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Detailed characterization of welding fumes in personal exposure samples

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Abstract. The objective of the project was to develop a method allowing for detailed characterization of welding particles including particle number concentration, size distribution, surface chemistry and chemical composition of individual particles, as well as metal concentration of various welding fumes in personal exposure samples using regular sampling equipment. A sample strategy was developed to evaluate the variation of the collection methods on mass concentration. Samples were collected with various samplers and filters at two different locations using our collection system. The first location was using a robotic welding system while the second was manual welding. Collected samples were analysed for mass concentration using gravimetry and metal concentration using ICP/OES. More advanced analysis was performed on selected filters using X-Ray Photoelectron Spectroscopy to determine surface composition of the particles, and X-Ray Diffraction to determine chemical composition of the fumes. Results showed that the robotic system had a lot of variation in space when the collection system was located close to the weld. Collection efficiency was found to be quite variable depending upon the type of filter. As well, metal concentrations in blank filters were dependent upon the type of filter with MCE presenting with the highest blank values. Results obtained with the XRD and XPS systems showed that it was possible to analyse a small of powdered welding fume sample but results on filters were not conclusive.

1. Introduction

It is well-known that exposure to welding fumes, especially in poorly ventilated areas, causes adverse pulmonary health effects. Metal fume fever, presenting with flu-like symptoms, is among the most common occupational illnesses in welders [1]. Other diseases include siderosis, which relates to the accumulation of iron oxide particles in the lungs [2, 3]. In addition, welders have an increased risk of developing chronic obstructive pulmonary disease (COPD) as well as occupational asthma and pneumonia [4, 5, 6]. Long-term exposure to high concentrations of welding fumes is associated with increased incidence of interstitial pulmonary fibrosis, where permanent tissue damage and scarring occurs in the lungs. This illness contributes to poor quality of life and premature death [7, 8]. The International Agency for Research on Cancer has classified welding fumes as possible carcinogens [9]. It is believed that lung exposure to metal oxide particles produced by the welding process induce pro-inflammatory cytokine production and oxidative stress. Indeed, increased levels of cytokines have been detected in the blood of welders exposed to zinc oxide welding fumes in controlled settings [1].

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It is well-known that the welding process produces particles ranging from few nanometers to few micrometers [10, 11, 12, 13, 14, 15]. Very detailed characterization of fumes have been done in controlled settings using scanning or transmission electron microscopy techniques coupled with energy dispersive spectroscopy (EDS), X-ray fluorescence (XRF), and X-ray photoelectron spectrometry (XPS) [16, 17, 18, 19, 20]. The main compound in welding fumes is iron oxide but manganese is present in significant concentrations. Carbon, oxygen, potassium, silica, and fluoride have also been found in particles produced by welding techniques using a flux [17, 18, 19]. It has been suggested that, in some cases, a core-shell structure is present for welding particles [18]. In addition, fume generation rate may vary with the welding technique [20]. The aim of the study was to develop a sampling and analytical technique, using common filters and samplers, to performed detailed characterization of welding fumes in personal exposure samples. It was important to find a filter suitable for the determination of both mass and metal concentration in one sample. It was also important to evaluate the efficiency of collection of various samplers.

2. Materials and methods

2.1. Sample collection
Welding fumes were collected at two different locations: the Canadian Centre for Welding and Joining (CCWJ), Department of Chemical Engineering, University of Alberta, and the Northern Alberta Institute of Technology (NAIT).

Since the aim of the project was also to test various samplers, a collection system was built to allow for multiple sample collection at once. Sampling assembly was based on a design from Zugasti et al. [21]. The assembly was composed of two vertical fiberglass rods inserted in two support stands and two horizontal fiberglass rods. Samplers were mounted as close as possible to each other on two separate rods. Five samplers were mounted per horizontal rod for a total of ten samplers, and the distance between the rods was kept at four to five inches. Personal sampling pumps Universal PCXR4 (SKC, Eighty Four, Pennsylvania) were used to collect samples. Pumps were calibrated prior to and after sampling and the average flow was used to calculate the sampling volume. All pumps were calibrated to run at a flow of 2 L/min since it is the recommended flow for the IOM sampler. For each sampling day, particle number concentrations were determined using the P-Trak (TSI, Shoreview, Minnesota). The P-Trak was located just next to the sampling assembly. All samplers were cleaned using a 2% detergent solution (Citranox, Alconox, White Plains, New York) prior to be used to avoid contamination of samples.

