

## A novel method for quick determination of trace amounts of vanadium (V) in fuel oil

N K Kshatriya

Research & Development, Bharat Aluminium Co Ltd, Korba (MP)  
and

Basant R Sahu

C M D Postgraduate College, Bilaspur (MP)

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A highly selective spectrophotometric method has been developed for the determination of vanadium (V) in fuel oil. Vanadium reacts with *n*-benzylbenzohydroxamic acid (BBHA) in hydrochloric acid medium to form a highly stable reddish violet complex, extractable into chloroform having the maximum absorbance at 510 nm. The system conforms to Beer's law over the concentration range of 2-8 ppm of vanadium. The results have been compared with phosphotungstate method.

The list of the metals found in petroleum is long<sup>1</sup>. The knowledge of the presence of metals in fuel oil is essential because metal oxides are highly corrosive and may lead to atmospheric pollution. Vanadium in petroleum ranges from 1-1000 ppm along with other heavy metals<sup>2</sup>. When the oils are burnt considerable amounts of vanadium oxide is carried away by the exhaust gas. The oxide of vanadium is highly corrosive<sup>3</sup>. Moreover, it causes health hazards like anemia, cough, emaciation, irritation of mucous membranes and gastrointestinal disturbances<sup>4</sup>. According to Fairhall<sup>5</sup> there is no question that vanadium compounds are toxic for even such material as crude or dust. The maximum allowable concentration reported is 0.5 mg of V<sub>2</sub>O<sub>5</sub> dust per cubic meter of air and 0.1 mg per cubic meter of vanadium in fume<sup>6</sup>.

Many chemical and instrumental methods have already been proposed. Neutron activation analysis (NAA), atomic absorption spectrometer (AAS) are powerful techniques but are limited in their wide application owing to be expensive required considerable floor space and high technical personnel. Spectrometry is still the preferred tool within the reach of the many of laboratories. The IP suggests sodium tungstate and haematoxylin reagents for vanadium estimation<sup>7</sup>. The ASTM has also prescribed atomic absorption and phosphotungstate method<sup>8</sup>. The IS has also given the phosphotungstate method<sup>9</sup>. The use of *n*-phenylbenzohydroxamic acid (PBHA) is also proposed for this purpose, but the method using these reagents suffer from the interferences of iron, molybdenum, zirconium and

titanium<sup>10</sup>. *n*-Benzylbenzohydroxamic acid (BBHA) is more selective than PBHA. BBHA has been reported to be a highly selective reagent for the determination of vanadium in biomaterials<sup>11</sup>. *n*-Benzylbenzohydroxamic acid has been employed for selective extraction and subsequent spectrophotometric determination of vanadium in fuel oil in the present communication.

### Experimental procedure

#### Apparatus

Absorption spectra were recorded on the Bausch & Lomb Spectronic-20 spectrophotometer, with 10 mm matched glass tubes.

#### Reagents and solutions

All the reagents used were of A R grade, *n*-benzylbenzohydroxamic acid (BBHA) was prepared and purified as described in the literature<sup>12</sup>. A 0.1% (w/v) solution of BBHA in chloroform was used for extraction purposes.

Standard solution of vanadium was prepared by dissolving ammonium meta vanadate (A R) and the solution standardised volumetrically<sup>13</sup>.

#### Procedure for testing sample

The samples were collected from various batches of fuel oil received at Korba Aluminium Project of M/s BALCO Ltd (MP). 25.00 g of fuel oil was taken into a 250 ml boro silicate glass beaker and added 2.5 g of potassium hydrogen sulphate. It was heated gently over a low flame till the oil fumes completely burnt. Heating was continued until the burning ceases.



Cooled and added mixture of 25 ml conc.  $\text{HNO}_3$  and  $\text{HClO}_4$  (5:1) digested until no black particle is left over. Evaporated to dryness and dissolved with 10 ml conc.  $\text{HCl}$ , filtered and diluted to 100 ml with distilled water in a volumetric flask.

#### Colour development

10 ml aliquot was placed in a separatory funnel. 10 ml potassium persulphate (1%) was added and acidity was adjusted between 3-7 M by adding conc.  $\text{HCl}$ . Then 10 ml chloroform solution of BBHA was added, shaken for two minutes and allowed to settle. The chloroformic layer was drained into a beaker containing anhydrous sodium sulphate. Extraction was repeated with further quantities (3, 2 ml) of the reagent solution. All the extracts were collected into 25 ml volumetric flask and the volume was made up to mark with chloroform. The absorbance was measured at 510 nm. The amounts of vanadium in sample was deduced from a calibration curve.

#### Results and discussion

The vanadium contents determined by phosphotungstate method and present extraction method are given below:

Sample No.	Present method	Std. deviation	Phosphotungstate method
	'V' in ppm		'V' in ppm
1	1.12	$\pm 0.007$	1.15
2	2.40	$\pm 0.009$	2.44
3	2.97	$\pm 0.009$	3.10

Results show that the proposed method matches well to phosphotungstate method recommended by IP, ASTM and IS. Further, the present method using BBHA is rapid, accurate and more selective as the method does not suffer interferences of Mo(V), Zr(IV), Ce(IV) and tolerates Fe(III) and Ti(IV) to a greater extent. No masking agent is required during extraction of vanadium (V) when extracted in presence of these metal ions.

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