SHIMADZU
Excellence in Science

GC-MS GCMS-QP™ 2020 NX
Thermal Desorption Autosampler TD-30R
Smart Aroma Database™

Application News

Evaluation of Aroma Characteristics Using the Smart Aroma Database —Simple Calculation of OAV—

Haruna Kawamitsu

User Benefits

- The Smart Aroma Database's semi-quantitative function (SQF) can be used to calculate approximate quantitative values.
- The SQF can calculate the Odor Activity Value, which evaluates the contribution of aromatic compounds to the overall aroma.
- The SQF can be used only by preparing two types of designated sensitivity-correcting reagents.

■ Introduction

Aroma is an important factor in determining the palatability of foods and beverages. Aromas are made up of many compounds, and the concentration of compounds at which are perceived as an aroma varies based on compounds. To determine the extent to which the constituent compounds contribute to an aroma, it is crucial to establish if they are present in concentrations that exceed the sensory threshold.

The Smart Aroma Database features a semi-quantitative function (SQF) that enables users to calculate approximate quantitative values (semi-quantitative concentrations) of an identified compounds without using a standard sample, so using the SQF enables more efficient workflows during quantitative analysis of aroma characteristics.

One indicator for evaluating the contribution of a particular aromatic compound to the overall aroma is the Odor Activity Value or OAV, which is calculated by dividing the aroma compound concentration by its sensory threshold value. However, calculating the OAV can be labor-intensive because it involves the following three steps: (1) identifying compounds using qualitative analysis, (2) generating a calibration curve and performing quantitative analysis, and (3) setting the sensory thresholds. Therefore, this study investigated the feasibility of efficiently evaluating aroma characteristics using the Smart Aroma Database's SQF.

■ Smart Aroma Database

The Smart Aroma Database, which contains registered information on more than 500 aroma compounds, is the only database of its kind that supports the entire workflow of aroma analysis. Using the results of total ion current chromatography (TICC) obtained with Scan Mode analysis, the Smart Aroma Database can automatically detect registered compounds, based on retention time, mass spectrum, and mass chromatogram information. One advantage of using the mass chromatogram information to search for compounds is that it enables searches for peaks buried in other compounds (Fig. 1).

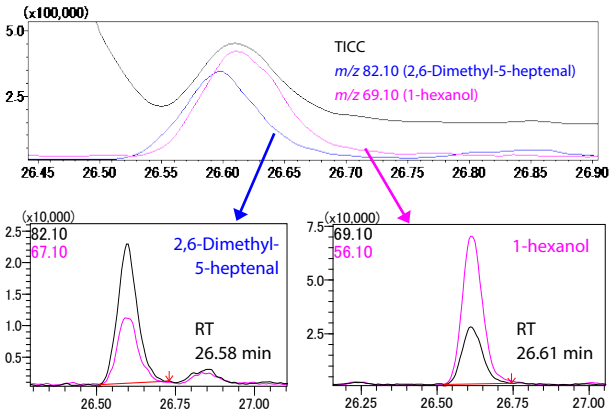

Fig. 1 Results of Automatic Separation and Detection of Overlapping Peaks

■ Semi-Quantitative Function

The SQF can calculate the semi-quantitative (SQ) concentration of a detected compound at the time of Scan Mode analysis without generation of a calibration curve.

The Smart Aroma Database contains registered information on response factors (which are calculated by dividing the peak area of a standard sample by the peak area of the internal standard) that can be used to calculate the SQ concentration of a compound.

An analysis workflow using the SQF is shown in Fig. 2. After analyzing the sensitivity-correcting sample, the resulting data is used to automatically generate the SQ analysis method. By using this method to analyze actual samples, the SQ concentrations are automatically calculated.

