



Trade Science Inc.

ISSN : 0974-7419

Volume 10 Issue 11

Analytical CHEMISTRY

An Indian Journal

Full Paper

ACAIJ, 10(11) 2011 [716-718]

Establishment of analytical method for analysis of IR 218 flare composition by accelerated solvent extractor (ASE) and atomic absorption spectrometer (AAS)

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Received: 15th April, 2011 ; Accepted: 15th May, 2011

ABSTRACT

IR decoy flares are used as countermeasure against IR guided missiles. Solid IR flare compositions consisting of Magnesium, Teflon and Viton, commonly known as MTV composition is being used in military aircrafts. An instrumental method for complete analysis of IR-218 flare has been established using Atomic Absorption Spectrometer (AAS) and Accelerated Solvent Extractor (ASE). The results are accurate and the method is time saving (89%) in comparison to the conventional method.

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KEYWORDS

Accelerated solvent extractor;
Atomic absorption
Spectrometer;
IR flare;
MTV flare.

INTRODUCTION

IR guided surface to air missiles (SAM) or air to air missiles (AAM) track the fighter aircrafts by sensing the hot gases evolved from the flames and hence they are a major threat to military aircrafts. Infra red (IR) Decoy flares serve as countermeasure against these heat seeking IR guided missiles and are usually ejected either individually or in salvoes by the pilot or automatically by tail-warning devices when an attack is imminent. Flares which are currently in use are made up of a solid pyrotechnic composition of magnesium, Teflon (PTFE) & Viton (vinylidene fluoride-hexa fluoroisopropene - copolymer) and are commonly called MTV flares. These flares produce relatively high heat ($\Delta H = -1438 \text{ kJ mol}^{-1}$) and give a great flame emission.

HEMRL has developed an improved infrared (IR) flare composition containing Magnesium, Teflon and Viton A (48:48:4), that possesses excellent tensile

strength and superior IR signature. The present study was under taken for the establishment of instrumental analytical technique for the detailed analysis of developed composition of IR 218 Flare. This paper describes the complete analysis method by instrumental as well as conventional techniques.

EXPERIMENTAL

Materials and Chemicals:

All chemicals and solvents were of analytical grade. Deionised water was obtained from the MILLIPORE water system (Elix-10 followed by Milli-Q 185 Plus and 0.22 μm filter unit). Standard Mg (1000ppm) from Merck was used for estimation of Mg by AAS.

Analysis Procedure

The process followed for the complete analysis of the composition has been summarised in the flow chart

(Figure 2).

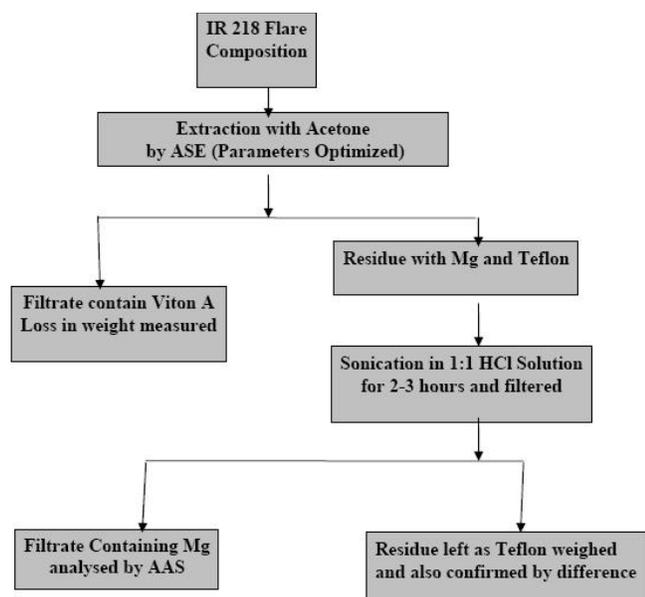


Figure 2 : Processes involved in complete analysis of IR 218 Flare Composition

Separation of Viton A by Accelerated Solvent Extractor (ASE) and Conventional Method.

Sample (1 g) was taken in a tarred G3 Gooch crucible. Using soxhlet apparatus, the sample was extracted with 250 ml of acetone for 6 hours. The acetone soluble ingredients were collected in an iodine flask. After thorough drying, the crucible was weighed. It showed the constant weight loss of 3.72%. For the extraction by accelerated solvent extractor (ASE), sample (0.4-0.5 g) was taken in high strength corrosion resistant stainless steel sample cells and the instrument was programmed for the extraction parameter such as pressure, temperature and time cycles. The parameters of temperature, time cycles and static time were optimised with acetone keeping pressure constant at 3000 psi. Initially temperature (5°C above the normal boiling point of acetone, 56°C) and static time were kept constant and time cycle was varied gradually using 10 ml cell until the specified weight loss was achieved. Then by keeping time constant, experiments were carried out with gradual increase of temperature with simultaneous decrease in number of cycles to get the same specified weight loss and optimised parameters were obtained. Results are given in TABLE 1. The total time for extraction comes out to be 40 minutes (4 cycles of 10 minutes each). Thus time saved for extraction is around

89% in comparison to soxhlet extraction. To check the reproducibility of the method, 5 different samples were extracted and percentage of R.S.D was calculated and found to be within 1.7% (TABLE 2).

