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Research Article

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The Contamination maybe from the Plastic Seal Identified in the Essential Oils of *Citrus Kinokuni* Tanaka

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ABSTRACT:

In the study of the essential oils of Citrus kinokuni Tanaka, a series of compounds which have strong characterization that their base peak is 149 (100), and the intensity of other peaks is low, usually lower than 15. Such kinds of compounds maybe from the plastic seal in the course of the analysis of the essential oil. These compounds are 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, Dibutyl phthalate, and 1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester. As a result, in the analysis of essential oil, if these four compounds were identified, they should be thought as contaminations.

Keywords: Essential oil; 149; 1,2-Benzenedicarboxylic acid, Bis(2-methylpropyl) ester; 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester; Dibutyl phthalate; 1,2-Benzenedicarboxylic acid; Mono(2-ethylhexyl) ester

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INTRODUCTION

In the study of the essential oils of *Citrus kinokuni* Tanaka, a series of compounds which have strong characterization that the base peak is 149 (100), and the intensity of other peak is low, usually lower than 15. Such kinds of compounds should not be from the essential oil of *C. kinokuni*, but maybe from the plastic seal in the injection of the essential oil. The base peak as 149 (100) is as the following formula (Figure 1).

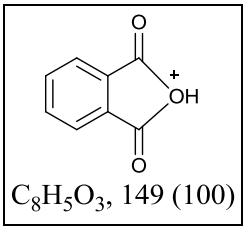

Figure 1: The base peak 149 (100)

EXPERIMENTAL SECTION

Materials

The peels of *C. kinokuni* Tanaka were collected each month from June, 2012 to November, 2012 from the same plant grown in Nancheng town, Fuzhou city, Jiangxi province of China.

Chemical Standards and Reagents

Hexane (HPLC grade) was purchased from Adamas Reagent Company, Ltd. *n*-Alkane standard solution of C_{10}–C_{25} purchased from Dr. Ehrenstorfer GmbH, Germany, was used to determine linear retention index (LRI).

EO Extraction

The distillation was used to extract the EOs in the peels. The peels of *C. kinokuni* Tanaka divided into about 0.4 cm × 0.4 cm sections weighed between 30.0-40.0 g were swollen with about 10 times volume (v/w) 250-450 mL of pure water in a 500 or 1000 mL round-bottomed flask, then soaked for 0.5 h at 40°C. The EO was extracted by Clevenger-type apparatus for 3-4 h. The EOs was prepared according to the procedure described in Chinese pharmacopoeia (Pharmacopoeia committee of the People's Republic of China, 2010). The EOs was stored in separate screw-capped vials in a refrigerator at 4 ˚C until needed. The EO was diluted as V_{oil}: V_{hexane} equal to 1: 50 for detection by GC-MS.

GC-MS analysis

An Agilent 7890B gas chromatograph (USA) matched with an Agilent 5977A mass spectrometer was used for GC-MS analysis. The gas chromatograph was coupled with an Agilent fused silica capillary column DB-5 (30 m × 0.25 mm i.d., 0.25 μm film thickness). The oven temperature was programmed from 60°C (3-min hold) to 270°C at 3°C min^{-1}, and then held for 8 min. Scan at 3.9 scans s^{-1} from m/z 30 to 400 amu. The injection volume was 1 μl of a 1: 50 n-hexane solution to obtain the appropriate peak intensity [1].

Component Identification

The peaks in the TICs obtained by GC-MS were identified by probability based matching (PBM) searching first. Then, the mass spectrum of these peaks was compared with the National Institute of Standard and Technology (NIST) 08 database. The LRIs of peaks were calculated and also compared with the LRIs of the corresponding matched chemicals provided by NIST 08 database or references.

RESULTS AND DISCUSSION

Four such kind compounds were identified in the essential oils of *C. kinokuni* Tanaka which can be seen in Table 1.

