Description

Technical Field.

This invention relates generally to a method and apparatus for measuring the amount of cyanide in a sample, and it more particularly relates to such a method and apparatus having improved cyanide separation capabilities.

Background Art.

The toxic compounds called cyanides threaten our health and environment. Harmful to man, and even more so to aquatic life, these compounds are produced and introduced into our environment in many damaging ways, so that it is important to understand and restrict this form of environmental pollution. Measuring the cyanides in a selected sample is one important step in the process of improving the environment.

Of the various existing types and kinds of analytical methology and related apparatus employed to measure cyanides, automated systems have acheived notable results. For example, refer to U.S. patent 4,265,857, and to a publication entitled "Chemistry Of Wastewater Technology" published by Ann Arbor Science Publishers, Inc. of Ann Arbor Michigan, Library of Congress Catalog Card No. 76-50991, ISBN 0 250 40185-1.

As described in Chapter 20 of "Chemistry Of Wastewater Technology," such an automated system involves the steps of separation, absorption, and measurement, with ultraviolet irradiation being employed to dissociate cyanides in the process of separation, along with thin-film distillation and chemical absorption techniques.

A typical ultraviolet radiation unit includes an ultraviolet lamp, surrounded by a quartz coil, through which is passed an acidic sample to be tested. The quartz coil passes all of the several energy levels, or spectral lines, of ultraviolet radiation produced by the ultraviolet lamp, of wave lengths between approximately 150 millimicrons and 400 millimicrons. This spectrum of ultraviolet radiation causes the break down of all cyanide complexes, including the strong iron and cobalt cyanide complexes. The cobalt complex is dissociated by the ultraviolet radiation both off approximately 255 millimicrons, and of approximately 310 millimicrons. In this manner, the cyanides are dissociated for subsequent measurement.

The thin-film distillation separates the resulting hydrogen cyanide gas, and the HCN gas is then absorbed in a sodium hydroxide solution for subsequent colorimetric measuring. Thus, all of the cyanides can be detected and measured in a large number of different samples in a continuous, automated manner.

The quartz coil in the conventional irradiation unit of the system is permeable to all, or at least substantially all, of the ultraviolet spectrum, thereby enabling the ultraviolet radiation to break down all of the cyanide complexes in the acidic sample. However, thiocyanate (SCN) is also dissociated, and therefore, is detected along with the cyanides, thereby causing somewhat inaccurate higher cyanide measurements. Thus, dissociation of the thiocyanate is unwanted and undesirable for most applications. Consequently, while the present cyanide detection measurement system is satisfactory for most applications, it would be highly desirable to have a cyanide measurement system, which would dissociate all the cyanide complexes, including the strong cobalt complex, without dissociating thiocyanate.

Disclosure of Invention

Therefore, the principal object of the present invention is to provide a new and improved measurement method and apparatus for indicating the amount of cyanide in a sample, without detecting or indicating the presence of thiocyanate, in a convenient and precise manner.

It is a further object of the present invention to provide such a new and improved method and apparatus, which enables dissociation of strong cyanide complexes, including cobalt and iron complexes, without the dissociation of thiocyanate.

Yet another object of the present invention is to provide such an improved method and apparatus, which requires only simple modifications of existing systems.

Briefly, the above and further objects of the present invention are realised by providing a new and improved system for measuring cyanides in liquid samples.

According to the invention a system for measuring cyanide in an alkaline sample has an ultraviolet irradiation unit for producing ultraviolet radiation to dissociate cyanides contained in the alkaline sample, and a thin-film distillation unit for separating cyanide from the irradiated sample to produce recovered cyanide for measurement purposes, characterised in that a clear glass filter is interposed between the irradiation unit and the sample for passing ultraviolet radiation having a wavelength between about 290 millimicrons and about 400 millimicrons to produce an irradiated sample in which cyanides have been dissociated, while blocking high frequency ultraviolet radiation, to inhibit thiocyanate dissociation, and a measuring device for receiving the recovered cyanide and generating a recordable signal indicative of recovered cyanide quantity.

