

Docket ID No. OW-2003-0070

Comments Related to the Proposed Cyanide Determination Methods

**Federal Register / Vol. 69, No. 66 / April 6, 2004 / Proposed Rules
pp. 18166 – 18226**

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Distillation-based methods for determination of operationally defined cyanide have very limited application because of the serious interferences from the species likely to be found in most of the cyanide containing effluents. To the best of our knowledge, there are no reliable spot tests for detecting the presence of some of these interfering species.¹ Hence, distillation-based methods should only be used for cyanide determination of samples that have well defined and constant matrix that do not contain interfering species. For example, distillation-based methods should not be used for cyanide determination of samples originating from precious metals cyanidation effluents, water treatment plants, petroleum refineries, coke producing plants, elemental phosphorous producing facilities, etc. In addition, these methods should not be used to monitor cyanide detoxification processes because cyanide and oxidant(s) (sulfite, thiosulfate, peroxide, etc.) can coexist in solution. The high temperature during distillation shifts the equilibrium towards the formation of the oxidation products (OCN⁻ and/or SCN⁻, etc.), which can result in serious underestimation of cyanide levels that are actually present in the original sample.

As several authors have demonstrated [1-10], distillation-based methods often achieve incomplete, species dependent cyanide recoveries and suffer from serious interferences. Depending on the relative concentrations of interferents and other matrix constituents, underestimation (ultimately *Type II errors* – probability of not detecting a constituent when it is actually present) or overestimation (ultimately *Type I errors* - probability of deciding a constituent is present when it is actually absent) of the cyanide levels present in a given sample may occur, causing in the first case false security and in the second case, excessive cyanide treatment costs.

The interference problems associated with the distillation-based total cyanide determination stem from the fact that the method utilizes extremely harsh

¹ If the spot test methods do exist, they are often not sensitive enough (e.g. tests for sulfide and oxidants).

analytical conditions ($t > 100\text{ }^{\circ}\text{C}$ and $\text{pH} < 0$). In addition, the actual chromogenic reaction in the spectrophotometric quantitation procedure involves the addition of a strong oxidant (chloramine-T). These conditions along with the complexity of the sample matrices, often produce complicated reaction pathways that can result in the production of cyanide (real or as an artifact) during distillation and/or associated colorimetric quantitation procedures. However, the consequences of the partial or complete destruction of cyanide during the distillation process are even more worrisome. In other words, it is possible that a water source may be deemed safe because the distillation-based analyses found no cyanide present even though high levels of cyanide are present in solution.

Some examples of both negative and positive biases of the distillation-based methods for cyanide determination follow.

Table I (adapted from references [8, 9]) shows concentration of cyanide found in NaCN standards containing interfering species.

TABLE I. *Concentration of CN^- in the Samples Containing 0.200 mg/L CN^- (added as NaCN) and 20.0 or 200 mg/L of Interferent Obtained with Method 335.4.*

Species	Ratio ^b	CN^- found (mg/L) ^a	Ratio ^b	CN^- found (mg/L) ^a
SO_3^{2-}	32	0.080 (10.0)	320	0
SCN^-	45	0.174 (8.0)	450	0.054 (1.0)
$\text{S}_2\text{O}_3^{2-}$	23	0.124 (4.3)	230	0.040 (3.2)

^a % Relative standard deviations ($n = 3$) are given in parentheses. ^b Molar concentration ratio: species/ CN^- .

For comparison purposes, the cyanide recoveries in the presence of the same sulfur-containing species by the flow injection mild UV digestion amperometric method (FI/UV) described earlier [8]² are shown in Table II.

² This method has significant advantages over the distillation-based methods for determination of total cyanide. The results of the round-robin testing in which eight laboratories analyzed nine samples each were very good. Samples in this study were obtained from diverse sources such as: 1, drum handling facility (filter effluent); 2, POTW (secondary effluent); 3, petroleum refinery (secondary effluent); 4, coke plant (secondary effluent); 5, rolling mill (filter effluent); 6, metals forming plant (primary effluent); 7, die casting plant (secondary effluent); 8, precious metal operation (reclaim water); 9, water treatment plant (filter effluent). The method was submitted to the EPA but was never approved.

