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# Linearity and Sensitivities of the Programmed GC-FID Method for Quality Control of Cinnamons

Khin Thantsin[1]


## Abstract

The developed Gas Chromatography Flame Ionization Detector (GC-FID) method was applied to determine camphora, cinnamaldehyde and eugenol in ten *Cinnamomum* species. In the recent research simple, sensitive and reliable programmed temperature capillary GC-FID method for simultaneous determination of camphora, cinnamaldehyde and eugenol was reported.


## Introduction

Medicinal and aromatic plants are important products found in forest area through out east and south Asia, from the plains to the high Himalayas, with the greatest concentration of in the tropical and sub-tropical belts. Cinnamon is one of the oldest known spices and remains an important mercantile commodity today. Its appealing characteristic aroma and flavor finds many uses in the food, cosmetics and pharmaceutical industries (Tainter et al 2001; Farrell, 1999).

The cinnamons of commerce are derived from the dried inner bark of several species of the genus *Cinnamomum*, a moderate sized ever green tree grown in tropical regions. In the United Kingdom and many of its former colonies and most of the Europe, cinnamon is defined as the dried inner bark of the species *Cinnamomum zeylanicum* Nees, indigenous to Sri Lanka and southern India, and for the most part now grown commercial in Sri Lanka, the Seychelles, and Madagascar. In the United States of America, the food, drug and cosmetic act of 1938 officially permits the term cinnamon to be used for a wider range of botanical sources including *Cinnamomum cassia* Blume, cultivate mainly in southern China, Burma(Myanmar) and Vietnam; *Cinnamomum burmannii* Blume, mainly from Indonesia and Sumatra (Tainter et al 2001). However, barks from other *Cinnamomum* species are frequently found as substitutes or adulterants. The uses of other *Cinnamomum* species may significantly affect its intended therapeutic values.

Most of the known compound responsible for the characteristic flavor and aroma of cinnamon are semivolatile compounds that can be isolated by method commonly employed for the analysis of essential oil [Farrell, 1999; Xiao and Satake, 1998; Jayatilaka et al 1995; Masada, 1976].

Essential oils basically consist of two classes of compounds, the terpenes and phenylpropenes. Depending on the number of 5-carbon building blocks (isoprene units); terpenes can be sub-divided into mono-, sesqui-, and di-terpene in which the numbers of isoprene units are 2, 3 and 4, respectively. Further derivatives of terpenes are typified by the presence or absence of a ring structure, double bond, addition of oxygen or stereochemistry. It is estimated that there are more than 1000 monoterpenes and 3000 sesquiterpenes. Phenylpropenes consist of a 6-carbon aromatic ring with a 3-carbon side chain ($C_6C_3$ compounds). Only approximately 50 phenylpropenes have been described. Eugenol and trans cinnamaldehyde are the important phenylpropene compounds (Lee et al 2004).

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The most important distinguishing feature of cinnamon oil was the cinnamaldehyde and eugenol content, typical levels being quoted as: cinnamon bark oil, cinnamaldehyde 55-75%, eugenol 5-18%, depending on the geographical source and method of production [Farrell, 1999; Ross, 1976].

The relatively non-specific methods may answer some questions but provide little information on the chemical signature that characterizes the quality of the spice and is required to determine its value and use. Hence, should evaluate the quality of those cinnamons and need to know simple, sensitive and reliable method to test their quality. The recent investigation indicates the sensitivities of the programmed GC-FID method.

## Materials and Methods

### *Materials used in the present study*

**Table 1** Sample of materials used for GC-FID analysis

| No. | Species | Localities | Voucher specimen number | Section |
|---|---|---|---|---|
| 1. | *C. cassia* Presl | Yunnan,, China | CLC 002 | Cinnamomum |
| 2. | *C. burmannii*(C.G. & Th. Nees.) Bl. | Yunnan, China | CLC 005 | Cinnamomum |
| 3. | *C. tamala* Fr. Nees. | Yangon, Myanmar | MLC501 | Cinnamomum |
| 4. | *C. multiflorum* Wight.. | Yangon, Myanmar | MLC502 | Cinnamomum |
| 5. | *C. obtusifolium* (Roxb.) Nees | Yangon, Myanmar | MLC503 | Cinnamomum |
| 6. | *C. inunctum* (Nees.) Meissn | Yangon, Myanmar | MLC504 | Camphora |
| 7. | *C. camphora* (L.) Presl. | Yangon, Myanmar | MLC505 | Camphora |
| 8. | *C. chartophyllum* H.W.Li | Yunnan, China | CLC006 | Camphora |
| 9. | *C. parthenoxylon* (Jack) Nees. | Yunnan, China | CLC007 | Camphora |
| 10. | *C. glanduliferum* (Wall.) Nees. | Yunnan, China | CLC008 | Camphora |

The origins of materials are shown in the Table 1. All of them were collected botanical origin from field during spring. The voucher specimens were deposited in the department of Chinese Materia Medica Analysis, China Pharmaceutical University.

