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(54) **Title**: COMPOSITION AND METHOD FOR MEASURING ORTHOPHOSPHATE CONCENTRATION IN WATER SYSTEM AND USE OF SAID COMPOSITION

(57) **Abstract**: A composition for measuring concentration of orthophosphate in water system comprising an acid which is in solid form at ambient conditions, a color development agent for developing the phosphomolybdic acid, molybdate and optionally a filler which is used to quickly and directly measure orthophosphate in water without dilution. A method for quickly measuring the concentration of orthophosphate in water without dilution, comprising adding the composition to a water sample to form a solution and perform coloration, measuring absorbance of the solution under UV-Vis light, and determining the concentration of orthophosphate in the water.

COMPOSITION AND METHOD FOR MEASURING ORTHOPHOSPHATE CONCENTRATION IN WATER SYSTEM AND USE OF SAID COMPOSITION

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to Chinese Patent Application Serial No. 201510084556.8 filed on February 16, 2015, the disclosure of which is incorporated herein by reference in its entirety.

TECHNICAL FIELD

[0002] The present invention relates to a composition in solid form for measuring orthophosphate in a water system, a method of using the composition to measure the orthophosphate concentration in the water system, and use of the composition for measuring the orthophosphate concentration in the water system.

BACKGROUND OF THE INVENTION

[0003] In industrial water systems such as cooling towers and steam boilers, phosphates, including orthophosphate, are widely used as corrosive inhibitors. When used as a corrosive inhibitor, the orthophosphate forms a film with cations such as iron and calcium on the surface of the material so as to protect the surface of the material from being eroded. However, if overdosed, orthophosphate may cause scaling, fouling and environmental issues. It is an important goal for industrial water treatment to control the orthophosphate concentration in an appropriate range. Therefore, there exists a need to regularly measure the concentration of orthophosphate in industrial water systems.

[0004] Among the current methods for measuring orthophosphate in a water system, especially in industrial water, the molybdenum blue method and the molybdenum yellow method are commonly used. The first step of the molybdenum blue method and molybdenum yellow method is to acidify the water sample and add a molybdate, where the orthophosphate and the molybdate react in the acidic aqueous solution to generate a solution that contains the phosphomolybdic acid (a heteropoly acid). In the second step of the molybdenum blue method, a reductant, for example, ascorbic acid, is added to the aforesaid phosphomolybdic acid containing solution to reduce the phosphomolybdic acid to produce an obvious blue color. In the second step of the molybdenum yellow method, a color development agent such as vanadate is added to the aforesaid phosphomolybdic acid containing solution to develop a yellow color. Subsequently, the absorbance of the water

sample is measured colorimetrically using a spectrometer, thereby determining the concentration of the orthophosphate contained in water. The molybdenum blue method is very sensitive and suitable for measuring orthophosphate in a low concentration. However, in industrial water system, sometimes the orthophosphate concentration may reach as high as tens of ppm. When the orthophosphate concentration in water is high, the molybdenum blue method is easily saturated, where the water sample needs to be diluted before the measurement can be done. Compared to the molybdenum blue method, the molybdenum yellow method has a wide range of measurement and is more suitable for industrial water samples.

[0005] However, in current molybdenum yellow methods, a liquid solution of a strong acid such as sulfuric acid or nitric acid is generally used. The liquid solution of the strong acid has strict requirements for transportation, complicated operation, lack of safety and a certain level of danger. Moreover, in current molybdenum yellow methods, it takes a long time for development and measurement.

[0006] It is an object of this invention to develop a composition in solid form that is easy to carry and transport and can be directly added into an industrial water system that contains orthophosphate for one step development. In the meanwhile, it is desired to rapidly detect the concentration of the orthophosphate contained in the water system and increase the detection efficiency by this solid form testing composition.

[0007] This solid form testing composition is stable for long term storage and poses little environmental and safety threats. When used to measure the orthophosphate in the industrial water system, this dry powder form composition is easy to formulate, safe and convenient to operate, and with low cost, and it can be easily employed by anyone, even those without analytical experiences.

