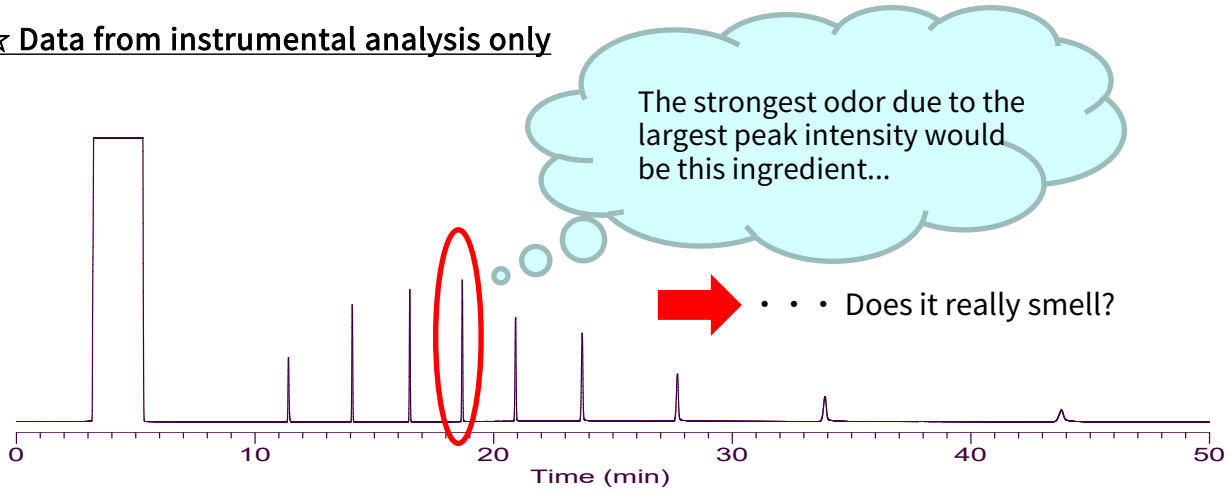
GC/O Analyses by Simplified Enrichment of Volatile Components in Children's Cold Syrups

We screened and analyzed the volatile constituents contained in commercial cold syrups, using HandyTD TD 265 and a simplified enrichment-tool MonoTrap RGC18 TD. Heatspace gas was collected by MonoTrap and then heat-introduced by HandyTD and analyzed by GC/FID/O as well as GC/MS. We also analyzed a sample of cold syrup supplemented with 2 and 6-Dichlorophenol, assuming a situation in which an offensive odor is present from cold syrup. (Sample concentration: 1 mg/L)

