Application Note
Food

Agilent
Trusted Answers

Determination of Dimethyl-polysiloxanes (DMPS) in Edible Fats and Oils by ICP-OES

A fast, stable, robust organic analysis method using an Agilent 5800 Radial View (RV) ICP-OES

Authors

Gaurav Kapadnis, Barun Ghosh, Vinay Jain, Prasenjit Kar, Nitin Vig, and Kannan Balakrishnan

Agilent Technologies, Inc.

Introduction

Ensuring the safety and quality of every-day foods and ingredients such as oils and fats is important for manufacturers and consumers. Edible fats and oils are composed of glycerides of fatty acids of vegetable, animal, or marine origin. They are widely used in frying, baking, and other types of cooking, as well as in the preparation of non-cooked foods and flavorings, such as salad dressings, salsas, and dips. Cooking oils are typically liquid at room temperature, although some oils that contain saturated fat, such as coconut oil, palm oil, and palm kernel oil are solid. Plant-based cooking oils include olive, palm, soybean, canola (rapeseed), corn, peanut, plus many others; animal-based oils include butter and lard; and marine-based oils include various fish oils.

Dimethylpolysiloxane (DMPS), which is also known as polydimethylsiloxane (PDMS), is a clear, colorless, viscous liquid that contains 37.3–38.5% silicon. DMPS is often added to edible oils and fats as an antifoaming agent as it prevents the formation of foam on the surface of liquids when heated by reducing the surface tension. As a food additive, DMPS is approved by the U.S. Food and Drug Administration (FDA), European Food Safety Authority (EFSA), Joint FAO/WHO Expert Committee on Food Additives (JECFA), and other authorities (*1*). Typically, the maximum concentration of DMPS that is allowed in ready-to-eat foods is 10 mg/kg. This is the limit set by the Food Safety Standard Authority of India (FSSAI) and Codex Standards for Fats and Oils Derived from Edible Fats and Oils for DMPS (and dimethyl silicone) (*2, 3*).

In this application note, the concentration of DMPS in edible oil was determined by measuring silicon using an Agilent 5800 Radial View (RV) ICP-OES.

Experimental

Instrumentation

The Agilent 5800 RV ICP-OES was used for the analysis because of its tolerance of high levels of total dissolved solids (TDS) and organic samples (*4*). The 5800 ICP-OES uses a solid-state RF (SSRF) system operating at 27 MHz to produce a robust plasma that remains stable over long-term analytical measurements of samples with complex matrices. The sample introduction system consisted of a double-pass glass cyclonic spray chamber, a conical nebulizer, and a 1.4 mm i.d. injector torch. To avoid build-up of carbon from the cooking oil matrix, a mixed gas of argon and oxygen (80:20) was introduced to the 5800 ICP-OES at a flow rate of 20% via the optional gas inlet. The high speed (1 MHz) VistaChip III charge-coupled device (CCD) detector of the 5800 ICP-OES enables fast warmup, fast analysis times, and high sensitivity. Instrument operating conditions are listed in Table 1.

Table 1. Agilent 5800 RV ICP-OES instrument and method parameters.

Parameter	Setting
Read Time (s)	5
Replicates	3
Sample Uptake Delay (s)	10
Stabilization Time (s)	10
Pump Speed (rpm)	12
Fast Pump (rpm)	80
RF Power (kW)	1.30
Aux Flow (L/min)	1.2
Plasma Flow (L/min)	12.0
Nebulizer Flow (L/min)	0.6
Oxygen Injection (%)	20
Viewing Mode	Radial
Viewing Height (mm)	8
Sample Pump Tubing	Black/black
Waste Pump Tubing	Gray/gray
Background Correction	FBC*
Internal Standard Pump Tubing	Orange/white

**FBC: fitted background correction (5).*

Preparation of sample and calibration standards

The oil samples, blank oil, and DMPS standard were supplied from a food manufacturer based in North India.

Edible oil solutions were prepared by mixing 4 g of soybean edible oil sample in kerosene (Agilent A-Solv ICP Solvent, part number 5190-8717) made up to 10 g. The solutions were then homogenized using a vortex mixer.

