



# The Analysis of Polyvinylchloride

## Application Note

Atomic Absorption

### Authors

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### Introduction

The presence of various metals in synthetic fibres and plastic materials can arise from catalyst residues, stabilizing agents, and other sources, however, excessive concentrations can have a deleterious effect upon the characteristics of such materials.

The technique of atomic absorption spectrophotometry is eminently suitable for the rapid and accurate analysis of metals in such polymeric materials.

The choice of solvents or the method used for dissolving polymeric materials depends on the type of polymer. Olivier [1] has described the determination of a wide range of trace metals in various polymeric materials, and the determination of germanium in synthetic fibres has also been reported [2].

A description is given of a method employed at Agilent Technologies, Inc. for the analysis of calcium, tin, titanium and zinc in a sheet of polyvinylchloride (P. V. C.) that contains a relatively high amount of titanium.



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## Experimental

The calibration graphs generated using the conditions of Table 1 are shown in Figure 1.

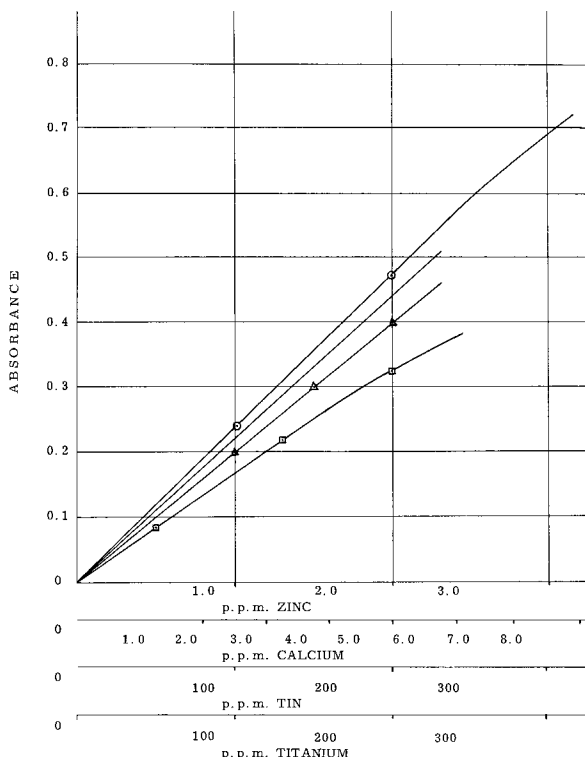


Figure 1. Calibration graphs for calcium, tin, titanium and zinc.

## Instrumentation

Table 1. Instrument Parameters

	Calcium	Tin	Titanium	Zinc
Line (Å)	4226.7	2246.1	3642.7	2138.6
Lamp Current	4 mA	8 mA	20 mA	6 mA
Spectral Band Pass (Å)	3.3 Å	3.3 Å	1.7 Å	3.3 Å
Flame	N <sub>2</sub> O-C <sub>2</sub> H <sub>2</sub>	N <sub>2</sub> O-C <sub>2</sub> H <sub>2</sub>	N <sub>2</sub> O-C <sub>2</sub> H <sub>2</sub>	Air-C <sub>2</sub> H <sub>2</sub>

## P. V. C. Sample Solutions

Approximately 2 g of P. V. C. are cut up and weighed out accurately into a 250 mL conical flask.

Twenty mL of concentrated nitric acid are added and the mixture is warmed on a hot-plate in order to initiate the oxidation.

Five mL of 60% v/v perchloric acid are carefully added and the mixture is boiled until white fumes of perchloric acid are evolved.

This procedure is repeated with another 10 mL of nitric and 4 mL of perchloric acids.

When all the nitric acid has been boiled off the mixture is heated strongly until all the organic matter has been removed and only a fine white precipitate of TiO<sub>2</sub> remains.

The contents are transferred quantitatively to a PTFE beaker and evaporated down to a volume of about 10 mL.

8 mL of nitric acid and 4 mL of hydrofluoric acid are added and the mixture is gently heated for one hour in order to complete the dissolution.

The clear solution is then transferred to a volumetric flask and made up to exactly 30 mL with distilled water.

## Standard Solutions

Standard solutions are prepared so as to cover the expected concentration of the metals in the sample solution.

## Notes on Individual Metal Determinations

### Calcium

The atomic absorption measurements are made at the 4226.7 Å resonance line, using a N<sub>2</sub>O-C<sub>2</sub>H<sub>2</sub> flame.