TABLE II. Concentration of CN^- in the Samples Containing 0.200 mg/L CN^- (added as NaCN) and 20.0 or 200 mg/L of Interferent Obtained with FI/UV method.

Species	Ratio ^b	CN^- found (mg/L) ^a	Ratio ^b	CN^- found (mg/L) ^a
SO_3^{2-}	32	0.199 (0.5)	320	0.175 (1.3)
SCN^-	45	0.208 (1.4)	450	0.211 (2.4)
$\text{S}_2\text{O}_3^{2-}$	23	0.196 (1.1)	230	0.218 (2.9)

^a % Relative standard deviations (n = 3) are given in parentheses. ^b Molar concentration ratio: species/ CN^- .

One of the primary problems with the standard methods for total cyanide determination is the interferences for which there are no reliable recognition tests ("spot tests"). That is the case, for example, with the SCN^- , SO_3^{2-} and $\text{S}_2\text{O}_3^{2-}$ ions. These are the species that are often found in the cyanide containing samples.³ From the results obtained (Table I), it is obvious that the sulfuric acid distillation procedure creates conditions that may result in the reaction of cyanide with either the reaction products of these species with acid or with the species themselves.

In the case of sulfite, recoveries of cyanide with the distillation procedure from 0.200 mg/L CN^- solutions containing 20.0 and 200 mg/L SO_3^{2-} are 40 and 0%, respectively. The concentrations of CN^- in the absorbing solutions found after distillation were determined with colorimetric as well as with amperometric detection methods. Since comparable results were obtained, it is clear that the low recoveries are caused by the oxidation of cyanide with sulfite and not because of the reaction of SO_3^{2-} with chloramine-T in the colorimetric quantitation step. On the other hand, corresponding recoveries obtained with the FI/UV method are 100 and 88%. Low recoveries of cyanide in the presence of sulfite obtained with the distillation procedure are probably due to the temperature enhanced sulfite oxidation of cyanide to cyanate.

As may be seen from Table I, incomplete cyanide recoveries were also obtained in the presence of thiocyanate with the Method 335.4. It has been established previously that decomposition of thiocyanate at elevated temperatures in the acidic solutions and in the absence of oxidants produces elemental sulfur, sulfur(IV) oxide, as well as carbonyl sulfide which is converted to sulfide upon adsorption in alkaline solution [1, 11]. Under similar conditions the thiosulfate ion may be converted to sulfur, sulfur(IV) oxide, hydrogen sulfide and polysulfide [12]. The high temperature distillation of cyanide containing samples in the

³ Sulfite and thiosulfate are used for cyanide detoxification and under certain conditions these species can coexist with cyanide in solution.

presence of thiocyanate and/or thiosulfate creates conditions conducive to the conversion of cyanide to other species, which in turn causes a decrease in cyanide recoveries. Hence, it is not surprising that the recoveries of cyanide in the presence of the 20.0 and 200 mg/L SCN^- obtained by the Method 335.4 are 87% and 27%, respectively. The corresponding recoveries in the presence of $\text{S}_2\text{O}_3^{2-}$ are 62% and 20%. In contrast, complete cyanide recoveries in the presence of the SCN^- and $\text{S}_2\text{O}_3^{2-}$ are obtained with the FI/UV method [8] (for details please see Tables I and II). It should be noted here that the standard distillation Method 4500-CN [13] recommends the addition of lead carbonate to the NaOH absorption solution prior to the distillation of the cyanide samples that may also contain various sulfur species. Our experiments [6], as well as the results obtained previously [3] show that the treatment is not effective in removing SO_3^{2-} , SCN^- and $\text{S}_2\text{O}_3^{2-}$ interferences, and may in fact be problematic. It is obvious that utilization of the distillation-based methods for determination of cyanide often produces underestimation of cyanide levels, **which may produce a dangerous sense of false security with a serious safety and health ramifications.**