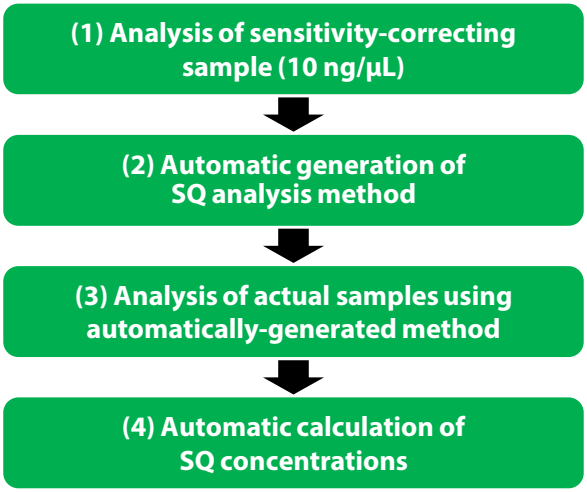

Fig. 2 SQF-Based Analysis Workflow Using Smart Aroma Database

■ Experiment 1: Analysis Using Aroma Capture and GC-MS

Samples were prepared using onion soup either cooked in a microwave or sauteed, and the compounds the contributed to the difference in aroma between the two soups were evaluated using OAV.

Onion only was extracted from each sample and placed in 40 mL vials (Fig. 3) after first measuring and recording the weight of each sample (Table 1). MonoTrap RGC 18TD sorptive media were fixed inside the vials, and the aroma that was emitted into the headspace was trapped for 60 minutes at 40 °C. The aromas of the MonoTrap RGC 18TD media and the samples were then smelled to confirm that the aromas matched (i.e., that the aromas of the samples had been trapped in the MonoTrap RGC 18TD media). The MonoTrap RGC 18TD media were then analyzed using TD-GCMS. The SQ analysis method generated by the Smart Aroma Database was used to perform the analysis. The analytical conditions are shown in Table 2.

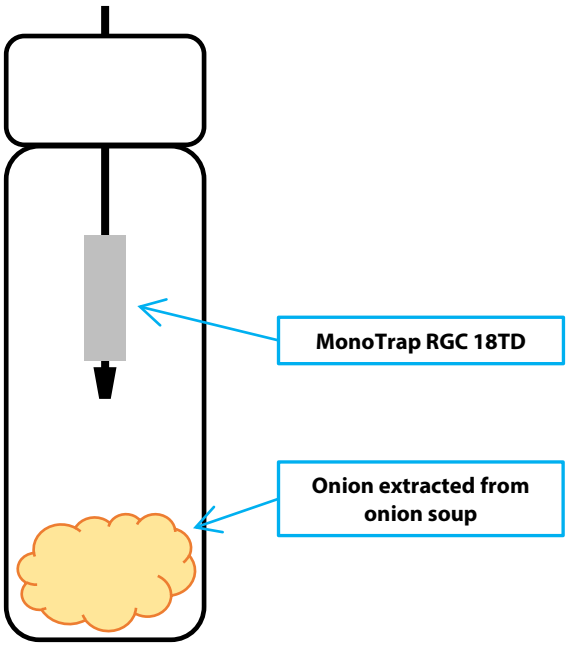

Fig. 3 Aroma Trapping Using MonoTrap RGC 18TD Sorptive Media

Table 1 Onion Sample Weights

Cooking Method	Onion Sample Weight (g)
Microwaved	3.82785
Sauteed	3.26747

■ Results of Experiment 1: Analysis Using Aroma Capture and GC-MS

The MonoTrap RGC 18TD sorptive media used to trap the aromas of the microwaved and sauteed onion soup samples were analyzed, and the resulting TICCs were compared (Fig. 4). Six different peaks were identified from the TICC comparison. They were then automatically identified (Table 3) and their SQ concentrations were calculated by the Smart Aroma Database. Based on the sample weights (Table 1), the SQ concentrations per 1 g sample were calculated and shown in Table 3.

