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Application Update 149



Determination of Metal Cyanide Complexes in Solid Wastes by Anion-Exchange Chromatography with UV Absorbance Detection

Metal cyanides are negatively charged ionic complexes represented by the general formula $[M(CN)_b]^{x-}$, where one or several cyanide ions are bound to a single transition metal cation such as Ag⁺, Au⁺, or Fe²⁺. The environmental impact of metal cyanides varies widely from one species to another depending on how readily they release free cyanide (HCN/CN⁻) into the environment. Several indirect methods are available for determining free and complexed cyanide.¹ However, anion-exchange separation of these complexes is the only method available that specifically identifies and quantifies each metal cyanide complex in a sample. Previous Dionex application documents describe the determination of metal cyanide complexes in metal finishing and mining wastes² and environmental waters at mg/L³ or μ g/L concentrations.⁴

This application update describes the determination of the metal cyanide complexes of iron, cobalt, silver, gold, copper, and nickel in solid wastes by anion-exchange chromatography with UV absorbance detection. Metal cyanide complexes are solubilized and recovered by an alkaline extraction procedure (SW846 Method 9013)⁵ prior to chromatographic analysis. Two analytical approaches are available depending on the concentration of metal cyanides expected in the leachate; the two methods differ only in how the sample is injected:

• Metal cyanide complex concentrations between 0.20–200 mg/L are determined by direct injection of the sample. This range is approximate and depends on the detection sensitivity, which varies among the analytes. Concentrations exceeding the linear calibration range may be determined after appropriate dilution.

 Metal cyanide complex concentrations less than 0.20 mg/L are determined by on-line sample preconcentration. This application update uses a simpler and more direct approach to preconcentration than that described in Dionex Application Note 161.⁴ The sample is directly applied to the concentrator column by the AS50 sample syringe, eliminating the sample loop, column valve, and extra pump required by Application 161.

This application update also summarizes expected method performance as determined during participation in an interlaboratory collaborative study to validate the use of anion-exchange chromatography for the determination of metal cyanide complexes in solid wastes. The method was evaluated for sensitivity, linearity, accuracy, precision, and spike recovery from various matrices.⁶

EQUIPMENT

Dionex ICS-2500 IC system consisting of: GS50 Gradient Pump AD25 Absorbance Detector AutoSelect[™] AS50 Autosampler (USB) AS50 Dual-Valve Needle Assembly (P/N 061267) Sample PREP Syringe, 10-mL (P/N 055068) Chromeleon[®] Chromatography Workstation (Version 6.6 or higher)

CONSUMABLES

Syringe filters (Gelman IC Acrodisk® 0.2-µm, PN 4483)
Storage bottles, amber HDPE (VWR IRN301-0125 or 16172-144)
Vial Kit 10-mL polystyrene (Dionex P/N 055058)

GM-4 Gradient Mixer (Dionex P/N 49135)

CONDITIONS

Columns:	Analytical: IonPac® AS11 2 × 250 mm (P/N 44077)					
		Guard: IonPac AG11 2 × 50 mm (P/N 44079)				
		ntor: IonPac mm (P/N 4	AG11 Guard 4078)			
	Trap: Ionl	Pac ATC-3 (P/N 059660)			
	Trap: MF	C-1 (P/N 37	017), 2			
Temperature:	35 °C (or	ambient)				
Injection:	•	r 5 mL conc AG11 colur				
Detection:	Absorban	ce at 215 nn	n			
System Pressure:	~850 psi					
Noise:	1–5 mAU					
Run Time:	32 min					
Flow Rate:	0.25 mL/	min				
Eluent A:	20 mM sodium hydroxide/150 mM sodium cyanide					
Eluent B:	20 mM sodium hydroxide /300 mM sodium perchlorate					
Eluent C:	20 mM sodium hydroxide					
Pump Program:						
Time (min)	%A	%B	%C			
Init.	10	10	80			
0.0	10	10	80			
18.0	10	45	45			
22.0	10	45	45			
25.0	10	10	80			
35.0	10 10 80					