At both locations, welding was performed on carbon steel (Wilkinson Steel and Metals, Surrey, British, Columbia). Metal concentration from the steel was obtained from the Material Safety Data Sheet and was 91-99 % iron, < 2% manganese, < 0.1% chromium, < 1% nickel, and < 1% copper.

2.1.1. CCWJ
Welding fumes were generated using an Arc Mate 100iC robot (Fanuc Robotics, Rochester Hills, Michigan). The power source was a Power Wave 455M/STT (Lincoln Electric, Toronto, Ontario) and the feeder was a 4R 220 Autodrive (Lincoln Electric, Toronto, Ontario). All welding fume generation was performed using a program developed for gas metal arc welding (GMAW) with a wire feed speed of 150 in/min and a voltage of 20 V. The wire was a 0.045 SuperArc L-56 (ER70S-6, Lincoln Electric, Toronto, Ontario) with a diameter of 0.045 in. The travel speed of the robot was 6.8 in/min and welding lasted about 10 min per experiment. Plates were grinded to remove rust prior to being used, and a new plate was used for each run. Sample collection was performed at CCWJ on 28 October and 7 November 2014.

The sampler assembly was put on a table located along the plate being welded, with the lowest rod mounted twenty centimeters higher than the plate to simulate the breathing zone of a welder. Local exhaust ventilation was installed 30 in behind the samplers at an angle of 45° (Figure 1).
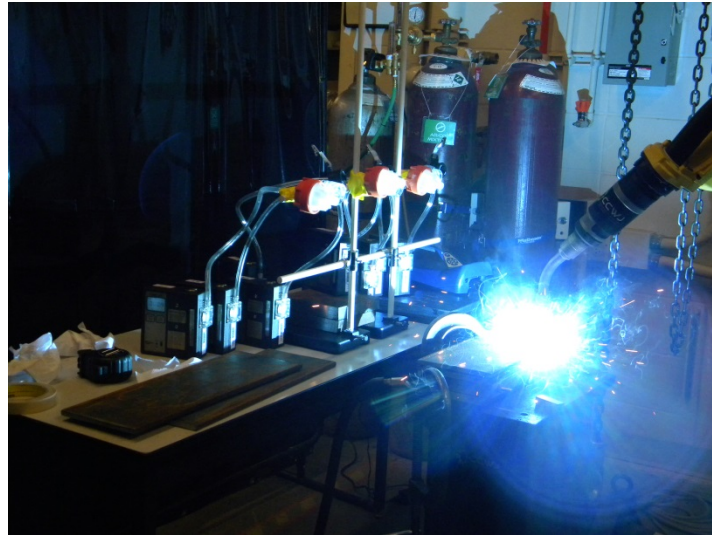

Figure 1. Sampling layout at CCWJ

On October 28th, three samplers were tested: the conductive plastic IOM sampler (SKC, Eighty Four, Pennsylvania), 37 mm total dust cassettes (SKC, Eighty Four, Pennsylvania), and a 47 mm Teflon PFA filter holder (SKC, Eighty Four, Pennsylvania). The testing was carried out in three runs with three samplers of each type attached to the rod for each run (Figure 1) for a total of 9 samples per sampler. Location of each sampler on the rod was rotated between runs to get all samplers in all positions. Sampling was carried out using 0.4 μm polycarbonate membranes (SKC, Eighty Four, Pennsylvania). On November 7th, sampling was carried out using only the IOM and the total dust cassettes (SKC, Eighty Four, Pennsylvania) in three different runs. For each run five of each sampler was tested. In the first run samplers were loaded with a 5 μm PVC filter (SKC, Eighty Four, Pennsylvania), and in runs 2 and 3 samplers were loaded with 0.4 μm polycarbonate membranes (SKC, Eighty Four, Pennsylvania) in order to verify the collection efficiency of different filters.

