# Fugitive Emission Measurements Above a Hard Chromium Plating Tank Using Spark-Induced Breakdown Spectroscopy (SIBS)

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This paper presents the results of total chromium fugitive emissions measurements above a hard chromium electroplating tank at the Marine Corps Logistics Base (MCLB) in Albany, GA. The measurements were made using OSHA Draft Method ID-215 and a newly developed realtime monitor based on spark-induced breakdown spectroscopy (SIBS). The SIBS monitor acquired data on a five-sec time scale and was used to survey the total chromium concentrations above the plating bath surface over a range of ventilation rates in conjunction with operation of an experimental liquid recirculation system. The SIBSmeasured concentrations varied from  $<10 \ \mu g/m^3$  (the lower limit of detection) to >1000  $\mu$ g/m<sup>3</sup>. The measured concentrations were well correlated to distance from the plating bath surface, smoke test indications of poorly ventilated areas and ventilation flow rate. The SIBS data quantitatively agree with simultaneous internal filter results and with the OSHA-approved air sampling filter measurements. These results indicate the SIBS monitor is capable of industrial hygiene monitoring of chromium in a plating environment.

Exposure to chromium is a known hazard associated with hard chromium electroplating,<sup>1-5</sup> abrasive blasting of painted surfaces,<sup>6-7</sup> and cement working.<sup>2</sup> The recognition of hexavalent chromium, Cr(VI), as a carcinogen, and therefore as one of the most toxic of the 189 hazardous air pollutants, has caused EPA and OSHA to react by placing severe restrictions on atmospheric release and worker exposure to fugitive emissions of this species, respectively. The EPA has set regulatory limits, in the form of a National Emission Standard for Hazardous Air Pollutants (NESHAP), at 15 and 30 µg/dscm for stack concentrations for large and small hard chromium electroplating facilities, respectively. The current American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit value time-weighted average for hexavalent chromium exposure is 50  $\mu$ g/m<sup>3</sup>, a value not to be exceeded on a time-weighted basis over an 8-hour shift. The OSHA PEL (ceiling) is 100  $\mu$ g/m<sup>3</sup> for chromic acid and chromates and the NIOSH REL (10-hour TWA) is 1  $\mu$ g/m<sup>3</sup> for all hexavalent chromium



High-Voltage Spark

Initiates Discharge



Discharge Vaporizes Ionizes Air and Aerosol Particles

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As Plasma Cools Atomic Emission Occurs

Fig. 1—Basis of spark-induced breakdown spectroscopy (SIBS).

compounds. OSHA is currently considering a revision of the PEL to an 8-hour TWA for Cr(VI) at a level of 0.5  $\mu$ g/m<sup>3</sup> with an action level of 0.25  $\mu$ g/m<sup>3</sup>.

This paper presents the results of total chromium measurements obtained by a new monitoring technology based on spark-induced breakdown spectroscopy (SIBS) above the hard chromium plating bath at the Marine Corps Logistics Base (MCLB) in Albany, GA. The results are used principally to evaluate the efficacy of a push/pull liquid flow system, developed by the U.S. Army Construction Engineering Research Laboratories, integrated with a modified ventilation system.<sup>8</sup> The goal of the test was to determine whether the liquid push/pull system has the potential to control worker exposure levels below the regulated limit with a reduced exhaust ventilation rate. The SIBS monitor has previously been used as a continuous emissions monitor (CEM) for lead and chromium in a simulated flue gas9 and for ambient lead in an indoor firing range.<sup>10</sup> This was the first field test of the SIBS monitor for ambient chromium in an industrial hygiene application.

#### Experimental Procedure

Basis of Spark-induced Breakdown Spectroscopy (SIBS) The basis of SIBS is the generation of a high energy electrical spark between two electrodes that vaporizes, ionizes and excites an air sample with metal-containing aerosols and particulates. A schematic of this process is shown in Fig. 1. Following the generation of the spark, optical detection of the atomic emission at characteristic optical wavelengths of the metal analyte species is performed. The concentration of the species of interest in the sample is proportional to the number



Fig. 2-Schematic of SIBS monitor.

of excited atoms, and to the intensity of the emission of the atomic features.

The apparatus required to perform this type of spectroscopy, shown schematically in Fig. 2, can be extremely simple. The necessary components are two electrodes with appropriate gap between them, a power supply and the optical collection equipment. For sampling a gas stream, the spark gap is situated such that the gaseous sampling stream can flow unimpeded between the electrodes. Because the number of aerosol particles encompassed is dependent upon the volume of the spark, the sensitivity of the technique is a function of electrode gap.