It is interesting to note that the SCN^- ion may also cause a positive interference in the determination of cyanide under the distillation protocols. At high acidity and in the presence of strong oxidants (for example, NO_3^-), the sulfur in the thiocyanate is oxidized with concomitant liberation of hydrogen cyanide [2]. Under the experimental conditions that exist during the FI/UV determination of cyanide, no production of HCN from SCN^- in the presence of nitrate occurs. For example, cyanide recovery from the sample containing 0.200 mg/L CN^- , 500 mg/L NO_3^- and 100 mg/L SCN^- was 102% [8]. The corresponding recovery with the distillation procedure was over 5000%.

Research by Carr et al. shows that various carbon sources produce cyanide during the low pH distillation processes [7]. For example, ~14 ppm of CN^- was produced when solution containing 5 mmol/L of *i*-propanol was distilled in the presence of sodium nitrate. Corresponding experiment in the presence of sodium nitrite produced ~120 ppm of cyanide. It is obvious that positive bias in the determination of cyanide may cause false alarms and unnecessarily high cyanide detoxification costs.

QuickChem® Method 10-204-00-1-X

DIGESTION AND DISTILLATION OF TOTAL CYANIDE IN DRINKING AND WASTEWATERS USING MICRO DIST AND DETERMINATION OF CYANIDE BY FLOW INJECTION ANALYSIS

Validation Study Report for Tier 3 for Modification of Part 136 Reference Method 335.2 and Part 141 Reference Method 335.4

[ATP Case nos. N98-0010 and D98-0005]

Unfortunately, in neither of the above named documents were we able to find how the method performs in the presence of potential interferences. Because the method utilizes 30 min distillation vs. 1 hour for the currently approved method, it is possible that interferences described earlier would be less pronounced.

The 1st Lachat document does state the following on page 3:

Thiocyanate will interfere if present. This method should not be used if sample thiocyanate concentrations exceed 0.002 mg/L. Instead, a method based on weak acid dissociable, or ligand-exchange digestion should be used.

Thiocyanate is often present in cyanide containing effluents. To the best of our knowledge there is no test sensitive enough to detect 2 ppb of SCN⁻. As was pointed out earlier in this text, depending on the sample matrix, thiocyanate can be positive or negative interference. Hence, in most instances the cyanide values determined with this method would not reflect the true cyanide value in the effluent sampled. It could be confusing that if thiocyanate is present, Lachat suggests that WAD method should be used instead. WAD and total cyanide methods are different in that they do not recover cyanide from the same species. In addition, WAD method was shown to have concentration dependent cyanide recoveries from the two Hg-cyano species [5].

This document also states the following:

Oxidizing agents, such as residual chlorine, decompose most of the cyanides. Test a drop of the sample with potassium iodide (KI)-starch paper at time of collection; a blue color indicates the need for treatment. Add ascorbic acid, a few crystals (about 0.6 g each) at a time, until a drop of sample produces no color in the indicator paper; then add an additional 0.06 g of ascorbic acid for each liter of sample volume. Sodium arsenite has also been employed to remove oxidizing agents.

Ascorbic acid should not be used for reducing oxidants if the sample is not going to be analyzed immediately upon sampling. Our investigations and other researchers have shown that ascorbic acid reduces cyanide levels as a function of storage time (probably due to the formation of cyanohydrins).

Next table summarizes some of the data that we obtained.