2.1.2. NAIT
Sample collection was performed while students were welding on December 3rd, 2014. Samples were collected while 3rd year students were working on a pipe using shielded metal arc welding (SMAW) using 0.0125 in diameter E6010 (Lincoln Electric, Toronto, Ontario) and 0.09375 in diameter E7018 electrodes (Blueshield, Edmonton, Alberta). Afternoon classes start at 11 am and generally end around 3 pm, therefore sampling was carried out for 2h45.

The sampler assembly was mounted on a mobile cart located 1.2 m from the weld with the lowest rod mounted 20 cm higher than the weld height to simulate breathing zone of the welder. Local exhaust ventilation was located behind the samplers (Figure 2).

Figure 2. Sampling layout at NAIT

Sampling was carried out using total dust cassettes (SKC, Eighty Four, Pennsylvania) loaded with 5 μm PVC filters (SKC, Eighty Four, Pennsylvania) and in total ten samples were collected. This sampling was performed to verify sampling variation for each sampler as results from CCWJ showed a lot of variation within samplers.

2.2. Analysis

All samples were analysed for mass concentration and blank filters were analysed for metal concentration. In addition, two bulk welding fume samples were collected at NAIT on the ground after class and sieved down to a fraction lower than 106 μm. These samples were used to develop analytical methods, mainly XRD and XPS. One of the samples was collected after shielded metal arc welding (SMAW) and the other one was collected after flux core arc welding (FCAW).

2.2.1. Mass concentration

All membranes were weighed pre and post-sampling to calculate the mass on the filter. Weighing was performed in a temperature and humidity-controlled room at the Department of Civil and Environmental Engineering (University of Alberta, Edmonton, Alberta). All filters were equilibrated with the room humidity for 24 hours prior to be weighed. Since indoor air is very dry in winter in Edmonton, static charging is an issue for weighing filters. All filters were passed through and antistatic device (Mettler Toledo, Mississauga, Ontario) prior to be weighed. All weighing was performed on a microbalance (Sartorius, Goettingen, Germany). To account for variation in weighing, blanks filters were weighed as samples for each sampling day.

2.2.2. Metal concentration

Metal analysis were performed on an ICP/OES iCAP 6000 (Thermo Fisher Scientific, Cambridge, UK) at the Natural Resources Analytical Laboratory (Faculty of Agricultural, Life and Environmental Sciences, University of Alberta, Edmonton, Alberta). The laboratory uses an Yttrium internal standard to correct for matrix interference and the software corrects for iron interference. All blank filters were digested using a 4:1 HNO_3:HCl solution in a microwave oven [22] and diluted to 25 mL with deionized water prior to analysis. The SMAW bulk sample was analysed as well to verify the suitability of the method. The analytical run included digestion blanks, instruments blanks, and an external reference check (200 ppb per metal). Samples were analysed for Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Ni, Pb, Si, V, and Zn. These metals were selected since they may be present in the steel or in the electrodes.

2.2.3. XRD and XPS analysis

X-Ray Diffraction analysis (XRD) was performed on a Rigaku XRD Ultima IV (Rigaku, Tokyo, Japan) at the Department of Chemical Engineering (University of Alberta, Edmonton, Alberta) on both bulk samples. X-Ray Photoelectron Spectroscopy (XPS) was performed on an AXIS 165 (Kratos, Manchester, United Kingdom) using a monochromatized Al Kα source (hν=1486.6 ev) at an emission current of 12mA and HT=14kV. The XPS is located the Department of Physics (University of Alberta, Edmonton, Alberta) and analysis was done only on the FCAW bulk sample. In all cases analyses were performed directly ion the powered sample.