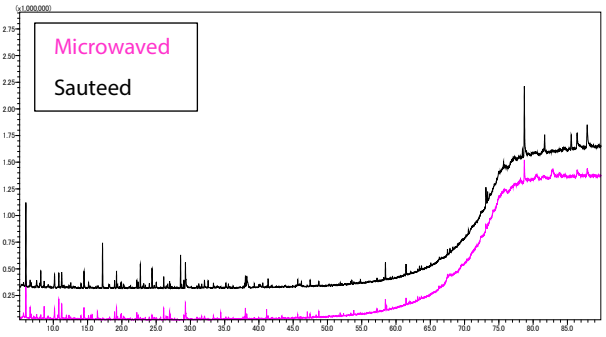

Fig. 4 Comparison of TICCs
Pink: Microwaved, Black: Sauteed

Table 2 Measuring Instruments and Analytical Conditions

■ Equipment and Reagents	
GCMS:	GCMS-QP2020 NX
Database:	Smart Aroma Database
Thermal Desorption Autosampler:	TD-30R
Column:	SH-PolarWax (Shimadzu Corporation) Length 60 m × 0.25 mm I.D., Df = 0.25 mm P/N 227-36247-02
Sensitivity Correcting:	EPA 524.2 Fortification Solution (Sigma-Aldrich)
Samples:	Acenaphthene-d10 (Sigma-Aldrich)
■ GC Conditions	
Injection Unit Temp.:	250 °C
Injection Mode:	Split (split ratio: 5)
Purge Flowrate:	3.0 mL/min
Control Mode:	Linear speed (25.5 cm/s)
Column Oven Temp.:	40 °C (5 min) → 3 °C/min → 250 °C (15 min)
■ MS Conditions	
Interface Temp.:	250 °C
Ion source Temp.:	200 °C
Ionization Method:	EI
Analysis Mode:	Scan *m/z* 35–400
■ TD Conditions	
Tube Purge Flowrate:	20 mL/min
Tube Purge Time:	0 min
Tube Desorption Temp.:	250 °C
Tube Desorption Flowrate:	20 mL/min
Tube Desorption Time:	10 min
Trap Cooling Temp.:	-20 °C
Trap Desorption Temp.:	250 °C
Trap Desorption Time:	5 min
Joint Temp.:	250 °C
Valve Temp.:	250 °C
Transfer Line Temp.:	250 °C
Trapping Agent:	MonoTrap RGC18 TD Cat No. 1050-74201

Table 3 Results of Analysis of Peaks with Differences between Two Cooking Methods Identified in TICCs
(SQ concentration/Sample weight = SQ concentration per 1 g sample)

Compound	SQ Concentration (ng/µL)		SQ Concentration /Sample Weight (ng/µL)	
	Microwaved	Sauteed	Microwaved	Sauteed
Dipropyl disulfide	0.085	9.7	0.022	3.0
Furfural	0.25	3.6	0.066	1.1
5-Methyl furfural	0.030	0.42	0.0078	0.13
Furfuryl alcohol	0.29	3.1	0.077	0.95
1-Methyl-2-pyrrolidinone	1.4	4.1	0.36	1.3
2-Acetylpyrrole	0.22	0.80	0.058	0.24

■ Experiment 2: Sensory Threshold Setting and OAV Calculation

In order to calculate the OAV, an experiment was performed to determine the sensory threshold of each compound identified in Experiment 1 (Table 3).

Standard samples of the six identified compounds and a 100 ng/µL sensory threshold assay reagent were prepared. An aqueous solution of 30 % propylene glycol was used as the diluent to improve the water dispersibility of highly hydrophobic compounds. (The propylene glycol was screened in advance to ensure that it did not have any perceptible odor.) The prepared sensory threshold assay reagent was serially diluted 10-fold, and each dilution was placed in a polypropylene drinking cup (Fig. 5). (The drinking cups were screened in advance to ensure they did not have any perceptible odor.) The odor of the sensory threshold assay reagent in each drinking cup was smelled, and the minimum concentration at which the odor quality could be determined was set as the sensory threshold. Odors were confirmed in both ascending and descending order of concentration before setting the threshold. There are various techniques for setting the sensory threshold, and the technique used in this experiment is described for reference purposes only.