Once the spark has been struck, optical detection is delayed to discriminate against broadband plasma emission caused by Bremsstrahlung radiation. Following this delay, the optical signals are collected radiometrically with a pair of filtered, miniature photomultiplier tubes (PMTs). The advantages of the filter and PMT approach include simplicity and low cost. The strategy behind the radiometer is to accumulate the signal associated with the atomic line (on-line) with one filter and to subtract any background signal with a nearby filter (off-line) that has no analyte atomic feature within its bandwidth.

The radiometer analog output signals are fed into a PC (Pentium<sup>TM</sup>, 100 MHz) with a 1.2-MHz A/D data acquisition board. The temporal traces are evaluated by routines developed within a LabWindows CVI<sup>®</sup> architecture. The on-line and off-line temporal traces are subtracted and integrated. Further experimental details of the SIBS technology have been published elsewhere.<sup>10</sup>

### MCLB Test Conditions

The monitor was calibrated in a dry aerosol generator<sup>9,10</sup> in the laboratory prior to shipment to the MCLB facility. ICP standards of chromium (III) nitrate, chromium (VI) nitrate and chromium (VI) chloride were used. The calibration curve encompassed seven points over the range 0 to 1500  $\mu$ g/m<sup>3</sup>. Identical sensitivities were observed for all three compounds. The 3 $\sigma$  (three times the S/N ratio of the baseline) detection limit of the monitor was determined to be 10  $\mu$ g/m<sup>3</sup>. Data measured below this value, therefore, are reported as <10  $\mu$ g/m<sup>3</sup>.

For the MCLB test, the spark was operated at 1 Hz and the data were recorded on a 5-sec, averaged basis. The concentration data for the tank scans were reported as averages (typically 3 min) for the full duration of the dwell at each specific position.

The sample chamber shown in Fig. 2 was constructed of plexiglas and was designed for easy removal and cleaning. Room air was drawn into the sampling chamber by a 1.3 cfm pump. Immediately downstream of the sampling chamber, the contaminated room air passed through a particulate filter\* offering greater than 99.98 percent particulate retention and DOP 0.3  $\mu$ m at 32 L/min. After the test, these filters were sent to an independent laboratory for analysis of total chromium for comparison with the monitor results.

Details of the test conditions are provided elsewhere<sup>8</sup> and are only summarized here. MLCB houses a hard chromium plating tank 3 ft wide, 8 ft deep and 11 ft long. The tank uses a reversible, two-busbar system. For this test, two large cylindrical parts were plated at 800 to 1100 amp (typical efficiencies for hard chromium electroplating are 10 to 20



Fig. 3—SIBS monitor sampling above plating tank.

percent). Each part was suspended separately in the plating tank by a rack with four surrounding anodes. The busbars were 20 in. apart and were positioned 3 in. from the front tank wall and 4 in. from the rear wall. The distance from the liquid level to the top of the tank was 12 in.

Emission control for the MCLB plating tank was performed exclusively by a forward and rear hood system. No surface tension-adjusting additives or tank covers were used. The only exception to this was a short (12 in. wide) PVC sheet installed halfway through the test to cover the rear area of the tank in a region not serviced by the hoods.

To acquire data above the surface of the plating tank, the spark power supply, pulse generator and pump were placed inside an iron mesh sample cage 3 ft on a side. The sample chamber was mounted beneath with the open end of the sampling chamber facing downward. A photograph of the SIBS monitor in this configuration is shown in Fig. 3. The radiometer and computer were placed on a nearby benchtop. The radiometer was connected to the sampling chamber via two optical fibers, 10 m in length each.

Plastic sheeting was used to protect the monitor components from chromic acid splashes that occurred during some of the tests. On some of the occasions when the sampling chamber was splashed, the instrument response became



Fig. 4—Sampling locations for chromium plating tank at MCLB. Positions P-T are aligned with A-E, F-J and K-O, but are shown offset to indicate that they are at an increased height above the tank.

<sup>\*</sup> Glass microfiber filter, Gelman Sciences, Ann Arbor, MI.