SUMMARY OF THE INVENTION

[0008] The present invention relates to a composition for detecting orthophosphate in a water system, especially an industrial water, a method for measuring the orthophosphate concentration in the water system using the composition, and use of the composition.

[0009] The invention provides a composition for measuring the orthophosphate concentration in a water system comprising

(a) about 75-98.5 % by weight of an acid in solid form;

(b) about 1-24% by weight of a water soluble molybdate; and

(c) about 0.05-1% by weight of a color development agent for developing the

phosphomolybdic acid.

[0010] The invention further provides a method for measuring the concentration of the orthophosphate in a water system comprising the steps of

(i) taking a water sample,

(ii) adding a composition according to any one of claims 1 to 15 into the water sample to form a solution and develop a color; and

(iii) measuring absorbance of the solution.

[0011] The invention further provides use of the composition of the invention for measuring the concentration of the orthophosphate in a water system.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] Figure 1 is a plot of absorbance vs. time at a wavelength of 365 nm and an optical path length of 1 cm for Example 1. It can be seen that within the range of the concentration suitable for the example, the coloration can be finished within one minute.

[0013] Figure 2 is a plot of absorbance vs. wavelength with an optical path length of 1 cm for Example 1.

[0014] Figure 3 is a calibration curve of absorbance vs. orthophosphate concentration at a wavelength of 380 nm and an optical path length of 1 cm for Example 1.

[0015] Figure 4 is a plot of absorbance vs. wavelength with an optical path length of 1 cm for Example 2.

[0016] Figure 5 is a calibration curve of absorbance vs. orthophosphate concentration at a wavelength of 365 nm and an optical path length of 1 cm for Example 2.

DETAILED DESCRIPTION OF THE INVENION

[0017] The present invention relates to a composition for measuring the concentration of orthophosphate in a water system. The composition provided in the invention is in solid form, especially in a form of a dried powder, to facilitate transportation, packaging and use.

[0018] In an embodiment, the invention provides a composition for measuring the concentration of orthophosphate in a water system comprising (a) an acid in solid form; (b) a water soluble molybdate; and (c) a color development agent for developing the phosphomolybdic acid.

[0019] The aforesaid acid in solid form refers to an acid which is solid under ambient conditions, wherein “ambient conditions” refer to temperatures and pressures occurring

indoors or outdoors. Preferably, the "ambient conditions" indicate a temperature between 16~35°C and a pressure of 1 atm.

[0020] The acid in solid form preferably has a pKa value of at most 1.5. The pKa value of the acid in solid form is more preferably between about -10 and 1.5, and most preferably between about -5 and 1.5.

[0021] The acid in solid form is preferably selected from a group consisting of sulfamic acid, benzenesulfonic acids, trichloroacetic acid, and mixtures thereof. The "mixtures" of the acids include mixtures comprising two, more or all selectable acids.

[0022] The benzenesulfonic acids include substituted benzenesulfonic acids, wherein the substituent is selected from C1-C4 alkyl such as methyl, ethyl, propyl or butyl and halogen selected from chloro, bromo and iodo. The substituted benzenesulfonic acid is selected from a group consisting of o-C1-C4 alkylbenzenesulfonic acids, m-C1-C4 alkylbenzenesulfonic acids, p-C1-C4 alkylbenzenesulfonic acids, o-chlorobenzenesulfonic acids, m-chlorobenzenesulfonic acids, p-chlorobenzenesulfonic acids, o-bromobenzenesulfonic acids, m-bromobenzenesulfonic acids, p-bromobenzenesulfonic acids, and mixtures thereof, and preferably p-toluenesulfonic acid.

[0023] The amount of the acid in solid form used in the invention is about 75-98.5% by weight; preferably about 78%-98% by weight, and more preferably about 80%-97.5% by weight.

[0024] The water soluble molybdate used in the invention is a molybdate that can react with the orthophosphate to form the phosphomolybdic acid under the acidic conditions described herein. The molybdate used in the invention includes those molybdate generally used in molybdenum yellow method. Preferably, the molybdate is ammonium molybdate, sodium molybdate, or potassium molybdate. A skilled artisan is able to determine the molybdate based on the prior art.

