METHODS AND MEANS FOR THE MONITORING OF AMMONIA IN WATER

An apparatus for monitoring the level of ammonia in water consists of a membrane (18, 20) that separates free ammonia gas dissolved in a sample of water from the water itself. The separated gases are transported to an ion mobility spectrometer (30), indicating the level of free ammonia gas, in filtered air. In one embodiment, the water is treated with hydroxide to convert ammonium ions into free ammonia, the spectrometer then indicating the level of total ammonia in the sample of water. In a second embodiment, the sample of water is left untreated and the spectrometer indicates the level of free ammonia in the sample only. In a third embodiment, part of the water is treated and part is untreated, and the spectrometer may selectively indicate either the level of total or free ammonia in the water. A method of measuring the level of ammonia in water is also disclosed.
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Methods and means for the Monitoring of Ammonia in Water

The present invention relates to a method and means for the continuous monitoring of ammonia in water. The method and means of the invention may be used to monitor either total ammonia in water or free ammonia in water, or both.

Ammonia occurs naturally in the environment as part of all life cycles using nitrogen, and industrially in the production of fertilisers. Ammonia is extremely soluble in water and as a gas dissolved in water has its greatest environmental impact. An essential part of sewage treatment is to reduce the ammonia level in the treated effluent so that animal and plant life in the receiving water are not harmed. In addition the presence of ammonia in water has an effect upon chlorination in potable water treatment, leading to the formation of products which make chlorination less effective or ineffective as a disinfecting process.

Although ammonia is a gas when it dissolves in water, it exists thereafter as both free ammonia and as ammonium ions. There is an equilibrium between the two which is dependent upon the water temperature, its pH and to a lesser extent, the total dissolved solids in the water. At pH 7 and 15 degrees C, less than 0.3% of the ammonia is present as free ammonia, but at pH 9.5 and 15 degrees C more than 50% is present as free ammonia. Although it is free ammonia which is toxic to fish, at the present time the water industry is only required to measure the total ammonia level, that is free ammonia and ammonium ion content together. The free ammonia level is calculated from
tables of available data.

One object of the present invention is thus the provision of an instrument able to monitor total ammonia in water or free ammonia in water, or both on a continuous basis.

According to one aspect of the present invention, there is provided an apparatus for measuring the level of ammonia in water comprising first separation means arranged to accept water to be measured and to permit gases dissolved in the water to be separated out, and detection means for detecting and measuring the level of free ammonia in the separated gases; the level of free ammonia in the separated gases being indicative of the level of ammonia in the water.

Preferably, the apparatus also includes treatment means arranged to convert ammonium ions in the water to free ammonia prior to its acceptance by the first separation means, the level of free ammonia detected and measured by the detection means being indicative of the level of total ammonia in the water.

The ammonium ions may be converted to free ammonia by treatment with a hydroxide. The treatment of the ammonium ions means that substantially all of the ammonia in the water is in the form of free ammonia, and the amount of gas detected by the detection means is therefore indicative of the level of total ammonia in the water.

Preferably, a combined apparatus is employed. In this, the water to be measured is split into two branches at the input to the apparatus, the water in one branch being treated to convert ammonium ions in it to free ammonia before being separated in first separation means, the water in the other branch being passed to second separation means without treatment to remove the ammonia
ions. Selection means, such as two-way valve, may be arranged to accept the outputs of the first and second separation means and selectively pass one or the other of these outputs to the detection means. Thus, the level of free ammonia or the level of total ammonia may be measured as desired. Subtracting means may be included in the detection means to subtract the level of total ammonia from the level of free ammonia to indicate the level of ammonium ions present in the water.

The detection means may be an ion mobility spectrometer or a mass spectrometer, for example. If an ion mobility spectrometer is employed it is preferable to stabilise the ion mobility spectrum by supplying the or each separation means with filtered air that has been doped.

According to a second aspect of the present invention, there is provided a method of measuring the level of ammonia in water, including the steps of separating out gases dissolved in a supply of water, and detecting and measuring the level of free ammonia in the separated gases to indicate the level of ammonia in the water.

