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**Abstract** □ Quantitative analyses of both camphor and parachlorophenol in camphorated parachlorophenol by high-pressure liquid chromatography is described. A 5% solution of phenol in the mobile phase (heptane–chloroform, 3:2) served as the internal standard; the assay involved addition of this phenol solution to the camphorated parachlorophenol, followed by further dilution using the mobile phase (heptane–chloroform, 3:2) and injection into the instrument.

**Keyphrases** □ Camphor—high-pressure liquid chromatographic analysis in pharmaceutical formulations of camphorated parachlorophenol □ Parachlorophenol—high-pressure liquid chromatographic analysis in pharmaceutical formulations of camphorated parachlorophenol □ High-pressure liquid chromatography—analyses, camphor and parachlorophenol in pharmaceutical formulations of camphorated parachlorophenol □ Anti-infectives, topical—camphorated parachlorophenol, high-pressure liquid chromatographic analyses of camphor and parachlorophenol, pharmaceutical formulations

Quantitative determinations of phenolic compounds (1–5) include bromination (1), bromination–UV spectroscopy (2), GLC (4, 5), and NMR spectroscopy (6).

None of these methods has been adapted to the simultaneous assay of the components of camphorated parachlorophenol. The USP XIX procedure involves separate assays for the two components. Parachlorophenol is assayed by oxidation followed by silver chloride formation, while the camphor assay involves hydrazone formation.

This paper reports the simultaneous quantitative determination of camphor and parachlorophenol in camphorated parachlorophenol by high-pressure liquid chromatography (HPLC) using heptane–chloroform (3:2).

## EXPERIMENTAL

**Reagents and Chemicals**—All chemicals and reagents used were USP, NF, ACS, or chromatographic grade.

**Preparation of Solutions**—A 5.0% (w/v) phenol solution was prepared in the mobile phase (heptane–chloroform, 3:2). A standard stock solution of camphorated parachlorophenol was prepared according to the USP. All dilutions were done on a weight per volume basis, using the mobile phase as the diluent.

**Preparation of Linearity Curve**—A standard stock solution containing 65.0% (w/w) camphor and 35.0% (w/w) parachlorophenol was prepared. To varying amounts of this stock solution, 2.50, 5.00, 7.50, and 10.00 g, was added 10 ml of a 5.0% phenol solution (internal standard). The solution was then diluted to 100 ml with the mobile phase. Each standard solution was injected (10 μl), and the peak height ratios of camphor to phenol and parachlorophenol to phenol were calculated. When the peak height ratios were plotted *versus* percent known concentration of camphor or parachlorophenol, the chromatographic response was linear for the tested concentrations of both camphor and parachlorophenol.

**Assay**—The camphorated parachlorophenol (5 g) was added to 10 ml of the 5.0% phenol solution (internal standard), and this solution was then diluted to 100 ml with the mobile phase. A 10-μl standard septum injection was made with a 25-μl syringe[1]. The 1:20 dilution of the standard stock solution of camphorated parachlorophenol served as the standard for determining concentrations. This solution contained 3.25% camphor and 1.75% parachlorophenol.

**Table I—Assay Results on USP Camphorated Parachlorophenol**

| Assay | Camphor, % | Parachlorophenol, % |
|---|---|---|
| 1 | 64.3 | 34.7 |
| 2 | 65.6 | 34.9 |
| 3 | 65.0 | 35.4 |
| 4 | 64.6 | 35.6 |
| *SD* | 0.56 | 0.42 |
| *CV* | 0.86 | 1.19 |
| Average deviation | 0.43 | 0.35 |

**Calculations**—The following formula was used for calculating concentrations:

$$\% \text{ sample} = \frac{R_S}{R_{std}} \% \text{ std} \times D \qquad \text{(Eq. 1)}$$

where % sample is the percent camphor or parachlorophenol in the sample, % std is the percent camphor or parachlorophenol in the standard, $R_{std}$ is the peak height ratio of standard camphor and phenol or standard parachlorophenol and phenol, $R_S$ is the peak height ratio of camphor sample and phenol or parachlorophenol sample and phenol, and $D$ is the dilution factor equal to 20.

**Instrument Parameters**—A high-pressure liquid chromatograph[2] was used with a solvent system of 60% heptane and 40% chloroform. A UV detector at 254 nm was used at an attenuation of ×8 for camphor and ×64 for parachlorophenol and phenol. The flow rate was 2.5 ml/min at an inlet pressure of 0.131 $Nm^{-2}$. The chart speed was 0.5 cm/min. The column[3] was 0.64 cm o.d. × 15 cm long, packed with 5-μm silica gel[4]. The injection volume was 7–15 μl, and the elution order was camphor (2.76 min), parachlorophenol (10.64 min), and phenol (12.61 min).

## DISCUSSION

The results in Table I indicate that camphor and parachlorophenol can be simultaneously assayed by simple dilution of the sample and injection into a high-pressure liquid chromatograph. The linearity curve indicated that the components being analyzed gave a linear response well beyond the limits expected in the sample.

This method allows for a much more rapid assay compared to the USP XIX procedure—only 20 min compared to approximately 5 hr.

## REFERENCES

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[1] Hamilton.

[2] Waters Associates model ALC 202.

[3] Hi-Eff Micropart.

[4] Applied Science Laboratories.