Sample ^a	Red/CN ⁻ ^b	CN ⁻ concentration (ppm)			
		0 h	24 h	48 h	72 h
0.200 ppm CN ⁻ + 0.010 g Sodium Arsenite	100	0.197	0.197	0.197	0.198
0.200 ppm CN ⁻ + 0.025 g Sodium Arsenite	250	0.198	0.196	0.198	0.196
0.200 ppm CN ⁻ + 0.050 g Sodium Arsenite	500	0.198	0.191	0.198	0.198
0.200 ppm CN ⁻ + 0.010 g Ascorbic Acid	74	0.200	0.181	0.176	0.173
0.200 ppm CN ⁻ + 0.025 g Ascorbic Acid	185	0.197	0.182	0.168	0.139
0.200 ppm CN ⁻ + 0.050 g Ascorbic Acid	370	0.196	0.172	0.141	0.063
2.00 ppm CN ⁻ + 0.010 g Sodium Arsenite	10	2.00	2.00	2.00	1.98
2.00 ppm CN ⁻ + 0.025 g Sodium Arsenite	25	2.01	2.02	2.01	1.96
2.00 ppm CN ⁻ + 0.050 g Sodium Arsenite	50	2.01	1.99	2.02	1.98
2.00 ppm CN ⁻ + 0.010 g Ascorbic Acid	7.4	1.98	1.96	1.93	1.89
2.00 ppm CN ⁻ + 0.025 g Ascorbic Acid	18.5	1.98	1.92	1.83	1.77
2.00 ppm CN ⁻ + 0.050 g Ascorbic Acid	37	1.99	1.88	1.74	1.54

^a samples were kept in a refrigerator at 4 °C;

sample volume 100 ml

^b molar ratio

In this study cyanide was determined with EPA Method OIA-1677. It is obvious that even large excess of arsenite does not decrease cyanide levels as a function of sample storage time, while ascorbic acid may be the cause of significant negative bias in cyanide determination.

Minor Comment:

On page 3 of the Lachat document there is a following statement:

Most non-volatile interferences are eliminated or minimized by the distillation procedure. Some of the known interferences are aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, thiosulfate, and sulfide.

Sulfide is not an oxidant.

Cyanide by UV-digestion/Flash Distillation/Colorimetry

Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821-B-01-009 (Kelada-01)

Why has EPA decided to consider the method that recovers cyanide from thiocyanate ion? One of the mechanisms that our body deals with cyanide induced poisoning is to convert CN⁻ to SCN⁻ ion. For example, 3-mercapto pyruvate sulfurtransferase, thiosulfate reductase, rhodanase and cystathionase are enzymes that convert cyanide to significantly less toxic thiocyanate [16]. One of the therapeutic antagonists for cyanide poisoning is the thiosulfate ion,

which also converts cyanide to thiocyanate. In addition, thiocyanate ion is the product of certain cyanide detoxification methods. If one wanted to include all the potential cyanogenic compounds in “total cyanide” method(s), the list would be very long and the analytical method(s) would be very difficult to develop. In addition, because of the toxicity differences between cyanide and other species (e.g. thiocyanate), in our opinion this would not be appropriate.

The following text is in the introduction of the Method Kelada-01:

Kelada-01 provides for rapid determination of total cyanide, acid dissociable cyanide, and thiocyanate in drinking waters, wastewater, ambient water, industrial wastes, and sludge, with minimal effects from interferences.

We were not able to find the information related to “minimal effects from interferences.” Has the author compared the recovery of cyanide obtained with the novel method for total cyanide determination and EPA Method 335.4 in the presence of potential interferences (e.g. sulfite, thiosulfate, thiocyanate, hydrogen peroxide, etc.)? This information would be very important because the method utilized a relatively short distillation step; hence, it is possible that both positive and negative interferences would be attenuated compared to Method 335.4. However, the quantification of these interferences is very important to make sure that there is no significant positive or negative bias in cyanide determination using the Kelada method(s).

On page 16 there is a following statement:

8.7.2 Acid Mixture for Acid Dissociable Cyanide – As directed in 8.7.1 ... or use Weak Acidic Acetate Buffer.

Earlier in the text (p. 2) it was stated that Acid Dissociable Cyanide recovers cyanide from $\text{Hg}(\text{CN})_2$ species. I could find no recovery data in the text. The Kelada method, if acetate buffer is used, is performed under conditions similar to the WAD distillation method except for the shorter distillation time. Our results [5] show that “classical” WAD procedure recovers less than 40 % of cyanide at the MCL and only about 20 % at 2.00 ppm CN^- added as mercury(II) cyanide. It is obvious that the recovery is concentration dependent; hence, the cyanide results may vary as a function of sample dilution prior to analyses.

