



Analysis of 95 % Methanol

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1 Introduction

95% methanol samples were presented to this laboratory for trace determination. Levels of the analytes in these samples were thought to be less than 100 ppm. This document gives the analytical conditions, a selection of the best wavelengths for the determination of analytes in these matrices, as well as an estimation of the detection limits.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). 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 Limits 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,
RSD₀ = 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 RSD₀ is evaluated by running the blank ten times.

3 Instrument specification

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

Table 1: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny-Turner
Focal length	1 m
Thermoregulation	Yes
Variable resolution	Yes
Nitrogen purge	Yes
Grating number of grooves	2400 gr/mm
1st order resolution	0.010 nm
2nd order resolution	0.005 nm
Order	2nd order

Table 2: Specification of RF Generator

Parameters	Specifications
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of pump flow	By computer
Cooling	Air



4 Operating conditions

The operating conditions are listed in table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1400 W
Plasma gas flowrate	18 L/min
Auxiliary gas flowrate	0.8 L/min
Sheath gas flowrate	0.15 L/min
Nebulizer gas flowrate	0.35 L/min
Nebulizer flowrate	1.2 bars (18 psi)
Sample uptake	0.3 mL/min
Type of nebulizer	Concentric
Type of spray chamber	Cyclonic
Argon humidifier	Yes
Injector tube diameter	3.0 mm

5 Wavelength selection and analytical conditions

The line with the highest sensitivity was used for analysis of all analytes, as there were no problems with interferences.

Table 4: Analysis conditions

Element	Slits μm	Analysis Mode	Integration Time (sec)
All elements	20 x 15	Direct Peaking	8

6 Discussion

6.1 Limits of Detection

The limits of detection have been calculated using the formula in paragraph 2.3. They are calculated in $\mu\text{g/L}$.

Table 5: Limits of detection ($\mu\text{g/L}$)

Element	Wavelength (nm)	LOD in 95 % Methanol
Al	167.020	5
Al	308.215	28
Al	396.152	7
B	249.773	0.2
Cr	267.716	2
Cu	324.754	1
Mn	257.610	0.2
Ni	221.647	2
Ni	231.604	2
Pd	340.458	3
Pd	360.955	11
Sn	189.989	58
Sn	242.949	20

7 Summary

To achieve the lowest detection limits, dilution is undesirable. The results show the HORIBA Scientific spectrometers perform the analysis very well and, even with a volatile solvent such as methanol, achieve the best limits possible.

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