[0025] The amount of the molybdate used in the invention is about 1-24% by weight, preferably about 2-20% by weight, and more preferably about 2-18% by weight.

[0026] The color development agent for developing the phosphomolybdic acid is a color development agent used in the molybdenum yellow method that develop a color for the phosphomolybdic acid formed from the reaction between the molybdate and the orthophosphate in the water system under the acidic conditions, for example, vanadates, preferably ammonium vanadate, sodium vanadate and potassium vanadate.

[0027] In the invention, the amount of the vanadate is about 0.05-1% by weight,

preferably about 0.1-0.8% by weight, and more preferably about 0.3-0.7% by weight.

[0028] In an embodiment, provided is a composition for measuring the concentration of orthophosphate in a water system comprising

(a) an acid in solid form which is about 75-98.5% by weight, preferably about 78%-98% by weight, and more preferably about 80%-97.5% by weight;

(b) a water soluble molybdate which is about 1-24% by weight, preferably about 2-20% by weight, and more preferably about 2-18% by weight; and

(c) a color development agent for developing the phosphomolybdic acid which is about 0.05-1% by weight, preferably about 0.1-0.8% by weight, and more preferably about 0.3-0.7% by weight.

[0029] In an embodiment, provided is a composition for measuring the concentration of orthophosphate in a water system comprising

(a) an acid in solid form selected from sulfamic acid, benzenesulfonic acids, trichloroacetic acid, and mixtures thereof, which is about 75-98.5% by weight, preferably about 78%-98% by weight, and more preferably about 80%-97.5% by weight;

(b) a water soluble molybdate, preferably ammonium molybdate, sodium molybdate sodium, or potassium molybdate, which is about 1-24% by weight, preferably about 2-20% by weight, and more preferably about 2-18% by weight; and

(c) a water soluble vanadate, preferably ammonium vanadate, sodium vanadate, or potassium vanadate, which is about 0.05-1% by weight, preferably about 0.1-0.8% by weight, and more preferably about 0.3-0.7% by weight.

[0030] In another embodiment of the invention, the composition comprises about 80~97.5% by weight of an acid in solid form such as p-toluenesulfonic acid, about 0.4~0.6% by weight of a vanadate such as ammonium vanadate, and about 2~20% by weight of a molybdate such as ammonium molybdate.

[0031] For the convenience of mixing and aliquoting (packaging), the composition of the invention may also comprise a filler to adjust the total amount of reagents so as to facilitate formulation and aliquoting. For example, when the composition is used in a very low amount, any minor mistake occurring during the aliquoting may cause a great error in the test result. Therefore, it is preferred to add a filler in the composition to facilitate the formulation and aliquoting of the composition. The filler can be a substance inert to the orthophosphate in the water system and other components in the composition, which does not affect the color change during the color development of the phosphomolybdic acid, and is usually a mineral salt such as sodium chloride, potassium

chloride, potassium nitrate, and the like. In an embodiment, the mineral salt is sodium chloride.

[0032] The filler can be added in any amount depending on the convenience of formulation, for example, one or more times of the weight of the composition, as long as it does not affect the measurement of the orthophosphate. This is apparent to a skilled artisan.

[0033] In an embodiment, a dried powder composition for measuring the concentration of orthophosphate in a water system is provided. The dried powder composition comprises (a) an acid in solid form, for example, selected from a group consisting of sulfamic acid, benzenesulfonic acid, trichloroacetic acid, and mixtures thereof; (b) a molybdate; (c) a color development agent for the phosphomolybdic acid, such as a vanadate; and (d) a filler, such as sodium chloride or potassium nitrate, wherein the filler is added in an amount according to the need on the basis of the existing composition, for example, more than 0 and up to 10 times the weight of the active ingredients of the composition.

[0034] In an embodiment of the invention, the composition comprises about 20~25% by weight of an acid in solid form such as benzenesulfonic acid, about 0.1~0.2% by weight of a vanadate such as ammonium vanadate, about 4~5% by weight of a molybdate such as ammonium molybdate, and about 70~75% by weight of a filler such as sodium chloride.

