

# The Determination of Metals in Welding Fume by X-Ray-Spectrometry

O V Kuznetsova <sup>1</sup>, L A Begunova <sup>1</sup>, S V Romanenko <sup>2, a</sup>, S A Solodsky <sup>2</sup>

<sup>1</sup> Irkutsk National Research Technical University, Irkutsk

<sup>2</sup> Tomsk Polytechnic University, Tomsk

E-mail: <sup>a</sup>svr@tpu.ru

**Abstract.** Analysis of the current hygienic situation in the welding production showed that the intensification of welding processes involves the deterioration of air quality, which negatively affects the welders health. Respiratory effects seen in full-time welders have included bronchitis, airway irritation, lung function changes, and a possible increase in the incidence of lung cancer. The metal concentration in the air of the working area have been determined using the photometric method of analysis, which involves the stage of decomposition of the sample material before analysis. However, losses of the analyzed elements are possible when the sample is decomposed. The X-ray fluorescence method of analysis has the advantage of being non-destructive. The investigations shown the data of photometric determination of metals in welding aerosols is 1.5÷2 times lower than the results of X-ray fluorescence analysis.

## INTRODUCTION

Welding is one of the main processes widely used to join metals in many industries, but this method of processing metals are the most unfavorable in terms of hygiene. Welding is one of the more important sources for fine and ultrafine particles the inhalation of which adversely affects human health. The particles of welding fumes may contain manganese, beryllium, cadmium, chromium, vanadium, antimony, zinc, nickel, molybdenum, mercury, lead, iron and cobalt [1, 2]. The chemical composition of the welding fumes depends upon various factors like type of welding, metal coating, material of the electrode and type of metal being weld. Welding fume pulmonary effects have been associated with bronchitis, metal fume fever, respiratory tract irritation, fibrosis, cancer and functional changes in the lung [1].

The data on the qualitative and quantitative composition of welding aerosols are needed to improve the working conditions of welders. It should be noted that the problem of quality laboratory monitoring of the air condition of the working area is very relevant and its role increases with the wear of technological equipment and the introduction of modern technologies.

A samples of welding fumes are collected on polyvinyl chloride filters or mixed cellulose ester filters. A number of analysis methods have been used to analyse welding fumes collected from workplace air in industrial enterprises. These have included atomic emission spectroscopy, atomic absorption spectroscopy, photometric method of analysis, X-ray fluorescence spectrometry and others. Photometric method (PhM) of analysis is mainly used, but one requires a large amount of time for preliminary sample preparation, while in the course of a long analysis changes in the structure of the aerosol can occur and loss of analyte is observed when the sample material is decomposed.

Non-destructive X-ray fluorescence (XRF) method of analysis is more perspective. The XRF method is the express and allows to study the welding aerosols immediately after its selection. However, the introduction of rapid XRF methods for determining the metal content in welding aerosols is limited to the absence of reference materials (RM) of composition of welding fumes samples collected on the filter. There are only a few copies of reference materials found in the foreign analytic practice, for example, BCR-545 (IRMM, Belgium), HSL MSWF-1 and HSL SSWF-1 (UK). Sample BCR-545 [3]



is a glass fiber filter loaded with welding dust; it is certified only for the content of Cr (VI) and total Cr. Samples HSL MSWF-1 [4] and HSL SSWF-1 [5] are welding fumes powder particles collected by welding of mild steel and stainless steel respectively, packed in glass bottles of 1 g each; they are certified for the content of Fe, Mn and Zn.

### PROBLEM

Particular complexity in the creation of CO of welding aerosols is represented by wide variations in particle size, as well as the complexity of the phase and chemical composition of TSSA. The solid particles of the welding spray are generally 0.1 to 1  $\mu\text{m}$  in size, regardless of the welding technique [6]. The phase and chemical composition of the TCCA depends on the welding conditions, therefore it is required to create standard samples for each type of welding separately.

The certification of RM as real materials collected on the filter is not possible as it is required to produce a large number of samples copies with the same composition, in order to use a part of them for certification analyses and to use the remaining ones as certified RM. However, the uneven distribution of pollutants in the monitored facility does not allow to obtain a samples group of the same chemical composition. Thus, the solution of the problem is seen only through the creation of synthetic reference materials, which would be adequate to real samples by their physico-chemical characteristics.

The X-ray fluorescence analysis of welding aerosols are graded most often using samples obtained by applying solutions of the analyzed elements to a filter. For example, a filter with a dried aliquot of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution is used in the determination of Cr (VI) [7]. The advantage of this method is the simplicity of the preparation, and the main drawback is the inadequacy of such samples to real samples of welding fumes by physico-chemical characteristics.