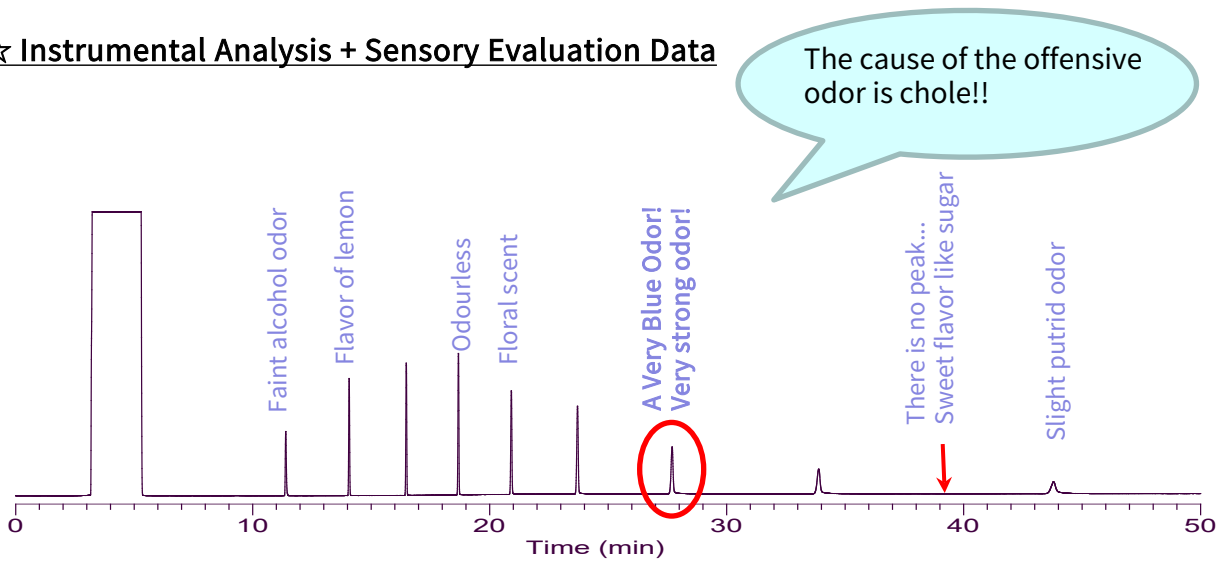
Why Sensory Evaluation is Necessary

In the event of odor-related problems such as an offensive odor claim, it is necessary to identify the compounds that caused the offensive odor. Most of the "odor" compounds that reach the nose are highly volatile compounds, so gas chromatographs are used as analytical instruments. However, such trace amounts of compounds that cannot be detected by gas chromatographs may be key components of the off-flavor, or the intensity balance between the chromatogram's peak intensity and odor may be different. Sensory assessment is also a very effective tool for filling the gap between the analyzer and the human nose.

☆ Data from instrumental analysis only



☆ Instrumental Analysis + Sensory Evaluation Data

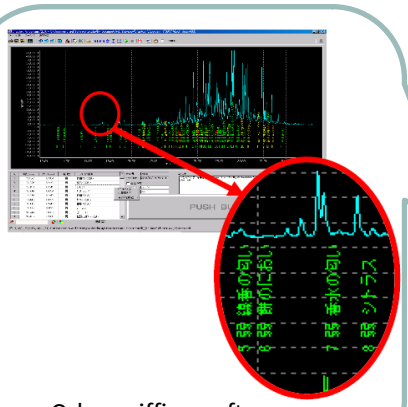


The actual smell of odors from peak to peak allows us to identify the compound with the greatest contribution or to feel the odor in the area without the peak.

Analysis System

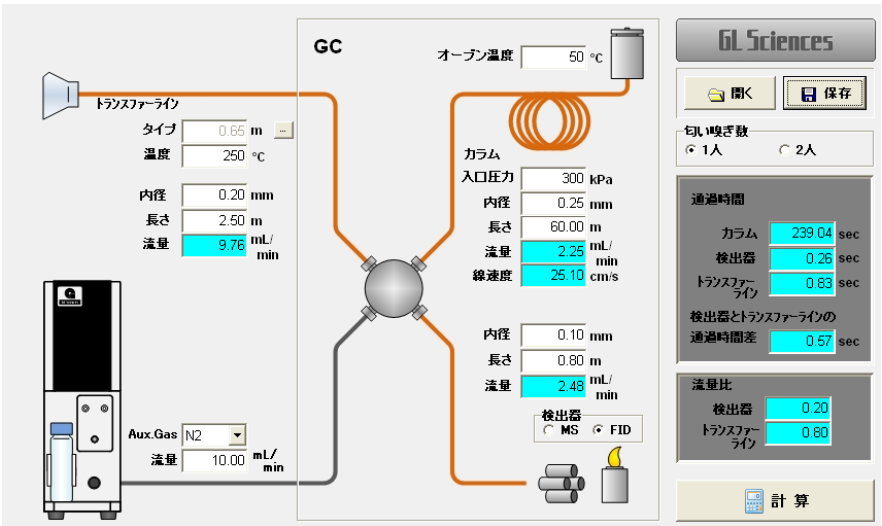
The method used for sensory assessment is GC/O [Gas Chromatography/Olfactometry. GC/O is a method of branching the analytical column outlet of GCs, connecting one of them to detectors such as FIDs and MSs for component analysis, while at the same time the operator sniffs the odor with the nose.