Preferably, the method includes the step of first treating the water to convert ammonium ions in it into free ammonia prior to separating out the dissolved gases, the step of detecting and measuring the level of ammonia in the separated gases indicating the level of ammonium ions in the water prior to its treatment.

In a preferred embodiment, part of the water is left untreated, the method including separating out gases disssolved in the said part, and detecting and measuring the said gases which have been separated out to indicate the level of free ammonia in the water.

These and other aspects of the present invention
will become apparent from the following description of a preferred embodiment of the invention, described with reference to the accompanying drawing which shows a schematic diagram of an instrument according to the invention.

Referring to the drawing, the instrument illustrated can be considered in two parts: the sampling system and the detection system.

The sampling system is concerned with the extraction of the water to be monitored from, for example, a sewage treatment process, and its filtration, and subsequent treatment with a biocide, prior to measurement of both free and total ammonia, and subsequent return of the residual sample to its source or to waste.

The detection system is concerned with the generation of a controlled atmosphere for the detector, the interface between this atmosphere and the water sample at which ammonia is transferred from the aqueous to the gaseous phase, the measurement of the ammonia levels and the processing and display of the information.

Referring first to the sampling system, a submersible pump 10 is used to extract water from the source to be monitored. The sampled water is fed to a cross-flow filter 14, through a pipe 12, where particles above a given size, for example 25 microns, are removed from a proportion of
the sample and the remainder of the sample is passed back to the source through outlet 16.

The filtered portion of the sample is treated with biocide at 17 to prevent biological growth in the sample and within the sampling system downstream of 17. A typical biocide treatment would consist of iodine or silver and copper ions in solution.

After biocidal treatment, the sample stream is split into two branches, one passing across one face of a membrane 18 and the other passing across one face of another membrane separator 20, after first passing through hydroxide treatment means 24 and through a heater 22.

Both sample streams are subsequently passed to outlet pipe 16.

The detection system comprises an ion mobility spectrometer detector 30 within a gas loop comprising the "dry" side of membrane separators 18 and 20, valve 32 and outlet filter 34, and an air inlet system comprising pump 36, filter 38 and dopant chamber 40.

Ambient air is drawn into the inlet 42 by pump 36 and filtered by an activated charcoal filter element in filter 38 in order to remove contaminants from the incoming air sample.

The filtered air passes into the thermostatically controlled dopant chamber 40 housing a permeation source containing an organic phosphonate for the purpose of stabilising the ion mobility spectrum and suppressing the formation of unwanted ion clusters.

The filtered phosphonate-doped air from the dopant
chamber 40 is fed directly to the IMS detector 30 through line 44, and across the membrane elements 18 and 20 through common line 46, and separate lines 48 and 50 respectively.

The membrane elements 18 and 20 each have a water sample flow across one face, and a doped air flow across the other. By employing as a membrane a material such as microporous ptfe with a pore size of 0.2 microns, free ammonia gas in the water flows is able to transfer across the respective membrane elements into the doped air flows.

Ammonia-doped air leaving membrane elements 18 and 20 is fed through lines 52 and 54 respectively to separate inlet ports 56 and 58 of an inert four-port two-way valve 32. By this means either ammoniated air from the untreated sample flowing through membrane 18, or ammoniated air from the treated water sample flowing through membrane 20, may be introduced into the sample inlet 62 of IMS detector 30, from outlet port 64 of valve 32, the other ammoniated air flow being exhausted from outlet port 66.

In the drawing, air from membrane 18 is being fed to the IMS detector 30, and air from membrane 20 fed to exhaust. Rotation of the valve 32 through 90 degrees causes air from membrane 20 via line 54 to be fed from port 58 to port 64 and to IMS detector 30, and air from membrane 18 via line 52, to be fed from port 56 to port 66 and to exhaust.

Exhaust air flow from outlet 68 of detector 30 is fed through filter 34 to exhaust line 70, as is the exhaust air flow from port 66 to valve 32.

IMS detector 30 is a commercially available ion
mobility detector, for example a Graseby Dynamics Limited Type HITIMS instrument, provided with the necessary power supplies and measuring, indicating and recording circuitry.