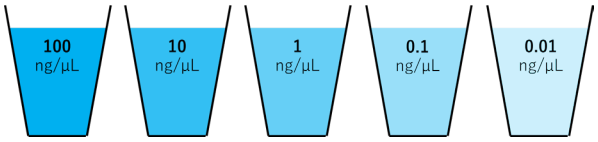

Fig. 5 Illustration of Prepared Sensory Threshold Assay Reagents

■ Results of Experiment 2: Sensory Threshold Setting and OAV Calculation

The results of the sensory threshold measurements and the subsequent OAV calculations are shown in Table 4. Based on the SQ concentrations determined in Experiment 1 and the sensory thresholds determined in Experiment 2, the OAVs of each compound were calculated and converted into log 10 values.

The OAV results suggest that among the six identified compounds, those compounds with large differences in their OAV values—specifically dipropyl disulfide and 5-methyl furfural—contributed significantly to the different aromas of microwaved and sauteed onions.

The procedures for calculating the OAV with and without the Smart Aroma Database are compared in Fig. 7. By using the Smart Aroma Database's semi-quantitative function, the steps for identifying compounds and calculating their SQ concentrations can be performed simultaneously, eliminating the need to generate a calibration curve and perform quantitative analysis. The results of this study therefore suggest that using the Smart Aroma Database can improve analytical workflow efficiency.

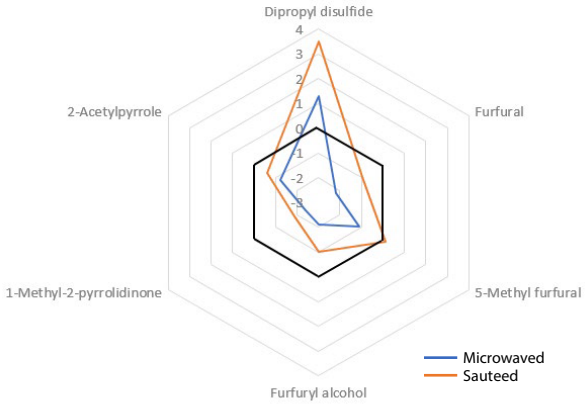

Fig. 6 Results of Comparing OAVs (Log 10)

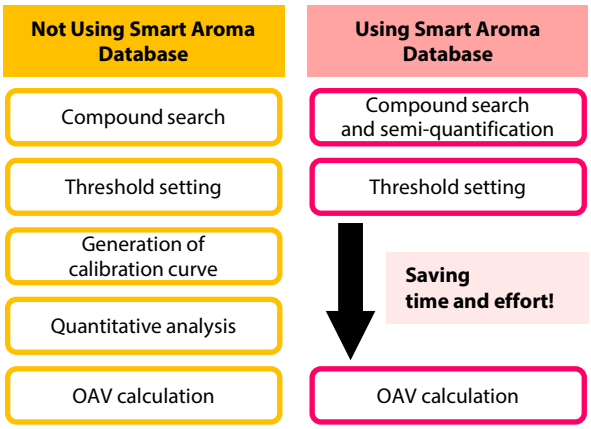

Fig. 7 Comparison of OAV Calculation Procedures

■ Conclusion

The results of this study demonstrate that using the Smart Aroma Database enables efficient calculation of OAV.

The Smart Aroma Database supports the quick acquisition of data required for aroma analysis.

Table 4 OAV Calculation Results

Compound	SQ Concentration /Sample Weight (ng/µL)		Sensory Threshold (ng/µL)	OAV		OAV (Log 10)	
	Microwaved	Sauteed		Microwaved	Sauteed	Microwaved	Sauteed
Dipropyl disulfide	0.022	3.0	0.001	22	3000	1.3	3.5
Furfural	0.066	1.1	10	0.0066	0.11	−2.2	−0.96
5-Methyl furfural	0.0078	0.13	0.1	0.078	1.3	−1.1	0.11
Furfuryl alcohol	0.077	0.95	10	0.0077	0.095	−2.1	−1.0
1-Methyl-2-pyrrolidinone	0.36	1.3	100	0.0036	0.013	−2.4	−1.9
2-Acetylpyrrole	0.058	0.24	1	0.058	0.24	−1.2	−0.62

Acknowledgments
We would like to acknowledge the highly valuable advice of Takehito Sagawa of S&B Foods Inc. in conducting the experiments in this study.

01-00742-EN First Edition: May 2024

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/