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THE ROLE OF CHROMIUM IN WHETLERITE ACTIVITY

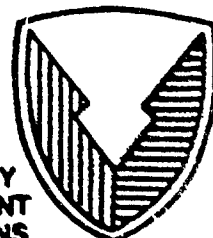
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PREFACE

The work described in this report was authorized under Project No. 1C162706A553, CB Defense and General Investigation. The work was started in May 1989 and completed in August 1989.

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THE ROLE OF CHROMIUM IN WHETLERITE ACTIVITY

1. INTRODUCTION

Impregnated activated charcoal is highly effective in removing gaseous toxic contaminants from breathable air. For example, activated charcoal impregnated with ammoniacal salts of copper, chromium, and silver has generally been used since World War I in military gas mask canisters as a standard filter for protection from toxic gases, such as cyanogen chloride (CK) and hydrogen cyanide (AC).¹ ASC whetlerite is a prototype of one such preparation that contains 6 to 8% copper, 2 to 3% chromium, and about 0.2% silver. The effectiveness of ASC whetlerite in removing toxic gases is believed to be due mainly to the physical adsorption, followed by one or more chemical reaction(s) between the toxicant and metal impregnants. For example, copper impregnant is believed to be essential for removing AC, and both copper and hexavalent chromium must be simultaneously present to destroy CK. The detoxification capabilities of the whetlerite have been carefully investigated in the past,² but very little information about the nature of chemical reactions occurring on the whetlerite is available at present. However, there is currently a great interest in identifying the mechanism(s) involved in the detoxification process. This interest stems from the fact that in recent years many of the chromium compounds were declared as potential carcinogens.³ Although there is no firm evidence confirming or denying the carcinogenicity of chromium in ASC whetlerites, the U.S. Army has put great emphasis on the need to remove chromium from ASC whetlerite and replace it with a nonhazardous impregnant.⁴ To accomplish this, one must understand the role of chromium on the whetlerite activity.

Impregnated activated charcoal is currently believed to promote chemisorption, which is followed by a transformation of the toxicants to nontoxic products. This process of detoxification could be either catalytic or stoichiometric. Most of the earlier studies seem to favor the catalytic process, in which the participating metal ion compositions are assumed to remain constant.^{5,6} However, there is strong evidence that a valence change from hexavalent chromium to trivalent chromium occurs during the process of detoxification. Uncertainty exists about whether this valence change is a parallel process unrelated to the proposed catalytic process transformation. In our earlier studies, we noticed a significant change in hexavalent chromium in the CK exposed ASC whetlerites.* We therefore decided to investigate if it is possible to discern the catalytic detoxification process from the stoichiometric transformations of the toxicants by accurately measuring the reduction in hexavalent chromium in a given ASC whetlerite sample.

This report details our experimental approach and observations along with a brief discussion of our results. Because hexavalent chromium seems to play a role in the detoxification of CK and AC, we have focused on the analysis of hexavalent chromium in the unexposed and exposed samples of whetlerites in this study.

*Krishanan, P.N., Birenzvege, A., Chemical Speciation of Copper and Chromium in Whetlerite Before and After Exposure to CK, U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, MD, unpublished data, 1989.

2. EXPERIMENTAL PROCEDURES

2.1 Sample Specifications.

The CK-exposed, AC-exposed, and corresponding unexposed ASC whetlerites were provided by the Quality Assurance Laboratory of the U.S. Army Chemical Research, Development and Engineering Center (CRDEC). Samples of the exposed and unexposed CK were taken from the same lot. The description of these samples is given in Table 1.

Table 1. Description of ASC Whetlerite Samples

Lot/Log	Description
1580/199	Unexposed
1580/199	CK exposed
1580/199	AC exposed
1581/200	Unexposed
1581/200	CK exposed
1581/200	AC exposed
1582/216	Unexposed
1582/216	CK exposed
1582/216	AC exposed
1578/200	Unexposed
1578/200	CK exposed
1578/200	AC exposed

Exposure to CK and AC was conducted under the following conditions:

CK exposure were done in canisters. Charcoal (250 mL) was challenged with CK at 4.0 mg/L and 50.0 L/min using intermittent flow until the breakthrough occurred. See the Appendix for breakthrough times and other details on these samples.

Exposure of AC was performed in tubes. Charcoal (12.0 g) was challenged with AC at 10.0 mg/L and 2.78 L/min using a constant flow until breakthrough occurred.

2.2 Reagents.

2.2.1 Sodium Carbonate (3%) - Sodium Hydroxide Solution (1%) (3-1 Solution).

To extract hexavalent chromium from ASC whetlerite, the following procedure should be followed:

Mix 30 g of sodium carbonate (Na_2CO_3 , Fisher Scientific Company, Silver Spring, MD) with 10 g of solid sodium hydroxide [NaOH , Fisher Scientific Company] in a large beaker and dissolve the chemical in 100 mL of distilled water. The volume was made to 100 mL, using a volumetric flask. This solution had a pH of 12.0.

