Determination of Total Sulphur in Geological Reference Materials by Non Dispersive Infra-Red Detector based Sulphur Analyser

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Precise data on sulphur in geological materials is essential to understand the crustal evolution, framework of magmatic process and identifying economic mineral deposits. The classical method for determination of sulphur using Eschka mixture and precipitation of total sulphur as $BaSO_4$ suffered with problems when dealing with rock samples having extremely low levels of sulphur, which is also very tedious and cumbersome. Although various models of automated micro elemental analysers for sulphur determination have been used since 1960, the determination at extremely low levels is a challenging task even today.

The present work is aimed at the determination of total sulphur contents of Geological Reference Materials (GRMs) from various international sources (USGS, GSJ, CCRMP) by sulphur analyser equipped with an automated Non Dispersive Infra-Red (NDIR) based detector following combustion at 1150 °C with influx of pure oxygen gas. Rock sample powders have been mixed with oxygen donors/combustion catalysts viz. WO_3 and disodium tetraborate (borax) for complete oxidative break down and the products of combustion were sent to the NDIR detector for measurement.

By plotting sulphur concentrations against the mass normalised peak intensity values using appropriate GRMs, two calibration curves (Fig.1 & 2) were constructed to cover dynamic range of absolute sulphur (as) concentration in rocks one with 0 to 267.6 (as) and the another with 263.2 to 3399.7 (as) to eliminate the ambiguity of measurement precision. Sample weights of test portions are varied from 0 to 202 mg for optimisation of sample quantity for different rock types.

From the preliminary work, it is observed that the values obtained in these investigations are in close agreement with the recommended values for various GRMs (Table). Further work is in progress to optimise the test portion weight for entire range of geological materials (igneous, sedimentary and metamorphic rocks) using a variety of oxygen donors/combustion catalysts.

Table

Sample	Sulphur Concentration (µg/g)	
	This Study (µg/g)	Recommended (µg/g)
SARM 39 (05)	1446 ± 113.3	1500
SARM 41 (08)	1423.3 ± 89.62	1500
SARM 45 (05)	512 ± 68.06	500
JSD 1 (21)	68.3 ± 0.57	68
JSD 2 (06)	13589 ± 1051	13000
SGR 1 (05)	15400 ± 529.15	15300
MRG-1 (13)	674± 71	610
GXR 1 (5)	2841.8±229.19	2500

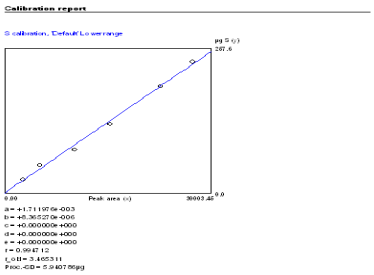

Figure 1

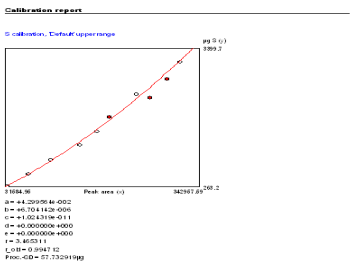

Figure 2