



# The Transfer of Some Fatty Acid Flavors in Cigarettes

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### Introduction

- Organic acids influence cigarette quality, the acid flavors are often added into cigarettes in order to improve aroma and taste of cigarettes.
- Over the years, papers about the transfer of flavors in cigarettes have been published, but study on the transfer of fatty acid flavors in cigarettes has not been reported yet.
- In our lab, 10 fatty acid flavors were injected into cigarettes and analyzed.



### Our objective

- To study the transfer behavior of some important fatty acid flavors in cigarettes
- To obtain some data which maybe helpful in using the flavors and developing low tar cigarettes



### Experiment

Materials

> cigarette sample

blank cigarettes: special 84-mm flue-cured cigarettes without flavored were prepared



## Ten standard compounds

No.	Standard compounds	Molecular weight	Boiling point
1	3-Methyllbutanoic acid	102.13	176.5
2	Valeric acid	102.13	185.5
3	3-Methylvaleric acid	116.16	198.0
4	Hexanoic acid	116.16	205.8
5	Heptanoic acid	130.19	223.0
6	Octanoic acid	144.21	239.7
7	Nonanoic acid	158.24	255.6
8	Decanoic acid	172.27	270.0
9	Dodecanoic acid	200.32	300.0
10	Tetradecanoic acid	228.38	326.0



## Reagents

- Extract Solvent:
  - > CH<sub>2</sub>Cl<sub>2</sub>, GC grade

- Internal standard:
  - > 2-phenethyl acetate, GC grade



#### **Apparatus**

20 channel linear automatic smoking machine

Simultaneous Distillation and Extraction Equipment (SDEE)

Agilent6890GC (FID) , Agilent 6890GC/ Agilent 5973MS



## Analysis procedure of 10 fatty acid flavors in flavored cigarettes

- Ten fatty acid flavors were dissolved in ethanol and then injected into cut tobacco
- Condition flavored cigarettes at 60% relative humidity and 22°C in airproof cigarette packets for 1 week
- The transfer ratios of 10 fatty acid flavors in flavored cigarettes were determined with SDEE, GC, and GC/MS.



# The transfer of 10 fatty acid flavors in flavored cigarettes

- Before smoking
  - > The retention ratios in cut tobacco
  - > The immigration ratios to filter tip
  - > The lost ratios to ambient
- After smoking
  - > The transfer ratios to particulate phase of mainstream smoke
  - > The absorption ratios of filter tip
- Flavored cigarettes were smoked according to ISO3402.



#### Separation scheme of testing sample

```
Testing sample
            SDEE with CH<sub>2</sub>Cl<sub>2</sub> (2 hours)
    Extract
            extracted with 5% NaOH
   Basic phase
            adjusted pH=1 ~ 2 with 20% HCl
            extracted with CH<sub>2</sub>Cl<sub>2</sub>
  CH,Cl, phase
            dried
            concentrated
GC, GC/MS analysis
```



#### GC conditions

- Agilent 6890GC
- Column: DB-WAXETR  $(30m \times 0.25mm \times 0.25\mu m)$
- Programming temperature:
  - 60°C (1min) to 210°C(30mins) at 2°C/min
- Detector : FID



#### GC/MS conditions

- Agilent 6890GC/Agilent 5973MS
- GC Conditions: same as above

Mass spectral library: NIST98



#### Results and discussion

#### **Method validation**

- The internal standard method was used for the quantitative determination of flavor compounds.
- The recovery and repeatability were determined.



#### Method validation

- Recovery
  - **>** 1 compound : 60.8%
  - > 9 compounds: 76.1% ~ 87.3%
- ◆ Variation coefficient <5%</p>