Points Raised in the Federal Register Text

Page 18176:

Recoveries of potassium nickel cyanide and mercury (II) cyanide, the two strongest available cyanide complexes, ranged from 89.9 to 99.6% and 82.9 to 99.3%, respectively (in samples fortified to 100 µg/L as CN^-).

Method D6888–03 states that either nickel cyanide or mercury (II) cyanide may be used to prepare quality control samples. However, for the purposes of NPDES compliance

monitoring, EPA is proposing that only mercury (II) cyanide be used. Mercury (II) cyanide is a stronger complex than nickel cyanide (as evidenced by the slightly lower recoveries cited above), and, therefore, provides the most rigorous test for method performance.

The recoveries of cyanide from tetracyanonickelate(II) and $\text{Hg}(\text{CN})_2$ species depend on the analytical method utilized. For example, at 2.00 ppm CN^- (added as mercury(II) cyanide) WAD distillation and CATC methods recover 20.7 and 85.4 %, respectively. On the other hand, corresponding recoveries at the same cyanide level from tetracyanonickelate(II) complex are 97.4 and 72.0 %. Release of cyanide from the coordination sphere of the metal is influence by both thermodynamic and kinetic stabilities. For the methods that utilize ligand-exchange reactions, one also has to consider the affinity of a particular ligand for a given metal ion. Hence, we suggest that for “available” cyanide methods both Ni and Hg-cyano species be used to determine method's performance.

Page 18176:

First, EPA has received information suggesting that sulfide at levels below those detected with the lead acetate paper may produce false positive signals on the amperometric detection systems used in D6888-03 and OIA-1677 (see Zheng *et al.* “Evaluation and Testing of Analytical Methods for Cyanide Species in Municipal and Industrial and Contaminated Waters,” Environ. Sci. Technol. 2003, 37, 107–115). Lead acetate paper generally recommended for screening for the presence of sulfide interferences in cyanide methods, but the paper will not detect sulfides below approximately 5 ppm. For this reason, analysts suspecting a sulfide interference should test their sample with a more sensitive sulfide procedure and treat the sample accordingly.

In 2001 [9] we have described a modified FI/LE (OIA-1677) method for on-line abatement of the sulfide interferent that utilizes $\text{H}_2\text{SO}_4/\text{Bi}(\text{NO}_3)_3$ reagent solution.

In order to test the effectiveness of sulfide attenuation using a system described, a series of sulfide standards were analyzed sequentially. The data obtained are summarized in the table below.

Apparent cyanide levels found in S^{2-} standards

Sample	Apparent CN^- Found (mg/L)
10.0 ppm S^{2-}	0.001
20.0 ppm S^{2-}	0.003
30.0 ppm S^{2-}	0.003
50.0 ppm S^{2-}	0.005

As may be seen, the $\text{H}_2\text{SO}_4/\text{Bi}(\text{NO}_3)_3$ system is very effective in suppressing the signal due to the sulfide ion. The selectivity factor of $\sim 10,000$ was obtained (it should be noted that in the absence of Bi^{3+} ion in the reagent stream, the signal due to the presence of 10 ppm S^{2-} would be off-scale due to detector saturation).

Cyanide recoveries in the presence of various amounts of sulfide are illustrated in the table below. As may be seen from the data presented in this table,

excellent cyanide recoveries were obtained even in the presence of 50.0 ppm S^{2-} . In addition, the values obtained for pure cyanide standard sandwiched between CN^-/S^{2-} mixtures gave excellent recoveries. This is important, since it was established previously that S^{2-} ion, due to the formation of Ag_2S , changes the surface of the silver-working electrode. The altered silver surface acts electro-catalytically in that the subsequent increase in current is observed for a given cyanide standard. Since no increase in CN^- values was observed, it is obvious that the sulfide on-line attenuation system functions very well.