REAGENTS AND STANDARDS

Copper cyanide (AlfaAesar 12135) Potassium dicyanoargentate (I) (AlfaAesar 12551) Potassium dicyanoaurate (I) (AlfaAesar 12552) Potassium ferrocyanide (II) trihydrate (Aldrich 22,768-4) Potassium hexacyanocobaltate (III) (AlfaAesar 23126) Potassium tetracyanonickelate (II) hydrate (Strem 93-2836) Sodium cyanide, 99.99 % (Aldrich 43,159-1) Sodium hydroxide solution, 50% (w/w) (Fisher SS254) Sodium perchlorate monohydrate, HPLC-grade (Fisher S490)

PREPARATION OF SOLUTIONS AND REAGENTS

Precaution: Sodium cyanide and some of the metal cyanide complexes are very toxic. Avoid contact with water or acid. Clean up and properly dispose of any spills.

Prepare all solutions from analytical reagent-grade chemicals. Use ASTM Type I reagent-grade water with a specific resistance of 18.0 M Ω -cm or greater. Filter the water through a 0.2-µm filter immediately before use and degas by sonicating under vacuum or sparging with helium for 10–15 min.

Always prepare sodium hydroxide eluents with 50% (w/w) sodium hydroxide solution. (Do not use sodium hydroxide pellets; they are covered with a thin layer of sodium carbonate that will cause irreproducible results). Keep all eluents blanketed under helium at 34–55 kPa (5–8 psi) after preparation. Properly dispose of old eluent and prepare fresh after one week.

Eluent Preparation

Eluent A (20 mM Sodium Hydroxide/150 mM Sodium Cyanide)

Place 14.70 g sodium cyanide into a 2-L volumetric flask containing 1.9 L of degassed reagent water. Use a plastic pipette to deliver 2.08 mL (or 3.2 g) of 50% (w/w) sodium hydroxide. Bring to volume with degassed reagent water. Cap and invert the volumetric flask eight times to mix.

Note: Do not mix excessively, as this will increase the carbonate ion in the solution by trapping carbon dioxide from the air.

Eluent B (20 mM Sodium Hydroxide/300 mM Sodium Perchlorate)

Place 84.20 g HPLC-grade sodium perchlorate monohydrate (NaClO₄ • H₂O) into an implosion-proof container that contains 1.9 L of reagent water and degas for 20 min by sonicating under vacuum. Transfer to a 2-L volumetric flask. Use a plastic pipette to deliver 2.08 mL (or 3.2 g) of 50% (w/w) sodium hydroxide. Bring to volume with degassed reagent water. Cap and invert the volumetric flask eight times to mix. Blanket with helium as described above.

Eluent C (20 mM Sodium Hydroxide)

Add 2.08 mL (or 3.2 g) of 50% (w/w) sodium hydroxide to a 2-L volumetric flask containing 1.9 L of degassed reagent water. Bring to volume with degassed reagent water. Cap and invert the volumetric flask eight times to mix. Blanket with helium as described above.

Standard Preparation

Store the metal cyanide reagent salts in a dessicator protected from the light. Prepare 1000 mg/L stock standards of each metal cyanide complex by consulting Table 1. Weigh the reagent salt into a 100-mL volumetric flask. Add enough 20 mM sodium hydroxide solution to dissolve, bring to volume with 20 mM sodium hydroxide solution, mix, and transfer to an amber HDPE bottle. Store at 4–6 °C. The individual stock standards are stable under these conditions for the periods shown in Table 1.

Prepare calibration standards spanning the range of interest for each analyte by diluting the 1000-mg/L stock standards with 20 mM sodium hydroxide solution. To prepare mixed standards, measure appropriate volumes of the 1000-mg/L standards into 100-mL volumetric flasks, bring to volume with a 20 mM sodium hydroxide solution, mix, and transfer to an amber HDPE bottle. These mixed calibration standards should be prepared fresh on the day of use.