[0035] In an embodiment of the invention, the composition comprises (a) an acid in solid form, for example, selected from sulfamic acid, benzenesulfonic acids, trichloroacetic acid, and mixtures thereof, which is about 90%-97.5% by weight; (b) ammonium molybdate, which is about 2-2.5% by weight; (c) a vanadate, for example ammonium vanadate, which is about 0.4~0.5% by weight; and a filler, for example sodium chloride, to 100%.

[0036] It has surprisingly been found that by specifically selecting the ratio of the acid in solid form, the molybdate, and the color development agent, rapid color development can be achieved, so as to enable more rapid detection of the concentration of the orthophosphate in the water system. It was surprisingly found that by adjusting the ratio of the various components in the composition so that after dissolved into the test sample, the molar concentrations of the acid in solid form, the vanadate, and the molybdate (calculated on the basis of the amount of single molybdenum) are about 50-100, about 0.02-0.06, about 5-10 millimolar per liter, preferably about 60-90, about 0.03-0.05, about 6-9 millimolar per liter, and more preferably about 70-85, about 0.032-0.040, about 6.5-8 millimolar per liter, respectively. When the composition is added into a water sample it

facilitates the rapid color development of the water sample, so as to enable more rapid detection of the concentration of the orthophosphate in the water sample. Preferably, the molar ratio of the acid in the form of a solid powder, the vanadate, and the molybdate (calculated on the basis of the amount of single molybdenum) is about 7-13:0.002-0.1:1.

[0037] The composition for measuring the orthophosphate in the water system, especially in the industrial water, is prepared by mixing the acid in solid form, the molybdate, the color development agent, and optionally the filler to form a uniform dried powder.

[0038] The composition in dried powder form of the present invention can be used to measure the concentration of the orthophosphate in a water system, especially in industrial water. At use, a water sample is taken, to which the solid composition is directly added and shaken to thoroughly mix the water sample for color development. The color of the water sample is measured using a spectrometer. The concentration of the orthophosphate in the water system is determined colorimetrically based on the developed color.

[0039] The water system referred to herein usually refers to an industrial water system, especially those industrial water systems in which the concentration of the orthophosphate needs to be determined, for example industrial water systems of, for example, cooling towers, steam boilers, and the like.

[0040] In an embodiment, a method for measuring the orthophosphate concentration in a water system is provided. The method generally comprises the steps of

(i) taking a water sample;

(ii) adding the composition of the invention into the water sample to form a solution and develop a color; and

(iii) measuring the absorbance of the solution.

[0041] In an embodiment, the method for measuring the orthophosphate concentration in a water system further comprises the steps of

(iv) obtaining an absorption curve of a standard solution that contains orthophosphates; and

(v) comparing the absorption measured in step (iii) with the absorption curve of the standard solution colorimetrically to determine concentration of the orthophosphate in water system.

[0042] Without wishing to be bound by a particular theory, in the method of the invention, it is believed that when adding the composition of the invention into a water sample, a reaction between the orthophosphate and the molybdate in acidic conditions

takes place, generating the phosphomolybdic acid, which is then reacted with the color development agent to develop a color.

[0043] A spectrometer (colorimeter) can be used to determine the UV-Vis absorbance of the water sample at 300-600 nm wavelength, preferably between 350 and 450 nm. Thethe concentration of the orthophosphate in the water system can be obtained colorimetrically, for example, by comparing with the absorption curve (calibration curve) of the orthophosphate-containing standard solution obtained in advance.

[0044] In order to obtain the aforesaid calibration curve, water samples of equal volume having different know concentration of the orthophosphate are placed under an UV-Vis light analysis to measure the absorption under each concentration. Subsequently, based on the measured data, a curve of absorption vs. orthophosphate concentration is plotted which is the aforesaid calibration curve.

[0045] In the invention, the water system tested using the dried powder composition may be a water system with very small orthophosphate content, for example, a water system with orthophosphate content down to about 0pmm, or a water system with large orthophosphate content, for example, a water sample with an orthophosphate content up to 100 ppm. Preferably, the composition of the invention can be used to test a water sample with an orthophosphate content of, for example, from 0 up to about 80 ppm, preferably from 0 up to 50 ppm, more preferably from 0 up to 30 ppm, and still more preferably from 0 up to 20 ppm.