≪GC/Olfactometry≫



Odor sniffing software
Olfactory Voicegram

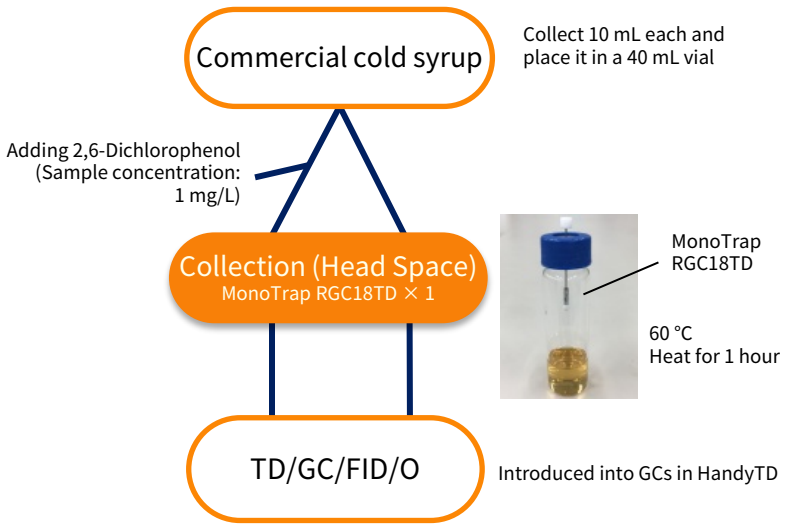
Sniffing port
OP275 Pro



Bifurcate the exit of the GC column inside the GC oven, carrying gas to the detector such as FID or MS, and simultaneously to the operator side (nose) on the other side.

In the present study, we conducted sensory evaluations by connecting the pipes so that the branching ratio of FID:transfer line = 2:8 according to the left panel (Split Manager).

Preliminary processing procedure



GC/FID/O Conditions

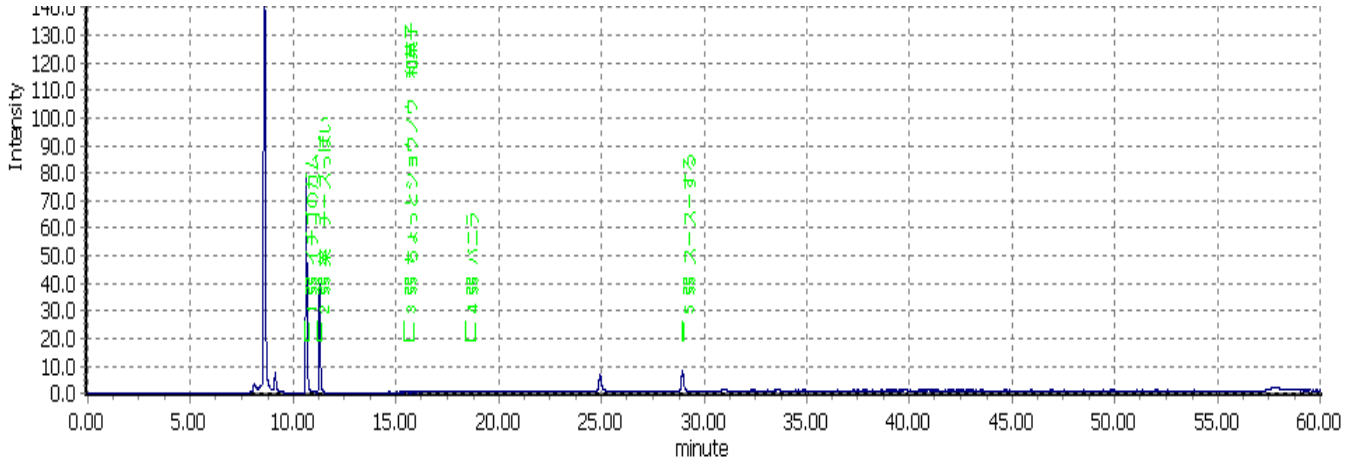
System	: Thermal Desorption-GC/FID/O (HandyTD TD265)
Column	: InertCap Pure-WAX 0.25 mm I.D. × 60 m, df = 0.5 µm
Col.Cat. No.	: 1010-68164
Col.Temp.	: 50 °C(1 min) - 10 °C/min - 240 °C
Carrier Gas	: He, 300 kPa
GC Inlet	: 250 °C, Split 5:1
Detection	: 300 °C

