



Determination of Elements in Glycerin and γ -Butyrolactone

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1 Introduction

This Application Note describes the analysis of Glycerin (Glycerol) and γ -butyrolactone (GBL). The difficulty of analysis of such matrices is their volatility. The conditions used to introduce the samples into the plasma are given and the limits of detection obtained are listed.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

2.3 Limit of detection estimation

The limits of detection are calculated using the following formula:

$$\text{LOD} = k \times \text{BEC} \times \text{RSD}_0$$

With:

LOD = limits of detection,
 $k = 3$ for the normal 3-sigma values,
BEC = Background equivalent concentration,
 RSD_0 = relative standard deviation of the blank.

To calculate the LOD, a calibration curve is constructed using two points, 0 ppm and 5 ppm, or some concentration where the calibration is linear; this gives the BEC. The RSD_0 is evaluated by running the blank ten times.

3 Sample Preparation

Samples of GBL were analyzed directly, while the Glycerin was diluted by a ratio 1:4 in deionized water.

4 Instrument specification

The work was done on a JY ULTIMA. The specifications of this instrument are listed below in Tables 1 and 2.

**Table 1: Specification of spectrometer**

Parameters	Specifications
Mounting	Czerny Turner
Focal length	1m
Nitrogen purge	Yes
Variable resolution	Yes
Grating number of grooves	2400 gr/mm
Order	2nd order

Table 2: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	by computer
Control of pump flow	by computer
Cooling	air

5 Operating conditions

The operating conditions are listed in Table 3 below. The conditions used are generally the conditions recommended for the analysis highly volatile samples. Notice that the nebulizer flow-rate and the sample uptake have been reduced to introduce these samples. The use of smaller pump tubing prevented low speed pulsations from the peristaltic pump.

For Cu and Na, the sheath gas flow was increased automatically by the software to 0.5 L/min and 0.8 L/min respectively. Due to the difficulty of analyzing the GBL solvent, an alternative is to make a 1:4 dilution with water or with a less volatile solvent such as kerosene. This may be useful where higher concentrations will be determined and the lowest LODs are not required.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1450 W
Plasma gas flowrate	16 L/min
Auxiliary gas flowrate	0.6 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer flowrate	2.1bars, 0.61 ml/min
Sample uptake	0.5 mL/min
Type of nebulizer	Meinhard
Type of spray chamber	Scott
Argon humidifier	No
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

The limits of detection were determined using the following parameters:

Mode of calculation: Mode maximum

Entrance slit: 20 μm

Exit slit: 15 μm

Integration time: 6 s

Replicates: 10



7 Results

The limits of detection are calculated using the formula in section 2.3 and are given in Tables 4 and 5 below.

7.1 γ -BUTYROLACTONE (direct aspiration)

Table 4: Limits of Detection for γ -butyrolactone

Element	RSDblank (%)	BEC (mg/L)	LOD (μ g/l)
Al 167.020 nm	5.13	0.026	4.04
Al 308.215 nm	0.32	1.33	12.7
Al 394.401 nm	0.40	1.64	19.5
Al 396.152 nm	0.29	1.26	11.1
As 189.402 nm	0.39	0.36	4.16
Cl 134.664 nm	3.76	34.1	3.85 mg/L
Cu 324.754 nm	0.35	0.0547	0.58
Fe 259.940 nm	0.35	0.0609	0.615
Hg 194.164 nm	0.61	0.201	3.67
Mn 257.610 nm	0.51	0.0138	0.21
Pb 220.353 nm	0.37	0.676	7.49
Sb 206.833 nm	0.23	0.634	4.43
Sn 189.989 nm	0.31	0.404	3.77
Te 214.281 nm	0.33	0.726	7.29
Zn 213.856 nm	0.40	0.334	0.40

7.2 GLYCERIN

Due to its high viscosity, the sample was diluted by a factor of 4 with deionized water.

Table 5: Limits of Detection for Glycerin

Element	RSDblank (%)	BEC (mg/L)	LOD (μ g/L)
As 189.402 nm	0.43	0.293	3.81
Cl 134.664 nm	3.13	22.3	2.1
Cu 324.754 nm	0.51	0.191	2.94
Fe 259.940 nm	0.50	0.0732	1.1
Hg 194.164 nm	0.36	0.0503	0.55
Mn 257.610 nm	0.59	0.0141	0.25
Na 589.592 nm	0.59	0.346	6.1
Pb 220.353 nm	0.29	0.497	4.34
Sb 206.833 nm	0.21	0.593	3.74
Sn 189.989 nm	0.26	0.241	1.91
Te 214.281 nm	0.28	0.550	4.63
Zn 213.856 nm	0.54	0.0299	0.48

8 Conclusion

This Application Note shows that it is possible to determine trace elements with good sensitivity and low LODs in difficult matrices such as Glycerin and GBL. The only precaution taken was reducing the sample uptake and the nebulizer flow-rate to ensure a stable plasma.



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