Report on the Results of the AES/EPA Sludge Characterization Project

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The disposal of waste treatment sludges has become of major concern to the metal finishing industry. This fact became evident at the first of the EPA/AES Conferences in Orlando, Florida in January 1978.

In many areas of North America, due to the lack of knowledge and understanding of the behaviour of these sludges in landfill sites, disposal had become extremely expensive, and, in fact, a virtual impossibility in many instances.

The problems associated with sludge disposal became exacerbated with the introduction by the EPA of a proposed test method, under the Resource Conservation and Recovery Act. This test was designed in such a way as to simulate the conditions under which different types of solid waste would behave, in conjunction with the codisposal of organic wastes, such as may be found in a sanitary landfill.

The test proposed by the EPA calls for filtering or centrifuging the sludge, and then agitating the remaining solid material for twenty-four hours, with a predetermined amount of water. This is maintained at a pH of 5, using acetic acid. The resultant extract is mixed with the original filtrate from the original sample, and the concentrations of various materials are compared to the limitations for these materials as prescribed by the EPA. These concentrations are generally established at ten times the drinking water standard.

The materials of concern to the metal finishing industry with the proposed limitations are shown in Table I.

T/ PROPOSED	ABLE I HAZARD	LIMIT
		mg/ 1
Chromium		0.5
Cadmium		0.1
Lead		0.5
Arsenic		0.5
Mercury		0.02
Silver		0.5

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Solid wastes that yielded results above the proposed limitations, would require a manifest system, and disposal must be made to an approved facility. While these facilities are not yet determined or generally available at present, it is possible that they would include some combination of fixation, landfill lining, and leachate collecting and treatment.

So obvious were the consequences of these added burdens that discussions ensued between the EPA and the AES, with a view to reconciling the behavior of electroplating waste sludges under the EPA extractions procedure, with their behavior under field conditions.

The proposal for a co-operative agreement evolved during 1978 and the final draft was presented at the second EPA/AES Conference in February 1979. Approval of the funding of 95% of the program by EPA came in March 1979, and the Task Force appointed by the board of directors of AES began its work with the author as technical director.

The Task Force members were Doug Thomas, chairman, Fred Steward, Irving Ireland, Richard Crain, and George O'Connor.

Howard Schumacher, the executive director of AES was also appointed program co-ordinator, and Centec Corporation of 11800 Sunrise Valley Drive, Reston, Virginia was chosen as the engineering and laboratory sub-contract or.

The method chosen by the Task Force called for the selection of twelve sludge sources that represented the spectrum of the metal finishing industry. These were to be subjected to a series of tests.

Phase I was designed to:

- •Characterize chemically and physically the solid and liquid portions of the sludges.
- Determine the effect of pH on results of the EPA extraction procedure (EP).
- •Determine the effect of interstitial water, (i.e. water in the sludge) on the EP results.
- . Determine the effect of sludge aging on EP results.

Phase II was designed to:

. Simulate more closely a segregated landfill containing hydroxide sludges, where there would not be severe agitation or exposure to low pH using a dynamic laboratory procedure.

Phase 111:

• was a field test, where soil samples were taken below a sludge bed that had been in use for ten-twelve years at the site of one of the plants from which current sludge samples were taken. (Plant #11).

TABLE II PLANT DESCRIPTION

Plant	% Solids	Primary Plating Process
1A	11	Segregated Zn
2A	6	Segregated Cd
3A	3	Zinc Plating and Chromating
4A	7	Cu-Ni-Cr on Zn
5A	17	Al Anodizing
6A	34	Ni-Cr on Steel
7A	15	Multi-process Job Shop
8A	29	Electroless Cu on Plastic, Acid Cu, Ni, Cr
9A	39	Multi-process with Barrel or Vibratory Finishing
10A	24	Printed Circuits
11A	19	Ni-Cr on Steel
12A	23	Cd-Ni-Cu on Brass and Steel

Phase I, description of the experiments, results, conclusions. and recommendations are contained in the Interim Phase J Report, which was submitted in September 1979.

The complete report, under the title "Interim Phase I Report, Electroplating Wastewater Sludge Characterization" is available at no cost from John Lehman, EPA, Office of Solid Waste (WH-565), Washington, D.C. 20460. A synopsis of this paper was published in Plating and Surface Finishing.

In the interest of time, I will touch on the highlights of that report and its conclusions.

Table H describes the plants sampled and the toxic materials, as defined by the proposed RCRA regulations that were present in the sludges as weight percentage of the dry solids.

Time and resources did not permit in-depth engineering studies of the plants being sampled, but with one exception, sufficient data was obtained to describe the nature of the treatment system and the volumes involved.