In order to prevent ionization of the calcium an excess of an ionization suppressant, for example, 5 000 ppm Na, is added to both sample and standard solutions. The standard solutions also contain approximately the same amount of perchloric acid as in the final diluted sample solution (approximately 1.5% HClO<sub>4</sub>).

Normal concentration range of standards:

0.5 to 8.0 ppm Ca, with 10 × scale expansion down to approximately 0.1 ppm Ca.

## Tin

The atomic absorption measurements are made at the 2246.1 Å resonance line. The use of the hotter, but less sensitive,  $\text{N}_2\text{O}-\text{C}_2\text{H}_2$  flame is preferred to the more sensitive Air- $\text{H}_2$  flame in order to eliminate or minimize possible chemical interferences.

Standard solutions containing the same amount of perchloric acid as in the sample solution are made up (approximately 25%  $\text{HClO}_4$ )

Normal concentration range of standards:

20 to 300 ppm Sn, with  $10 \times$  scale expansion down to approximately 4 ppm Sn.

## Titanium

Titanium absorbance is measured at the 3642.7 Å resonance line, using a  $\text{N}_2\text{O}-\text{C}_2\text{H}_2$  flame.

It has been observed that titanium absorbance is enhanced in the presence of hydrofluoric acid, therefore both sample and standard solutions should be fairly closely matched for hydrofluoric acid content.

It has also been found that sodium causes an interference on titanium absorbance in the presence of hydrofluoric acid (Figure 2), therefore, if sodium is present in the sample solutions it should also be present at the same concentration in the standard solutions.

Normal concentration range of standards:

20 to 300 ppm Ti, with  $10 \times$  scale expansion down to approximately 4 ppm Ti.

## Zinc

The atomic absorption measurements are made at the 2138.6 Å resonance line, using an air- $\text{C}_2\text{H}_2$  flame.

The standard solutions should contain approximately the same amount of perchloric acid as in the final diluted sample solution (approximately 1.5%  $\text{HClO}_4$ )

Normal concentration range of standards:

0.2 to 3.0 ppm Zn, with  $10 \times$  scale expansion down to approximately 0.04 ppm Zn.

The lowest given metal concentration is not the detection limit, but a concentration at which the precision of the determination is still fairly high.

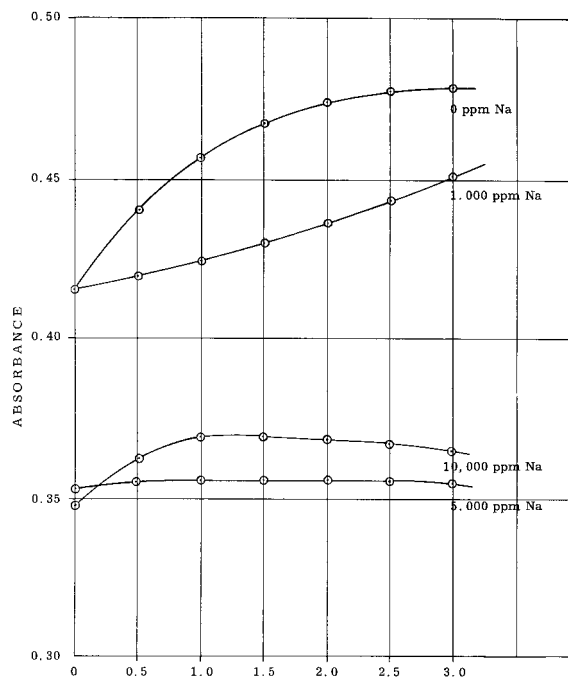


Figure 2. Effect of sodium on titanium absorbance in the zinc presence of hydrofluoric acid.

## Results and Discussion

Some typical results are shown in the table.

Table 2. Typical Results

Ca	0.015	ppm
Sn	0.023	ppm
Ti	2.01	ppm
Zn	0.024	ppm

## Conclusion

The atomic absorption spectrophotometric technique is highly suitable for the rapid and accurate determination not only of these four elements but other elements as well.

For the determination of much lower levels of these metals in polyvinylchloride a chelation-organic solvent extraction-concentration procedure would have to be carried out prior to the atomic absorption measurements.

## References

1. M. Olivier, Z. Anal. Chem., 248, 145-148 (1969)
2. M. Yanagis, M. Suzuki, T. Takeuchi, Anal. Chim. Acta, 46(1), 152-154 (1969)

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