### *Extraction*

The fresh samples were washed thoroughly with water and air dried. The extraction from 5g air-dried and grounded stem-barks was carried out with 50mL diethyl ether as organic solvent, using an ultrasonic bath for 30 min to ensure an exhaustive extraction and then filtered. The soluble fraction 25mL was concentrate to 1mL. All samples were extracted by the same procedure and stored at -4°C for further use.

### *GC analysis*

The quantification of the components of the extraction was accomplished with a Hewlett-Packard gas chromatograph model 6890 series, equipped with an FID. A fused-silica capillary column (30 m long, 0.32 mm i.d., 0.25 $\mu$m thickness), coated with a nonpolar stationary phase (Hewlett-Packard-5, crosslinked 5% phenyl methyl silicone) was used. The

temperature program began at 50°C with an increase of 5°C $min^{-1}$ up to 250°C (15min). Nitrogen was used as the gas carrier. Injector and detector temperatures were 250 and 280°C, respectively. The injection technique used was split with a split ratio 10:1, and the injection volume was 1*u*L. Identification of the three major components of cinnamon barks oils were confirmed by comparison with standards. Chinese National Institute for the Control of Pharmaceutical and Biological Products was purchased as references standards.

## Results and Discussion

***Linearity and sensitivities of the programmed GC method***

Under the chosen programmed temperature GC conditions, good linearity was observed for camphor, cinnamaldehyde and eugenol. Detection limits (DLs), defined as signal-to-noise ratio of 3 objective compounds under the chosen GC conditions. The DLs were approximate to or better than that of most of the reported HPLC and GC methods (Smith et al 2001; Usta et al 2003).

The developed GC method was applied to determine camphora, cinnamaldehyde and eugenol in ten *Cinnamomum* species samples. Figure 2 to Figure 11 were the typical chromatograms of recent investigated samples, respectively.

***Study of three compounds detected in the extracts of ten Cinnamomum spp.***

The concentration of camphor, cinnamaldehyde and eugenol were calculated. Retention time ($t_R$) of camphor, cinnamaldehyde and eugenol were 7.9 min, 9.0 min and 9.8 min respectively.

Compares values across categories of compound concentrations of those three compounds are shown with 3D column in Figure 1 *C. pathenoxylon* possessed the highest concentration of camphor. While *C. cassia* possessed the highest concentration of cinnamaldehyde and the highest eugenol concentration was found in *C. tamala* extract. In the investigated species, *C. glanduliferum* was obviously poor in those three compounds.

The present programmed temperature capillary GC method for simultaneous determination of camphora, cinnamaldehyde and eugenol is rather simple, sensitive and reliable. It was satisfactorily applied to simultaneously measure the 3 compounds in ten different species. The method is promising for use in the quality control of the pharmaceutical preparations.

