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# Determination of Dimethylpolysiloxanes in Oils
# Atomic Absorption Spectroscopy

Dimethylpolysiloxanes or methyl silicones are commonly added to fats and oils to prevent oxidative foaming in products used in frying applications. To measure the amount of DMPS, a direct aspiration, flame atomic absorption method is demonstrated.

## APPARATUS

1. Shimadzu model AA-7000F spectrophotometer – with Silicone hollow-cathode lamp.
2. A nitrous Oxide – acetylene flame
3. Volumetric Flasks 25 mL.
4. 100 – 1000 uL volumetric Pipetter

| **Optics Parameters** | **Atomizer/Gas Flow Rate Setup** |
|---|---|
| Element: Si | Fuel Gas Flow Rate(L/min): 7.5 |
| Lamp Current (Peak)(mA): 20 | Support Gas Flow Rate (L/min): 11.0 |
| Wavelength(nm): 251.6 | Flame Type: N2O-C2H2 |
| Slit Width(nm): 0.7 | Burner Height(mm): 11.0 |
| Lamp Mode: BGC-D2 | |

Table 1. Instrument Parameters

## REAGENTS

1. Dow Corning 200 or equivalent. Dimethylpolysiloxane standard.
2. DMPS-free hydrogenated oil-to match viscosities of the standards to the samples.
3. Conostan® PremiSolv solution

## STANDARDS

Calibration standards were prepared dispensing 1, 2, 5 mL of 20 ug/mL DMPS standard in PremiSolv into 25 mL volumetric flasks that contained 10 g of hydrogenated DMPS-free oil and brought to volume with PremiSolv.

## PROCEDURE

1. Sample solutions were prepared by bringing 10 g of oil to 25 mL with PremiSolv.
2. Sample spikes were prepared adding 1 mL of 20 ug/mL DMPS solution to the 10 g of oil then brought to 25 mL total volume with PremiSolv.
3. Solutions were homogenized with a vortexer.

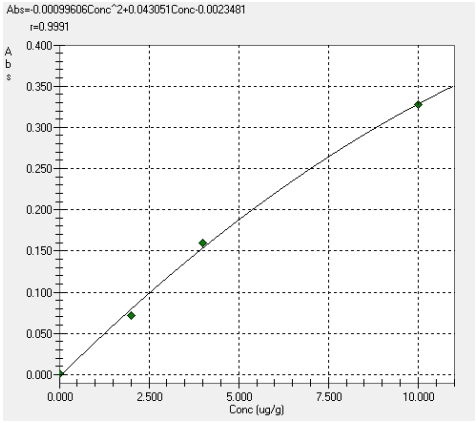


Figure 1. Calibration curve generated from DMPS standards

INSTRUMENT CALIBATION

The instrument was calibrated by aspirating 0, 2, 4 and 10 ug/g solutions and plotting the response versus their concentration. Figure 1 above shows the calibration solution responses and their plotted regression.

SAMPLE ANALYSES AND RECOVERIES

Two samples were analyzed to which known amounts of DMPS were added and were also analyzed. The result and repeatability of each analysis is show in table 2.

Table 2. Results and Repeatability

| Rep # | Sample Name | Conc ug/g | Abs. |
|---|---|---|---|
| 1 | Sample A | 0.1293 | 0.0032 |
| 2 | Sample A | 0.1293 | 0.0032 |
| | | | |
| 1 | Sample A 2ug SPK | 2.1318 | 0.0849 |
| 2 | Sample A 2ug SPK | 2.1369 | 0.0851 |
| | | | |
| 1 | Sample B | 0.3241 | 0.0115 |
| 2 | Sample B | 0.3241 | 0.0115 |
| | | | |
| 1 | Sample B 2ug SPK | 2.1911 | 0.0872 |
| 2 | Sample B 2ug SPK | 2.2040 | 0.0877 |

The recovery results for the spiked samples are shown table 3.

| Sample Name | Result (ug/g) | Recovery (%) |
|---|---|---|
| Sample A | 0.13 | |
| Sample A +2 ug SPK | 2.13 | **100.23** |
| Sample B | 0.32 | |
| Sample B +2 ug SPK | 2.20 | **94.50** |

Table 3 Sample Results and Recoveries

The analysis of the samples yielded 0.13 ug/g and 0.32 ug/g DPMS concentrations for the two samples analyzed. Additionally each sample had known amounts of DPMS added. Each sample was spiked to give a 2 ug/g concentration difference. The recoveries for added amounts of DPMS were 100.23% and 94.50%.


Picture 1. Shimadzu AA-7000 Atomic Absorption Spectrophotometer.