

Determination of Elemental Sulfur from S-Rich Soil and Fertilizer by Titrimetric Method

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Abstract

In the present study the emphasis was given on two points, one is find out the effective extractants solvent and another is method of determination. Importance also was given on extraction ratio between solvent used as extractants and the amount used or dilution of the sample, shaking time and the incubation period on the basis of which after shaking how much time is required for settle down the solution just before titration/determination. The better result was obtained in 45 minutes to one hour after extraction was completed. Among three extractants, determination of elemental sulfur (E^S), acetone can be used as an effective extractant for both S-rich fertilizer and soil sample (% recovery values obtained were 99.0 and 97.0% respectively). On the contrary, the % recoveries with toluene and xylene the values obtained from S-rich fertilizer and S-rich soils were 97.0 and 93.0 and 92.0 and 87.0% respectively. Determination was done with titrimetric method. Colorimetric always time consuming, values obtained with spectrophotometer is sometimes variable and expensive due to using of power supply. In titrimetric method where a high degree of accuracy and precision is not required. It is rapid and suitable for routine analysis in laboratories without using specialized equipment.

Keywords: Elemental sulfur; Colorimetric; Spectrophotometer; Extractants; Solvent

Introduction

Elemental sulfur (E^S) is employed to produce acidity in the soil and as well as reclaim alkaline lands and as a fertilizer for crops. One pound of sulfur added to the agricultural land will makes three pounds of sulfuric acid. On the other hands, one part of sulfur being equal to five parts of gypsum in reclamation. Early attempts at analyzing soils and fertilizer for sulfur content were confined to the determination of sulfate ions, later total sulfur were made and organic sulfur was reported as the difference between total and sulfate ions. For elemental sulfur determination of fertilizer very few works has been reported although so many procedures have been developed for the soil related methods.

Elemental sulfur (E^S) from soil

So many methods have been prepared by different analyst in different time extraction methods based on using different type of organic solvent (without considering whether it is costly or volatile) in different ratio between the extractants and the amount of sample to be used for bringing E^S in solution with shaking period in various incubation time and the method of evaluation (colorimetric or titrimetric) is very important for this determination. E^S can be extracted from soils with acetone, ether, pyridine, with steam of distillate with chloroform; Schoenberg reagent, Ref. [1] isolated free sulfur from soil with steam and extracted the distillate with chloroform. In Ref. [2,3] used a modification of Morris method to determine E^S that is based upon the reaction of sulfur with sodium sulphite to give thiosulfate. The determination of E^S in acetone extracts originally prepared by Ref. [4] involved the direct precipitation of E^S by solvent exchange. When applied to the analysis of soil extracts the method is subject to interference from acetone-soluble organic matter and the procedure should not be used uncritically for highly organic soils. Preliminary investigations were conducted to determine i) the most suitable extractant for E^S polluted forest floor materials, and ii) the effect of soil moisture and the extraction time on the recovery of added E^S .

Temperature may also affect in the recoveries of E^S . Drying soil at

temperature greater than 25°C has reported to show lower recoveries of E^S [5]. According to Ref. [3] 5 minutes are adequate for the removal of E^S from soil with acetone, but Ref. [6] have suggested that complete extraction of sulfur was achieved only after 3 hour. But in solubility tests approximately 45 minutes were required to dissolve pure sulfur in acetone. Amount of soil used varied between 0.2 and 0.1 g (oven-dry weight basis), depending on the expected E^S concentration. The minimum extraction ratio between extractants and the sample should be 100:1 or depending on sample containing more than 50% of E^S (then ratio should be 175:1). E^S in soils is generally determine only those soils having E^S less than 100 mg Kg^{-1} and sulfur concentration in excess of 50000 mg Kg^{-1} could be measured. Lower ratios produced excessive color interference even with the use of a color blank. Increasing the extractant to soil ratio did not affect the recovery of E^S .

E^S of soil

In the present inquisition 0.2 g sample was mixed to the 20 ml of each extractant, i.e., acetone, toluene and xylene at the ratio of 1:100. The solution was shaken thoroughly on suitable shaking machine for 45 minutes to 1 hour and allowed to stand for overnight. Then the solution was filtered through Whatman no.44. Now 2 ml of aliquot was taken in 50 ml white porcelain basin, 2 ml of Potassium cyanide (KCN, 0.1M: 0.6519 g/100 ml) was added to the sample solution to give thiocyanide which was then titrated with 0.025M $AgNO_3$ (4.2467 g/L) in presence of 2 ml of 0.33 M formalaldehyde (1 ml of Conc.HCOH/100 ml) to bind residual cyanide ions.. For this reason same amount of KCN was added in the solution as to the amount of aliquot in the procedure to

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Extractants	Highly-S contained Polluted soil. Original E ^s amount=28.66%		Highly-S contained Fertilizer. Original E ^s amount=90.58%	
	Amount of E ^s in % by the present method	% recovery	Amount of E ^s in % by the present method	% recovery
Acetone	27.66	96.54	89.92	99.02
Toluene	26.53	92.56	87.52	96.62
Xylene	25.00	87.26	83.13	91.78

Table 1: Original E^s contained in soil and fertilizer and % recovery through the titration methods by three extractants procedures.

achieve the maximum recovery from the sample. Care must be taken during titration, pour down each drop from burette containing 0.025M AgNO₃ to the mixture after 4 to 5 seconds. At the end of titration the color should give bright coffee color. The titration reading (R) was note down. Average value of three readings was used in the following calculation after standardize or correction the 0.025M AgNO₃ strength with 0.025M NaCl solution (f).

Calculation:

$$\text{Elemental-sulfur in \%} = \frac{32.06 \times 35 \text{ ml} \times 6 \text{ ml} \times 100 \times \text{Xf} \times \text{XR}^*}{1000 \times 0.2\text{g} \times 2\text{ml}}$$

From the Table 1 out of three extractants (i.e., acetone, toluene and xylene) acetone as an extractants gave the best results. The values obtained were about 97, 93.5 and 87% respectively. So, suggestion can be made for E^s of soil, acetone can be use as an extractant for soil E^s safely.

E^s of high content –S in fertilizer

Before determination of E^s the extraction was made at the ratio of 175:1, three extractants were used in this determination i.e., 35 ml of acetone, toluene and xylene: 0.1 g of sample and then all steps of above procedures up to calculation were followed accordingly.

Among three extractants acetone and toluene were found to be significant for E^s of high content –S in fertilizer determination. In Table

1 revealed that the % recovery of E^s was obtained best with acetone and toluene the values were found to be 99.0 and 97.0 respectively, whereas xylene showed about 92%. Acetone and toluene have more or less same capacity in extracting E^s but toluene has some advantages over acetone regarding cost of the chemicals. So, simultaneously acetone and toluene could be used for this purpose.

Calculation:

$$\text{Elemental sulfur in \%} = \frac{32.06 \times 35 \text{ ml} \times 6 \text{ ml} \times 100 \times \text{Xf} \times \text{XR}^*}{1000 \times 0.2\text{g} \times 2\text{ml}}$$

*Strength of 0.025N AgNO₃

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