2.3. Statistical analysis

All statistical tests were performed on SPSS Statistics software version 22 (IBM, Armonk, New York). Mann-Whitney U test was used to compare samplers or filters. Averages and standard deviations were calculated from Excel (Microsoft, Redmond, Washington).

3. Results and discussion

3.1. Mass concentration

Blanks filters for all day of analysis showed differences between pre and post weighing from 0.001 to 0.002 mg. Therefore weighing was considered stable between pre and post weighing days.

Results from CCWJ showed a lot of variation for both days of analysis within and in between samplers (Figures 3 and 4) and results from November 7th are shown in Table 1.
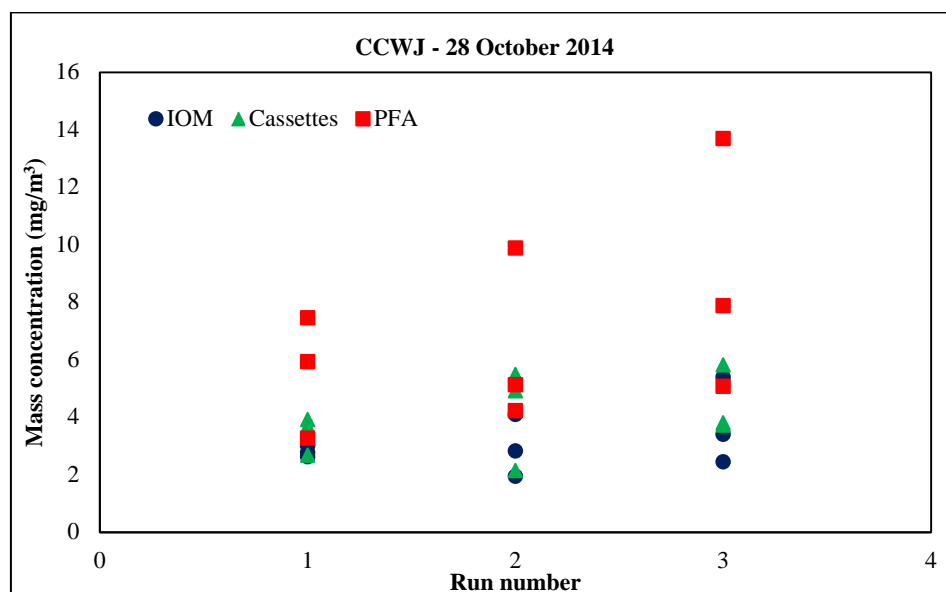

Figure 3. Comparison of three samplers loaded with 0.4 μm polycarbonate membranes
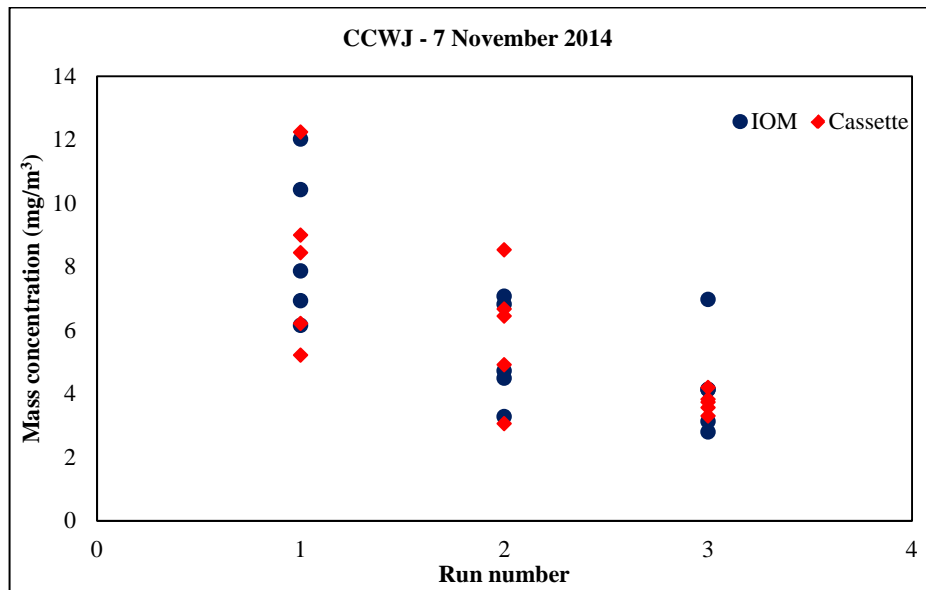
Figure 4. Comparison of the IOM sampler and the total dust cassette loaded with PVC filters (run 1) and polycarbonate filters (runs 2 and 3)

