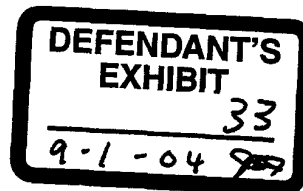


WGK ANALYTICAL RESEARCH AND DEVELOPMENT REPORT No. 1813, December 30, 1970

OTHER LOCATIONS	GENERAL OFFICE	W. G. KRUMMRICH PLANT
G. L. Jesse - Anniston T. Bell - Anniston E. G. Wright - Anniston M. W. Dietrich - JFQ	P. B. Hodges	D. W. Jackson R. M. Blowers File



TITLE: Analysis of Anniston Plant Waste Streams for Metals

PERSONNEL:

L. Briggs, D. Arras, K. Schutzenhofer, M. Webb (H. J. Horner)

PROBLEM:

Determine levels of metallic elements, cyanide and phenols in plant waste streams.

JUSTIFICATION:

The levels of potentially toxic materials entering the environment must be known in order to define any problems that exist in meeting government water quality standards.

SUMMARY:

A preliminary analytical survey of waste stream samples from the Anniston Plant has been completed. The samples were analyzed for arsenic, cadmium, chromium, lead, mercury, copper, selenium, zinc, cyanide and phenols. These are the Priority I & II items as outlined in P. Hodges' memo of Oct. 6, 1970, "Analysis of Waste Samples for Metals".

A summary of analytical results are given in the attached table. A brief description of the analytical method used in this work is included in the Detail Section.

Possible problem areas in meeting the standards for public water supplies exist with Lead, Mercury, Selenium and Phenols.

by

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M. E. Webb
M. E. Webb

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Details

A brief description of the analytical methods used in this work follows:

1. Cyanide (W GK Method A-1008)

This method is based on the FWPCA method from the Nov. 1969 issue of "FWPCA Methods for Chemical Analysis of Water and Wastes." Cyanide ion and complex cyanides are converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium and mercuric ions. The cyanide is absorbed in a solution containing sodium hydroxide. The cyanide ion is then determined colorimetrically using the pyridine pyrozone reaction.

2. Phenols (W GK Method 12,791)

This is a colorimetric method based on the formation of a color by the reaction of 4-aminoantipyrine with phenolic compounds in the presence of an alkaline oxidizing agent. The determination is done automatically with a Technicon Autoanalyzer. This method is calibrated against phenol. The method is non-specific and can vary significantly depending on the type of phenolic compounds present.

3. Arsenic (W GK Method A-1005)

The sample is digested, if necessary, with sulfuric and nitric acid to destroy organic matter. The arsenic in the sample is reduced to arsine gas which is absorbed in a pyridine solution of silver diethyldithiocarbamate. The resulting red colored complex is measured colorimetrically.

4. Mercury (W GK Method A-1002)

Samples are treated with $\text{KMnO}_4\text{-H}_2\text{SO}_4$ to destroy complexes and place all mercury in the inorganic form. Mercury salts are reduced to elemental mercury by stannous chloride and the mercury vapor passed through an absorption cell with a stream of air. The light absorption by the mercury is measured with an atomic absorption spectrophotometer.

5. Selenium (W GK Method A-1010)

Samples are oxidized by an acid-permanganate digestion. Selenates are then reduced and reacted with 3,3'-diaminobenzidine hydrochloride forming a yellow colored complex. The colored complex is extracted with toluene and the color intensity is measured with a spectrophotometer.

6. Cadmium (Modification of W GK Method A-1006)

Samples are digested by boiling after addition of nitric and hydrochloric acid. The pH is adjusted to 6.5 and the cadmium is complexed by the addition of sodium diethyldithiocarbamate.

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The complex is extracted with methyl isobutyl ketone and measured by atomic absorption spectrophotometry.

7. Copper, Lead, Zinc and Chromium (Modification of WCK Method A-1007)

Samples are digested by boiling after addition of nitric and hydrochloric acid. The pH is adjusted to 4.0 and the metals are complexed by the addition of ammonium pyrrolidine dithiocarbamate. The complexes are extracted with methyl isobutyl ketone and measured by atomic absorption spectrophotometry.

Lab Notebook Ref: 14544-14551
14712-14714
14720-14723
13934-13972

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Anniston Plant Waste Streams

	Pit Effluent 10-12/10-18 0.005	Pit Effluent 10-19/10-25 0.005	Plant Effluent 10-26/11-1 0.005	Plant Effluent 11-2/11-9 0.005
Arsenic	< 0.005	< 0.005	0.005	< 0.005
Cadmium	< 0.005	< 0.05	< 0.05	< 0.05
Chromium	< 0.05	< 0.05	0.11	< 0.05
Lead	< 0.05	< 0.005	0.012	< 0.005
Mercury	< 0.005	< 0.025	0.075	< 0.025
Copper	0.054	0.008	0.011	< 0.002
Selenium	0.13	0.13	0.10	0.10
Zinc	< 0.005	< 0.005	< 0.005	< 0.005
Cyanide	2	5	6	1
Phenols				

Results are in parts per million

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