[0046] Preferably, the measurement is conducted by add the composition into the water sample at a ratio of 10~100 miligram composition per 1 mililiter water sample.

EXAMPLES

[0047] The invention is not limited by the following examples. The following examples are provided for the purpose of illustration, rather than limiting the scope of the invention, which is based on the appended claims. The numeric values in the invention, unless defined otherwise, are all weight percentage based on the total weight of the composition.

[0048] Example 1

[0049] In this example, 6.35 grams of dry sulfamic acid, 42 milligrams of ammonium vanadate, 1.27 grams of ammonium molybdate and 20 grams of sodium chloride were grinded together into 27.662 grams of solid homogeneous powder. The powder was

evenly distributed among 1,000 disposable bags, with each bag containing 27.7 milligrams of said homogeneous powder. One bag of the homogeneous powder was then dissolved in 1 milliliter of the orthophosphate-containing water sample to be tested under ambient conditions to form a sample solution. The concentration of components of the testing reagent in the sample water is listed below.

Table 1

Reagent Components	Concentration (mg per ml of water sample)
Sulfamic Acid	6.35
Ammonium vanadate	0.042
Ammonium molybdate	1.27
Sodium chloride filler	20.0

[0050] After the color is fully developed, an UV-Vis analysis (using Shimadu spectrometer) was conducted on the water sample to acquire the absorbance of the sample. The optical path length that the light must travel through during the UV-vis analysis of the sample solution is 1 centimeter.

[0051] Utilizing the testing composition, orthophosphate concentrations of 30, 10 and 1 ppm were tested. The results are shown in Figure 1. The color change of the sample solution was monitored by absorbance at wavelength of 365 nm. Time 0 was the time that the solid testing composition was fully dissolved in the water sample and mixed thoroughly.

[0052] As can be seen from figure 1, full color development of the sample solution was achieved within 1 minute from full dissolving and thoroughly mixing of the testing reagent.

[0053] Figure 2 depicts an UV-Vis spectrum for various orthophosphate concentrations at 1 cm optical path length, under wavelengths between 300 and 600 nm. As can be seen from figure 2, absorbance vs. wavelength curves for the various concentrations are clearly distinguishable within the wavelength range of 350 to 450 nm.

[0054] Figure 3 is a calibration curve showing absorbance at wavelength of 380 nm at optical path length of 1 cm vs. orthophosphate concentration in the sample. As can be seen from Figure 3, linearity of the absorbance vs. concentration curve is maintained from 0 ppm to at least 20 ppm. Maintenance of this linear relationship means that at least where orthophosphate concentration is between 0 and 20 ppm, the concentration can

simply be interpolated from the absorbance at 380nm wavelength.

[0055] Example 2

[0056] In this example, p-toluene sulfonic acid was used instead of sulfamic acid. 50.64 grams of sulfamic acid, 0.25 grams of ammonium vanadate, and 1.11 grams of ammonium molybdate were homogenously mixed together and evenly distributed in 1000 disposable bags, with each bag containing 52 milligrams of the dry solvant homogeneous powder, which was dissolved in 1 milliliter of sample water under ambient conditions to form a sample solution. The composition of the homogeneous solvant powder is listed below.

Table 2

Reagent Components	**Concentration (mg per ml of water sample)**
p-toluenesulfonic acid	50.64
Ammonium vandate	0.25
Ammonium molybdate	1.11

[0057] When full color change of the sample solution was achieved, UV-Vis analysis was conducted on the sample solution to acquire the absorbance of the sample. The light used for the UV-Vis analysis was of a wavelength between 350 and 450 nanometers. The optical path length that the ultraviolet light must travel through during the UV-vis analysis of the sample solution is 1 centimeter.

[0058] The composition was used to measure various orthophosphate concentrations at 1 cm optical path length. Figure 4 is an UV-Vis spectrum for various orthophosphate concentrations, under UV wavelength between 350 and 450 nm.