2.2.2 Phosphoric Acid - Sulfuric Acid Mixture.

For chromium titration:

Phosphoric acid (65 mL) [H_3PO_4 (Fisher Scientific Company, 86% assay)] was carefully added to approximately 100 mL of distilled water in a large beaker. Concentrated sulfuric acid (83 mL) was then added to the diluted phosphoric acid. The solution was allowed to cool for 1 hr and then diluted to 500 mL in a volumetric flask using distilled water.

2.2.3 Mohr's Salt Solution (0.1 N Ferrous Ammonium Sulfate).

For chromium titration:

Ferrous ammonium sulfate (39.3 g) [$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)2.6\text{H}_2\text{O}$] - Fisher Scientific Company] was dissolved in distilled water, treated with 5 mL of concentrated sulfuric acid, and diluted to a final volume of 1,000 mL using distilled water.

2.2.4 Diphenylamine Sulfonate Indicator.

For chromium titration:

Barium diphenylamine sulfonate (0.1238 g) was transferred to a 25-mL volumetric flask, dissolved in about 15 mL of distilled water, and the resulting solution was made up to 25 mL with distilled water.

2.2.5 Potassium Dichromate (0.05 g) ($\text{K}_2\text{Cr}_2\text{O}_7$ Fisher Scientific) Solution.

For chromium titration:

Potassium dichromate (1.2431 g) was dissolved and diluted to 500 mL in a volumetric flask.

2.2.6 Diphenyl Carbazide Solution.

For colorimetric determination of chromium:

Diphenyl carbazide (100 mg) was dissolved in 20 mL of acetone in a graduated cylinder and then diluted to 40 mL using distilled water.

2.3 Determination of Hexavalent Chromium Content.

The currently established extraction procedures reveal that all the hexavalent chromium from ASC whetlerites can be extracted at pH = 12 in 3% sodium carbonate-1% sodium hydroxide solvent without interfering with the trivalent chromium present.⁷ The procedure was adopted for determining hexavalent chromium in most cases. In a few cases (AC exposed) where the concentration of hexavalent chromium was too low, an additional colorimetric test using diphenyl carbazide chromophore was also employed.⁸

The Procedure for chromium speciation follows:

The charcoal samples were dried for 2 hr at 110 °C and kept in a desiccator to protect them from moisture. The 250-mg specimen was weighed into a 50-mL Griffin beaker. A 3% sodium carbonate-1%-sodium hydroxide solvent (10 mL) was added to the contents of the beaker. The beaker was covered with a watch glass and heated to just below the boiling point for 60 min. The contents of the beaker were allowed to cool. The liquid phase was aspirated into a 250-mL Erlenmeyer flask. The residue in the beaker was rinsed with water, and the washing was aspirated into the Erlenmeyer flask. Phosphoric acid/sulfuric acid (2.5 mL), 0.1 M Mohr's salt solution (exactly 5.00 mL), and diphenylamine sulfonate indicator (5 drops) were added to the contents of the Erlenmeyer flask. The contents of the flask were titrated with 0.05000 N potassium dichromate solution until the green-purple color transition persisted for 30 s. The volume of potassium dichromate required for the titration was recorded. The blanks were prepared by starting with 10 mL of the sodium carbonate-sodium hydroxide solvent following the above procedure. Each run was repeated 10 times.

The colorimetric method used for the AC-exposed samples follows:

The specimen of ASC whetlerite (250 mg) was extracted with 8 mL of 3% sodium carbonate-1% sodium hydroxide as described above. The liquid phase was decanted into a 10-mL volumetric flask. The residue was washed with distilled water, and the washings decanted into the flask. The contents of the flask were diluted to 10 mL using distilled water. Exactly 0.5 mL of the diluted extract was pipetted into a second 10-mL volumetric flask. Diphenylcarbazine reagent (0.5 mL) and 1 N sulfuric acid (0.5 mL) were added to the contents of the second flask and the volume was diluted to 10 mL using distilled water. After mixing, the solution was transferred to testtube cuvettes; 2 min later, the absorbance was measured at 540 nm. The standards were prepared, and the absorbance of the standards was measured simultaneously. The standards contained 0.0, 0.5, 1.0, 1.5, 2.0, 3.0, and 5.0 µg/mL.

3. RESULTS AND DISCUSSION

Hexavalent chromium contents of various samples of ASC whetlerites, both exposed and unexposed to CK and AC, were determined using titrametric and colorimetric procedures.⁹ These samples were extracted with a 3% sodium carbonate-1% sodium hydroxide solution, and the extracts were used for assays. All assays were done in deciplicates. The accuracy and reproducibility is in the range of 2%. The results are shown in Table 2.

Table 2. Hexavalent Chromium Content of Whetlerite

Lot/Log	Mean % (Standard Deviation)		
	Unexposed	CK Exposed	AC Exposed
1578/200	2.12 (0.03)	1.42 (0.03)	0.13 (0.008)
1580/199	2.06 (0.04)	1.30 (0.08)	0.08 (0.007)
1581/200	2.14 (0.02)	1.17 (0.06)	0.12 (0.001)
1582/216	2.11 (0.04)	1.11 (0.04)	0.12 (0.008)

As seen in Table 2, there is a profound reduction in the hexavalent chromium upon exposure to AC and CK. In the case of AC, the reduction in hexavalent chromium is over 90%. Cyanogen chloride also shows a significant reduction of about 30-50%.