TABLE 1 : Optimisation of Extraction Parameters for Viton A with solvent acetone using ASE

Pressure (psi)	Temperature (°C)	Static Time(min)	Cycles (Nos.)	Weight Loss
3000	60	10	3	1.62%
			5	2.35%
			7	3.14%
	70	10	2	2.28%
			4	3.31%
			6	3.83%
75	10	2	2.56%	
		4	3.94%	

TABLE 2 : Quantitative Estimation of Viton A using optimized ASE condition

Sample	Weight Loss (%)	R.S.D	Total Time	Remark
I	4.04	1.7%	40 minutes	89 % time is saved by ASE
II	3.94			
III	4.10			
IV	4.11			
V	3.98			
Conventional Extraction	3.72	----	6 hours	

Separation of Teflon from the residue

The residue left after extraction of Viton A by ASE which contained Mg and Teflon was subjected to sonication in 1:1 HCl (100 ml) solution until the Mg trapped in the Teflon matrix dissolved completely (3 hours). The solution was filtered using G-3 crucible and residue (as Teflon) was dried and weighed and its weight was compared with that obtained by the difference of Viton A and Mg (determined quantitatively by AAS) from the total weight of the sample.

Quantitative Estimation of Mg by AAS

Standard solution of Mg (1000ppm) has been used for analysis. Standard solutions of Mg in the range 1-10 ppm concentration were freshly prepared by diluting standard of 1000 ppm by deionised water before each analysis.

TABLE 3 : Instrumental Analysis Result of IR 218 Flare

Ingredients	Specified Values	Results (%)					Mean	RSD
		I	II	III	IV	V		
Mg Gr. V (Imported)	48%	47.36	47.71	47.82	47.25	47.86	47.60	0.59%
Viton A	04%	4.04	3.94	4.10	4.11	3.98	4.03	1.7%
Teflon (ByDifference)	48%	48.60	48.35	48.08	48.64	48.16	48.37	0.51%

All experiments were carried out with AA800 atomic absorption spectrometer equipped with Deuterium as well as Zeeman correction background correction.

All the absorption measurements of Mg were carried out at 202.6 nm in the instrumental conditions that provide the best sensitivity. These conditions were 15mm burner height with 10 cm burner head, at 1.0 nm spectra bandwidth and 0.7 nm slit width and with Air-acetylene flame having 0.9 L/min C₂H₂ flow.

The quantitative estimation of Mg in the solution was done by aspirating the standard solutions prepared in the range of 1-10 ppm into the flame and measuring the absorbance. The calibration graph of absorbance versus concentration was then plotted (Figure 1). Similarly the absorbance of sample solutions was also measured and the concentration of Mg in sample was then calculated using the calibration graph. Results are given in the TABLE 3.

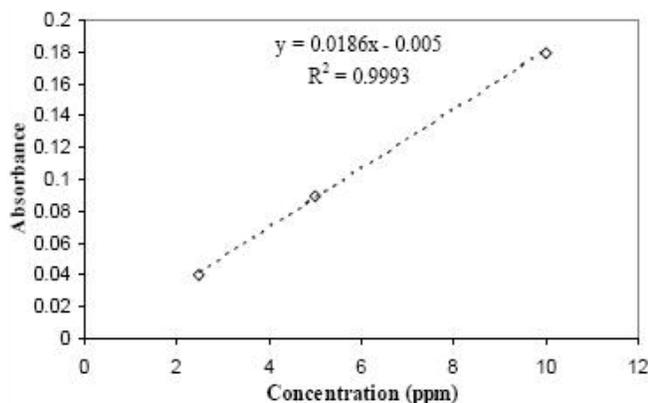


Figure 1 : Calibration graph for Mg

RESULT AND DISCUSSION

The analysis results of five different samples of the same batch of the IR 218 Flares are shown in TABLE 3.

It is evident from the results that all the ingredients (Mg, Teflon & Viton A) are within the specified values. This method is more accurate (RSD < 2% for all the three

ingredients). The IR flare composition is analysed completely by instrumental techniques (ASE and AAS) which saves a lot of time. In comparison to Soxhlet extraction of Viton A from the composition, ASE saves around 89% of time.

CONCLUSIONS

The instrumental method for complete analysis of indigenously developed IR 218 Flare MTV composition (Mg, Teflon, Viton A) by using Accelerated Solvent Extractor (ASE) and Atomic Absorption Spectrometer (AAS) has been established which is otherwise difficult to analyse by conventional method. The method is accurate and saves time up to 89%.

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