HandyTD Conditions

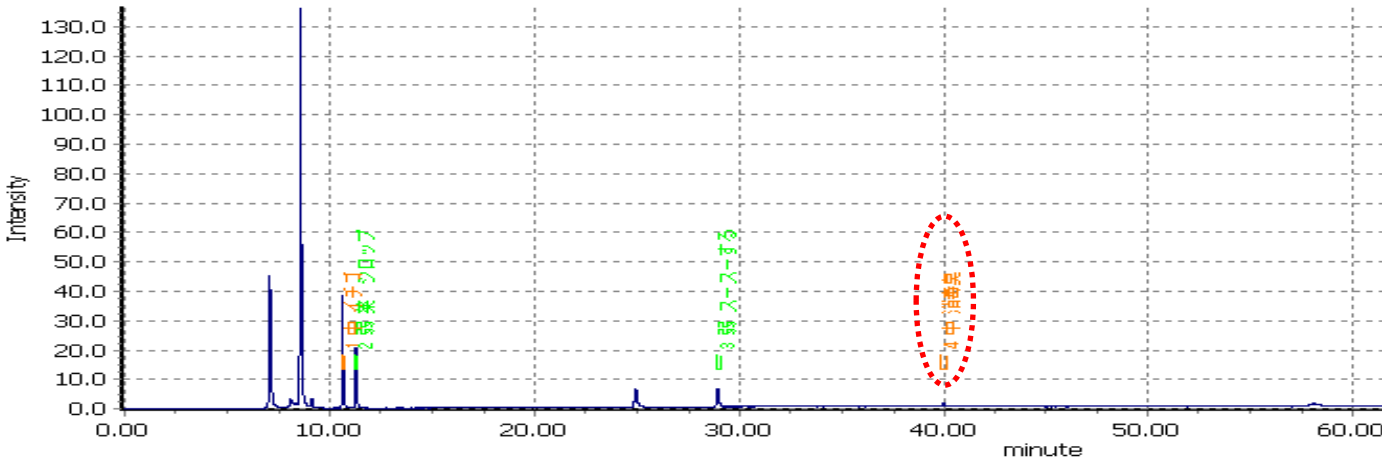
Desorb Temp.	: 40 °C-45 °C/sec-250 °C(5 min)
Desorb Press.	: 330 kPa

Sensory Evaluation Results (strawberry taste)

● Cold syrup (strawberry taste) only



● Cold syrups (strawberry taste) + 2, 6-Dichlorophenol



● Cold syrup (strawberry taste) only

No.	Initiation (mim)	Termination (min)	Intensity	Type
1	10.536	10.791	Weak	Strawberry gum
2	11.192	11.367	Weak	Cheese-like medicine
3	15.41	15.891	Weak	Mildly pepper confectionery
4	18.399	18.855	Weak	Vanilla
5	28.95	29.037	Weak	Sooth

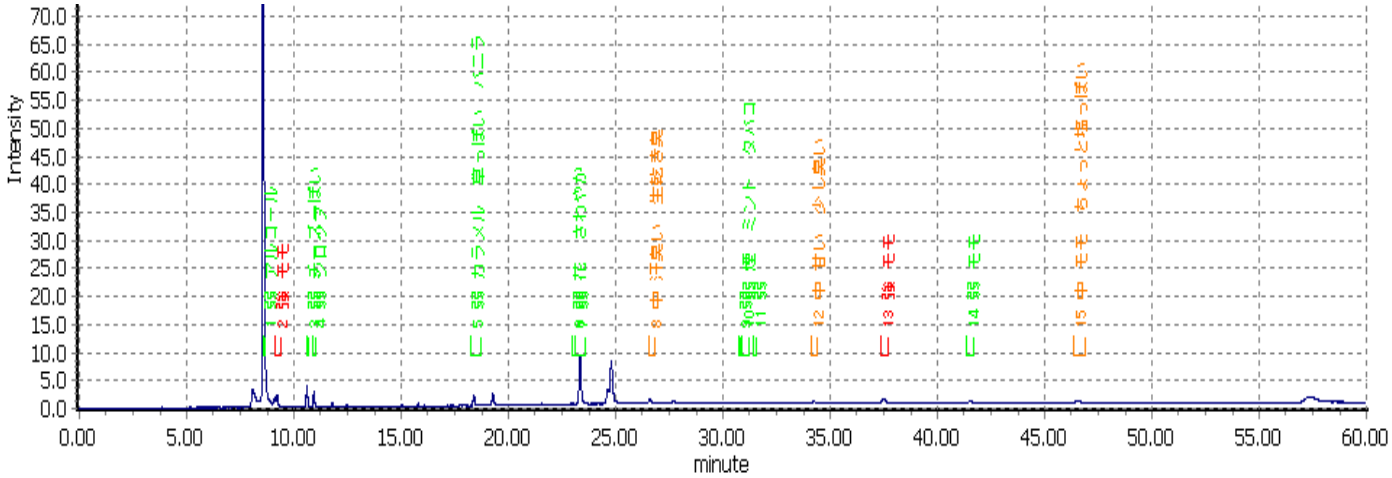
● Cold syrups (strawberry taste) + 2, 6-Dichlorophenol