The results show that there was a lot of variation when sampling at CCWJ as compared to what was obtained in the study done by Zugasti et al. were they reported coefficient of variation close to 4% [21]. In fact, coefficients of variations were 30-35% for each sampler except for the PFA sampler that showed a coefficient of variation of 46%.

Average results for each samplers obtained on October 28th were respectively 3.16 mg/m^3 (standard deviation = 1.03 mg/m^3, n = 9) for the IOM sampler, 4.01 mg/m^3 (standard deviation = 1.21 mg/m^3, n = 9) for the total dust cassette, and 6.94 mg/m^3 (standard deviation = 3.24 mg/m^3, n = 9) for the PFA filter holder. Mann-Withney U tests were run to verify if results were significantly different between samplers. Results from the statistical tests showed that the IOM was not significantly different than the cassette (U = 23; Z = -1.545; p = 0.122), the IOM was different than the PFA filter holder (U = 6; Z = -3.046; p = 0.002), and the total dust cassette was different than the PFA (U = 14; Z = -2.340; p = 0.019). Since the PFA filter holder gave higher mass concentrations than the IOM sampler or total dust cassette, it was decided not to use it in further testing.

Table 1. Results from November 7th 2014 using PVC and polycarbonate filters

		IOM sampler	Total dust cassette
PV filter	Average (mg/m^3)	8.7	8.2
	Standard deviation (mg/m^3)	2.5	2.7
	Coefficient of variation (%)	26	33
Polycarbonate membrane	Average (mg/m^3)	4.8	4.9
	Standard deviation (mg/m^3)	1.6	2
	Coefficient of variation (%)	34	39

In the experiment from 7 November, results with both PVC filters and polycarbonate filters showed no difference between the IOM sampler and the total dust cassette. However a difference was noted between the two types of filters with averages for PVC and polycarbonate respectively at 8.45 and 4.79 mg/m^3. This difference was found to be significant when using the Mann-Whitney U test (U =

23; Z = -3.388; p = 0.001). Therefore the polycarbonate filters are not suitable to determine accurately the mass concentration in air samples since they underestimate the results.

Since results obtained from CCWJ were very variable, another test was performed at NAIT on December 3rd to verify the variation in mass concentration. Results are shown in table 2.

Table 2. Results obtained at NAIT with total dust cassettes and PVC filters 5 μm

Sample number	Mass concentration (mg/m^3)
1	0.438
2	0.478
3	0.487
4	0.510
5	0.528
6	0.438
7	0.487
8	0.535
9	0.494
10	0.540
Average	0.493
Standard deviation	0.036

Results obtained at NAIT show much better reproducibility of the sampling and analytical method with a coefficient of variation of 7%, which was closer to what had been previously published [21]. Various reasons can explain the variability obtained when sampling at CCWJ. The samplers were mounted very close to the area being welded. It is possible the welding process produces a lot of spatial variations which would have been recorded by our sampling design. In addition, sampling at NAIT was carried out for a longer time therefore any error in the timing would result in a lower error on the calculation of the total volume sampled.

3.2. Metal concentration
Instrument blank was negligible for all metals analysed. External reference check gave average recovery values from 82% (silica) to 102% (zinc). Results for digestion blanks and various filters are shown in Table 3.

