APPLICATION NOTE

F&F-S-002-2018/A1

Sulphur Determination in Fertilizer

according to the Randall method

Tested with **VELP Scientifica SER 158/6 Solvent Auto Extractor** (Code S303A0380)

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SULPHUR DETERMINATION IN FERTILIZER
RANDALL METHOD

Introduction

Sulphur is one of the 17 essential plant nutrients. It is essential for the growth and development of all crops, without exception. Like any essential nutrient, sulphur also has some key functions in plants:

- Formation of chlorophyll that permits photosynthesis through which plants produce starch, sugars, oils, fats, vitamins and other compounds.
- Protein production. Sulphur is a constituent of three S-containing amino acids (cysteine, cystine and methionine), which are the building blocks of protein. About 90% of plant S is present in these amino acids.
- Synthesis of oils. This is why adequate sulphur is so crucial for oilseeds.
- Activation of enzymes, which aid in biochemical reactions in the plant.
- Increases crop yields and improves produce quality, both of which determine the market price a farmer would get for his produce.
- With reference to crop quality, S improves protein and oil percentage in seeds, cereal quality for milling and baking, marketability of dry coconut kernel (copra), quality of tobacco, nutritive value of forages, etc.
- It is associated with special metabolisms in plant and the structural characteristics of protoplasm.

For all these reasons it is very important to monitor and if necessary increase the amount of sulfur in the soil using appropriate fertilizers.

Gravimetric Sulphur Determination in Fertilizer

Hot solvent extraction process with SER 158 Series can be summed up in 5 steps, for a fully unattended operation:

During IMMERSION the sample is immersed in boiling solvent. Then the REMOVING step automatically lowers the level of the solvent to below the extraction thimble. During WASHING the condensed solvent flows over the sample and through the thimble to complete the extraction process. The fourth step involves solvent RECOVERY. Approximately 90% of the solvent used is collected in the internal recovery tank. The final step is the COOLING of the extraction cups containing the extracted matter. The extraction cups containing the extract are placed in a drying oven, cooled in a desiccator and weighed for the extract percentage calculation.

Sample

Fertilizer Expected Sulphur: 42.0 ± 0,5%

Chemicals and Equipment Required

- Analytical balance, 3 decimals
- Extraction thimbles 33x80 mm (Code A00000295)
- Glass extraction cups Ø56x120mm (Code A00000290)
- Vaflon seals (Code A00000288)
- Dichloromethane (DCM) as solvent
- Fan convection oven
- Dessiccator

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Sample Preparation

In order to obtain reliable and repeatable results it is necessary to homogenize the sample before starting the analysis. Fix every Extraction thimbles with the Extraction thimbles holders (Code A00000312). Weigh 2.5g of sample directly in the VELP extraction thimbles using the Thimble weighing cup (Code A00000310) (the quantity of sample weighed is linked to the percentage of sulfur to be extracted).

Glass Extraction Cups Preparation

Position the empty extraction cups in an drying oven (105 °C) for 1 hour.
Cool them in a desiccator until constant weight of the tare (*Tare*).
The extraction cups containing the extraction thimble con now be placed on the ultra-fast heating plate of SER158.

Extraction Procedure with SER 158

On the ControlPad select “Analysis”, and then create a new method setting the following parameters:

- Immersion Time: 60 minutes
- Removing Time: 10 minutes
- Washing Time: 180 minutes
- Recovery Time 20 minutes
- Cooling Time: 4 minutes
- Solvent: Dichloromethane (*), 100 ml
- Extraction cups: Standard Ø56x120mm
- Thimble: 33x80 mm

(*)Dichloromethane requires the energy level 4 and the **Vaflon seals** (Code No. A00000288), please select them in the appropriate customized solvent menu.

Close the safety guard and add the solvent using the automatic solvent dispensing system SolventXpress™ to minimize exposure to the solvent ensuring operator safety.
Press START to begin the extraction process. At the end of analysis position the extraction cups containing the extract in a drying oven (1 hour at 105 °C), cooled them in a desiccator to room temperature and record the accurate weight (*Total*).

Typical Results on Fertilizer

Analysis results are calculated automatically and stored in the ControlPad when entering the weights into the software (manually or automatically through a balance). The extract percentage calculation is performed by using the following formulas:

$$Extract\ (g) = (Total - Tare)$$
$$Extract\ (\%) = Extract \times 100 / (Sample)$$

Where:
Sample = sample weight (g)
Tare= weight of the empty extraction cup (g)
Total = weight of the extraction cup + extract (g)

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Sample (g)	Extract (g)	Extract (%)
2,501	1,0459	41,82%
2,501	1,0463	41,83%
2,501	1,0457	41,81%
2,502	1,0461	41,82%
2,501	1,0460	41,82%
2,505	1,0476	41,82%
	Average ± SD%	**41.82 ± 0.01**
	RSD% **	**0.02**
Expected Sulphur: 42.0 ± 0,5%		

* RSD% = (Standard Deviation x 100) / Average

Conclusion

The results obtained are reliable and reproducible in accordance with the expected values, with a low relative standard deviation (RSD<1%), that means high repeatability of the results.
The SER 158 Solvent Extractor is the ideal solution for environmental analysis such as the determination of Sulphur in fertilizer samples.

The Benefits of hot solvent extraction (Randall) by using 158 Automatic Solvent Extractor are:
- up to 5 times faster than Soxhlet (hot solvent vs. cold solvent)
- low solvent consumption (high solvent recovery, approximately 90%) - limited cost per analysis
- no exposure to solvent
- worldwide official method
- full traceability with automatic result calculation and on-board archive

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