Figure 1 is a schematic of the extraction procedure. The experiments conducted included:

- •Effect of pH on extraction.
- . Effect of volume of extraction water.
- . Reproducibility.
- •Effect of temperature.

TABLE III EFFECT OF PH mg/l							
1A (11% Solids)	pH 5	1.22	0.23	0.041	0.073		<0.01
2A (6% Solids)	рН 5	1.89	126	<0.001	0.005		<0.01
3A (3% Solids)	pH 5	85.0	6.00	0.009			
4A (7% Solids)	pH 5	21.8		0.038			.02
5A (17% Solids)	pH 5	<0.01		<0.001			<0.01
6A (34% Solids)	pH 5	25.4	<0.01	.001			
7A (15% Solids)	pH 5	0.24	2.16	.003			
8A (29% Solids)	pH 5	400		0.032	0.0450	.02	
9A (36% Solids)	pH 5	0.32	0.03	.010			
10A (24% Solids)	pH 5	0.12		0.88			
11A (19% Solids)	pH 5	4.22	<0.01	0.004	0.035		
12A (23% Solids)	pH 5	4.85	263	0.031	<0.001		

Blanks, Ba, and Se were at or below detection limits.



Figure I. Extraction Apparatus

- Comparison with ASTM extraction procedure,
- Effect of sludge aging,
- Total metal content.
- Anion content.
- X-ray diffraction.
- Filtrate analysis and washing.
- Filtration versus centrifuging.

In Table HI, the results of the execution of the extraction procedure at a maintained pH of 5, as proposed by the EPA, and at four other pH's is shown.



Figure II. Changes in Cadmium Concentration with pH



Figure III. Changes in Chromium Concentration with pH

Figures 11 and III show the results in graph form for typical plants with significant cadmium and chromium content.

Table IV shows the effect of aging on the EP at three months, compared with the original extraction.

The conclusions that were reached concerning Phase I were:

- EP is sensitive to pH.
- EP exaggerates the leachability y of the sludge since it includes the effect of metal levels in interstitial (i.e. associated) water.
- . Aging greatly decreases leachability.

TABLE IV EFFECT OF AGING SLUDGE						
EP Results mg/1 Plant 7A						
	Cd	Pb	Cr			
pH 5 Fresh Aged (3 months)	2.16 0.30	0.003 0.002	0.24 <0.05			
pH 7 Fresh Aged (3 months)	0.04 0.01	0.005 0.001	0.50 0.15			

The interstitial water is simply the supernatant and entrapped water mixed with the sludge. Its concentrations of metallic ions will be essentially the same as the plant's effluent as discharged to a water body or a municipal sewer. It is possible that a plant could be meeting an effluent discharge regulation, but would be beyond the proposed hazard limit for sludges, because of the metal content of the interstitial water alone. This point is further developed in the interim report, and I would recommend it to your attention.

Surprisingly, and in spite of the presence of some interstitial water, Plants #5 and #9 passed the very aggressive EP test.

When the distorting effect of the interstitial water is removed, the sludge then behaves in the extraction as one would expect from the laws of chemistry.

Using the results of Phase I as a guide, we were able to enter Phase II work, knowing much of the nature of the sludges with which we were dealing.

Phase II was the dyanamic testing designed to simulate segregated landfill conditions.



Figure IV

A reduced number of plant sludges was used for this Phase, in order to maximize the number of experiments. Plants 4, 5, 6, 8, 11, and 12 were chosen as being widely representative of those with hazardous materials, as defined by RCRA.

The complete description of the procedure will appear in the final report, which will be available this summer. However, the schematic drawing in Figure IV encompasses some pertinent data.

Samples are poured into weighed Buchner funnels seated with filter paper. D.I. water is fed from a container above the filter. A vacuum is applied to remove the interstitial waters and these are then added back to the top of the sample cake, so that it can be leached through at a more normal rate.

The leachates were analyzed on days 1, 2, 3, 5, 7, and every seventh day thereafter for the metals expected to be **most predominant**.



Figure V. Dynamic Testing of Chromium & Cadmium Versus Days Leached.

TABLE V PHASE II DYNAMIC LEACHING								
Days Leached Chromium mg/1								
Plant Sample Code	EP Test Results	1	2	3	5	7	14	21
4 A	21.8	1.01	0.14	0.12	0.12	0.08	0.08	0.08
5A	<.01	< 0.05	< 0.05	0.06	0.06	0.07	0.05	<0.05
6A	184.0	< 0.05	< 0.05	< 0.05	<0.05	<0.05	< 0.05	<0.05
8A	400.0	*		*	*	*	*	*
11A	4.2	< 0.05	0.05	*	٠	*	*	
12A	4.6	0.34	0.21	0.15	0.12	<0.05	0.07	0.05
Cadmium mg/l								
12A	268	0.24	0.18	0.16	0.12	0.11	0.09	0.09
* = <0.05								

The results are shown in Table V.