No.	Initiation (mim)	Termination (min)	Intensity	Type
1	10.588	10.726	Middle	Strawberry
2	11.233	11.39	Weak	Drug syrup
3	28.914	29.125	Weak	Sooth
4	**39.803**	**40.128**	**Middle**	**Disinfection smell**

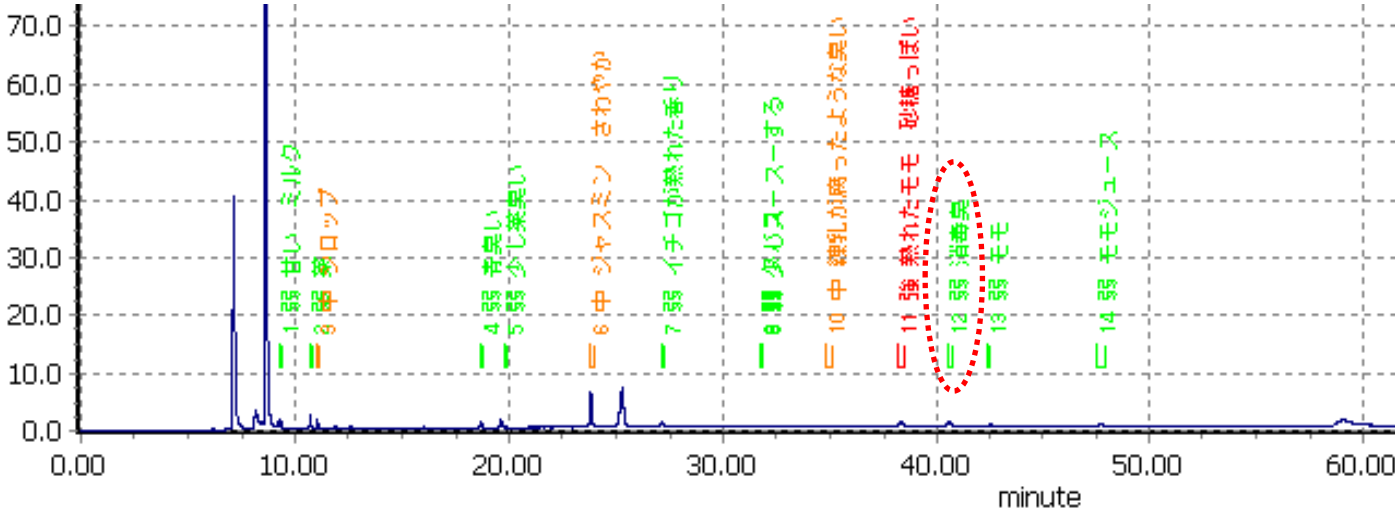
Besides the fragrance, which seems to be derived from fragrances,
We were able to feel firmly the disinfectant odor derived from 2,6-Dichlorophenol.

Sensory evaluation results (peach taste)

● Cold syrup (peach) only



● Cold syrups (peach)+2, 6-Dichlorophenol



● Cold syrup (peach) only

No.	Initiation (mim)	Termination (min)	Intensity	Type
1	8.586	8.648	Weak	Alcohol
2	9.144	9.395	Strength	Peach
3	10.58	10.671	Weak	Cheesey
4	10.872	11.014	Weak	Syrup
5	18.265	18.721	Weak	Caramel grassy vanilla
6	22.939	23.096	Weak	Flower pomace
7	26.567	26.811	Middle	Sweat-smelling live dry odor
8	30.898	31.215	Weak	Smoke mint tobacco
9	34.128	34.387	Middle	Slightly sweet odor
10	37.352	37.698	Strength	Peach
11	41.366	41.716	Weak	Peach
12	46.378	46.918	Middle	A peach that is slightly salty