Table 3. Metal results for acid blanks and selected filters

Filter type	Al (ng)	Ba (ng)	Be (ng)	Ca (ng)	Cd (ng)	Co (ng)	Cr (ng)	Cu (ng)	Fe (ng)
Digestion Blank	169	23	ND	752	ND	ND	ND	196	134
MCE[a]	988	113	ND	47291	ND	1	357	305	1730
PVC	ND	29	ND	907	ND	ND	41	ND	72
Fluoropore[b]	ND	28	ND	ND	ND	ND	7	ND	99
Omnipore[c]	ND	18	ND	583	ND	ND	20	ND	ND
Polycarbonate	ND	23	ND	494	ND	ND	19	ND	159

Filter type	K (ng)	Mg (ng)	Mn (ng)	Mo (ng)	Ni (ng)	Pb (ng)	Si (ng)	V (ng)	Zn (ng)
Digestion Blank	ND	61	4	ND	ND	2	365	ND	105
MCE[a]	1685	11614	81	2	20	13		24	
PVC	1180	56	2	ND	27	ND	1546	ND	75
Fluoropore[b]	600	ND	1	ND	2	ND	246	8	3
Omnipore[c]	ND	52	ND	ND	ND	ND	368	ND	28
Polycarbonate	ND	11	4	ND	74	ND	334	ND	16

[a] Mixed cellulose ester
[b] Hydrophobic PTFE membranes (Millipore, Billerica, Massachusetts)
[c] Hydrophilic PTFE membranes (Millipore, Billerica, Massachusetts)

Both calcium and potassium gave blank values quite high in the microgram range. MCE filters gave blank values higher than the digestion blanks for Al, Cr, Cu, Fe, Mn, Ni, Pb, and V. Since these metals are of interest in welding fumes, MCE filters were not used in our experiments. For elements other than Ca and K, blank values for filters were similar to digestion blanks suggesting than most of the contamination was coming from the acid digestion. It is unknown why polycarbonate membranes gave a higher value for nickel as compared to PTFE membranes. The Omnipore hydrophilic PTFE membranes (Millipore, Billerica, Massachusetts) showed to be suitable for metal analysis. Further investigation will determined if the membrane is also suitable for the determination mass concentration in samples.

Detection limit was calculated as three times the standard deviation of the digestion blank [23] and quantification limit was calculated as five times the detection limit. Detection limits and quantification limits are given in Table 4. These values are considered sufficient for the analysis of welding fumes.

Table 4. Detection limits and quantification limits for selected metals

	Al (µg)	Cd (ng)	Co (ng)	Cr (ng)	Cu (µg)	Fe (µg)	Mn (ng)	Mo (ng)	Ni (ng)	Pb (ng)	V (ng)	Zn (µg)
Detection limit	1.7	1	11	19	1.3	0.5	9	9	18	52	10	0.4
Quantification limit	8.9	6	56	94	6.6	2.3	46	47	88	261	48	1.8

Further work will involve the analysis of real welding fume samples using various instruments to confirm measurements.