Thus, the method and apparatus of this invention alleviates the above-mentioned drawbacks of existing techniques. The filter blocks ultraviolet radiation in the range of approximately 150 millimicron to approximately 290 millimicrons, to inhibit thiocyanate dissociation, while passing the remaining longer ultraviolet wavelengths.

The pH of the sample is raised to enable the strong cobalt cyanide complexes to be completely broken up as well. In this regard, in prior known systems, the samples are acidic, and are irradiated with non-filtered quartz irradiation units to provide a complete dissociation of the cyanide complexes, as well as the release of the unwanted thiocyanate. However, it has been discovered that an alkaline sample combined with the filtered irradiation, achieves total break down of cyanide complexes, without dissociating thiocynate. In this manner, more convenient cyanide measurements are provided, with only a simple modification of existing systems.

Brief Description of Drawings

The above and other objects and features of this invention and the manner of attaining them will become apparent, and the invention itself will be best understood, by reference to the following description of an embodiment of the invention taken in conjunction with the accompanying drawings, wherein:

FIGURE 1 is a partially schematic, block diagram of a cyanide measuring system constructed according to the present invention;

FIGURE 2 is schematic diagram of an ultraviolet irradiation unit of the system of Fig. 1; and

FIGURE 3 is a block diagram of a two-channel system for detecting and measuring thiocyanate in samples, according to the present invention.

Best Mode for Carrying Out the Invention

Referring now to the drawings, and more particularly to Fig. 1, there is shown a cyanide measuring system 10, which is constructed according to the present invention, and which is used to measure the different amounts of different cyanides, contained in a series of different water samples admitted seriatim to the system 10 at an inlet 12B. The system 10 generally comprises a separation section including a photoillumination irradiation unit 11A for causing the break down of cyanide complexes in the water samples being pumped through it. A thin-film distillation unit 11B separates cyanide from the irradiated samples to produce recovered cyanide for measuring purposes. An absorption section 11C provides for the sodium hydroxide absorption of the hydrogen cyanide gas evolved from the samples. A measurement section 11D automatically measures the cyanide in the samples from the section 11C.

A segmented flow pump 12 transfers the samples and reagents to the system 10 at inputs 12A-12N and 12P to outputs, 13, 15-21. The samples (usually made alkaline for preservation) are mixed with a suitable quantity of nitrogen by admitting them to an inlet 13 to a sample mixing coil 25, to provide the vehicle for the segmented flow of the series of water samples to be tested. In the system 10, air can be used in place of nitrogen.

The photoillumination unit 11A is in the form of a filtered ultraviolet irradiation unit 30, which irradiates the series of segmented samples flowing through it from the coil 25, to dissociate the cyanide complexes contained in the samples. The unit 30 emits light in only a limited portion of the ultraviolet spectrum, namely, between approximately 290 millimicrons and approximately 400 millimicrons wavelengths of ultraviolet radiation, for dissociating the cyanides from their cyanide complexes, without dissociating thiocyanate.

The irradiated samples flowing continuously from the outlet of the irradiation unit 30 are mixed with a suitable acid mixture via a line 15, as well as air under pressure received via a line 16. The continuous thin-film evaporation unit 11B receives the aerated acidified samples for separating the cyanides from the samples. The evaporation unit 11B is disclosed more fully in the foregoing mentioned U.S. patent. The thin-film evaporation unit 11B causes hydrogen cyanide gas to be released, together with some water, from the acidified samples in a continuous segmented flow basis. The water is condensed back, and the hydrogen cyanide gas is absorbed via the absorption section 11C.

The segmented samples flowing from the outlet of the thin-film evaporation unit are mixed with sodium hydroxide from a line 17 in the section 11C, which is an absorption coil 50. In this regard, the hydrogen cyanide gas is forced with air through the coil, simultaneously with sodium hydroxide, as more fully described in the foregoing mentioned publication, to recover the hydrogen cyanides in each sample.

