



DAIKIN Advanced Analysis Method for C6/C8 **(DAIKIN AAM)**

1. Introduction

This analysis method is designed for simple and accurate determination of whether a fabric is treated with C6 or C8 PFC product.

Traditionally, a difficult extraction/LC-mass analysis method has been used as C6/C8 identification method. This traditional method, however, is most useful to quantify impurities (PFOA etc.) in PFC products before and after application to final products like fabric, and is not well suited to simple confirmation of whether articles have been treated with C6 or C8 product.

This new method can be applied, not only to distinguish C6/C8, but also to determine C4, or fluoro-chains longer than C8.

2. Apparatus and Materials

- Head Space Vial (5ml)
(PTFE/Silicone septum, Aluminum cap)
- GCMS or GC
(Note; There is the possibility that C8 or longer fluoro-chain cannot be detected by GC only (FID) due to its detection limit. GCMS is recommended for correct analysis.
- Headspace Autosampler (Perkin-Elmer and Agilent are available)
or
Gas-Tight Syringe and Oven (100- 200C)
- Standard Reagent
Available from Sigma-Aldrich.
 - C6: Perfluorohexylethanol (CAS# 647-42-7)
CF3CF2CF2CF2CF2CF2-CH2CH2-OH
 - C8: Perfluorooctylethanol (CAS# 678-39-7)
CF3CF2CF2CF2CF2CF2CF2CF2-CH2CH2-OH



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3. Method description

The method is based on 1) significantly degrading fluorotelomer monomers and polymers to alcohol by severing the ester-bond with heat, and 2) measuring the resulting alcohol.

<Sample preparation>

- 1) Prepare a 100 mg fabric sample, or in any case one larger than 3cm x 3cm.
- 2) Place the fabric sample into headspace vial.
- 3) Seal the headspace vial with septum and cap.

<Headspace GC analysis>

- 1) Heat the headspace vial at 200C for 30min in oven. (Use of an autosampler is preferred.)
- 2) Take 2ml of gaseous phase sample from headspace vial and inject into GC.

<GC conditions>

See 5. Actual Results below. The conditions are not limited to our conditions.

4. Data analysis

The signal for identification C6/C8 is detected as Perfluorohexylethanol (C6)/ Perfluorooctylethanol (C8).

<In case of mass available>

- C6: $\text{CF}_3\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{-CH}_2\text{CH}_2\text{-OH}$:MW 364
- C8: $\text{CF}_3\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{-CH}_2\text{CH}_2\text{-OH}$:MW 464

<In case of mass unavailable>

Compare the retention time with a standard reagent.

5. Actual Result

<Equipment>

GC/MS: Perkin-Elmer GC Auto System XL + Turbomass
Column: DB624 60m x0.32mm, 1.8mm (Agilent Technologies)
Initial 50C for 5min, 10C/min to 250C (hold for 5min)
INJ: 250C
Transfer line Temp.: 250C
Carrier: He, 1.4ml/min constant flow
Split: 1.4:20
Sample quantity: 2ml
Ionization: Electron impact (EI)
Ionization Voltage: 70V