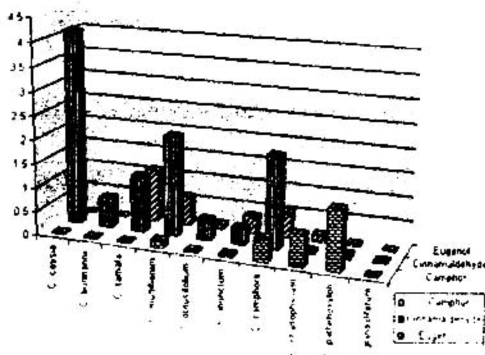


**Figure 1** Relative peak areas (RPA) of the GC-FID fingerprints of 10 different species samples

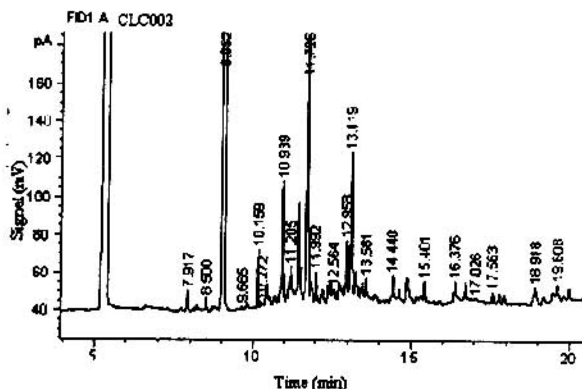

**Figure 2** Chromatogram of *C. cassia* sample under the chosen GC condition.

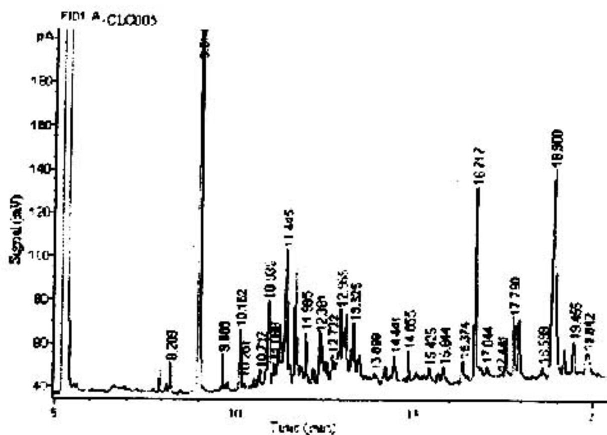

**Figure 3** Chromatogram of *C. burmannii* sample under the chosen GC condition

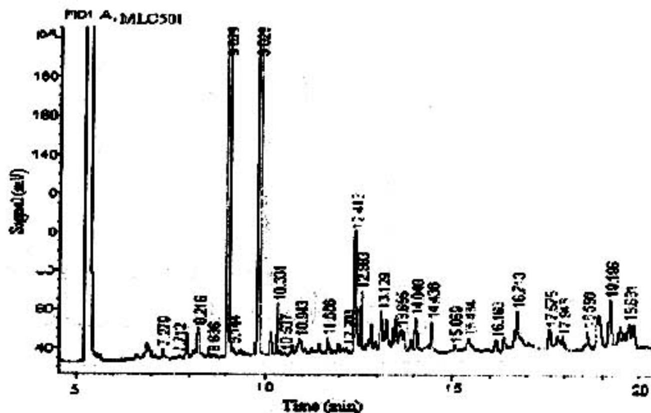


**Figure 4** Chromatogram of *C. tamala* sample under the chosen GC condition

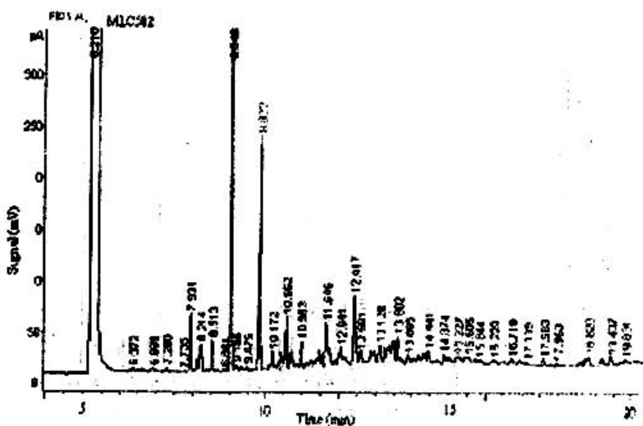

**Figure 5** Chromatogram of *C. multiflorum* sample under the chosen GC condition

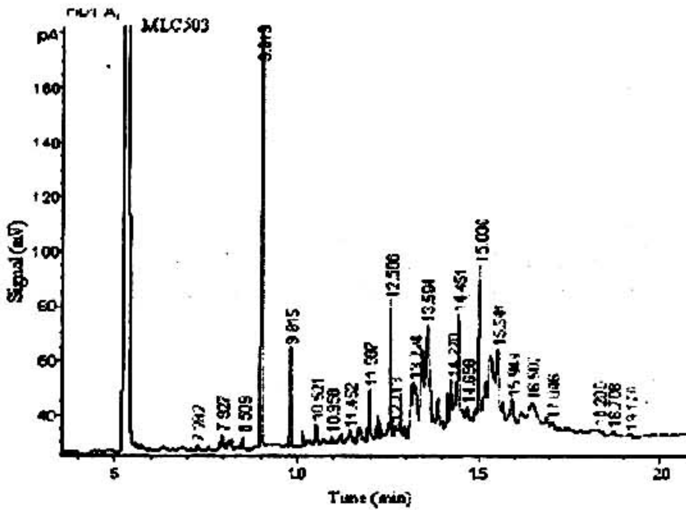