The outlet of the absorption coil 50 is pumped back through the pump 12 via a return line 18, to be mixed with a suitable buffer and segmented by air, to a sample mixing coil 55.

The outlet of the sample mixing coil 55 is mixed with chloramine-T from a line 19, in a mixing coil 57, where it is mixed with a color reagent from a line 20 from the pump 12.

The outlet of the mixing coil 57 is connected through a mixing coil 60 to a colorimeter 65 and discharged through a line 66, with the pump 12

driving the system and exhausting it from the line 21.

The colorimeter provides a recordable signal indicative of the recovered cyanide quantities to an input of a recorder 70, connected to a printer 75.

Thus, the method and system of this invention provides dissociation of all cyanide complexes, including cobalt complexes, without thiocyanate interference. It achieves this by employing an alkaline sample with a new and improved ultraviolet irradiation unit, which filters selected ultraviolet energy levels to inhibit the thiocyanate break down.

It is a feature of the present invention to acidify the samples with the acid mixture via line 15, after the irradiation, rather than prior to it, for the purpose of causing the stronger cyanide complexes, such as the cobalt complexes, to break down, without the break down of thiocyanate.

Considering now the filtered ultraviolet irradiation unit 30 with reference to Fig. 2 unit 30. An opague metal housing 31 confines therein a vertically disposed ultraviolet lamp 32 for emitting a full spectrum of ultraviolet radiation. A clear glass tube 33 surrounds the lamp 32 and is supported vertically by first and second rings 34 and 35. The rings 34 and 35 are supported respectively, in turn, by a plurality of support rods 36 and a support screen 37.

A clear glass coil 38 is supported by a plurality of upright support rods 39 and surrounds the ultraviolet lamp 32. In this position, the segmented samples flowing through the glass coil 38 are irradiated by ultraviolet radiation, a fan 40 cools the unit 30.

The glass tube 33 is clear glass and serves as a filter to pass ultraviolet wavelengths of between about 290 millimicrons and about 400 millimicrons, and to block the shorter ultraviolet wavelength. The result is that all of the cyanide complexes including iron complexes are dissociated, except the strong cobalt complex is only partially dissociated. By using the alkaline samples, instead of acidified ones, for irradiation, even the cobalt complex breaks down.

In operation, samples are passed through the glass tube 38 to produce the irradiated samples in which cyanides have been dissociated, without dissociating thiocyanate.

Figure 3 is a block diagram of a two-channel system 100 for detecting thiocyanate in the samples. In one channel, the glass system 10 is employed to provide output signals indicative of the complete cyanides. The second channel includes a conventional quartz system 110, as disclosed in the foregoing mentioned publication, to provide an indication of the complete cyanides as well as thiocyanates from the same samples.

Samples are communicated through line 101, and reagents are communicated through line 102, and are coupled to inputs 111 and 112 of quartz UV cyanide system 110 and glass UV cyanide system 10. System 110 is similar to system 10, and employs a conventional quartz ultraviolet irradiation unit (not shown), in place of the filtered ultraviolet irradiation unit 30, to provide a full spectrum of ultraviolet radiation, thereby dissociating all of the cyanides and thiocyanates in each sample.

The output of system 110 is an electrical signal 113 indicative of all of the dissociated cyanide and thiocyanate for each sample. The output of system 10 is an electrical signal 114 indicative of the dissociated cyanides without any thiocyanate (SCN). These two output signals may be produced by the output of a colorimeter, such as a colorimeter 65 in Fig. 1. Other conventional techniques may also be employed for generating the signals. The signals are coupled to a suitable conventional subtraction circuit 130, which produces an electrical output signal 131 indicative of the difference between the input signals 113 and 114. Thus, the output signal 131 indicates the amount of thiocyanate (SCN), in the samples under test.

Thus, the method and apparatus of this invention provides convenient measurements of complete cyanides and also of thiocyanate contained in the samples, if desired.