Cyanide levels found in the CN^-/S^{2-} mixtures

Sample	CN^- Found (mg/L) ^a
0.100 ppm CN^-	0.105 (0.5)
0.100 ppm CN^- + 0.100 ppm S^{2-}	0.101 (0.8)
0.100 ppm CN^-	0.104 (0.8)
0.100 ppm CN^- + 1.00 ppm S^{2-}	0.101 (1.2)
0.100 ppm CN^-	0.105 (0.5)
0.100 ppm CN^- + 10.0 ppm S^{2-}	0.102 (0.5)
0.100 ppm CN^-	0.105 (1.0)
0.100 ppm CN^- + 30.0 ppm S^{2-}	0.104 (1.0)
0.100 ppm CN^-	0.105 (1.0)
0.100 ppm CN^- + 50.0 ppm S^{2-}	0.103 (0.9)
0.100 ppm CN^-	0.104 (1.6)

^a Calibration was performed with the following CN^- standards: 0.000, 0.050, 0.100 and 0.200 ppm; %-Relative standard deviations (n=3) are given in the parentheses.

We will supply further details of this modification of the Method OIA-1677 to anyone interested.

Total cyanide Draft Method OIA-1678 described earlier [8, 15] already has an on-line sulfide abatement procedure incorporated into the method.

Pages 18176 – 18177:

Second, EPA's National Enforcement Investigation Center (NEIC) laboratory has found that when samples that contain significant solids are analyzed by OIA-1677, particles in the sample can settle out in the sample apparatus and also can clog the flow-injection system. As a result, measurements of cyanide in samples containing particulates decrease as the samples are allowed to stand in the sample tubes, and decrease as the system clogs. This decrease could be due to a number of factors, such as adsorption of released cyanide onto particulate or re-complexation of released cyanide with metals. This settling and clogging may be a problem in other similar cyanide-measurement systems that contain a rack of sample tubes, because the particles can settle during the time that the samples sit in these tubes. Suggested solutions to the problem are to (1) limit methods that use a rack of sample tubes to measurement of dissolved cyanide only; *i.e.*, samples that have been filtered through a 0.45-micron filter to remove particles, (2) to limit these methods to analysis of one sample at a time so that the settling cannot occur, (3)

to limit the time between addition of the ligand-exchange reagents and the time of analysis to preclude settling, or (4) to require sample agitation during storage in the sample rack.

Method OIA-1677 was developed and only tested for analyses of dissolved cyanide. While the method can probably tolerate small particles, the performance of the method (spike recovery and interferences) should be evaluated under these conditions. We agree that the time between addition of the ligand-exchange reagents and the time of analysis should be limited to less than 30 minutes to preclude settling in some samples. In preparation for the manufacturing and testing of the automated process cyanide analyzer based on OIA-1677 technology, we developed a method for the automated on-line addition of the ligand-exchange reagents, which limits the time between addition of the reagents and cyanide determination step to less than two minutes. Upon request, we will provide the instruction on how to modify the method to incorporate the on-line addition of the ligand-exchange reagents.

CONCLUSION

We strongly believe that methods proposed for determination of the operationally defined cyanide should include complete concentration and species dependent studies, as well as interference investigations similar to those performed in the development of Method OIA-1677 and USEPA Draft method OIA-1678 [5, 8, 14, 15].

If this is not carried out, depending on the relative concentrations of interferents and other matrix constituents, underestimation (ultimately *Type II errors* – probability of not detecting a constituent when it is actually present) or overestimation (ultimately *Type I errors* - probability of deciding a constituent is present when it is actually absent) of the cyanide levels present in a given sample may occur, causing in the first case false security and in the second case, excessive cyanide treatment costs. Of course, cyanide underestimation is more worrisome because **it may produce a dangerous sense of false security with a serious safety and health ramifications.**

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