# Determination of Cr in carbon steel of outlet headers

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## 1. Introduction

Flow-accelerated corrosion (FAC) is a pipe degradation process that is a chemical corrosion accelerated by flowing water or a water steam mixture in pressure vessels of fossil and nuclear power plants [1-3]. It is known that even lower amounts of chromium (as low as 0.1%) in carbon steel may significantly reduce the FAC rates. Therefore, the contents of chromium in used piping systems made from low alloy steel must be known for an evaluation for reusing pressure vessels.

In this study, we examined a method of a sample preparation for a chromium determination using a file, and the determinations of chromium in used outlet headers of nuclear power plants were carried out by a inductively coupled plasma atomic emission spectrometry (ICP-AES) for an evaluation of the FAC.

## 2. Experimental and Results

### *2.1 Sample dissolution*

About 0.1 g of a sample was transferred to a glass beaker, and then 12 ml of concentrated aqua regia was added to the beaker. The sample was heated on a hot plate, and then 0.5 ml of $H_2O_2$ was added to the beaker. The solution was made up to 50 ml with distilled de-mineralized water in a glass volumetric flask.

### *2.2 ICP-AES system*

The inductively coupled plasma atomic emission spectrometry system (Activa M, Horiba Jobin Yvon) was used to determine the amounts of chromium in the piping of the nuclear power plants. The measurement was carried out with two wavelengths of chromium. The incident power of ICP was 1.0 kw.

### *2.3 Effect of Fe on the Cr measurement*

Although ICP-AES has an advantage such as a relative freedom from matrix interferences, we examined the effect of the Fe concentration on the Cr intensity because of the high concentration ratio of Fe to Cr. As shown in Table 1, the Cr emission intensity was not severely influenced up to 500 mg/L of Fe at both Cr wavelengths.

| Fe(mg/L) | Cr(mg/L) | Net intensity of Cr | |
|---|---|---|---|
| | | 205.571 nm[1] | 206.164 nm[2] |
| 0 | 0.2 | 5975 | 4785 |
| 250 | 0.2 | 5940 | 4779 |
| 500 | 0.2 | 5816 | 4720 |
| 1000 | 0.2 | 5650 | 4659 |
| 2000 | 0.2 | 5544 | 4638 |

1. Background position: +0.03259 nm
2. Background position: −0.04018 nm

Table 1. Effect of Fe concentration on Cr intensity

### *2.4 Using the file for sample collection*

To obtain samples of pipe systems made from carbon steels in nuclear power plants, a file was chosen as a tool. An envelope taped to the pipe just below the sampling location was used to collect the filings.

To check on the chemical composition of the file and to know the toughness of the abrasive wear for the used file, we carried out preliminary tests. After the form of a disk of SRM 1227 was ground with the file, Cr content in the filings was determined by ICP-AES. As shown in Table 2, the result of Cr was much higher than the certified values. Thus we determined the contents of Cr in the file made of chrome steel. It was found to be 0.63%. Therefore we concluded that the file can not be used for a sample preparation.

| N | Cr (μg/g) | | |
|---|---|---|---|
| | Certified | Found | |
| | | 205.571 nm | 206.164 nm |
| 1 | | 1189 | 1189 |
| 2 | | 1127 | 1124 |
| 3 | | 1082 | 1082 |
| X | 190 | 1133 | 1132 |
| SD | 30[1] | 54 | 54 |
| RSD | | 4.7 | 4.8 |
| Relative Error (%) | | 496 | 496 |

1. Uncertainty

Table 2. Analytical results for a SRM 1227 ground using a file

We also determined the amount of Cr in the filings after the disk form of SRM 1227 was ground with a diamond file. It was found to be 265 (μg/g) which is 1.4 times higher than the certified value. Hence this file can not be used for a sample collection.

*2.5 Coating of the file with tungsten carbide*

Because of the high amounts of Cr in the file, we coated the file with tungsten carbide to prevent a contamination of Cr. Using the coated file, the results for Cr determined using ICP-AES in a SRM 1227 were in good agreement with the certified values, as shown in Table 3. Thus the contamination problem from the chromium steel file can be solved.

| Wavelength (nm) | Cr (μg/g) | |
|---|---|---|
| | Certified | Found[1] |
| 205.571 | 190 | 194±2 |
| 206.164 | 190 | 195±2 |

1. N=3

Table 3. Analytical results for a SRM 1227 ground using a file coated with tungsten carbide

*2.6 Determination of Cr in outlet header*

Each outlet header consists of one main header body and two branch lines. The nozzles, to which the outlet feeders are welded, are extruded from these pieces. Hence, the nozzles have the same chemical composition as the material from which they are pulled. Therefore, a sample should be collected from the thick-walled main header body and from the thick-walled branch line. No samples shall be collected from the nozzles. As shown in Table 4, chromium contents in the filings collected from the used outlet headers of the nuclear power plants were in the range of 0.11 to 0.19% using the file coated with tungsten carbide for the collection of the samples.

| Sample | Cr (%)[1] | SD | RSD (%) |
|---|---|---|---|
| 1-1 | 0.113 | 0.003 | 2.7 |
| 1-2 | 0.190 | 0.002 | 1.1 |
| 1-3 | 0.119 | 0.003 | 2.5 |
| 3-1 | 0.114 | 0.001 | 0.88 |
| 3-2 | 0.112 | 0.002 | 1.8 |
| 3-3 | 0.112 | 0.004 | 3.6 |

1. N=3

Table 4. Analytical results for Cr contents in filings collected from header at nuclear power plants using a file coated with tungsten carbide

## 3. Conclusion

The contamination problem caused by a chromium steel file can be solved by coating it with tungsten carbide using high velocity oxygen-fuel spraying techniques. It was found that the amounts of chromium in the outlet header were more than 0.11% with relative standard deviations from 0.88 to 3.6%.

## REFERENCES

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