

## Atomic Absorption



## Determination of Nickel in Fats and Oils

### Scope

Triglyceride-based vegetable fats and oils can be transformed through partial or complete hydrogenation to fats and oils of greater molecular weight. The hydrogenation process involves sparging the oil at high temperature and pressure with hydrogen in the presence of a catalyst, typically a powdered nickel compound. Atomic Absorption Spectrometry is commonly used to estimate the amount of nickel left in the vegetable oils.

### Typical Analytical Procedure

#### Materials and Methods

The following reagents and equipment are used for the measurement:

- Atomic absorption spectrometer
- Nickel metal
- Conc. nitric acid
- Conc. hydrochloric acid
- Double distilled water

## Solution Preparation

**Preparation of nickel standard solution:** Weigh accurately 1.00 g of nickel metal into a clean beaker. Add 25 mL aqua regia (3 mL concentrated hydrochloric acid: 1 mL concentrated nitric acid) and heat until it dissolves. Transfer the solution into a one-liter volumetric flask and dilute to volume with distilled water. This is the 1000 mg/L nickel standard solution.

**Preparation of the working standards:** Transfer 10 mL of 1000 mg/L standard solution into a 100-mL volumetric flask and dilute to volume with distilled water. This is the 100 mg/L standard solution. Prepare 1.0, 2.0, 3.0 and 4.0 mg/L of the nickel standard solutions by transferring 1, 2, 3 and 4 mL of this 100 mg/L nickel standard solution in different 100-mL volumetric flasks and dilute to volume with distilled water.

**Preparation of the stock sample solution:** Weigh accurately 1.9094 g of fat in a clean beaker. Add 10 ml of 1:1 hydrochloric acid. The contents are heated to boiling, then add 10 mL of 1% dimethylglyoxime in ethyl alcohol to acidify the fat in hot condition, stirring thoroughly. 1:1 ammonia is added in slight excess and stirred. The final contents are transferred into a separating funnel. The aqueous phase is separated into a 100-mL volumetric flask. The fat is washed with 25 mL of hot distilled water and the aqueous phase is taken into the volumetric flask. This is repeated twice to extract total amount of nickel from the fat sample. Add 5 mL of concentrated hydrochloric acid to the flask and dilute to volume with distilled water.

## Analysis Procedure

- Element - Ni
- Flame – air-acetylene flame
- Slit width – 0.15 nm
- Wavelength – 232.1 nm
- Detector gain – 575 V
- Lamp current – 7.0 mA
- BGC – off
- Mode – concentration mode
- Type of fit – least squares

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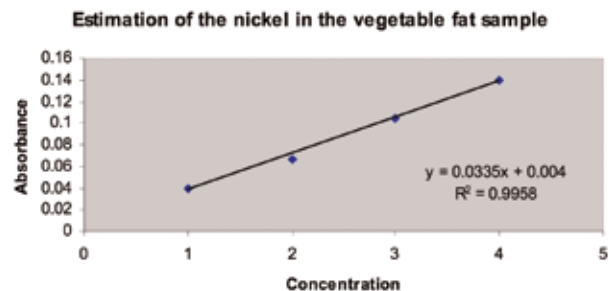
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Aspirate the nickel standard solutions of 1, 2, 3, 4 mg/L into air-acetylene flame to create the calibration curve and then analyze the samples. The absorbance and concentration readings of the standard and sample solutions are below.

## Results

### Calibration Curve for the Standards



Std no.	Conc. in mg/L	Absorbance
1	1.0	0.040
2	2.0	0.067
3	3.0	0.105
4	4.0	0.139

### Sample Data

Sample no.	Sample name	Absorbance	Conc. in mg/L
1	Fat solution	0.035	1.03
2	Fat solution	0.035	1.03

### Calculations

$$\% \text{ of nickel in the sample} = \frac{100 \times [\text{Ni}] \text{ in mg/L}}{[\text{sample}] \text{ in mg/L}}$$

**Report:** The nickel content in the sample is 0.54%.

### References

W.J. Price, J.T.H. Roos and A.F. Clay, Rapid determination of nickel in edible fats by atomic-absorption spectrophotometry, Analyst, 1970, 95, 760-762.

ASTM Method Ref. E-63-86, IS 504, 1989.