The quartz system 110 differs from the system 10, only by the fact that the glass tube 33 is replaced with a quartz tube (not shown), and that the acid mixture is added with the nitrogen to the samples prior to irradiation. Thus, only minor differences exist between the two systems.

Therefore, the signal 131 is the difference between the two measurements made by the two channels, and provides an indication of the thiocyanate contained in each sample under test.

An advantage of the two-channel technique for measuring thiocyanate, is that color and turbidity of the samples, does not interfere with the measurements. Conventional direct colormetric measurements of thiocyanate are subjected to interferences from color and turbidity, when present in the samples. With the automated system of this invention, total cyanide, which completely includes all the strong cyanide complexes (even the strong cobalt complex), is conveniently measured without the presence of thiocyanate.

Claims

1. A system for measuring cyanide in an alkaline sample having an ultraviolet irradiation unit (30) for producing ultraviolet radiation to dissociate cyanides contained in the alkaline sample, the system further having a thin-film distillation unit (11B) for separating cyanide from the irradi-

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driving the system and exhausting it from the line 21.

The colorimeter provides a recordable signal indicative of the recovered cyanide quantities to an input of a recorder 70, connected to a printer 75.

Thus, the method and system of this invention provides dissociation of all cyanide complexes, including cobalt complexes, without thiocyanate interference. It achieves this by employing an alkaline sample with a new and improved ultraviolet irradiation unit, which filters selected ultraviolet energy levels to inhibit the thiocyanate break down.

It is a feature of the present invention to acidify the samples with the acid mixture via line 15, after the irradiation, rather than prior to it, for the purpose of causing the stronger cyanide complexes, such as the cobalt complexes, to break down, without the break down of thiocyanate.

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An advantage of the two-channel technique for measuring thiocyanate, is that color and turbidity of the samples, does not interfere with the measurements. Conventional direct colormetric measurements of thiocyanate are subjected to interferences from color and turbidity, when present in the samples. With the automated system of this invention, total cyanide, which completely includes all the strong cyanide complexes (even the strong cobalt complex), is conveniently measured without the presence of thiocyanate.

Claims

1. A system for measuring cyanide in an alkaline sample having an ultraviolet irradiation unit (30) for producing ultraviolet radiation to dissociate cyanides contained in the alkaline sample, the system further having a thin-film distillation unit (11B) for separating cyanide from the irradi-

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ated sample to produce recovered cyanide for measurement purposes, characterized by a clear glass filter (33) interposed between the irradiation unit (30) and the sample for passing ultraviolet radiation having a wavelength between about 290 millimicrons and about 400 millimicrons to produce an irradiated sample in which cyanides have been dissociated, while blocking high frequency ultraviolet radiation, to inhibit thiocyanate dissociation, and a measuring device (11D) for receiving the recovered cyanide and generating a recordable signal indicative of recovered cyanide quantity.

2. The system recited in claim 1, further characterized in that the irradiation unit (30) includes an ultraviolet lamp (32).

3. The system recited in claim 1, further characterized in that the glass filter is a glass tube (33) disposed around the irradiation unit (30).

4. The system recited in claim 2, further characterized in that the glass filter (33) includes a glass coil (30) for passing the sample to be tested there through and disposed around the lamp (32).

5. A method of measuring cyanides in a sample characterized by:

using photoillumination components for producing ultraviolet radiation to dissociate cyanides and thiocyanates contained in a first portion of an alkaline sample;

separating cyanide from the irradiated sample produced by the dissociation of cyanides and thiocyanates to produce total recovered cyanide for measurement purposes;

measuring the total recovered cyanide to generate a first recordable signal indicative of total recovered cyanide quantity;

interposing a filter between the photoillumination means and a second portion of the alkaline sample to be tested for passing ultraviolet radiation having a wavelength between about 290 millimicrons and about 400 millimicrons to produce an irradiated sample in which cyanides have been dissociated, while blocking high frequency ultraviolet radiation to inhibit thiocyanate dissociation;

separating cyanide from the irradiated sample to produce recovered cyanide for measurement purposes;

measuring the recovered cyanide to generate a second recordable signal indicative of recovered cyanide quantity; and

using a subtraction circuit responsive to the first and second recordable signals to generate a recordable signal indicative of the difference between the first and second signals this being the thiocyanate quantity.