Suitably doping the air supply through dopant chamber 40 to the detector 30, enables the detector to respond selectively to ammonia in the presence of otherwise interfering compounds which may be present in the air flow at the inlet of the detector.

In operation, free ammonia present in the incoming water sample will be present in the untreated water sample flow across membrane 18 at the same temperature and a portion of the free ammonia will be transferred through membrane 18 into the air flow in line 52.

Free ammonia related to the total ammonia content of the incoming water sample will be present in the water sample flow across membrane 20 after pH adjustment at 24 and heating by heater 22, and will be transferred through the membrane 20 into the air flow in line 54.

Either the free ammonia or total ammonia in the original incoming sample water at 12 may thus be measured by appropriate positioning of valve 32, after equilibration of detector 30, which will typically be within seconds. The valve may be rotated automatically with an appropriate duty cycle to enable both free and total ammonia levels effectively to be continuously monitored.

Whilst the means of detection is shown as an ion mobility spectrometer, it will be apparent that other means of measuring the ammonia content of the untreated and treated sample streams across membranes 18 and 20
respectively, for example a mass spectrometer could be used, although an ion mobility spectrometer is possessed of a number of features making it a preferred choice for the present application e.g. simple logistics, ruggedness and low maintenance requirements and costs.
CLAIMS

1. An apparatus for measuring the level of ammonia in water comprising first separation means arranged to accept water to be measured and to permit gases dissolved in the water to be separated out, and detection means for detecting and measuring the level of free ammonia in the separated gases; the level of free ammonia in the separated gases being indicative of the level of ammonia in the water.

2. An apparatus as claimed in claim 1, further including treatment means arranged to convert ammonium ions in the water to free ammonia prior to its acceptance by the first separation means, the level of free ammonia detected and measured by the detection means being indicative of the level of total ammonia in the water.

3. An apparatus as claimed in claim 2, including second separation means arranged to accept a part of the water to be measured not having been treated by the treatment means, the second separation means permitting gases dissolved in the water to be separated out and selection means for selectively supplying the outputs of the first and second separation means to the detection means, the detection means selectively indicating the level of free ammonia in the water and the level of total ammonia in the water.

4. An apparatus as claimed in claim 3, in which the selection means is a two-way valve.
5. An apparatus as claimed in any one of the preceding claims, in which the or each separation means includes a membrane having a pore size which allows passage of free ammonia gas from the water.

6. An apparatus as claimed in any one of the preceding claims, in which the water is first treated with a biocide to prevent biological growth.

7. An apparatus as claimed in any one of the preceding claims, in which the detection means is an ion mobility spectrometer.

8. An apparatus as claimed in any one of the preceding claims, in which the or each separation means is further supplied with air that has been doped to stabilise the ion mobility spectrum and to suppressed the formation of unwanted ion clusters.

9. An apparatus as claimed in any one of claims 2 to 8, in which the treatment means includes hydroxide treatment means.

10. Apparatus constructed and arranged substantially as specifically described with reference to and as shown in the accompanying drawing.

11. A method of measuring the level of ammonia in water, including separating out gases dissolved in a supply of water, and detecting and measuring the level of free ammonia in the separated gases to indicate the level of ammonia in the water.

12. A method as claimed in claim 11, including first
treatment of water to convert ammonium ions in it into free ammonia prior to separating out the dissolved gases, the step of detecting and measuring the level of free ammonia in the separated gases providing an indication of the level of ammonium ions in the water prior to treatment.

13. A method as claimed in claim 12 in which a part of the water is left untreated, the method including separating out gases dissolved in the said part and detecting and measuring the said gases which have been separated out to indicate the level of free ammonia in the water.

14. A method as claimed in any one of claims 11 to 13, further comprising the steps of treating the water supply with a biocide to prevent biological growth.

15. A method as claimed in any one of claims 11 to 14, including supplying air to carry the separated-out gases to be detected and measured.

16. A method substantially as specifically described with reference to and as shown in the accompanying drawing.
FIG. 1.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 G01N33/18 G01N33/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Date of the actual completion of the international search 30 May 1996

Date of mailing of the international search report 7.06.96

Authorized officer Zinngrebe, U

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