Tables 2 and 3 show the concentrations of calibration standards used for this document.

Table 1. Preparation of Metal Cyanide Stock Standards						
Anion	Compound	mpound Mass (g)				
[Ag(CN) ₂]-	KAg(CN) ₂	0.1244	1			
[Au(CN) ₂]-	KAu(CN) ₂	0.1157	30			
[Cu(CN) ₃] ²⁻	Cu(CN) and NaCN	0.0632*	1			
[Ni(CN) ₄] ²⁻	$K_2Ni(CN)_4 \cdot H_2O$	0.1591***	1			
[Fe(CN) ₆] ⁴⁻	K ₄ Fe(CN) ₆	0.1993	30			
[Co(CN) ₆] ³⁻	K ₃ Co(CN) ₆	0.1546	30			

*Dissolve the CuCN with 0.138 g sodium cyanide in a 100-mL volumetric flask containing a 50-mL 20 mM sodium hydroxide solution. Bring to volume with 20 mM sodium hydroxide solution.

**Stability in number of days when stored in amber HDPE at 4–6 °C. Prepare fresh stock standards as needed according to this table.

***Dissolve (1.4806 + 0.1107 × n) g of potassium nickel cyanide mono- or polyhydrate, [K₂Ni(CN)₄ + nH₂O, where n = number of water molecules of hydration.

Table 2. Linear Ranges and MDLs for Metal Cyanide ComplexesDetermined by Preconcentration of 5 mL									
Anion		Concentration (µg/L)							
	Level 1	Level 2	Level 3	Level 4	Level 5	r ²	MDL Standard (µg/L)	MDL* (µg/L) (n = 5)	
[Ag(CN) ₂]-	125	62.5	31.25	15.63	7.81	0.99979	15	1.92	
[Cu(CN) ₃] ²⁻	5.0	2.50	1.25	0.63	0.31	0.99893	0.8	0.74	
[Au(CN) ₂]-	100	50.0	25.0	12.5	6.25	0.99999	10	2.19	
[Ni(CN) ₄] ²⁻	100	50.0	25.0	12.5	6.25	0.99995	50	4.84	
[Fe(CN) ₆] ⁴⁻	20	10.0	5.0	2.50	1.25	0.99999	1.0	0.23	
[Co(CN) ₆] ³⁻	200	100	50.0	25.0	12.5	0.99991	10	0.85	

*MDL = (*l*) x (S) where *t* = Student's *t* value for a 99% confidence level and a standard deviation estimate with n - 1 degrees of freedom (*t* = 3.75 for five replicates of the MDL Standard), and S = standard deviation of the replicate analysis.

Determined by Direct Injection of 100 μ L								
Anion	Concentration (mg/L)							
	Level 1	Level 2	Level 3	Level 4	Level 5	r ²	MDL Standard (mg/L)	MDL* (mg/L) (n = 5)
[Ag(CN) ₂]-	100	25.0	6.25	2.5	0.50	0.99998	1	0.14
[Cu(CN) ₃] ²⁻	2.00	0.50	0.12	0.05	0.01	0.99997	0.2	0.048
[Au(CN) ₂]-	50.0	12.5	3.12	1.25	0.25	0.99997	1	0.16
[Ni(CN) ₄] ²⁻	200	50.0	12.5	5.00	1.00	0.99997	1	0.34
[Fe(CN) ₆] ⁴⁻	20.0	5.0	1.25	0.50	0.10	0.99999	0.5	0.10
[Co(CN) ₆] ³⁻	100	25.0	6.25	2.50	0.50	1.00000	1	0.064

Table 3. Linear Ranges and MDLs for Metal Cyanide Complexe

*MDL = (t) x (S) where t = Student's t value for a 99% confidence level and a standard deviation estimate with n - 1 degrees of freedom (t = 3.75 for five replicates of the MDL Standard), and S = standard deviation of the replicate analysis.