[0059] Figure 5 shows a calibration curve showing absorbance at wavelength of 365 nm at optical path length of 1 cm vs. orthophosphate concentration in the sample.

[0060] As can be seen from Figure 5, linearity of the absorbance vs. concentration curve was maintained from 0 ppm to at least 20 ppm. Maintenance of this linear relationship means that at least where orthophosphate concentration is between 0 and 20 ppm, the concentration can simply be interpolated from the absorbance at 380nm wavelength.

[0061] Example 3

[0062] The repeatability of the testing method was examined using the testing

composition of Example 2. Two orthophosphate concentrations (2 ppm and 20 ppm as PO_4^{3-}) were tested under UV-vis analysis with UV wavelength of 365nm. The tests were conducted 17 times and the results are summarized in the table below.

Table 3

Concentration	Average absorbance at 365 nm	Standard deviation	Relative standard deviation
2 ppm	0.31	0.02	6.5 %
20 ppm	1.40	0.04	2.8 %

[0063] As can be seen, for both low and high orthophosphate concentrations, the absorbance values measured in all of the trials did not deviate much from the mean, indicating good precision of the detection within a broad range of orthophosphate concentrations.

[0064] Example 4

[0065] The effect of the variation in weight of the testing composition itself on the orthophosphate concentration measurement was examined. Nine samples of the testing reagent powder samples of Example 2, with weight ranging from 98 to 108 mg, are each dissolved in 2 milliliters of water sample (each water sample is of consistent orthophosphate concentration) to measure the absorbance under UV-vis analysis with 365 nm UV wavelength.

Table 4

Weight of the reagent (mg)	105	100	104	104	108	99	98	101	103
Absorbance at 365nm	0.621	0.663	0.697	0.635	0.674	0.625	0.691	0.686	0.716
Average absorbance	0.668								
Standard Deviation	0.034								
Relative standard deviation	5.1%								

[0066] As can be seen from the table above, slight variations in the total weight of the reagent powder did not significantly affect the absorbance measurements and concentration determinations. Hence the method is relatively consistent with respect to the changes in the weight of the reagent, and exhibits reliable sensitivity and accuracy.

[0067] Stability Studies: Stability tests found that the powder stored for at least 6 months under ambient conditions did not show any noticeable degradation.

[0068] In view of the above, it is clear that the powdered testing composition is stable in storage, easy to carry and transport, easy to operate even by a person with no special training in this area. Moreover, it is clear that the method is convenient to measure the orthophosphate concentration with only one step of mixing the dry chemical powder composition with the water sample, while not requiring the step of diluting the water sample nor requiring adding the molybdate and the color development agent in two steps. Meanwhile, rapid color development can be achieved using the composition of the invention which reduces the development time from about 7 minutes by the conventional molybdenum method to 1-2 minutes which efficiently reduces the detection time and improves the detection efficiency. Moreover, high detection sensitivity and accuracy are obtained using the composition of the invention.

[0069] Based on the above, it is clear that the testing composition in solid form according to the invention is stable in storage, easy to carry and easy to operate. Solid form of the composition rapidly develops a color with high repeatability, allows testing the orthophosphate in a water system with excellent sensitivity and accuracy, and can be effectively used to measure the orthophosphate concentration in a water system, especially in industrial water, so as to increase the monitor efficiency.

CLAIMS

1. A composition for measuring concentration of orthophosphate in a water system comprising:

(a) about 75-98.5 % by weight of an acid in solid form;

(b) about 1-24% by weight of a water soluble molybdate; and

(c) about 0.05-1% by weight of a color development agent for developing the phosphomolybdic acid.

2. The composition according to claim 1, wherein the acid in solid form has a pKa value of about -10 ~ 1.5.

3. The composition according to claim 1 or 2, comprising about 78-98% by weight of the acid in solid form.

4. The composition according to any one of claims 1 to 3, comprising about 2-20% by weight of the water soluble molybdate.

5. The composition according to any one of claims 1 to 4, comprising about 0.1-0.8% by weight of the color development agent.