**Figure 6** Chromatogram of *C. obtusifolium* sample under the chosen GC condition

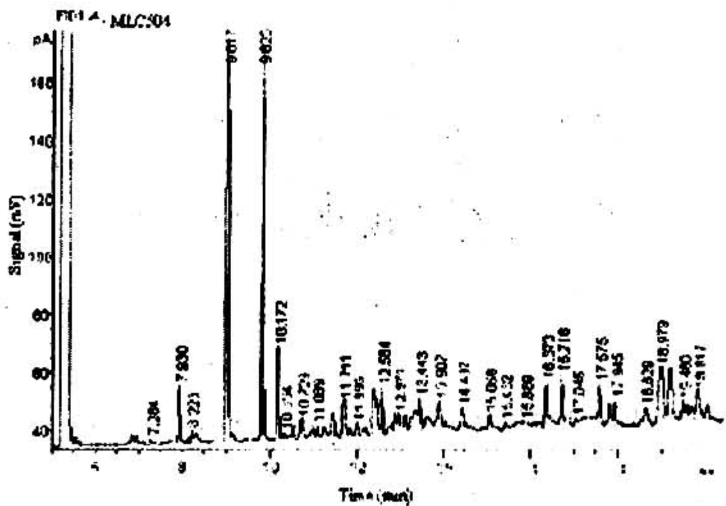


**Figure 7** Chromatogram of *C. inunctum* sample under the chosen GC condition

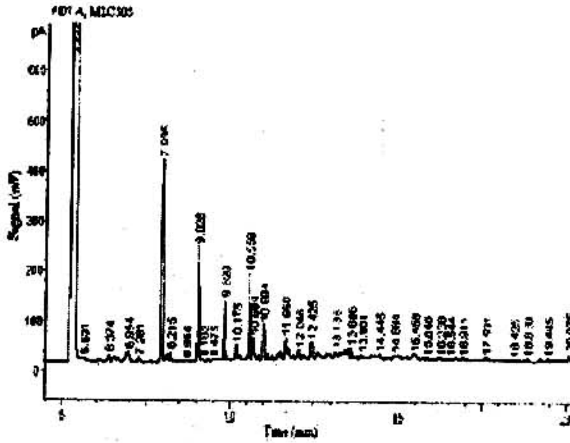

**Figure 8** Chromatogram of *C. camphora* sample under the chosen GC condition

**Figure 9** Chromatogram of *C. chartophyllum* sample under the chosen GC condition

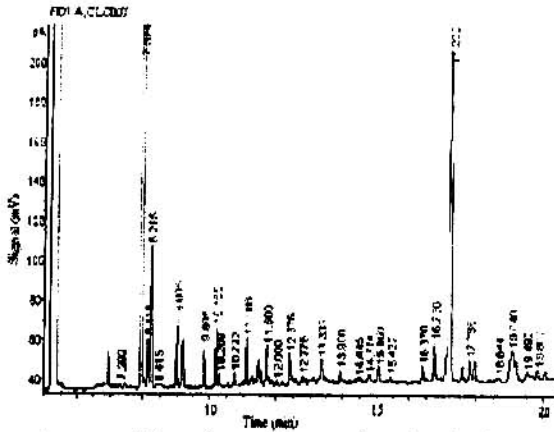


**Figure 10** Chromatogram of *C. pathenoxylon* sample under the chosen GC condition

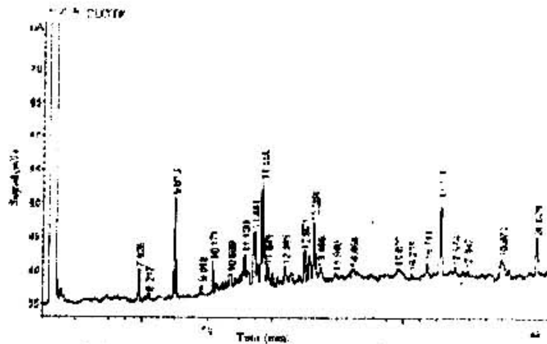

**Figure 11** Chromatogram of *C. glanduliferum* sample under the chosen GC condition

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