SAMPLE PREPARATION

This section gives a brief summary of the sample preparation procedure followed in the EPA OSW study. The study organizers provided extracts from various matrices. The four matrices analyzed for the high-level study (mg/L concentrations by direct injection) were labeled, "Clean Ottawa Sand Leachate", "Manufactured Gas Plant Soil Leachate" (two samples), and "Aluminum Reduction Plant Soil Leachate". The single matrix analyzed for the low-level study (µg/L concentrations by sample preconcentration) was labeled, "Clean Ottawa Sand Leachate".

The samples were extracted in accordance with SW 846 Method 9013.⁵ Briefly, up to 25 g of the solid sample are combined with 500 mL of water plus 5 mL of 50% (w/w) NaOH in a bottle and extracted by tumbling continuously for 16 h. The pH is maintained above 10 throughout the extraction step. The extract is filtered and the filtrate (leachate) stored in amber bottles at 4–6 °C until the time of analysis, which should be within 14 days.

On the day of analysis, the leachate samples were brought to room temperature and spiked with the concentrated spiking solutions provided by the study organizers. Each matrix was spiked at six levels. The six levels consisted of three pairs of closely spaced concentrations (Youden Pairs). After spiking, the samples were filtered through 0.22-µm IC syringe filters into the autosampler vials.

SYSTEM PREPARATION AND SETUP

Assemble and configure the ICS-2500 system modules. Verify that the pump flow rate is within specifications and recalibrate if necessary. Verify that the UV-Vis Absorbance Detector wavelength accuracy is within specifications and recalibrate if necessary. (Both the pump flow rate and detector wavelength accuracy can be verified by performing the Instrument OQ/PQ per Document No. 031726). To aid in troubleshooting, it is good practice to periodically record the visible lamp output (i.e., the reference cell current in nA) and elapsed time. Consult the pump or detector manuals for procedural details.

Install an IonPac ATC-3 between "Eluent Reservoir B" and the pump inlet. The ATC-3 removes metal cyanide impurities present in the sodium cyanide solution that would otherwise cause elevated background noise. Regenerate the ATC-3 as needed by using a Trap Column/Suppressor Cleanup Kit (P/N 059659) according to the *Installation and Instruction Manual* (Document No. 031835). Install a GM-4 gradient mixer and an MFC-1 column between the pump outlet and injection valve. The GM-4 reduces background noise by ensuring an adequate mixing of eluents. The MFC-1 traps metal cations that could combine with the eluent to create interferences. If reagent water blanks indicate contamination by the eluent, regenerate the MFC-1 according the *Installation and Instruction Manual* (Document No. 034990). Prepare the eluents and prime the pump, eluent lines, and trap columns with eluent.

For samples containing metal cyanide complexes above 0.2 mg/L install a 100- μ L sample loop between ports 1 and 4 of the injection valve and enter a "Sample Loop Size" of 100 μ L in the AS50 Plumbing Configuration Screen. When a sample volume of 100 μ L is entered into the sequence editor, the AS50 makes a 100- μ L fullloop injection.

For samples containing metal cyanide complexes below 0.2 mg/L, install and configure the autosampler with the AS50 dual-valve needle assembly (P/N 061267). Install a 10-mL sample syringe and enter a "Sample Syringe Volume" of 10 mL in the AS50 Plumbing Configuration Screen. Set the "Syringe Speed" to 2. (Important—setting the Syringe Speed too high may cause the inject port to leak during loading of the sample loop.)

Install a 4-mm AG11 concentrator column between ports 1 and 4 of the injection valve and enter a "Sample Loop Size" of 5 mL in the AS50 Plumbing Configuration Screen. When a sample volume of 5000 µL is entered into the sequence editor, the AS50 directly concentrates 5 mL of sample onto the AG11 concentrator column. Refer to the *AutoSelect AS50 Autosampler (USB) Operator's Manual* (Document No. 031935) for details.