6. A method as recited in claim 5, further comprising:

acidifying the irradiated sample and subjecting the acidified sample to thin-film distillation.

7. A method as recited in claim 6, further comprising:

absorbing HCN from the output of the thin-film distillation.

Patentansprüche

1. System zum Messen von Zyanid in einer alkalischen Substanzprobe mit einer Ultraviolettbestrahlungseinheit (30) zur Herstellung ultravioletter Strahlung, um in der alkalischen Substanzprobe enthaltene Zyanide zu dissoziieren, wobei das System weiterhin eine Dünnschichtdestilliereinheit (11B) zum Trennen des Zyanids von der bestrahlten Substanzprobe zur Bereitstellung zurückgewonnenen Zyanids für Messungszwecke umfaßt, gekennzeichnet durch ein zwischen der Bestrahlungseinheit (30) und der Substanzprobe zwischengeschaltetes Klarglasfilter (33) zum Durchlassen ultravioletter Strahlung mit einer Wellenlänge zwischen etwa 290 Millimikron und etwa 400 Millimikron, um eine bestrahlte Substanzprobe zu erstellen, bei welcher die Zyanide dissoziiert worden sind, bei gleichzeitiger Blockierung der Hochfrequenzultraviolettstrahlung zur Verhinderung von Thiozyanatdissoziation, und eine Meßeinrichtung (11D) zur Aufnahme des zurückgewonnenen Zyanids und zur Erzeugung eines speicherbaren, die Menge zurückgewonnenen Zyanids anzeigenden Signals.

2. System nach Anspruch 1, weiterhin dadurch gekennzeichnet, daß die Bestrahlungseinheit (30) eine Ultraviolettlampe (32) umfaßt.

3. System nach Anspruch 1, weiterhin dadurch gekennzeichnet, daß das Glasfilter von einem um die Bestrahlungseinheit (30) angeordneten Glasrohr (33) gebildet ist.

4. System nach Anspruch 2, weiterhin dadurch gekennzeichnet, daß das Glasfilter (33) eine um die Lampe (32) angeordnete Glasspule (30) zur Durchleitung der zu untersuchenden Substanzprobe umfaßt.

5. Verfahren zum Messen von Zyaniden in einer

Substanzprobe, gekennzeichnet durch:

die Benutzung von Photoilluminationskomponenten zur Erzeugung ultravioletter Strahlung, um in einer ersten Menge einer alkalischen Substanzprobe enthaltene Zyanide und Thiozyanate zu dissoziieren;

die Trennung von Zyanid aus der bestrahlten Substanzprobe durch Dissoziation von Zyaniden und Thiozyanaten zur Bereitstellung insgesamt zurückgewonnenen Zyanids für Messungszwecke;

die Messung des insgesamt zurückgewonnen Zyanids zur Erzeugung eines ersten speicherbaren, die Menge an insgesamt zurückgewonnenem Zyanid anzeigenden Signals;

die Zwischenschaltung eines Filters zwischen die Photoilluminationsvorrichtungen und eine zweite Menge der zu untersuchenden alkalischen Substanzprobe zum Durchlassen von Ultravioletttstrahlung mit einer Wellenlänge zwischen etwa 290 Millimikron und etwa 400 Millimikron zur Herstellung einer bestrahlten Substanzprobe, in welcher die Zyanide dissoziiert worden sind, bei gleichzeitiger Blockierung der Hochfrequenzultravioletttstrahlung zur Verhinderung von Thiozyanatdissoziation;

die Trennung von Zyanid aus der bestrahlten Substanzprobe zur Bereitstellung von zurückgewonnenem Zyanid für Messungszwecke;

die Messung des zurückgewonnenen Zyanids zur Erzeugung eines zweiten speicherbaren, die Menge zurückgewonnenen Zyanids anzeigenden Signals; und

die Benutzung eines auf die ersten und zweiten speicherbaren Signale reagierenden Subtraktionsschaltkreises zur Erzeugung eines speicherbaren, die Differenz zwischen den ersten und zweiten Signalen anzeigenden Signals, welches die Menge an Thiozyanat darstellt.

