# VALIDATION PROCEDURE FOR TOTAL SULPHUR DETERMINATION IN COMPLEX FERTILIZER MATRICES USED IN ORGANIC FARMING

**Violeta Alexandra ION[1], Andrei MOŢ[1], Roxana Maria MADJAR[1,2], Liliana BADULESCU[1,3]**

[1]Research Center for Studies of Food Quality and Agricultural Products, University of Agronomic Sciences and Veterinary Medicine of Bucharest, 59 Marasti Blvd, District 1, Bucharest, Romania

[2]Faculty of Agriculture, University of Agronomic Sciences and Veterinary Medicine of Bucharest, 59 Marasti Blvd, District 1, Bucharest, Romania

[3]Faculty of Horticulture, University of Agronomic Sciences and Veterinary Medicine of Bucharest, 59 Marasti Blvd, District 1, Bucharest, Romania

**Keywords**: sulphur, method validation, dry combustion, CHNS, organic inputs.

## INTRODUCTION

- In general, sulphur determination methods are tedious and time consuming.
- This study proposes a dry combustion method for sulphur determination, which can gain ground due to the speed and ease of application.
- This elemental analysis method is sensitive and accurate, and can be applied for the determination of total S content in many types of sample matrices, including fertilisers.

## MATERIALS AND METHODS

### Sulphur analysis parameters:
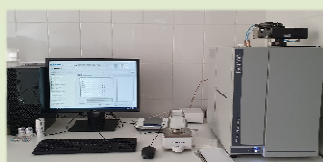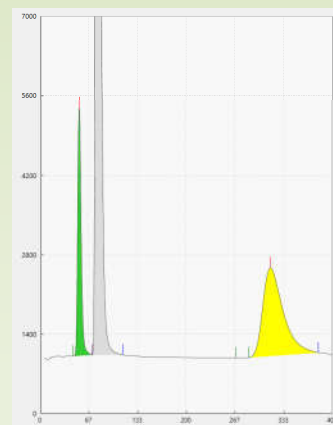


**Eurovector EA 3100 Elemental Analyzer**

| Parameter | Value |
|---|---|
| Carrier Pressure | 90 kPa |
| Reference Pressure | 10 kPa |
| Furnace #1 | 950 °C |
| GC Oven | 110 °C |
| Transfer Line | 100 °C |
| Run Time | 400 s |
| Sample Delay | 6 s |
| $O_2$ Volume | 20 mL |
| O2 Injection Rate | slow |

### Materials

Calibration standard: Cystine (Elemental Microanalysis certif. no. 347115)



Tin capsules (8x5 mm)





**Typical chromatogram of separated gases (N-green, C-grey, S-yellow)**

## RESULTS AND DISCUSSIONS

**Linearity:**
Calibration curve:



| Equation | ax+b |
|---|---|
| Slope, a | 485873 |
| Correlation coef. R | 0.9999 |
| Regression coef. $R^2$ | 0.9999 |
| y intercept, b | 22535 |

- Accurate measurement of total sulphur in any material has been shown by interlaboratory comparisons to be a problem with considerably more variation for sulphur than for other element measurements (N, C);
- Calibration curve: 3 points, 2.5 – 4.5 mg cystine (0.667 – 1.2 mg S);
- More accurate results were obtained when the linear regression coefficient was $R^2$> 0.9999, therefore we would recommend removing point from the curve in order to obtain higher regression coefficient;
- Accuracy of the method based on the standard cystine was tested several days with obtained results < 10% (from 95.61 % to 101.78 %);
- Hydrogen trap is recommended for when analyzing sulfur samples;
- When real samples reference materials were analyzed the accuracy decreased considerably (less that 80%, mainly due to pour homogeneity of the samples;

## CONCLUSIONS

- Accurate measurement of total sulphur in different types of material has been shown.
- The method can be optimised for other type of samples, such as soils, plants, and processed food.

## ACKNOWLEDGEMENTS

## REFERENCES

- Leitão, J. M. M., Costa, F. S. and Tack, F. M. G. (2001) 'Determination of Total Sulfur in Soils and Plants by an Automated Dry Combustion Method', International Journal of Environmental Analytical Chemistry, 80:3, 219 – 226
- C. Grant Kowalenko (2000) Influence of sulphur and nitrogen fertilizer applications and within-season sampling time on measurement of total sulphur in orchard grass by six different methods, Communications in Soil Science and Plant Analysis, 31:3-4, 345-354
- EURACHEM Guide - The Fitness for Purpose of Analytical Methods (2014).