Intermediate solutions of DMPS were prepared at 100 and 10 ppm by diluting the 100% pure DMPS stock standard to 100 and 10 ppm in kerosene. Matrix matched calibration standards were then prepared at 0.2, 0.5, 1.0, 2.0, 5.0 and 10 mg/kg by adding the intermediate DMPS stock standards and soybean blank oil (free from DMPS), up to 4 g. Each standard was made up to a final weight of 10 g with A-Solv solvent.

The calibration graph and signal spectrum for silicon is shown in Figure 1. Linear calibration was acquired as indicated by the correlation coefficient of greater than 0.999 (Figure 1).

A separate soybean oil sample, which was free from DMPS, was used as the method blank.

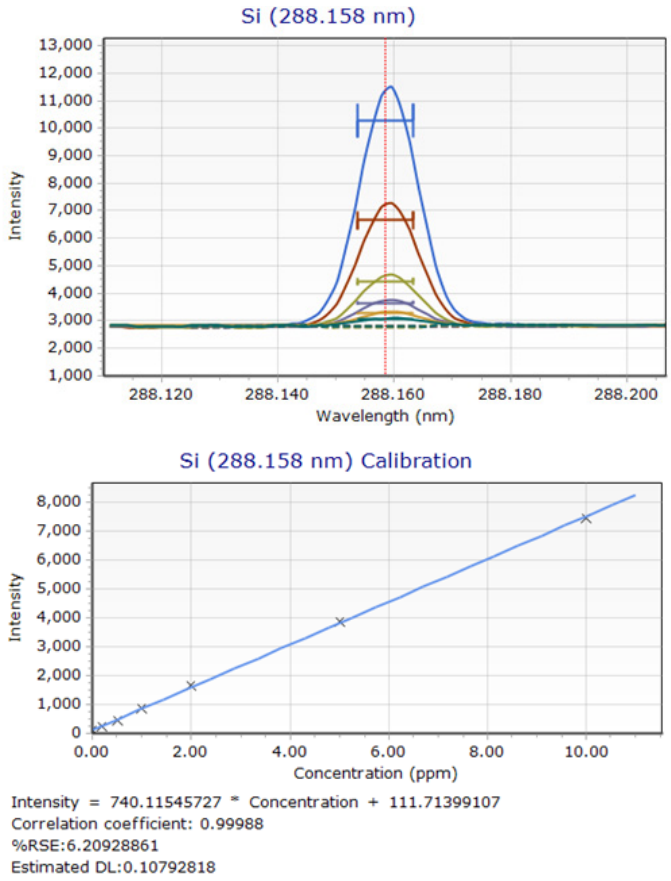

Figure 1. Overlay of Si 288.158 nm signals for each calibration standard (top) and calibration plot (bottom), showing good linearity up to 10 ppm.

For the spike recovery test, the edible oil sample was spiked at 0.2 and 1 mg/kg by adding the required amount of DMPS stock solution to 4 g of oil. The A-Solv solvent was then added to a final weight of 10 g. A solution containing 10 mg/kg yttrium prepared in A-Solv was used as the internal standard to correct for matrix interferences. The internal standard was introduced to the ICP-OES through a Y connector. The sample preparation workflow is shown in Figure 2.

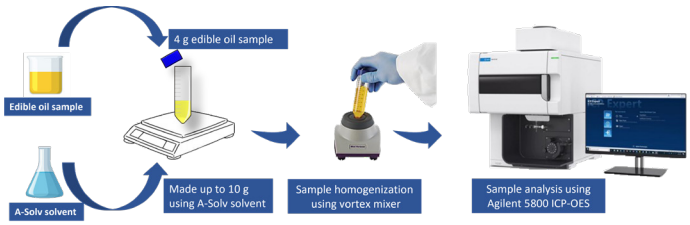

Figure 2. Preparation of edible oil samples for the analysis of DMPS by ICP-OES.