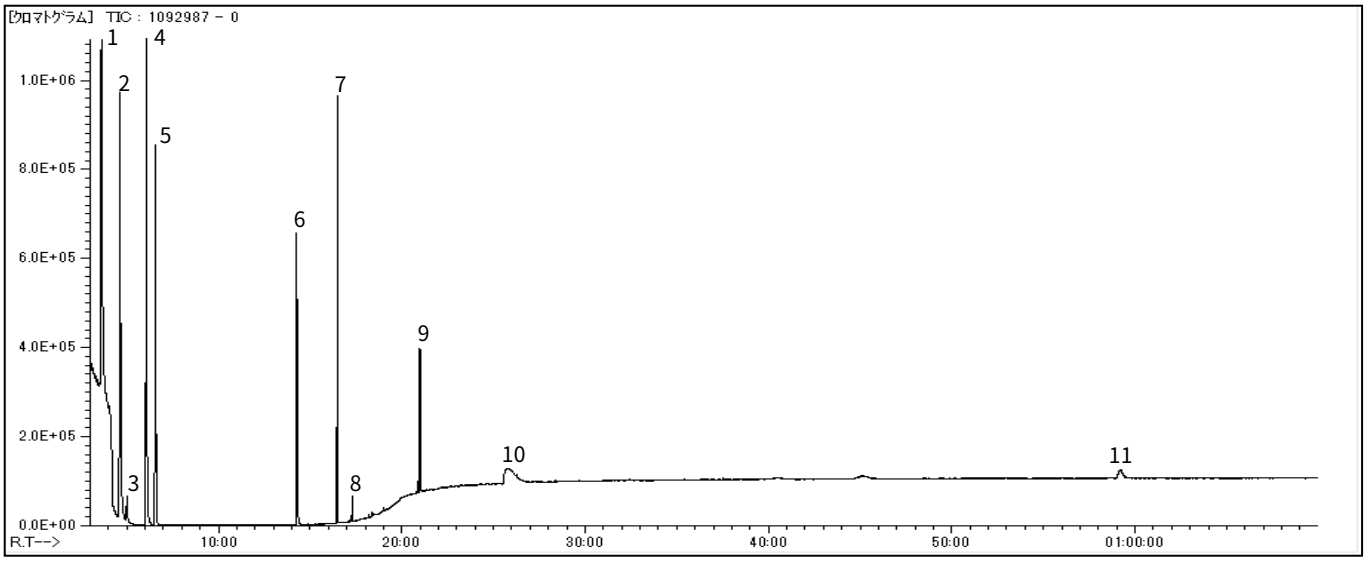
● Cold syrups (peach)+2, 6-Dichlorophenol

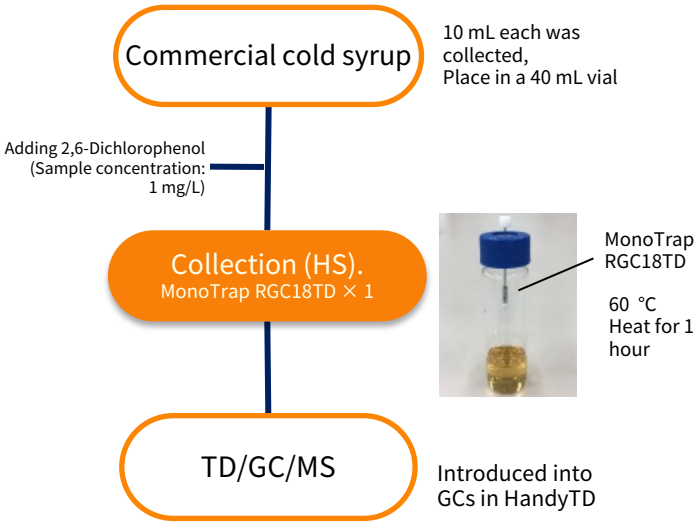
No.	Initiation (mim)	Termination (min)	Intensity	Type
1	9.297	9.391	Weak	Sweet milk
2	10.705	10.788	Weak	Drug
3	11.011	11.127	Middle	Syrup
4	18.674	18.842	Weak	Sophomoric
5	19.851	19.906	Weak	Slightly druggable odor
6	23.767	23.971	Middle	Jasmine wasp
7	27.128	27.31	Weak	A ripe scent of strawberries
8	31.725	31.805	Weak	Tobacco
9	31.831	31.911	Weak	Sooth a little
10	34.85	35.174	Middle	Smell of decayed milk
11	38.2	38.51	Strength	Ripe peach sugary
12	**40.53**	**40.716**	**Weak**	**Disinfection smell**
13	42.375	42.535	Weak	Peach
14	47.508	47.96	Weak	Modulus

We were able to feel firmly the disinfectant odor derived from 2,6-Dichlorophenol. They also felt more flavored than strawberry-tasted cold syrup.