Table 1 Tank Survey Chromium Concentrations

Ventilation Measured Concentration, µg/m <sup>3</sup> Flow Rate																
scfm	Note	А	В	С	D	F	G	Н	I	K	L	М	N	Q	R	S
6830	Conv.	<10	<10	<10	<10	<10	<10	<10 0.072 ±0.018	<10	<10	<10	<10	<10	<10 0.054 ±0.038	<10 0.031 ±0.015	<10 0.033 ±0.042
4827	P/P on					<10	<10	<10	<10							
3263	P/P on	<10	<10	<10	<10	<10	<10	<10	<10 0.12 ±0.02	<10	<10	<10	<10	<10	<10 0.045 ±0.017	<10 0.036 ±0.016
2249	P/P on	<10	25 ±16	<10	<10	<10	<10	10±14	<10	<10	<10	13 ±14	<10	<10 0.25 ±0.15	<10 0.28 ±0.14	<10 0.31 ±0.12
1845	P/P on	<10	<10	11 ±20	72 ±55	<10	<10	13 ±10	$186\pm50$	18±12	<10	15 ±14	27 ±22	15 ±13	<10	13 ±10
1704	P/P on	<10	<10	<10	57 ±37	93 ±70	47 ±41	<10	85 ±57	27 ±9	<10	36 ±34	69 ±64	$14\pm10$	11±3	16±8
1493	P/P on	32±32	481 ±163	125 ±35	259 ±114	116 ±80	384±101	152 ±74 228 ±158	448±110	144 ±77	89 ±74	138 ±74	190 ±93	<10 4.6 ±0.4	17 ±4 4.2 ±1.1	16±24 5.2±1.0
1200	P/P on													880 ±302		
Where there are multiple entries, the first is for the SIRS monitor: the second is from OSHA Draft method ID-215																

saturated from overexposure to acid mist. When this occurred, the plexiglas housing was removed and cleaned. The electrodes were cleaned alternately with dilute nitric acid, water and methanol, followed by sanding with a mild abrasive to remove chromium residue. The entire cleaning procedure was performed in less than an hour.

Surveys of fugitive chromium emissions above the tank were performed in a scan and hold pattern with the iron mesh cage containing the SIBS monitor mounted on a crane. The crane was used to position the sampling chamber inlet at each desired location for sampling. After a period of three min, the chamber inlet was moved to the next position. A schematic of the plating tank with approximate sampling locations is shown in Fig. 4. The positions A-O were located 20 in. above the plating solution surface. The positions P-T were located 48 in. above the plating surface solution plane immediately outside the tank. These positions were representative of the breathing air space of the workers. As a result of physical limitations caused by the ventilation ducts, positions E, J, O and T could not be sampled by the monitor.

Industrial hygiene monitoring was performed using OSHA Draft Method ID-215. Air samples were collected at posi-



Fig. 5—SIBS sampling system after being splashed by chromic acid.

tions H, Q, R, and S on 37-mm diameter, 5-micron pore, mixed-cellulose ester filters at a nominal flow rate of 4 L/min. Sampling duration was 50 to 75 min. Most samples were collected in a series of three consecutive samples at the same location and under the same conditions. Filters were analyzed for chromium (VI) at an independent laboratory.

## Results and Discussion

The SIBS monitor operated for 12 hr each day for the full duration of the three-day test. During the test, the monitor required no maintenance or repair (other than cleaning after the sampling system was splashed by chromic acid as previously noted). The environment in the plating area was challenging for an ambient monitor. The temperature was elevated with high humidity and the ambient air contained dust from plating operations, nearby paint stripping and cleaning operations, and from outside air that served as the principal ambient ventilation. The SIBS monitor sampled the ambient air as encountered without any purification or removal of extraneous particulate or atmospheric contamination.



Fig. 6—Sample raw data on SIBS instrument at two locations above plating bath.

The temperature of the plating bath was typically 140 °F. Chromic acid mist rising from the surface and being drawn away by the ventilation system was always visible. When the SIBS monitor was placed within a few inches of the surface, the air temperature may have reached the bath temperature and the concentration of chromic acid rose to very high levels (some short-term concentrations exceeded 1400  $\mu$ g/m<sup>3</sup>). The additional hazard the instrument faced near the bath surface was direct contact with large droplets of chromic acid. A photograph of the SIBS sampler after such an event is shown in Fig. 5. Direct contact with large droplets on the electrodes caused instrument saturation at the end of this particular test. Immediately after cleaning, the instrument was acquiring data again.

#### Tank Survey Results

Sample raw data from the SIBS instrument are shown in Fig. 6. The data are shown on a five-sec time scale responding to individual particles and groups of particulate that drift toward the sampling inlet from the bath surface. Data from two sampling locations are shown, one at 20 in. above the bath surface and another 48 in. above the bath surface. When the data are converted to concentration units and averaged over the sampling times, the concentrations correspond to 93  $\mu$ g/m<sup>3</sup> and less than 10  $\mu$ g/m<sup>3</sup>, respectively.