6. The composition according to claim 5, wherein the color development agent is a vanadate.

7. The composition according to any one of claims 1 to 6, wherein the acid in solid form, the vanadate and the molybdate are present in a molar ratio of about 7-13:0.002-0.1:1, calculated on the basis of single molybdenum.

8. The composition according to any one of claims 1 to 7, wherein the acid is selected from a group consisting of sulfamic acid, benzensulfonic acids, trichloroacetic acid, and mixtures thereof.

9. The composition of claim 8, wherein the benzenesulfonic acid is a substituted benzenesulfonic acid, wherein the substitutent is selected from C1-C4 alkyls and halogens.

10. The composition of claim 8, wherein the acid is sulfamic acid or trichloroacetic acid.

11. The composition according to any one of claims 1 to 10, wherein the molybdate is ammonium molybdate, sodium molybdate, or potassium molybdate.

12. The composition of claim 6, wherein the vanadate is ammonium vanadate.

13. The composition according to any one of claims 1 to 12, further comprising a filler.

14. The composition according to any one of claims 1 to 13 comprising

(a) 80-97.5% by weight of an acid in solid form, preferably selected from sulfamic acid, benzensulfonic acids, trichloroacetic acid, and mixtures thereof;

(b) 2-20% by weight of molybdate, preferably ammonium molybdate;

(c) 0.1-0.8% by weight of vanadate, preferably selected from ammonium vanadate, sodium vanadate and potassium vanadate;

(d) a filler as a balance.

15. The composition according to claim 14, wherein the vanadate is ammonium vanadate.

16. A method of measuring the orthophosphate concentration in a water system comprising the steps of

(i) taking a water sample,

(ii) adding a composition according to any one of claims 1 to 15 into the water sample to form a solution and develop a color; and

(iii) measuring absorbance of the solution.

17. The method according to claim 16, further comprising the steps of

(iv) obtaining an absorption curve of a standard solution that contains orthophosphates; and

(v) comparing the absorption measured in step (iii) with the absorption curve of the standard solution colorimetrically to determine concentration of the orthophosphate in

water system.

18. The method according to claim 16, wherein the composition is added at a ratio of about 10 to about 100 milligrams of the composition per 1 milliliter of the water sample.

19. Use of the composition according to any one of claims 1 to 15 for measuring concentration of orthophosphate in a water system.

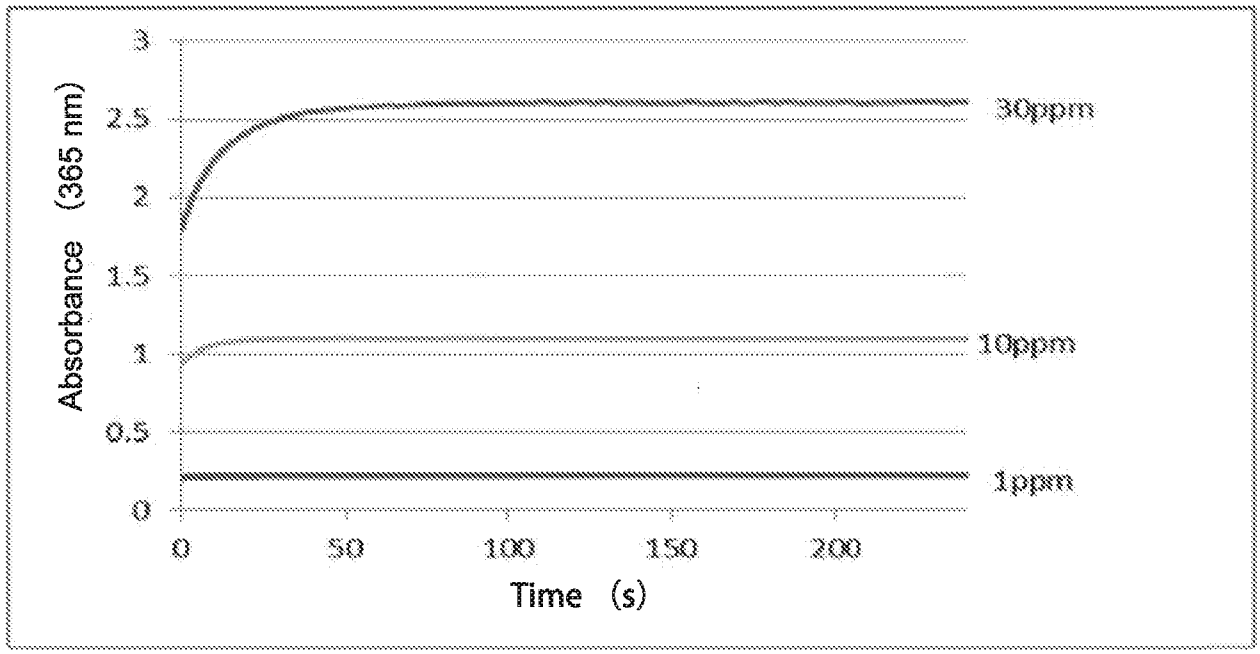
Absorbance (365 nm)
3
2.5
2
1.5
1
0.5
0
30ppm
10ppm
1ppm
0
50
100
150
200
Time (s)