Figures V and VI show graphically and rather dramatically how quickly the interstitial water is flushed through, and how little material is leached thereafter,

The flow rates involved were approximately equal to one half inch of rain per day for the entire thirty-five days of the test.

It will be noted that these leaching results are approximately the same as the results obtained from the extraction made in Phase I at a pH of 7.

Although nickel is not on the list of hazardous metals as defined by RCRA, we have included it to show that it behaves in a similar manner.

The conclusions that may be drawn from the Phase 11 work are:

- . Dynamic tests show drastically reduced levels of metal concentrations in the leachate, compared to the EP.
- This work confirms the indications from Phase I that interstitial water can be the major contributor to metals in leachate.

I would like to repeat that this interstitial water is essentially the same as the effluent and is a very small percentage of the total metal content of the sludge. A calculation of this percentage is in the Phase I report.

Also established is the fact that for a sludge source to apply stringent dewatering to his settled solids in order to pass an EP test would not, indeed, make a contribution toward improving the environment. The decision as to how much to dewater should be based on the pragmatic



Figure VI. EPA-AES Nickel (Ni) Leachate vs Days Leached

economics of shipping and handling, space limitations, etc.

While building the information bases in Phase I and II, we had largely exhausted our available funds. However, we did manage to have sufficient means for one field trip to the location of Plant #11.

This plant had begun to dispose of metal finishing waste treatment sludges twelve years ago in a pond created by excavation on their property. This pond became dormant six years ago and a new pond was developed some miles away that was dug to a limestone base.

In order to gain access to the soil under the pond, (there was actually two ponds), the company pumped it out just before our arrival. Samples were taken at two depths of soil below the interface and at the interface. A background sample was taken from the soil at a distance sixty feet from the pond.

During the pumping-out procedure, the suction end of the hose kept plugging with bullfrog tadpoles. Some of these hundreds of tadpoles were still alive when we arrived.

Samples were also taken from the newer limestone based pond and from a drainage ditch at a point thirtyfive feet below the pond.

The results of these samplings are shown in Table VI. Each sample, except the water sample, was subjected to the EP test at a pH of 5, and some were completely digested and analyzed.

While this Phase III work was limited, (more sampling work should be done in the field) there was no result which disproved the outcome of Phases I and 11, but the evidence was, rather, to the contrary. There appears to be no harmful effect on the environment when metal hydroxide sludges are disposed of in a segregated landfill.

The landfill need not be elaborately prepared, such as having lining or limestone base, and without the necessity of paying for expensive fixation treatment. This information, in turn, will permit more flexibility in the location of these landfill sites, which could also receive other sludges such as 'fly-ash, without disturbing the plating waste sludges.

Some unfinished work, however, still remains. There is always the possibility y that' a malfunction of a waste treatment system may produce a sludge that would not be compatible with other sludges in a segregated landfill. It

TABLE VI PHASE III — PLANT SITE II							
New Land Fill mg/1							
Plant Sample Code	Ag	As	Cr				
Leachate	0.01	0.004	< 0.05				
Surface Sludge	0.02	0.006	3.58				
Subsurface Sludge	0.01	0.005	4.37				
Older Subsurface Sludge	<0.01	0.002	3.35				
(Old Land mg/1	Fill					
11" Below Sludge	0.03	0.022	<0.05				
17 ["] Below Sludge	0.02	0.011	<0.05				
Sludge	0.19	0.002	0.09				
Background Soil	<0.0!	0.004	<0.05				
(Samples were also analyzed detection limits.)	d for Ba, C	d, Pb and Se. A	ll were below				

is important that the sludge generator knows this before he ships that particular sludge, and for the landfill operator to know that he is receiving a "safe" sludge.

To help minimize this potential problem and to simplify test procedures, future work should include:

- . Development of a quick test which would separate the effects of interstitial water from sludge leachability, and would confirm the sludge's suitability for disposal in a segregated landfill.
- Obtaining data which would provide guidance to the plater which will ensure him that his sludge will be suitable for disposal in a segregated landfill.

Much of the success of the project has resulted from the very high level of technical direction received from the Task Force and the very professional performance of Centec Corporation and its personnel,

The Task Force that volunteered its time, put more of it into this project than originally was anticipated. It always responded to the requests of the chairman, Doug Thomas, Howard Schumacher, and myself.

The co-operation of the Metals and Inorganic Chemicals Branch of EPA's Industrial Environmental Research Laboratory in Cincinnati, and George Thompson, and Fred Craig in particular were vital to the completion of this work.