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**MINES BRANCH INVESTIGATION REPORT IR 65-54**

**THE DETERMINATION OF SULPHUR IN  
METEORITIC MATERIALS AND BASIC ROCKS**

by

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**MINERAL SCIENCES DIVISION**

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SUMMARY

Sulphur in meteoritic materials and basic rock was determined by a combustion-titrimetric method and also by the classical gravimetric procedure. The results by the two methods agreed satisfactorily between themselves and also with figures obtained by the gravimetric procedure in another laboratory.

The methods used are described briefly.

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## INTRODUCTION

The Geological Survey of Canada requested a referee analysis for the determination of sulphur in three samples of meteoritic material and one sample of basic rock.

Dr. J.A. Maxwell, of the Geological Survey had informed the authors that his laboratories had experienced a discrepancy between the results for the total sulphur content of these materials by two different methods. The classical gravimetric procedure, preceded by a preoxidation treatment with a carbon tetrachloride-bromine mixture had given consistently higher sulphur values than the other method namely a combustion-titrimetric procedure developed in the Geological Survey laboratories for the determination of low sulphur in basic rocks. These differences were as high as 0.7% for sulphur content in the 2% range. Each method, however, was precise.

It was decided that essentially the same two procedures would be used in the Mineral Sciences Division. The determination was done by a combustion-titrimetric technique, as adapted and used in the Mineral Sciences laboratories, and also by the classical gravimetric method. Quadruplicate determinations were done for each sample by each method. J.C. Hole performed the combustion determinations and D.J. Charette the gravimetric. Details of the methods are as follows.

## COMBUSTION-TITRIMETRIC METHOD

A combustion-titrimetric procedure similar to the method used for the determination of sulphur in iron, steel and iron ores was employed. The empirical nature of this technique, which, although suitable for the low sulphur contents of the materials for which it was developed, is such that its accuracy depends entirely on the use of suitable standards. In this particular case, because no meteoritic materials with a standardized sulphur value were available, NBS cast iron standards were used.

The weighed sample is transferred to a combustion boat equipped with a cover, and a suitable fluxing agent is added. The boat is then placed in the combustion tube of a Leco furnace maintained at 1500°C, and ignited for five minutes in a stream of oxygen flowing at approximately one litre per minute. The combustion gases are carried along with the oxygen and bubbled through an absorption cylinder containing silver nitrate solution and a suitable acid-alkali indicator. The nitric acid produced by the reaction of the SO<sub>2</sub> and AgNO<sub>3</sub> is titrated with a sodium hydroxide solution standardized in terms of sulphur.

## GRAVIMETRIC METHOD

The classical gravimetric procedure for the determination of sulphur as barium sulphate, preceded by treatment with a carbon tetrachloride-bromine mixture, was used.

The weighed sample (one gram) is transferred to a beaker and a mixture of carbon tetrachloride and bromine is added. After standing for one half hour nitric acid, saturated with potassium chlorate, is added and the solution is evaporated to dryness. The dried residue is treated with HCl and again evaporated to dryness. This step is repeated and the residue finally treated with a few ml of HCl and water, heated to boiling, and filtered to remove the insoluble residue.

The filtrate is heated to boiling, ammonia is added to just precipitate the iron followed by sufficient barium chloride solution to precipitate the sulphate as barium sulphate. The solution is then acidified to dissolve the iron precipitate and an excess of one ml of HCl per 100 ml of solution added. It is boiled and set aside to cool for 30 minutes. The solution is then filtered, washed until free of chloride, keeping the volume of water as low as possible. The paper and precipitate are transferred to a tared crucible and ignited carefully to a final temperature of approximately 900°C (1).

## RESULTS

A comparison of the results showed good precision for each method and acceptable agreement between the two methods, although the combustion procedure gave consistently higher values. This is in direct contrast to the experience of the Geological Survey laboratories. The largest difference was 0.16% which is definitely lower than the differences reported by Dr. Maxwell. The values obtained in the Mineral Sciences laboratories agreed well with the results obtained by the gravimetric procedure in the Geological Survey laboratories.

The table presents the results of the determinations done in the Mineral Sciences laboratories and the average gravimetric figure supplied by Dr. Maxwell.

## REFERENCES

1. G.E.F. Lundell, H.A. Bright and J.I. Hoffman, "Applied Inorganic Analysis", 2nd edition, p.702, 1953.

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DJC:JCH:ST

TABLE 1

Lab No.	Sample Description	Combustion- titrimetric J.C. Hole	Per Cent Sulphur	
			Gravimetric D.J. Charette	Gravimetric Geological Survey
1028	966-64 meteorite, non-magnetic part same as 965-64 except that metal is reduced to 1%	2.39	2.22	
		2.39	2.22	
		2.29	2.21	
		<u>2.35</u>	<u>2.22</u>	
		Av. 2.36	2.22	2.19
1029	965-64 meteorite, non-magnetic part 92% silicates, 2% metal, 6% sulphide (approx. 2% S)	2.38	2.20	
		2.37	2.17	
		2.28	2.16	
		<u>2.28</u>	<u>2.15</u>	
		Av. 2.33	2.17	2.17
1030	C-856 meteorite 92% silicates, 1% metal (Fe <sub>1</sub> Ni) and 7% sulphide (FeS), (approx. 2% S)	3.18*	2.60	
		2.77	2.62	
		2.65	2.61	
		<u>2.65</u>	<u>2.62</u>	
		Av. 2.69	2.61	2.58
* omitted from average				
1031	EHO 672 amphibolite silicate, high magnesium, with total sulphur of approximately 0.2%	0.232	0.202	
		0.212	0.199	
		0.228	0.195	
		<u>0.214</u>	<u>0.192</u>	
		Av. 0.222	0.197	0.20