6. Verfahren nach Anspruch 5, weiterhin umfassend:

die Ansäuerung der bestrahlten Substanzprobe und die Dünnfilmdestillation der angesäuerten Substanzprobe.

7. Verfahren nach Anspruch 6, weiterhin umfassend:

die Absorption von HCN aus dem Ausstoß der Dünnfilmdestillation.

Revendications

1. Système pour mesurer du cyanure dans un échantillon alcalin ayant une unité d'irradiation par ultraviolets (30) destiné à produire un rayonnement ultraviolet pour dissocier des cyanures contenus dans un échantillon alcalin, le système conportant encore une unité de distillation en couche mince (11B) pour séparer le cyanure de l'échantillon irradié afin de produire un cyanure récupéré dans le but de mesure, caractérisé en ce qu'un filtre de verre transparent (33) est interposé entre l'unité d'irradiation (30) et l'échantillon pour faire passer le rayonnement ultraviolet ayant une longueur d'onde comprise entre 290 millimicrons environ et 400 millimicrons environ afin de produire un échantillon irradié dans lequel ont été dissociés des cyanures, tout en bloquant le rayonnement ultraviolet de haute fréquence pour inhiber la dissociation du thiocyanate, et un appareil de mesure (11D) pour recevoir le cyanure récupéré et engendrer un signal pouvant être enregistré et indicatif de la quantité de cyanure récupéré.

2. Système selon la revendication 1, caractérisé encore en ce que l'unité d'irradiation (30) comprend une lampe ultraviolette (32).

3. Système selon la revendication 1, caractérisé encore en ce que le filtre en verre est un tube de verre (33) disposé autour de l'unité d'irradiation (30).

4. Système selon la revendication 2, caractérisé en ce que le filtre en verre (33) comprend un serpentin de verre (30) pour y faire passer l'échantillon à tester et qui est autour de la lampe (32).

5. Méthode de mesure des cyanures dans un échantillon caractérisé en ce que:

on utilise des composants de photoillumination pour produire un rayonnement ultraviolet afin de dissocier cyanures et thiocyanates contenus dans une première partie d'un échantillon alcalin;

on sépare du cyanure de l'échantillon irradié produit par la dissociation des cyanures et des thiocyanates afin de produire le total du cyanure récupéré dans le but de mesure;

on mesure le total du cyanure récupéré afin d'engendrer un premier signal d'enregistrement indicatif de la quantité totale de cyanu-

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re récupéré;

on interpose un filtre entre le moyen de photoillumination et une seconde partie de l'échantillon alcalin à tester pour faire passer le rayonnement ultraviolet ayant une longueur d'onde comprise entre 290 millimicrons environ et 400 millimicrons environ afin de produire un échantillon irradié dans lequel des cyanures ont été dissociés, tout en bloquant le rayonnement ultraviolet de haute fréquence pour inhiber la dissociation de thiocyanate;

on sépare du cyanure de l'échantillon irradié afin de produire du cyanure récupéré dans le but de mesure;

on mesure le cyanure récupéré pour engendrer un second signal d'enregistrement indiquant la quantité de cyanure récupéré; et

on utilise un circuit de soustraction recevant les premier et second signaux d'enregistrement pour engendrer un signal d'enregistrement indiquant la différence entre les premier et second signaux, qui est la quantité de thiocyanate.

6. Méthode selon la revendication 5, comprenant encore:

on acidifie l'échantillon irradié et on soumet l'échantillon acidifié à la distillation en couche mince.

7. Méthode selon la revendication 6, comprenant encore:

on absorbe HCN à la sortie de la distillation en couche mince.