Install a 2 \times 50 mm IonPac AG11 and a 2 \times 250 mm IonPac AS11 column. Rinse the column with the ending eluent composition (10:45:45) for 30 min. Equilibrate the column with the initial eluent composition (10:10:80) for 10 min before analyzing a system blank of deionized water. In an equilibrated system, the background shift during the gradient run should be less than 100 mAU. The peak-to-peak noise and drift should not exceed 5 mAU/min. There should be no significant peaks eluting within the retention time windows of the metal cyanide analyte anions.

Inject a mid-level standard. The column is equilibrated when two consecutive injections of the standard produce the same retention time (\pm 0.2 min) for the analyte anions. Confirm that the resulting chromatogram resembles the chromatogram of the standard shown in Figure 1.

Calibrate the system by injecting one blank and at least five standards for every two decades of the calibration range. Plot the peak area for each metal cyanide complex versus the concentration injected and use a linear regression to fit the data.

RESULTS AND DISCUSSION

Table 2 summarizes the calibration data obtained by preconcentrating 5 mL of standards in the μ g/L concentration range. Table 3 summarizes the calibration data obtained by directly injecting 100 μ L of standards in the mg/L concentration range. Both calibration curves are linear over about one and one-half orders of magnitude for each of the complexes. On the AS11, the copper cyanide, gold cyanide, and nickel cyanide complexes begin to coelute at higher concentrations. Samples that contain higher concentrations of these metal cyanide complexes can be diluted with 20 mM NaOH as needed to resolve these peaks. If necessary, experienced chromatographers can modify the eluent gradient program to optimize the separation for a particular analysis.

Figure 1 shows the high-level (mg/L) method detection limit (MDL) standard analyzed by directly injecting 100 μ L of the standard. Four replicates of this standard were analyzed and the resulting concentration statistics were used to calculate the MDLs given in Table 2. Figure 2 shows the low-level (μ g/L) MDL standard analyzed by preconcentrating 5 mL of the standard. Five replicates of this standard were analyzed and the resulting concentration statistics were used to calculate the MDLs given in Table 3. The MDL standards were prepared by diluting a 100× limit of detection (LOD) stock standard provided by the collaborative study organizers. In some cases, the resulting concentrations were higher than the usual recommendation of 3–5 times above the expected MDL.

Figures 3–5 are typical chromatograms obtained for several of the matrices analyzed in the EPA OSW collaborative study. Each figure displays the matrix blank along with the matrix spiked with metal cyanide complexes at concentrations approximating a mid-level standard. The samples analyzed by direct injection were spiked at mg/L concentrations whereas the samples

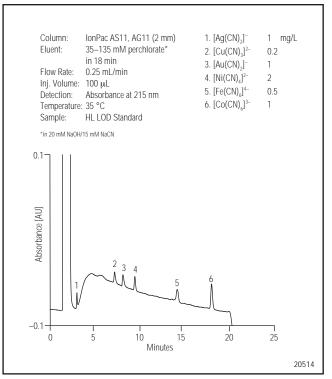


Figure 1. Anion-exchange separation with absorbance detection at 215 nm of metal cyanide complexes in reagent water. Highlevel (mg/L) limit of detection (LOD) standard analyzed by directly injecting 100 µL of the standard.

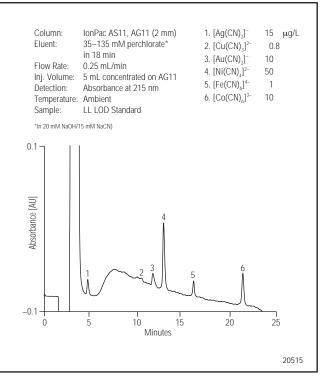


Figure 2. Anion-exchange separation with absorbance detection at 215 nm of metal cyanide complexes in reagent water. Low-level (μ g/L) LOD standard analyzed by preconcentrating 5 mL of the standard.

analyzed by preconcentration were spiked at μ g/L concentrations. A few of the chromatograms are discussed below.