3.3. XRD and XPS analysis
Results for XRD showed that both FCAW and SMAW bulk sample contained iron and manganese oxides. Silica was also found in both fumes while titanium was found only in the SMAW sample. An example of graph is given in Figure 5.
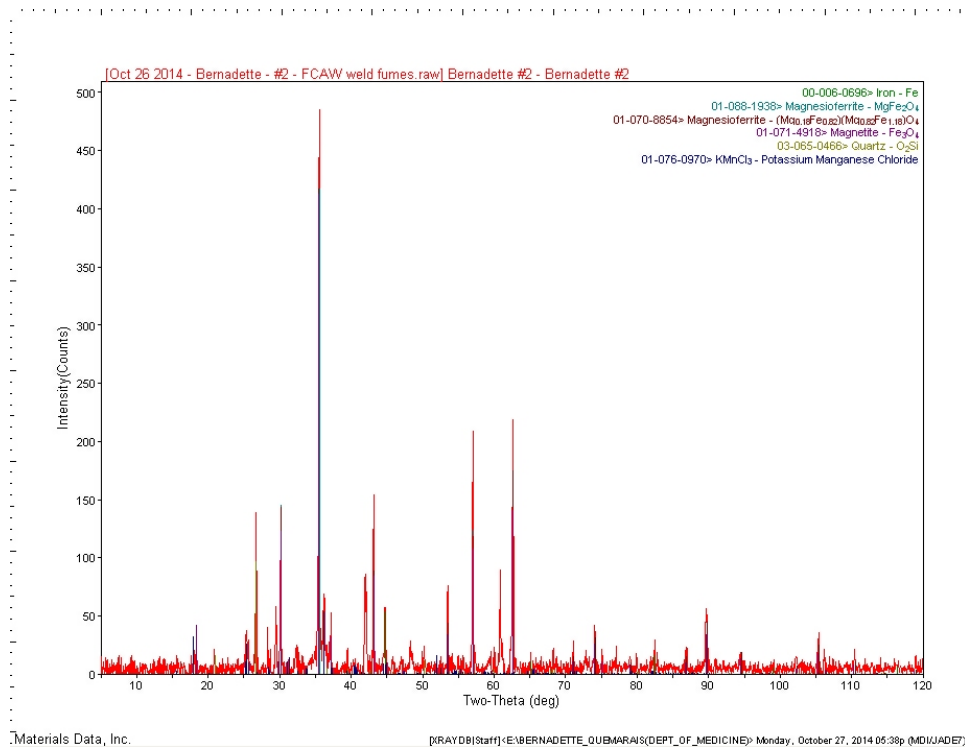
Figure 5. XRD analysis on FCAW bulk sample.

Results for XPS are shown in figure 6. The surface chemistry of the FCAW samples shows a lot of different chemical species due to the presence of a flux on the FCAW electrode.
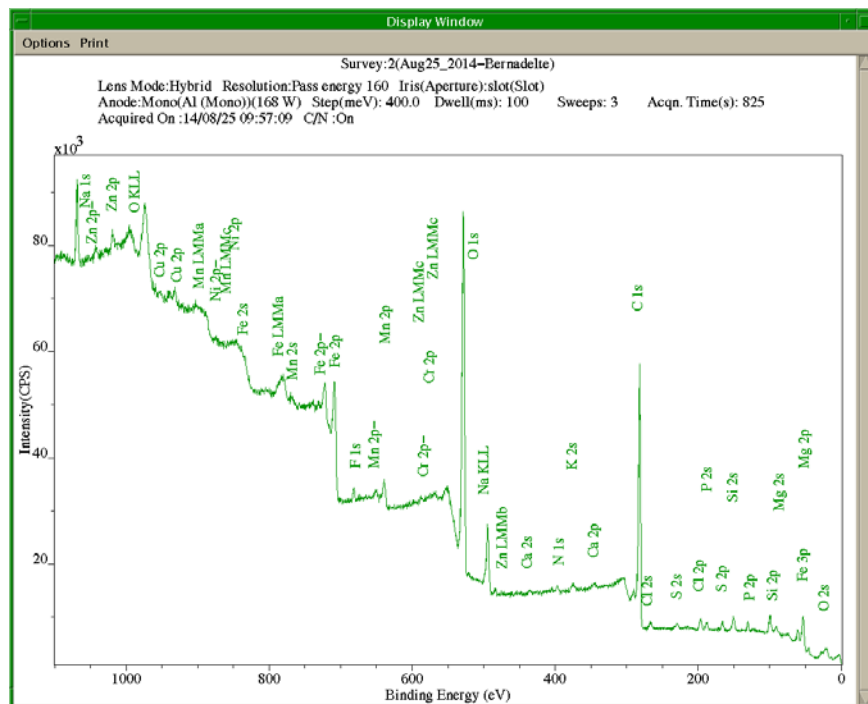

Figure 6. Analysis of a FCAW bulk sample using X-Ray Photoelectron Spectroscopy

Results obtained with XRD and XPS showed that it is possible to perform this type of analysis on small welding fume bulk samples. Unfortunately analysis on filters was not successful because the filters presented some white surfaces. Further investigations are necessary to improve this type of analysis.

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