Results and discussion

Method Detection Limits

The Method Detection Limits (MDLs) shown in Table 2 were based on three sigma of 10 replicate measurements of the method blank (DMPS-free soybean oil) diluted in A-Solv solvent.

Table 2. Method detection limit and calibration correlation coefficient for Si.

Element	Wavelength (nm)	MDL in Solution (mg/kg)	Correlation Coefficient
Silicon	288.158	0.0329	0.99988

Sample analysis and spike recoveries

A spike recovery test was carried out to check the accuracy of the 5800 RV ICP-OES method. Table 3 shows the concentration of Si in the unspiked edible oil sample (0.29 mg/kg, which is well below the maximum limit of approximately 3.73–3.85 mg/kg Si in 10 mg/kg DMPS). The measured spiked concentration data shows the contribution of Si from DMPS, which was spiked at two concentration levels (0.200 and 1.00 mg/kg) in the same edible oil sample. The recoveries for Si in the edible oil sample were within ±5% at both spike-levels.

Table 3. Sample analysis result and recovery results for edible oil samples spiked at two concentration levels with DMPS.

Element and Wavelength (nm)	Measured Sample Conc (mg/kg)	Spiked Conc (mg/kg)	Measured Spiked Conc (mg/kg)	Recovery (%)	Spiked Conc (mg/kg)	Measured Spiked Conc (mg/kg)	Recovery (%)
DMPS based on Si 288.158	0.29	0.200	0.500	105	1.00	1.30	101

Long-term stability data

A long-term stability test was carried out by analyzing an edible oil sample spiked at 0.2 mg/kg over three hours. The percent relative standard deviation (% RSD) was only 2.1%, demonstrating the robustness and precision of the method over a three-hour run of organic samples (Table 4). The excellent performance of the 5800 RV ICP-OES for the analysis of complex samples is due to the vertically oriented plasma and the SSRF system.

Table 4. Long-term stability results (% RSD) for an edible oil spiked sample analyzed over three hours.

Element	Wavelength (nm)	% RSD
Silicon	288.158	2.1

Sample analysis time

Sample-to-sample analysis time was only 35 seconds using the 5800 RV ICP-OES. The fast analysis time and low argon gas consumption of the 5800 ICP-OES is due to different technologies, including the high-speed Vista Chip III charge-coupled device (CCD) detector. Considering all gas flows into the ICP-OES, not just the torch gas flows, the total argon consumption was less than 8.5 L per sample.

Conclusion

The Agilent 5800 RV ICP-OES with oxygen injection was used for the rapid determination of DMPS in edible oil samples. The accuracy of the method was demonstrated by the excellent spike recovery test results of edible oil samples spiked at 0.2 and 1 mg/kg with DMPS. The vertical plasma and robust 27 MHz SSRF system of the 5800 ICP-OES delivered excellent stability over three hours, with 2.1 %RSD for an oil sample spiked with DMPS at 0.2 mg/kg.

References

1. Food Additives, What is Dimethylpolysiloxane (E900) in Food and What are the Uses? December 2019, accessed November 2021, https://foodadditives.net/antifoaming-agent/dimethylpolysiloxane/
2. Food and Agriculture Organization of the United Nations (FOA), Codex Standards for Fats and Oils Derived from Edible Fats and Oils, Section 4, accessed November 2021, http://www.fao.org/3/Y2774E/y2774e06.htm
3. Food Safety and Standards Authority of India (FSSAI), Chapter 3, Substances added to foods, section 3.1.8, Antifoaming agents in edible oils and fats, accessed November 2021, Food Safety and Standards (Food Product Standards and Food Additives) Regulation, 2011
4. Resilient Vertical Torch Means Less Cleaning, Less Downtime, and Fewer Replacements—Even for Tough Samples, Agilent publication 5994-1514EN
5. Fitted Background Correction (FBC)—Fast, accurate and fully automated background correction, Agilent publication, 5991-4836EN

www.agilent.com/chem/5800icp-oes

DE21948909

Printed in the USA, December 8, 2021
5994-4440EN

Agilent
Trusted Answers