The samples prepared using a specially designed air samplers or aerosol generators are more closely approximated to real samples of welding fumes loaded onto aspiration filters. In the works [8, 9] a large number of welding aerosols samples were collected in parallel to prepare the samples of welding fumes composition, then some samples were analyzed by different methods and the contents of the components on the filter were determined. The remaining part of samples was used as RM. The authors [8] have found that there are small differences in the mass of the aerosol on the filter and, consequently, the content of the elements in the sample, and therefore, there was an additional analysis of the loaded filters was carried out using an X-ray fluorescence spectrometer with wave dispersion to exclude different specimens. In [10, 11] for the control of the metal content in the air of the working zone when welding steel structures using an generator of fumes, synthetic samples were obtained from artificial fume from metal wire using a robotic system. It should be noted that these techniques make it difficult to prepare a large number of identical samples with different variations of chemical composition typical for samples of welding fumes.

The technology of manufacturing synthetic RM of the atmospheric aerosols composition in the form of a polyvinyl alcohol polymer film containing a finely divided powder carrier of the identified components is proposed in the work [12]. The developed technology makes it possible to prepare the required number of identical RM samples with wide variations in the chemical composition and mass of welding fumes particles. The error in determining the content of elements in RM is 5÷18 % depending on the element, it meets the requirements for the analysis of welding aerosols.

The purpose of this study is to investigate the possibility of using the XRF technique for control of the metal content in the welding fumes and the air of the working area.

### EXPERIMENT

The studies were carried out using synthetic samples based on polyvinyl alcohol films [13, 14] containing powder material of welding fumes collected on the process of welding.

The content of metals in the samples was determined using by RFA and PhM techniques. X-ray fluorescence measurements were performed on an energy-dispersive X-ray fluorescence spectrometer "EDX-8000" (Shimadzu, Japan) based on silicon drift detector with thermoelectrical cooling; an X-ray tube with an Rh-anode (work mode: air cooling, voltage 4÷50 kV, current 1÷1000  $\mu\text{A}$ ).

Photometric measurements were performed on a spectrophotometer “PromEcoLab PE-5400B” (Shanghai Mapada Instruments, China). Preparation of samples for photometric analysis includes ashing of samples in a muffle furnace at the temperature of 750÷800 °C during 40÷60 min. The fly ash residues were fused with the smelt (Na<sub>2</sub>CO<sub>3</sub> and KNO<sub>3</sub> in a ratio of 2:1) the weighting 0.5÷1 g at 800÷850 °C during 25÷30 min. The fusion were leached with 25 ml of 10% H<sub>2</sub>SO<sub>4</sub> [15]. The calibration characteristics of the PhM technique were determined from the series of single-element standard solutions. The optical density of the solutions was measured in cuvettes with an absorbing layer thickness of 10 mm at room temperature (20 ± 5) °C.

The table 1 provides a comparison of XRF and PhM results of determination of iron content in the samples.

Table 1

Comparison of XRF and PhM results

| Sample | Content of Fe, % |               |
|--------|------------------|---------------|
|        | XRF              | PhM           |
| 1-1    | 0.380 ± 0.020    | 0.320 ± 0.030 |
| 1-2    | 0.330 ± 0.017    | 0.270 ± 0.027 |
| 2-1    | 0.340 ± 0.017    | 0.250 ± 0.025 |
| 2-2    | 0.340 ± 0.017    | 0.240 ± 0.024 |
| 3-1    | 0.71 ± 0.04      | 0.60 ± 0.06   |
| 3-2    | 0.69 ± 0.03      | 0.59 ± 0.06   |
| 4-1    | 0.76 ± 0.04      | 0.60 ± 0.06   |
| 4-2    | 0.74 ± 0.04      | 0.57 ± 0.06   |
| 5-1    | 1.46 ± 0.07      | 0.81 ± 0.08   |
| 5-2    | 1.44 ± 0.07      | 0.79 ± 0.08   |
| 6-1    | 1.26 ± 0.06      | 0.89 ± 0.09   |
| 6-2    | 1.20 ± 0.06      | 0.85 ± 0.09   |

The analysis of the samples showed that the results of the photometric determination of metals in solutions after digestion the sample material according to the methodical instructions [15] are 1.5÷2 times lower than the results of X-ray fluorescence analysis of samples without digestion. Underestimation of the results of photometric measurements may be due to the analyte losses on digestion process or incomplete decomposition of some analyte compounds. Sources of the PhM results errors are discussed in detail in [16].

### CONCLUSION

XRF spectrometry is a simple, accurate and reliable method for the evaluation of metals concentration in weld fume samples and require little sample preparation. The energy dispersive XRF method allows to carry out non-destructive rapid definition of a wide range of elements.

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