Figure 3A is the chromatogram of a manufactured gas plant (MGP) soil leachate blank and Figure 3B is MGP soil leachate fortified with metal cyanides. The matrix blank shows traces (<0.04 mg/L) of copper and iron cyanide complexes. The metal cyanides spiked into this matrix were all quantitatively recovered, as summarized in Table 4 for all of the matrices evaluated.

Figure 4A is the chromatogram of an aluminum reduction plant (ARP) soil leachate blank and Figure 4B is ARP soil leachate fortified with metal cyanides. The matrix blank is free of metal cyanide complexes. The metal cyanides spiked into this matrix were all quantitatively recovered, as summarized in Table 4.

Figure 5 shows the result of an Ottawa sand leachate analyzed by preconcentrating 5 mL of sample on the AG11 column. Figure 5A is the matrix blank and 5B is the matrix spiked with µg/L concentrations of metal cyanide complexes. Although the silver cyanide complex shows evidence of additional band broadening during the preconcentration step (compared to Figure 2), the peak is well resolved from the matrix ions peak and—along with the other metal cyanide complexes quantitatively recovered from the matrix. The resolution and recovery of the silver cyanide complex should be carefully monitored in leachates from other sample types for evidence of matrix effects.

Interferences

Before analyzing a matrix that has not been previously characterized, perform an evaluation of spike recovery and precision to rule out matrix effects.

This method assumes that the alkaline conditions used will reduce iron (III) cyanide (ferricyanide) to iron (II) cyanide (ferrocyanide), resulting in a single peak for $[Fe(CN)_6]^{4-}$. Calibration is based on the iron (II) cyanide complex. To represent the sum of ferrocyanide and ferricyanide, report results as mg/L of $[Fe(CN)_6]^{3-/4-}$. If unreduced $[Fe(CN)_6]^{3-}$ is present, it will elute as a tail on the iron ferrocyanide peak.

The sodium cyanide used to prepare Eluent A may contain metal cyanide complex impurities. Reduce the level of these impurities by using 99.99% sodium cyanide to prepare the eluent, and by installing an ATC-3 anion trap column as described in this application update.

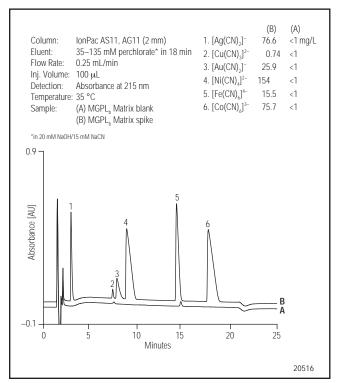


Figure 3. Anion-exchange separation with absorbance detection at 215 nm of metal cyanide complexes in a manufactured gas plant soil leachate (MGPL). (A) MGPL_b matrix blank and (B) matrix spiked with metal cyanide complexes as shown.

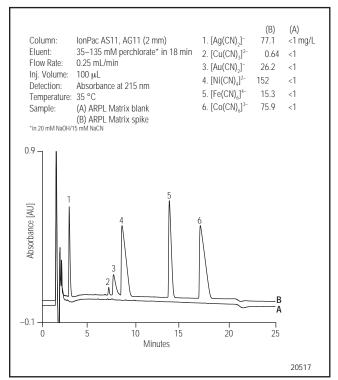


Figure 4. Anion-exchange separation with absorbance detection at 215 nm of metal cyanide complexes in an aluminum reduction plant soil leachate (ARPL). (A) ARPL soil leachate matrix blank and (B) matrix spiked with metal cyanide complexes as shown.

Precautions

Some metal cyanide complexes will decompose when exposed to light. Protect samples and standards from UV light by storing them in amber HDPE containers.

Prepare all cyanide-containing solutions within a ventilation hood. Wear gloves, avoid inhalation, and avoid skin or eye contact. Do not let acid contact any of the cyanide-containing samples, standards, or eluents. Such contact will liberate hydrogen cyanide gas, an extremely toxic substance. Dispose of the eluent waste in accordance with applicable laws.