The instrument response shown in Fig. 6 indicates the source (particles emitted from the plating bath surface) to be highly variable as constant steady-state signals are recorded by the SIBS instrument from stable sources. The large source variability leads to high standard deviations in the measured concentrations. For example, the averaged data in Fig. 6 for 20 in. above the bath surface has a standard deviation of 63  $\mu$ g/m<sup>3</sup>, or 70 percent of the average value. Standard deviations of this magnitude are typical of the database obtained from the test.

A complete summary of the tank survey data is presented in Table 1. Where there is more than one entry, the first represents the SIBS measurement and the second the result of the OSHA Draft Method ID-215 sampling method. All the results are shown with one standard deviation uncertainty. As noted previously, the SIBS measurements were generally three-min averages, whereas the OSHA sampling method



Fig. 7—SIBS measured chromium concentrations at positions M, I and G as a function of ventilation rate.

was performed typically for more than an hour. A total of six complete tank survey scans were performed at six different ventilation flow rates. Partial scans were also performed at two ventilation rates. The ventilation rates ranged from 1200 to 6830 scfm.

Baseline data were collected at the unmodified system flow rate of 6830 scfm with the liquid push/pull system off. A survey scan of the SIBS instrument produced no measurable results. This is consistent with the OSHA Draft Method ID-215 which measured  $\leq 0.072 \ \mu g/m^3$  at positions H, Q, R, and S. The SIBS monitor did measure 63  $\pm 12 \ \mu g/m^3$  at position H when it was lowered several inches closer to the plating bath surface.

The remainder of the test was performed with the push/pull liquid flow system operational. A goal of this exercise was to identify an optimum ventilation exhaust flow rate, that in combination with the push/pull system, keeps the ambient chromium concentration below the regulated exposure specification.

Only a partial scan of the tank was performed at 4800 cfm. The measurements at this ventilation rate showed no measurable chromium concentrations. The survey at 3263 scfm produced results similar to 6830 scfm with no measurable chromium detected by the SIBS instrument. This result is confirmed by the low concentrations determined by the OSHA sampling method. OSHA Draft Method ID-215 measured  $0.12 \,\mu$ g/m<sup>3</sup> at position H and even lower concentrations in the breathing air space positions Q-R.

At 2249 scfm, the SIBS instrument began to measure chromium concentrations above the detection limit. At position B, the SIBS instrument measured  $25 \pm 16 \,\mu\text{g/m}^3$  and  $13 \pm 14 \,\mu\text{g/m}^3$  was measured at position M. A measurement at position H was not performed in this survey. The OSHA measurement at point H is  $10 \pm 14 \,\mu\text{g/m}^3$ . Measurements at positions Q-R are still very low, less than  $0.31 \,\mu\text{g/m}^3$ , but the OSHA-measured levels are significantly higher than those measured at 3263 scfm. The SIBS data are consistent with data obtained with the OSHA method.

Measurable chromium concentrations were observed from several positions around the tank at 1845 scfm ranging from 11 to 186  $\mu$ g/m<sup>3</sup>. Chromium concentrations were measured just above the instrument detection limit at two of the worker breathing positions (Q and S). The SIBS data correlated well with the smoke tests here. This test identified positions D and I to be "hot spots," where fugitive emissions were poorly captured by the ventilation system. These positions were also near active electroplating (near an anode or cathode rack). Subsequent measurements employed a PVC sheet to cover positions E, J, and O (12 in. wide, 1/2 in. thick and long enough to fit across the tank width on top of the hoods). This improved the capture of emissions from positions D and I.

At 1704 scfm, data from only five of the fifteen measurement locations showed no measurable chromium above the monitor detection limit. These were at positions A-C, at the rear of the tank, position H in the tank center, which was a quiescent area with no active electroplating, and position L at the front of the tank. The highest concentrations were measured at positions F and I. The effect of the special cover at the right of the tank is clearly evident in the data for positions D and I as these concentrations decreased from the levels measured at 1845 scfm.

The chromium concentrations increased markedly at 1493 scfm with the highest at positions B, G and I. The latter two

were located on the tank centerline and were expected to be significantly affected by decreasing the ventilation rate. Position B was identified as a "hot spot" at this ventilation rate. The chromium concentrations for these positions were in the range 380 to  $500 \,\mu\text{g/m}^3$ . Increased concentrations were also observed at positions R and S. Comparisons with the OSHA sampling method data are good considering the different sampling time scales involved. Consistent with measurements at other positions, the concentrations observed at these positions are highly variable on a three-min time scale. The data for position H was  $152 \pm 74 \,\mu\text{g/m}^3$  as determined by the SIBS instrument and  $228 \pm 158 \,\mu\text{g/m}^3$  by the OSHA sampling method.