LOQ: 0.02 ~ 0.18 μg/cig

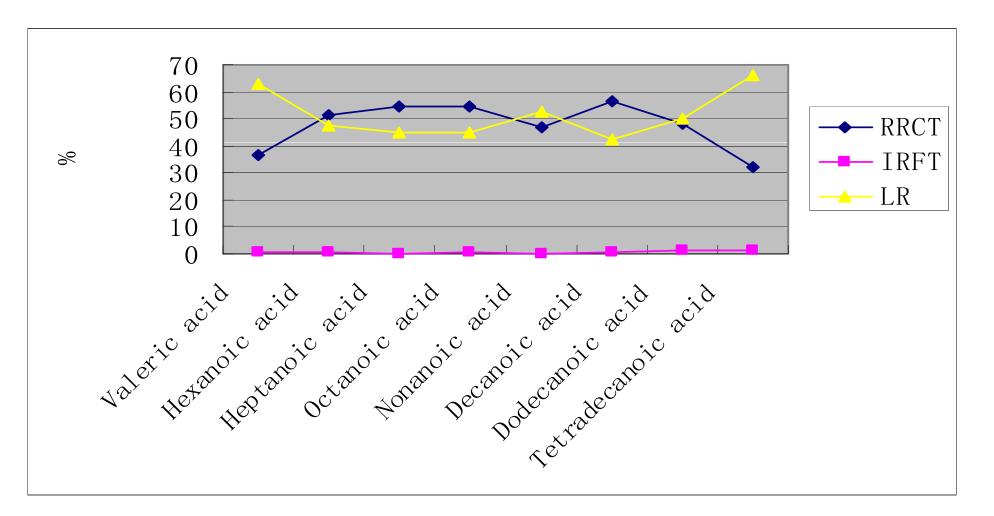


## The distribution of fatty acid homologous compounds before smoking

**RRCT:** the retention ratios in cut tobacco

LR: the lost ratios to ambient

IRFT: the immigration ratios to filter tip



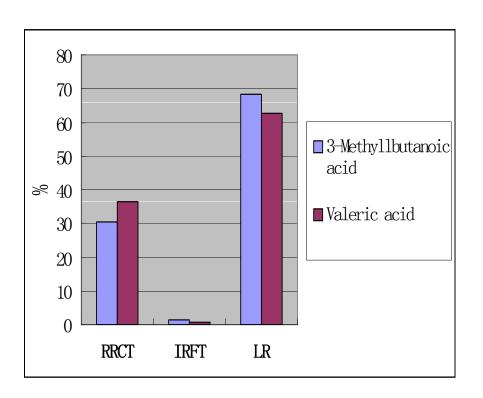


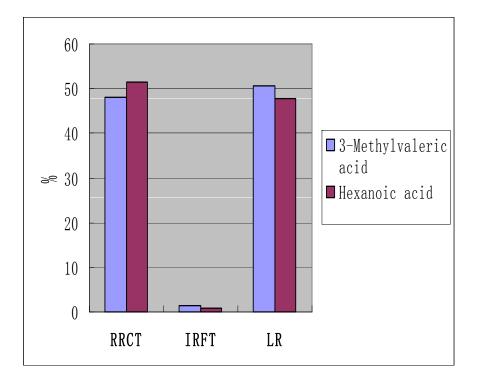
## The distribution of fatty acid isomers before smoking

**RRCT:** the retention ratios in cut tobacco

LR: the lost ratios to ambient

IRFT: the immigration ratios to filter tip





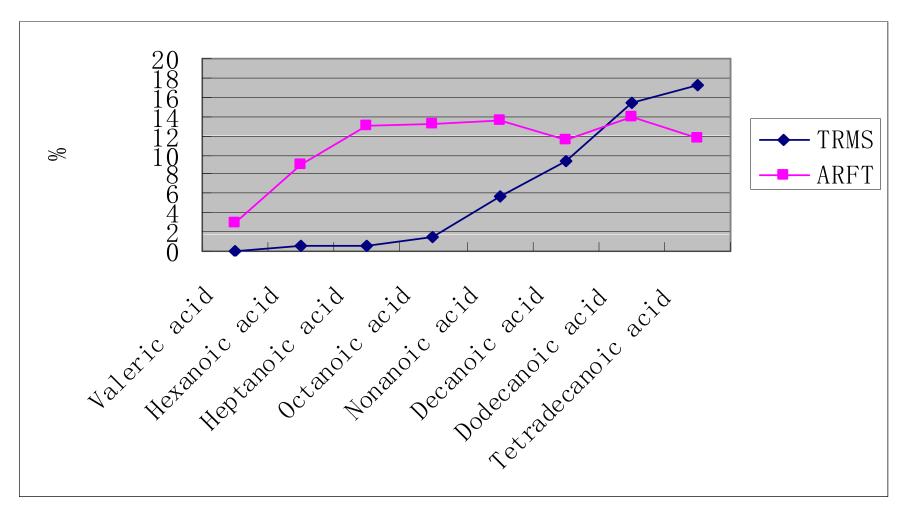
The boiling point: 3-Methyllbutanoic acid < Valeric acid
3-Methylvaleric acid < Hexanoic acid



## The transfer of fatty acid homologous compounds after smoking

TRMS: the transfer ratios to particulate phase of mainstream smoke

ARFT: the absorption ratios of filter tip

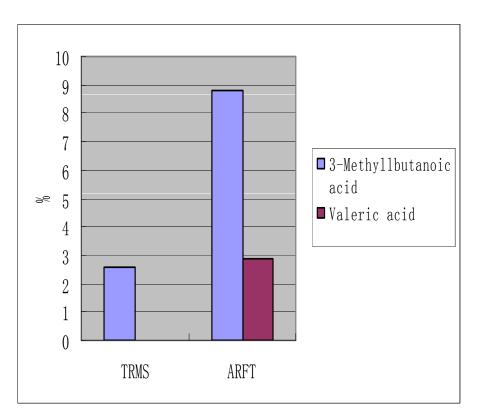


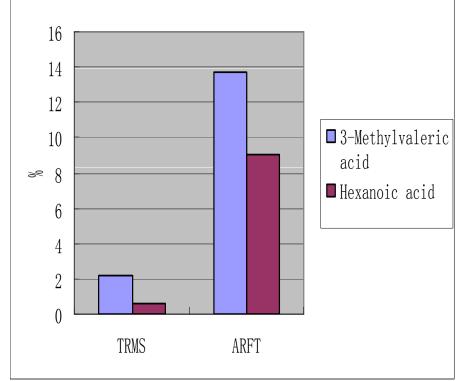


## The transfer of fatty acid isomers after smoking

TRMS: the transfer ratios to particulate phase of mainstream smoke

**ARFT:** the absorption ratios of filter tip







### **Conclusions**

- For fatty acid homologous compounds flavors
  - > the larger molecular weight and higher boiling point were, the higher the immigration ratios to filter tip and the transfer ratios to particulate phase of mainstream smoke would be
  - > the retention ratios in cut tobacco, the lost ratios and the absorption ratios of filter tip changed irregularly



### Conclusions

- two pairs of fatty acid isomers had similar transfer behavior
  - > for the same molecular weight fatty acids, the lower boiling point generally associated with lower retention ratio in cut tobacco, and relatively high the lose ratio, transfer ratio to particulate phase of mainstream smoke, immigration ratio and absorption ratio of filter tip.
  - > Moreover, the transfer ratios to mainstream smoke, and the absorption ratios of filter tip significantly differed between the two isomers.





## Thanks for your attention!