Chromatogram (strawberry taste)

The fragrance components of cold syrups as well as GC/O were collected by MonoTrap and also analyzed by GC/MS.
Besides esters, Butylparaben, which is used as a preservative for food and pharmaceuticals, was also detected.





GC/MS Conditions

System	: Thermal Desorption-GC/MS (HandyTD TD265)
Column	: InertCap Pure-WAX 0.25 mm I.D. × 60 m, df = 0.5 µm
Col.Cat. No.	: 1010-68164
Col.Temp.	: 50 °C (1 min) - 10 °C/min - 240 °C
Carrier Gas	: He, 295 kPa
GC Inlet	: 250 °C, Split 5:1
Detection	: MS Scan (*m/z* 30-450) 250 °C

HandyTD Conditions

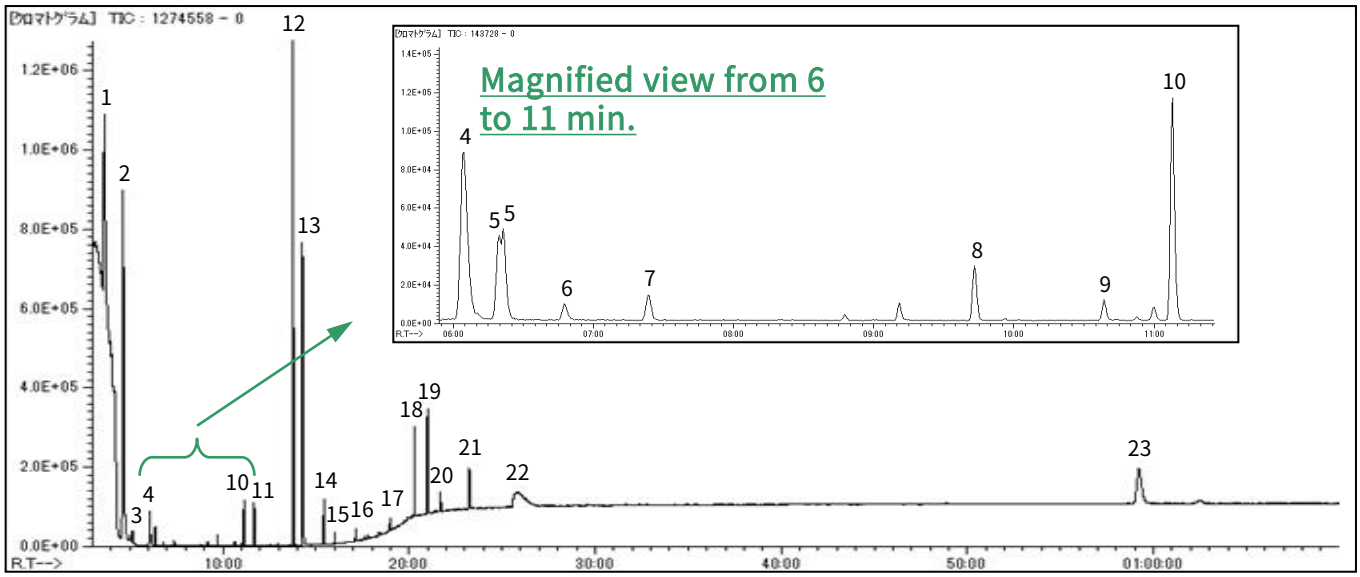
Desorb Temp.	: 40 °C - 45 °C/sec - 250 °C (5 min)
Desorb Press.	: 330 kPa