Only a single measurement was performed at 1200 scfm. It was done at position M and a concentration of  $880 \pm 302 \,\mu g/m^3$  was measured. The chromium concentration in the breathing air space exceeded the regulated PEL, requiring respirators to be worn during this measurement for those personnel remaining in the area; all other personnel were required to exit.

#### Tests of the Push/Pull System

One of the purposes of the test was to evaluate the effectiveness of the push/pull liquid circulating system. During electroplating, by-product gases rise and disperse across the surface of the plating solution. These bubbles eventually burst and eject small droplets of solution into the air above the tank. The push/pull system is operated by injecting plating solution through a pipe near the bath surface at the front of the tank, toward the rear of the tank, where it is drawn in by another pipe. The rapid flow across the surface forces the majority of the bubbles to the rear of the tank near the ventilation inlet. This increases the droplet capture efficiency and decreases the chromium concentration above the tank.

The effectiveness of the push/pull system was evaluated by placing the SIBS monitor in a fixed position in an area of active electroplating and turning the liquid flow pump on and off. The results of these tests are shown in Table 2. Experiments were performed at 3263 and 1845 scfm ventilation flow rates and at two positions, G and I. Both were on the tank centerline and both were in areas of active electroplating. A second position several inches closer to the surface was also measured at position G. In all three measurements, the chromium concentrations with the pump off exceeded those with the pump on, often by a factor of two or more. This verified that the push/pull system successfully reduced the concentrations in these areas.

#### Data Correlations

The data correlate well with the ventilation rate. A plot of the data from positions M, I and G as a function of the ventilation flow rate is shown in Fig. 7. The measured concentrations uniformly decrease as the ventilation rate increases. The only

Table 2 Test of Push/Pull System								
Ventilation Ra	te Position	Avg. Conc. Pump on μg/m³	Avg. Conc. Pump off μg/m <sup>3</sup>					
3263	G1	58 ±24	121 ±27					
1845	I1	165 ±123	$259 \pm 102$					
1845	G1 (low)	576 ±397	1223 ±291					

exception is position I, where the use of the special cover substantially reduced the chromium concentrations at ventilation flow rates of 1704 scfm and below. The cover was not installed at the higher flow rates.

Correlation of instrument data to NIOSH- or OSHAapproved methods is a critical factor in determining regulatory acceptance of the data and in obtaining NIOSH recognition of the instrument or method. We have performed a correlation of the data in Table 1 for the SIBS monitor against the results of Draft Method ID-215. The resulting plot has a correlation coefficient ( $\mathbb{R}^2$ ) of 0.984, indicating excellent correlation.

A comparison of the SIBS monitor data has also been made against the total mass of chromium collected on the internal filters. During the tests, these filters were periodically removed, labeled and placed in plastic bags for temporary storage and shipment. A total of seven samples was sent to an independent laboratory for total chromium mass analysis. These results were compared to the monitor data by integrating the measured concentrations. The correlation, encompassing nearly two orders of magnitude of measured mass, is shown in Fig. 8. Two of the seven points were excluded, as the monitor had saturated as a result of exposure to extremely high concentrations. For the five valid points, the correlation is excellent with a correlation constant (R<sup>2</sup>) of 0.995. Additionally, four of the five SIBS monitor measurements are within ±20 percent of the filter measurements. The NIOSH guidelines<sup>11</sup> for acceptance of new air sampling and analytical methods require that 75 percent of the data agree to within  $\pm 25$  percent of the true value. Although the database here is too small to begin the acceptance process, it does demonstrate an important capability.

## Conclusions

The SIBS monitor data correlate well with distance above the bath surface, ventilation rate, areas of active electroplating, "hot spots" identified by the smoke test, chromium masses from the monitor filters, and chromium concentrations determined by OSHA-approved sampling methods. To our knowledge, this is the first instance of real-time chromium survey concentrations being measured above an electroplating tank. The results of this test indicate that the fugitive emissions from plating baths exhibit a high degree of spatial and temporal variability. Technologies aimed at reducing emissions are thus best evaluated with a portable, real-time instrument. A NIOSH-approved commercial product release of the SIBS instrument is anticipated in 1999.



Fig. 8—Comparison of the SIBS monitor data with internal filter measurements (best fit).

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