Determination of Sulfur in Fertilizers

LECO Corporation; Saint Joseph, Michigan USA

Instrument: S832DR

Introduction

Sulfur is considered an essential mineral that is important in the development of plants. Sulfur plays key roles in the formation of enzymes, proteins, and a constituent of Coenzyme A, as well as the vitamins biotin and thiamine within the plant. Plants will leach sulfur from the soil, with sulfur historically being replenished in the soils by sulfur-containing rainfall (acid rain). As sulfur levels in rainfall are reduced with more stringent sulfur emission regulatory restrictions, the prevalence of sulfur deficiency in plants has increased, leading to the introduction of sulfur into commercial fertilizers for use in arable soils. Sulfur deficiency in cultivated crops will typically lead to stunted growth, light yellow appearance, delayed maturity, and reduction in yield. Sulfur measurement in fertilizer is used in the manufacturing and blending of the fertilizer products, as well as in generating a guarantee valu e for the commercial fertilizer products.

Reference

AOAC First Action Method 2017.08 Total Sulfur in Fertilizer by High-Temperature Combustion.

Accessories

528-203 Ceramic Crucible, 502-321 Com-Cat™, 769-608-HAZ Antimony Metal, 769-610-HAZ F-Cl Absorbent.

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

Method Parameters

General Parameters

Furnace Temperature	1350 °C
Lance on Delay	20 s
Nominal Mass	1.0000 g

Element Parameters

Wait for Baseline Stability	Yes
Starting Baseline	2 s
Use Comparator	Yes
Comparator Level	0.30 %
Minimum Integration Time	120 s
Maximum Integration Time	360 s
Range Select	Auto
Range Lower Limit	975
Range Upper Limit	1000

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Replace Secondary Anhydrone Filter with Chlorine Trap. Refer to Bulletin #202-001-315.
3. Condition the Chlorine Trap by analyzing 3 to 5 replicates of a chlorine-containing material following the procedure outlined in step 6.

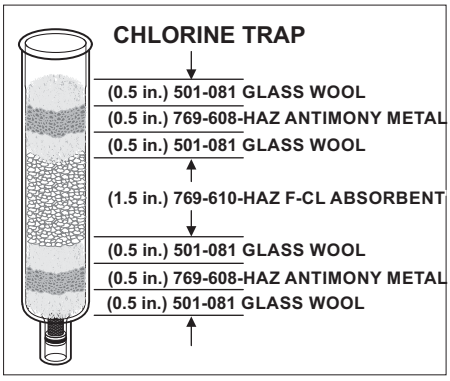
   NOTE: The trap has been successfully conditioned when stable sulfur values are obtained for three sequential replicates of the chlorine-containing material. The trap has been depleted when the F-Cl Absorbent has turned a dark brown and/or the bottom layer of the antimony metal becomes tarnished.

4. Determine instrument blank.
   a. Login a minimum of three blanks.
   b. Add ~1.0 g of 502-321 Com-Cat™ to a 528-203 Crucible.
   c. Place the crucible in front of the furnace entrance or in the appropriate autoloader position.
   d. Initiate the analysis by pressing the Analyze button.
   e. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
   f. Repeat steps 4b through 4e a minimum of three times.
   g. Set the Blank according to the procedure outlined in the operator's instruction manual.
5. Instrument calibration/drift correction.

   Note: This application requires that samples and reference materials are concentration matched. In cases where the sulfur concentrations are known, the samples should be sorted and analyzed to best match the reference material calibration range. In cases where the sulfur concentration is unknown, it is necessary to perform a test analysis to determine the approximate sulfur content. Please refer to Table 1 for recommended reference materials.

   a. Login a minimum of five Standard reps for each calibration/drift reference material to be used for calibration/drift.
   b. Weigh ~0.03 to 0.15 g of the suggested reference material from Table 1 into a 528-203 Crucible.

   Note: To meet the precision requirements for AOAC First Action Method 2017.08 Total Sulfur in Fertilizer by High-Temperature Combustion, a minimum of five calibration points are required for a linear curve. It is recommended to select five sample masses within the specified range of step 5b to establish the instrument calibration.



Organic Application Note

c. Enter the mass and reference material identification into the Standard login.
d. Add ~1.0 g of 502-321 Com-Cat™ on top of the sample and thoroughly mix.
e. Place the crucible in front of the furnace entrance or in the appropriate autoloader position.
f. Initiate the analysis by pressing the Analyze button.
g. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
h. Repeat steps 5b through 5g a minimum of five times for each reference material.
i. Calibrate/drift correct by following the procedure in the operator's instruction manual.

6. Sample Analysis.
   a. Login a Sample with the desired amount of reps.
   b. Weigh ~0.05 to 0.10 g of a fertilizer sample into a 528-203 Crucible.
   c. Enter the mass and sample identification into the Sample login.
   d. Add ~1.0 g of 502-321 Com-Cat™ on top of the sample and thoroughly mix.
   e. Place the crucible in front of the furnace entrance or in the appropriate autoloader position.
   f. Initiate the analysis by pressing the Analyze button.
   g. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
   h. Repeat steps 6b through 6g as necessary.

Table 1. Recommended Reference Materials

Expected Sulfur Conc.	Recommended Reference Material	Calibration Type	Unknown Sample Analysis Mass (mg)
<1%	BBOT	Linear, Forced Through Origin	100
1-7%	BBOT	Linear, Full Regression	100
7-35%	Ammonium Sulfate	Linear, Full Regression	100
>35%	Sublimed Sulfur	Linear, Full Regression	50

Typical Results

Name	*Mass (g)*	*% Sulfur*
Magruder 2011-05	0.1011	1.49
Organic Poultry Litter	0.1064	1.47
	0.1017	1.48
	0.1032	1.45
	0.0999	1.47
	Avg =	***1.47***
	s =	***0.02***
KMAG Fertilizer, Langbenite	0.1058	21.48
	0.1025	21.53
	0.1071	21.53
	0.1042	21.58
	0.1055	21.53
	Avg =	***21.53***
	s =	***0.04***
Calcium Sulfate Dihydrate	0.1021	18.51
Sigma-Aldrich	0.1064	18.52
NF Grade Reagent	0.1017	18.54
	0.1059	18.51
	0.1037	18.53
	Avg =	***18.52***
	s =	***0.01***

Name	*Mass (g)*	*% Sulfur*
Ammonium Sulfate Fertilizer	0.1043	23.96
	0.1028	23.90
	0.1052	23.89
	0.1065	23.94
	0.1057	23.97
	Avg =	***23.93***
	s =	***0.04***
Elemental Sulfur Fertilizer	0.0517	82.56
	0.0502	82.49
	0.0520	83.17
	0.0513	82.72
	0.0510	82.49
	Avg =	***82.69***
	s =	***0.29***

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