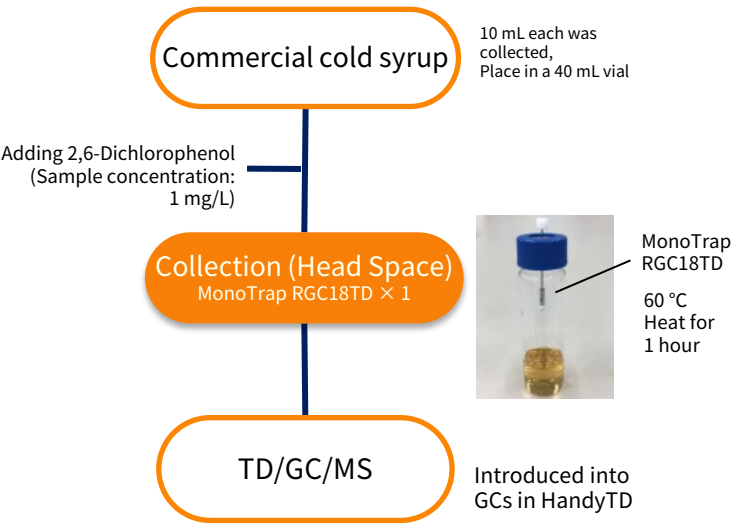
1. Acetone
2. Ethyl alcohol
3. Ethyl propionate
4. Ethyl butanoate
5. Ethyl isovalerate
6. Propylene glycol
7. Benzylcarbamate
8. Methyl salicylate (compress odor)
9. 2,6-Dichlorophenol (disinfectant odor)
10. Benzenecarboxylic acid
11. Butylparaben

※ No qualitative tests have been performed on standard samples. Library search results.

Chromatogram (peach taste)

Many esters and lactones, known as characteristic flavors of peaches, have been detected.
We also detected Butylparaben, which is used as a preservative, as well as strawberry taste.





GC/MS Conditions

System	: Thermal Desorption-GC/MS (HandyTD TD265)
Column	: InertCap Pure-WAX 0.25 mm I.D. × 60 m, df = 0.5 µm
Col.Cat. No.	: 1010-68164
Col.Temp.	: 50 °C (1 min) - 10 °C/min - 240 °C
Carrier Gas	: He, 295 kPa
GC Inlet	: 250 °C, Split 5:1
Detection	: MS Scan (*m/z* 15-450) 250 °C

HandyTD Conditions

Desorb Temp.	: 40 °C-45 °C/sec-250 °C (5 min)
Desorb Press.	: 330 kPa

1. Acetone
2. Ethyl alcohol
3. Ethyl isobutyrate
4. Ethyl butanoate
5. Ethyl 2-methylbutanoate
6. Hexanal
7. Isoamyl acetate
8. Isoamyl butylate
9. 4-methyl-2-pentadecyl-1,3-Dioxolane
10. 2-Isopropyl-4-methylthiazole
11. Cyclohexanol
12. β-Linalool
13. Propylene glycol
14. 2-Methylbutanoic acid
15. Terpineol
16. Pentanoic acid
17. 4-Hydroxyoctanoic acid lactone
18. 4-Hydroxynonanoic acid lactone
19. 2,6-Dichlorophenol (disinfectant odor)
20. γ-Decalactone
21. γ-Decalactone
22. Benzenecarboxylic acid
23. Butylparaben

※ No qualitative tests have been performed on standard samples. Library search results.

For sample introduction Here!

Products used

MonoTrap RGPS TD

Cat.No. :1050-74202
* It is individually packaged and delivered to ampoules.

InertCap Pure-WAX

Cat.No. : 1010-68164
0.25 mm I.D. × 60 m, df = 0.5 µm

Portable Thermal Disorver HandyTD TD265

Cat.No. :2709-80000

Speech recognition odor sniffing system

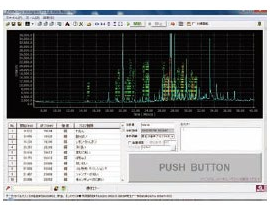
The speech recognition odor sniffing system consists of a sniffing port and speech recognition software.

Sniffing port

OP275Pro

Speech Recognition Softfair Olfactory Voicegram

Analysis screen



Aroma Palate

Description	Transfer line		Number of admissions
	Length	Temperature increase or not	
Speech-recognition odor-sniffing systems OPV277 Pro L	1000mm	Allowable	Equation 1

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