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Type of experiment: Scan

Scan Rang: 10~600

<Result>

Retention Time: C6 15.4-15.5min
C8 17.4min
C10 19.0min

<Fabric>

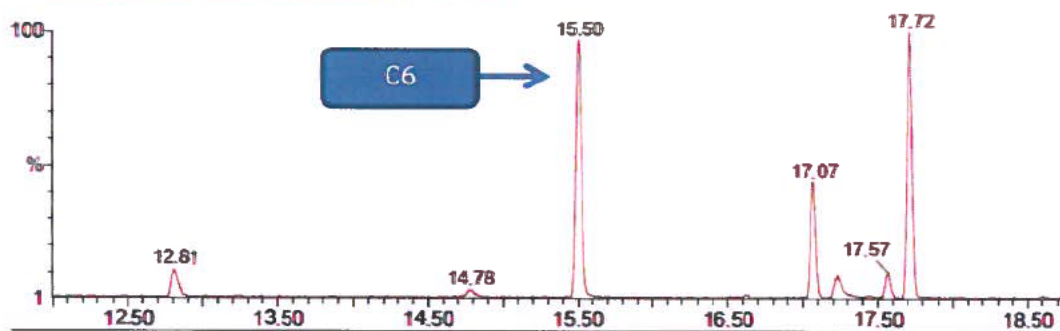
Nylon 15d Taffeta

<Recipe>

C6 FC (30%solid) : 2%

WPU: 32%

Weight of 3cmX3cm analysis sample: 33mg



<Fabric>

Polyester 20d Dobby

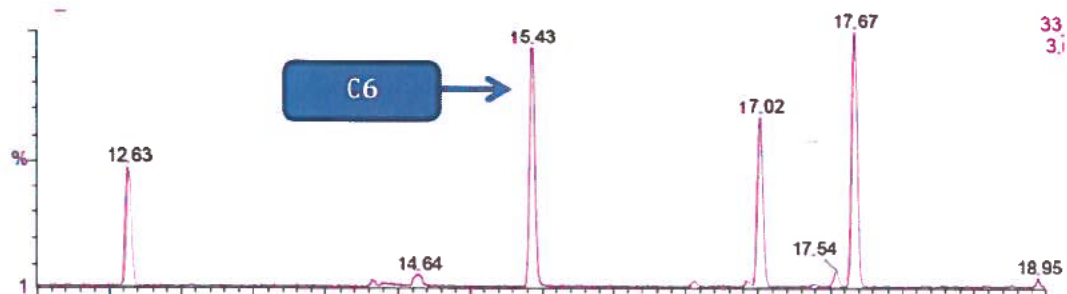
<Recipe>

C6 FC (30%solid): 4%

Si softener: 2%

WPU: 44%

Weight of 3cmX3cm analysis sample: 38mg



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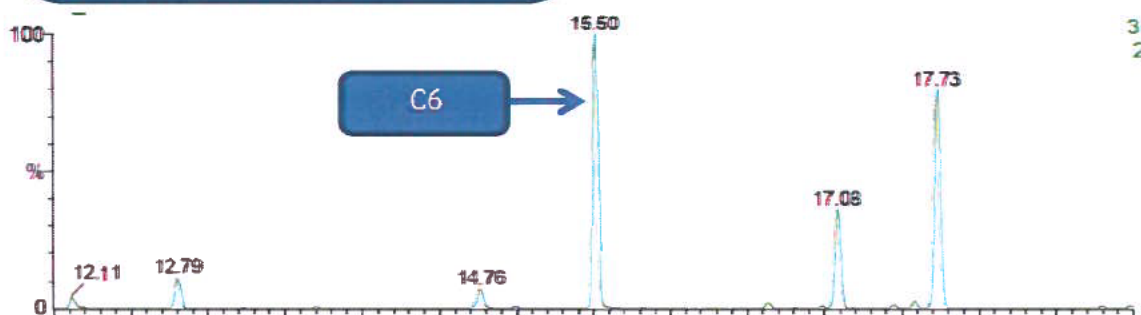
<Fabric>
Nylon 30d Dobby (Acrylic coating)