Table 1: The information about the four components

Component	Formula	Molecular Weight	CAS	Characterized ion peaks (M/W, %)	LRI (NIST08 or Ref.)	RI	RT
1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester	$C_{16}H_{22}O_4$	278	84695	149 (100) 57 (12) 150 (9.3) 104 (6.0) 223 (5.7) 41 (4.7) 76 (4.0) 167 (3.0) 56 (3.0) 93 (1.9) 65 (1.9) 121 (1.6) 205 (1.5) 132 (1.4) 224 (0.7) 225 (0.1)	1863[N]	1861	43.663
1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester	$C_{16}H_{22}O_4$	278	17851535	149 (100) 150 (9.1) 57 (6.2) 223 (5.4) 104 (4.9) 41 (4.4) 76 (4.0) 205 (3.0) 56 (2.4) 105 (2.2) 65 (2.1) 93 (1.6) 121 (1.7) 167 (1.2) 132 (0.9) 224 (0.6)	1900[N]	1906	45.133
Dibutyl phthalate	$C_{16}H_{22}O_4$	278	84742	149 (100) 150 (8.7) 223 (3.9) 104 (3.8) 205 (3.5) 76 (3.1) 41 (2.9) 56 (2.0) 105 (1.8) 65 (1.8) 93 (1.7) 121 (1.5) 224 (0.5) 160 (0.5) 278 (0.2) 279 (tr)	1957[2]	1954	46.665
1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester	$C_{16}H_{22}O_4$	278	4376209	149 (100) 167 (29) 57 (21) 71 (17) 70 (13) 150 (11) 43 (11) 55 (8.6) 41 (8.6) 113 (7.8) 279 (7.3) 112 (5.9) 132 (2.1) 280 (1.4) 93 (1.2) 65 (1.2)	2550[N]	2541	63.188

Note: tr (Trace) means the value is less than 0.05. [N] after the LRI value means it is provided by NIST 08 MS database. Ref. equal reference.

To correct analysis the essential oil of *C. kinokuni*, these kinds of compounds should be identified and be thought as contaminants. From Figure 2, it can be seen that the structure of compounds as 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, and Dibutyl phthalate, are very alike.

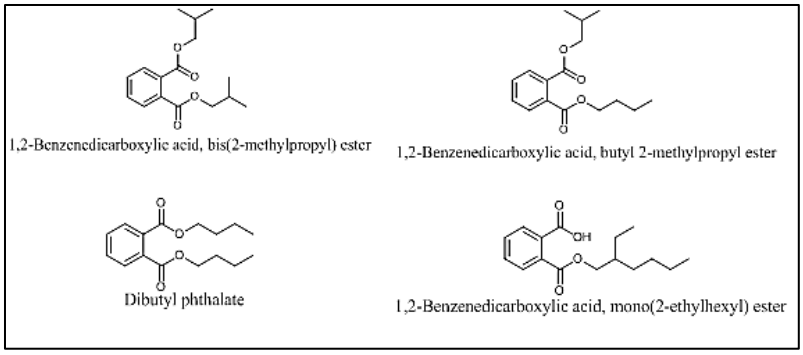

Figure 2: The structure of four compounds

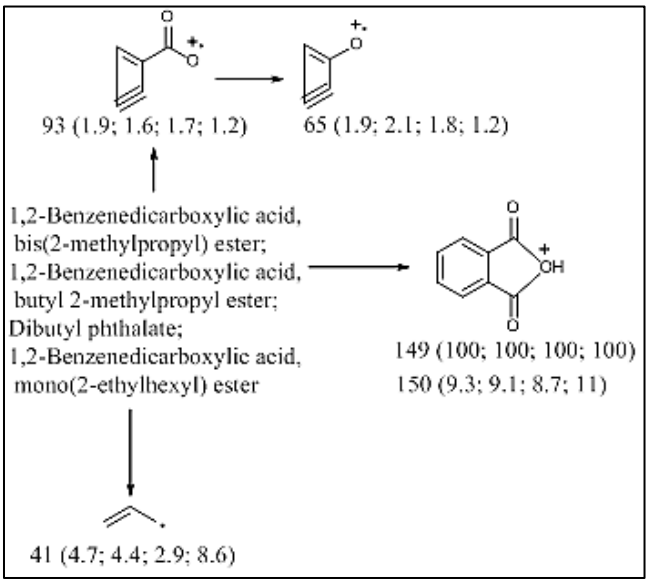

Figure 3: The common dissociation pathway of 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, Dibutyl phthalate, and 1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester

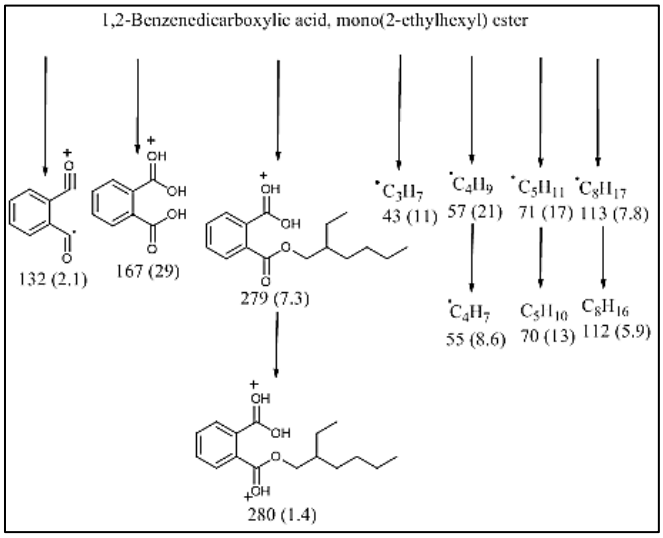

Figure 4: The other dissociation pathway of 1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester

The dissociation pathway of 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, Dibutyl phthalate, and 1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester can

be seen in Figure 3-6. The dissociation pathway of 1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester is different from that of the other three compounds greatly which can be seen in Figure 4, while the dissociation pathway of 1,2-Benzenedicarboxylic acid, bis (2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, and Dibutyl phthalate are more alike which can be seen in Figure 5.

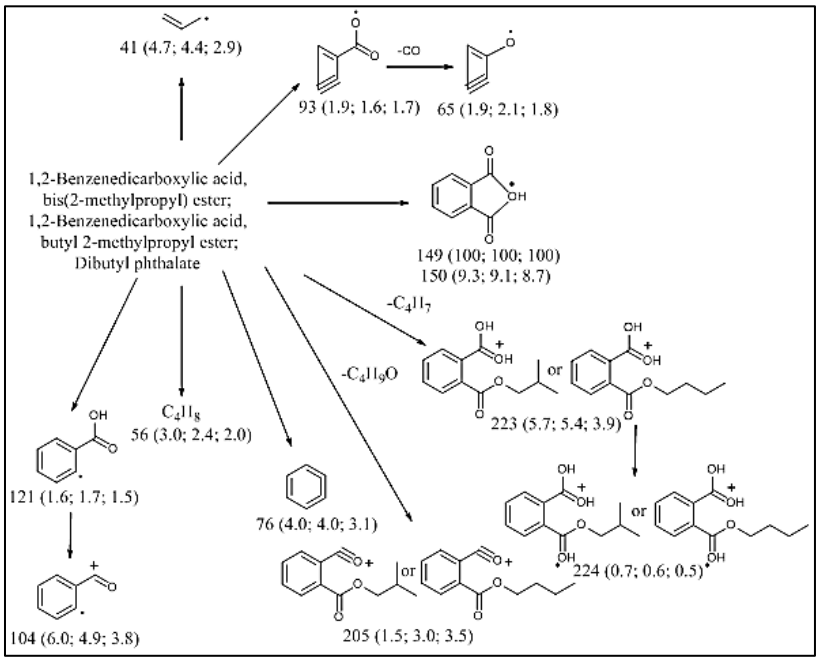

Figure 5: The common dissociation pathway of 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, and Dibutyl phthalate

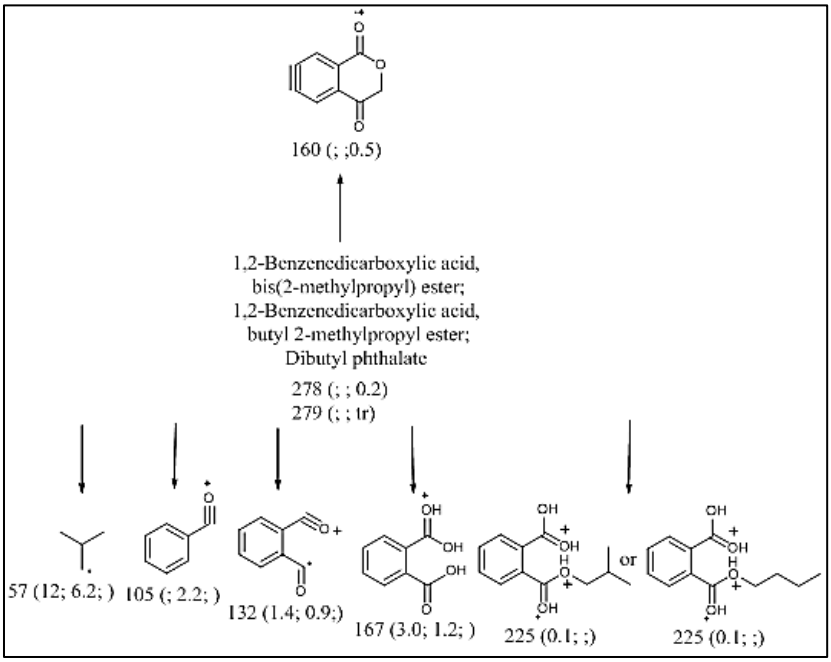

Figure 6: The other dissociation pathway of 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester, 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester, and Dibutyl phthalate

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