Strongly retained compounds from injected samples can accumulate on the column and degrade its performance. Signs of a fouled column include loss of capacity, loss of resolution, shortened retention times, higher noise and background, spurious peaks, and peak tailing. The AS11 column can be flushed with up to 100% acetonitrile to help remove contaminants from the column. For more information on column troubleshooting and cleanup, see the *Installation Instructions and Troubleshooting Guide for the IonPac AS11 Analytical Column* (Document No. 034791).

Some samples contain particulates that will plug the column and increase the backpressure. Use a guard column to protect the analytical column; change the guard column if such a sample causes a sudden increase in total backpressure greater than 3000 psi.

REFERENCES

- Standard Methods for the Examination of Water and Wastewater. 17th Edition 1989; APHA-AWWA-WPCF; 4500-CN B; pp. 4–38.
- Dionex Corporation. Application Note 55; Sunnyvale, CA.
- Dionex Corporation. Application Update 147; Sunnyvale, CA.
- Dionex Corporation. Application Note 161; Sunnyvale, CA.
- Cyanide Extraction Procedure for Solids and Soils: Test Methods for Evaluating Solid Wastes SW-846, Third Edition; EPA Method 9013; United States Environmental Protection Agency, Washington, DC; 1986.
- EPA Inter-Laboratory Collaborative for Office of Solid Waste, Sharon Drop, Task Group Chairperson.

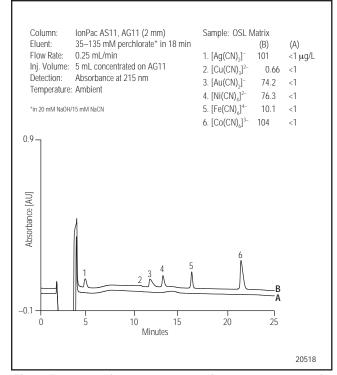


Figure 5. Anion-exchange separation with preconcentration and absorbance detection at 215 nm of metal cyanide complexes in an Ottawa sand (OS) leachate. (A) OS leachate matrix blank and (B) matrix spiked with metal cyanide complexes as shown.

Anion	% Recovery						
	Ottawa Sand 21–90 mg/L	Manufactured Gas Plant Soil _a 21–90 mg/L	Manufactured Gas Plant Soil₅ Plant Soil 21–90 mg/L	Aluminum Reduction Plant Soil 21–90 mg/L	Ottawa Sand 20–115 μg/L		
[Ag(CN) ₂]-	106.5	104	102.9	102.2	100.9		
[Cu(CN) ₃] ²⁻	108	106.1	128	112.6	109.9		
[Au(CN) ₂]-	106.1	102.8	103.7	102.3	100		
[Ni(CN) ₄] ²⁻	103.2	102.5	101.8	100.2	91.1		
[Fe(CN) ₆] ⁴⁻	102.2	101.5	103.8	99.5	102.7		
[Co(CN) ₆] ³⁻	104.6	103.3	102.5	101.7	103.3		

Table 4. Recovery of Metal Cyanide Complexes from Solid Waste Leachate

LIST OF SUPPLIERS

- Alfa Aesar, 30 Bond St., Ward Hill, MA 01835 USA, Tel: 800-343-0660, www.alfa.com.
- Fisher Scientific, 2000 Park Lane, Pittsburgh, PA 15275-1126 USA, Tel: 800-766-7000, www.fishersci.com.
- Sigma-Aldrich Chemical Company, P.O. Box 14508, St. Louis, MO 63178 USA, Tel: 1-800-325-3010, www.sigmaaldrich.com.
- Strem Chemical, 7 Mulliken Way, Newburyport, MA 01950-4098 USA,

Tel: 1-800-647-8736, www.strem.com.

VWR International, 1310 Goshen Parkway, West Chester, PA 19380 USA, Tel: 800-932-5000, www.vwr.com.







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