<Recipe>
C6 FC (30%solid): 4%
Cross Linker: 2%

After FC treated, acrylic resin was coated

WPU: 52%

Weight of 3cmX3cm analysis sample: 132mg

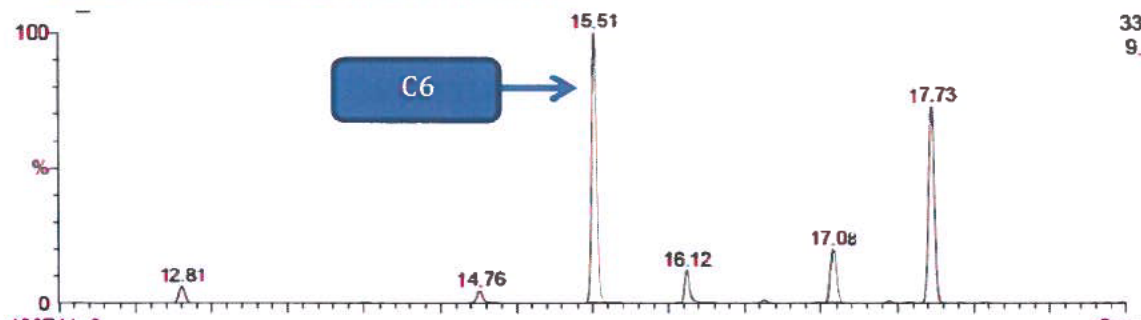


<Fabric>
Cotton Twill

<Recipe>
C6 FC (30%solid): 4%

WPU: 65%

Weight of 3cmX3cm analysis sample: 219mg





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<Fabric>

Cotton Twill

<Recipe>

C8 FC (30%solid) 6%

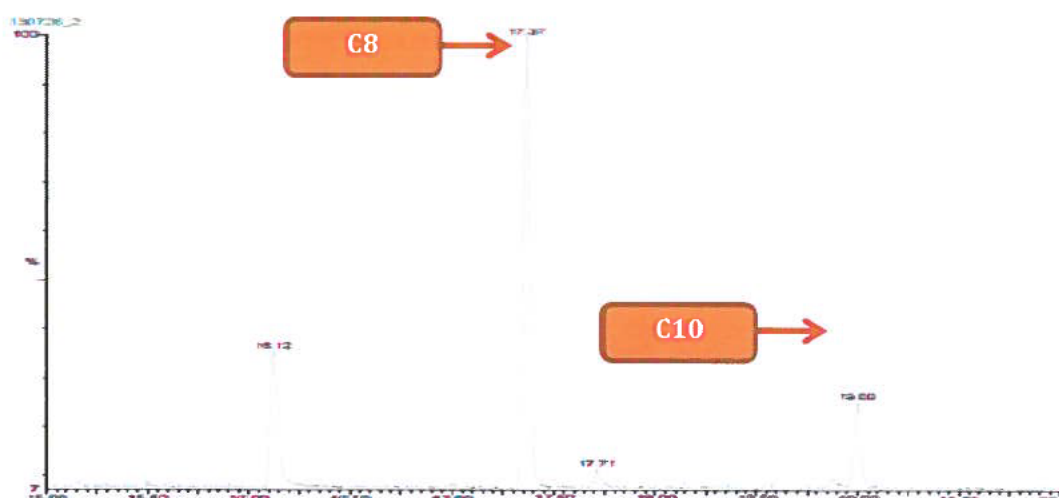
Glyoxal Resin 4%

PE softener 2%

Cross Linker 1%

WPU: 65%

Weight of 3cmX3cm analysis sample: 204mg



<Fabric>

Nylon 30d Dobby (Acrylic coating)

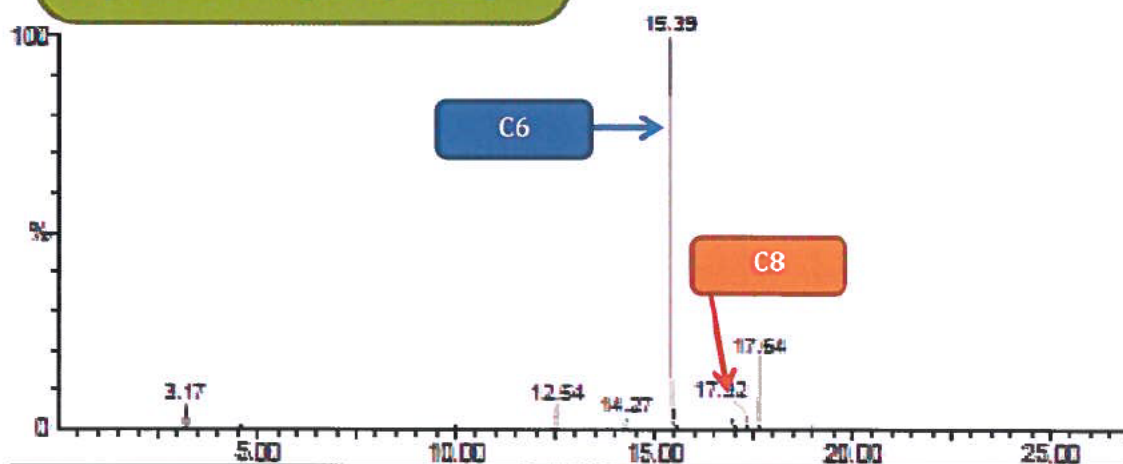
<Recipe>

C6 FC (30%solid): 1.8%

C8 FC (30%solid): 0.2%

WPU: 52%

Weight of 3cmX3cm analysis sample: 128mg



DAIKIN Patent pending.



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<Misleading Peak>

Retention Time:	C6	15.4-15.5min
	C8	17.4min
	C10	19.0min

rt (min)	Derivation
15.1	8:2-Cl (C8), Derivative from the FC polymer that is co-polymerized with vinyl chloride
15.5	6:2-OH (C6)
17.1	Derivative from Non FC co-monomer
17.4	Derivative from blocked isocyanate that was used with FC as cross-linker
17.5	8:2-OH (C8)
17.7	Derivative from Non FC co-monomer. This peak is shown with 17.1min peak

6. Test Report

Report "Positive" or "Negative" that are chosen from complying with following requirement

- Positive : > 5ppm*
 - Negative : Non detected
- * ppm: against fabric weight

7. MeOH Washing Method

This method is the pre-treatment for verification when C8 and/or C8 related compound is detected and reported as "Inconclusive".

<MeOH washing>

- 1) Put 3cm x 3cm fabric sample into 9ml of glass vessel
- 2) Charge 5g of MeOH in the vessel
- 3) Place the vessel inside an ultrasonic bath for 10min
- 4) Get the fabric out of the vessel and take off extra MeOH with Kimwipe.
- 5) Repeat 2) - 4)
- 6) Put the fabric sample in vacuum dryer

8. Safety

<Potential Health Effects>

Vapors and fumes liberated during heat processing (above 200 C) with the material.
Above 280 C, hydrogen fluoride and other toxic gas may be produced.
Harmful if thermal decomposition products are inhaled.



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- Use suitable, heatproof/pressure resistance, headspace vial
- Use personal protective equipment
 - Protective glove (Heatproof, -200C)
 - Safety glasses
 - Respirator (organic vapour/acid gas respirator)

9. Contact Information

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