ASC whetlerites were equilibrated with 80% relative humidity (RH) at 80 °F for 4 hr prior to exposure to AC and CK. Therefore, the exposed samples contained a larger amount of water. High water content in ASC whetlerites promotes aging, during which time a partial reduction of hexavalent chromium species to trivalent chromium species might take place.¹⁰ To assess whether the observed reduction in our studies was due to humidity, we analyzed the samples for their water content. The results are shown in Table 3.

Table 3. Analysis of Water Content in Samples 1578, 1580, 1581, and 1582

Sample ID	Mean Percent (Standard Deviation)		
	Unexposed	CK Exposed	AC Exposed
1578/200	1.57 (0.15)	14.8 (0.5)	12.5 (0.3)
1580/199	1.56 (0.24)	15.1 (0.1)	12.3 (0.2)
1581/200	1.84 (0.46)	15.2 (0.2)	9.3 (0.1)
1583/216	2.00 (0.08)	15.8 (0.1)	12.5 (0.3)

As seen from this analysis, the water content of both CK-exposed and AC-exposed ASC whetlerite is considerably higher than the unexposed ASC whetlerites. This result suggests that perhaps the observed valence change in chromium may be due to the increase in water content. To analyze this possibility, we took a sample of unexposed ASC whetlerite (1580/199 unexposed) and exposed that to 80% RH for 16 hr. The sample showed an increase in water content of approximately 25%.¹⁰ Analysis of these samples showed only 7% reduction of the hexavalent chromium content. Hence, the observed reductions in hexavalent chromium in both AC- and CK-exposed samples are likely to be related to the chemical process involved in the detoxification.

Taken together, these findings strongly argue against a catalytic role of hexavalent chromium in the detoxification process. If the mechanism of detoxification of either of the CK or AC toxicants were catalytic, one should not see such a dramatic reduction in hexavalent chromium content. Hence, it may be stated that perhaps the process of detoxification has a non-catalytic, stoichiometric involvement of hexavalent chromium. However, our results are still only suggestive. To be definitive, it is necessary to correlate the change in hexavalent chromium to the rate of formation of the detoxified products. For example, it has been suggested that in the case of AC, the cyanogen (CN)₂ is formed as an initial product and then undergoes subsequent hydrolytic transformation to oxamide.^{5,6} Measurement of the amount of oxamide formed and its correlation with the reduction of hexavalent chromium may provide further evidence for the oxidative transformation involved in the detoxification.

The difference between the behavior of CK and AC towards ASC whetlerite deserves further investigation. In these studies, we observed over 90% reduction of hexavalent chromium in AC-exposed samples, while CK-exposed samples showed a reduction of about 40%, suggesting that the mechanism of detoxification may be different for these toxicants. It is known that copper is required for AC destruction. It is believed that the initial interaction of AC with copper leads to the formation of cyanogen gas and that the role of chromium is to promote the hydrolytic transformation of cyanogen to oxamide. Therefore, it is believed that it would be of interest to expose

ASC whetlerite samples to cyanogen gas and correlate the reduction in the hexavalent chromium with the ASC whetlerite samples exposed to hydrogen cyanide. Such studies would then establish the role of chromium in the detoxification of cyanide ions.

4. CONCLUSIONS

Exposing ASC whetlerite samples to AC produced a very large reduction (over 90%) in the hexavalent chromium content. Likewise, exposing the samples to CK also resulted in a significant reduction (about 40%) in the hexavalent chromium. This reduction in the hexavalent chromium was not due to humidity. These facts suggest that the catalytic process proposed earlier is not acceptable and that an oxidative process is indeed prevailing.

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APPENDIX

Quality Control/Quality Assurance (QC/QA)

To validate the experimental procedure, we conducted the Quality Control/Quality Assurance (QC/QA) program. Samples of ASC whetlerite of a particular lot were extracted and analyzed for Cr^{+6} . The experimental procedures were such that the Cr^{+6} content was analyzed to show: (1) homogeneity, (2) preservation with time, and (3) completeness of recovery. Each test was conducted on the following four different samples.

Trial	Sample Description	Cr^{+6} Content (Standard Deviation)
1	250 mg	2.05 (0.03)
2	250 mg - 3 days later	2.05 (0.02)
3	125 mg	2.05 (0.03)
4	1 mg Cr^{+6} spiked to 250 mg ASC whetlerite	97.5% recovered

In each case, the four samples were withdrawn and extracted for Cr^{+6} content. Trial 2 was analyzed 3 days after being extracted and showed that the hexavalent chromium content did not deteriorate with time. Agreement between Trials 1 and 3 shows that the Cr was homogeneously dispersed through the sample, and the result did not depend on sample size. Trial 4 showed the completeness of recovery.

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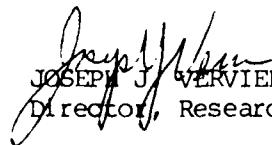
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