



Atomic Absorption Spectrophotometer AA-7800F

# Impurity Evaluation of Gelatin Using Atomic Absorption Spectrophotometer

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### **User Benefits**

- Impurities in gelatin can be easily evaluated using an atomic absorption spectrophotometer (AA).
- Efficient measurement is possible by using the flame method (air-acetylene flame).
- High sensitivity data can be obtained from gelatin measurement using the AA-7800.

# Introduction

Gelatin is used for gelification of solutions and to add viscosity. Therefore, it is widely used as a gelling agent, thickening agent, and stabilizer in various fields such as food and medicine, and its usage rate is increasing, especially in Japan.

The Japanese Pharmacopoeia specifies evaluation methods for strength, identification, purity, and conductivity of gelatin. This article describes purity tests on gelatin using the flame method of atomic absorption spectrophotometer, and the effectiveness of this method is discussed.

# Pretreatment of Measurement Samples

Commercially available gelatin was used for the samples. The flow chart of sample pretreatment is shown in Fig. 1.

Japanese Pharmacopoeia states that 5 g is processed to a content of 100 g. Here, half this quantity of sample was pretreated.

```
(Sample Solution)
Add 2.5 g of gelatin to a glass-stoppered flask
 \downarrow Add 5 mL HCl, place the stopper
Heat in a water bath at 75 °C to 85 °C for 2 hours
 \downarrow
 T
After all solids are dissolved, cool down
 \downarrow
 T
Add pure water up to 50 mL
(Standard Addition Sample)
Add 2.5 g of gelatin to a glass-stoppered flask
 \downarrow
 \downarrow (Same treatment as the sample solution)
 \downarrow
Add the appropriate concentration of the standard
solution
 \downarrow
 .1.
Add pure water up to 50 mL
```

# Measurement Conditions

The Shimadzu Atomic Absorption Spectrophotometer AA-7800F was used. The main conditions for the spectrometer and atomization are shown in Table 1.

Measurements were performed by the standard addition method.

| Table 1 Measurement Conditions          |           |                                   |           |
|---|-----------|-----------------------------------|-----------|
| Elements                                | Cr        | Fe                                | Zn        |
| Analysis Wavelength                     | 357.9 nm  | 248.3 nm                          | 213.9 nm  |
| Slit Width                              | 0.7 nm    | 0.2 nm                            | 0.7 nm    |
| Lighting Mode                           |           | BGC-D2                            |           |
| Lamp Current                            | 10 mA     | 12 mA                             | 8 mA      |
| Height of Burner                        | 9 mm      | 9 mm                              | 7 mm      |
| Type of Flame                           |           | Air-C <sub>2</sub> H <sub>2</sub> |           |
| C <sub>2</sub> H <sub>2</sub> Flow Rate | 2.8 L/min | 2.2 L/min                         | 2.0 L/min |
| Integration Time                        |           | 3 seconds                         |           |
| Repetition                              |           | 3 times                           |           |

# Measurement Results

The calibration curves for Cr, Fe and Zn obtained by the standard addition method are shown in Tables 2 to 4. The concentration of the standard addition solution was adjusted according to the device sensitivity. Each calibration curve is shown in Figs. 2 to 4. All the elements showed good linearity.

Table 5 shows the measurement results and the limit of quantification (LOQ) by the standard addition method. All the measured elements in the samples were below the upper limit (Cr: 10 ppm, Fe, Zn: 30 ppm, converted to concentration in solid). The LOQ was calculated from the value of  $10\sigma$  calculated from the standard deviation (SD) obtained from 10 times repeated measurements of non-addition samples. Furthermore, the LOQ in solids is expressed in terms of the actual sample from the LOQ in solution.

| Concentration<br>Added (ppm) | Absorption<br>(Abs) | %RSD<br>(n = 3) | SD<br>(n = 3) |
|------------------------------|---------------------|-----------------|---------------|
| 0                            | 0.0012              | 9.90            | 0.0001        |
| 0.25                         | 0.0254              | 1.20            | 0.0003        |
| 0.5                          | 0.0493              | 1.72            | 0.0009        |
| 1                            | 0.0971              | 0.63            | 0.0006        |

Fig. 1 Flow Chart of Sample Pretreatment

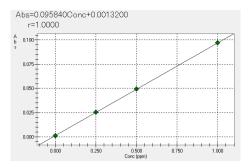


Fig. 2 Calibration Curve of Cr

Table 3 Measurement Result of Standard Addition Sample of Fe

| Concentration | Absorption | %RSD    | SD      |
|---------------|------------|---------|---------|
| Added (ppm)   | (Abs)      | (n = 3) | (n = 3) |
| 0             | 0.0488     | 0.83    | 0.0004  |
| 0.25          | 0.0745     | 1.36    | 0.0010  |
| 0.5           | 0.1054     | 0.40    | 0.0004  |
| 1.5           | 0.2079     | 0.60    | 0.0012  |

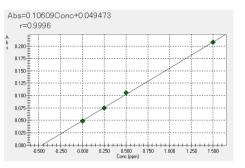


Fig. 3 Calibration Curve of Fe

| Concentration<br>Added (ppm) | Absorption<br>(Abs) | %RSD<br>(n = 3) | SD<br>(n = 3) |
|------------------------------|---------------------|-----------------|---------------|
| 0                            | 0.0466              | 0.54            | 0.0003        |
| 0.1                          | 0.1016              | 0.43            | 0.0004        |
| 0.25                         | 0.1814              | 0.17            | 0.0003        |
| 0.5                          | 0.3333              | 0.24            | 0.0008        |

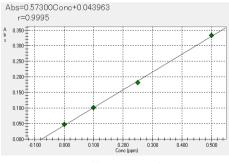


Fig. 4 Calibration Curve of Zn



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| Table 5 Measurement Results of Samples |                            |   |  |
|--|----------------------------|---|--|
| Cr                                     | Fe                         | Zn  |  |
| N.D.*                                  | 0.47                       | 0.077   |  |
| N.D.                                   | 9.3                        | 1.5   |  |
| 0.3                                    | 2                          | 0.3   |  |
| 10                                     | 30                         | 30  |  |
|  | Cr<br>N.D.*<br>N.D.<br>0.3 | Cr         Fe           N.D.*         0.47           N.D.         9.3           0.3         2 |  |

\* Under I OO

# ■ Conclusion

The impurity levels of gelatin can be evaluated with AA by simple pretreatment to analyze trace amounts of Cr, Fe and Zn at several ppm levels. In addition, by using the flame method, the measurement time is shorter, and the work is more efficient. In addition, data can be obtained with high sensitivity in measurements with reference to Japanese Pharmacopoeia.

The AA-7800 series is an atomic absorption spectrophotometer that can be used safely and securely thanks to advanced safety technologies such as automatic fire extinguishing with a vibration sensor, automatic gas leak inspection, and acetylene regulator failure detection.

The AA-7800 series can also be upgraded with additional units, allowing the system to evolve based on what is being analyzed.



Flame Model AA-7800F



AA-7800F Dual Atomizer System

# Reference

1) Japanese Pharmacopoeia 18th Edition (the Ministry of Health, Labour and Welfare Notification No. 220 of June 7, 2021)