Fig. 1

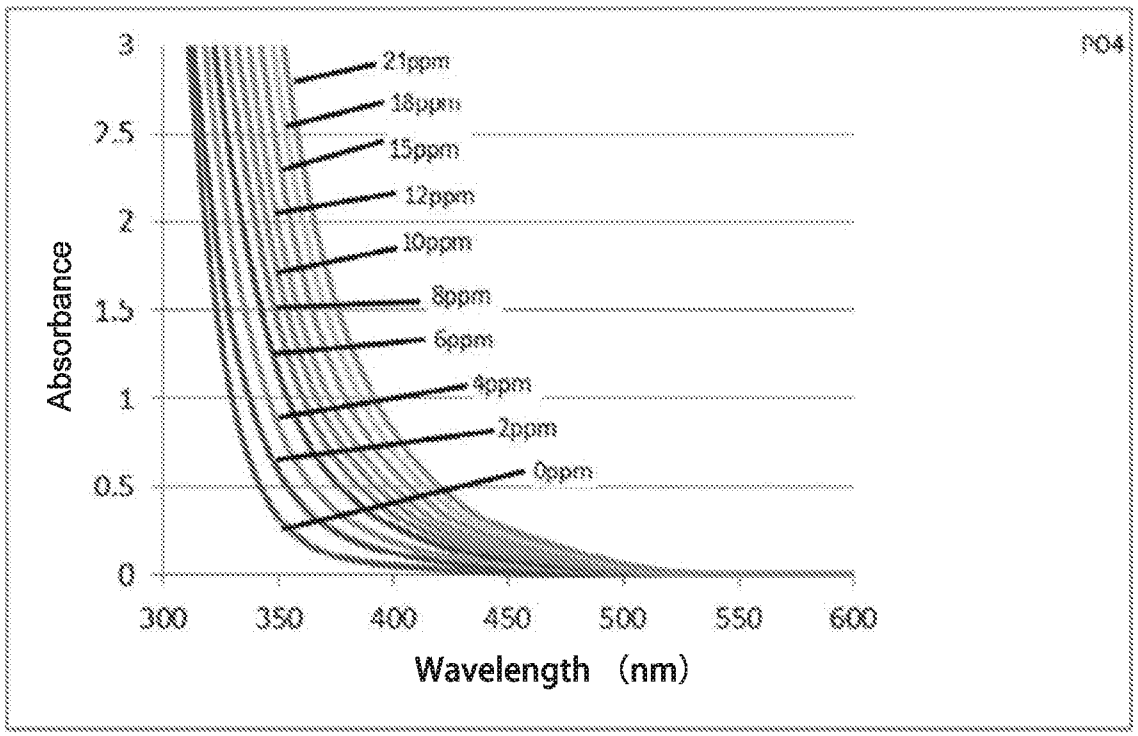
NO4
Absorbance
3
2.5
2
1.5
1
0.5
0
21ppm
18ppm
15ppm
12ppm
10ppm
8ppm
6ppm
4ppm
2ppm
0ppm
300
350
400
450
500
550
600
Wavelength (nm)

Fig. 2

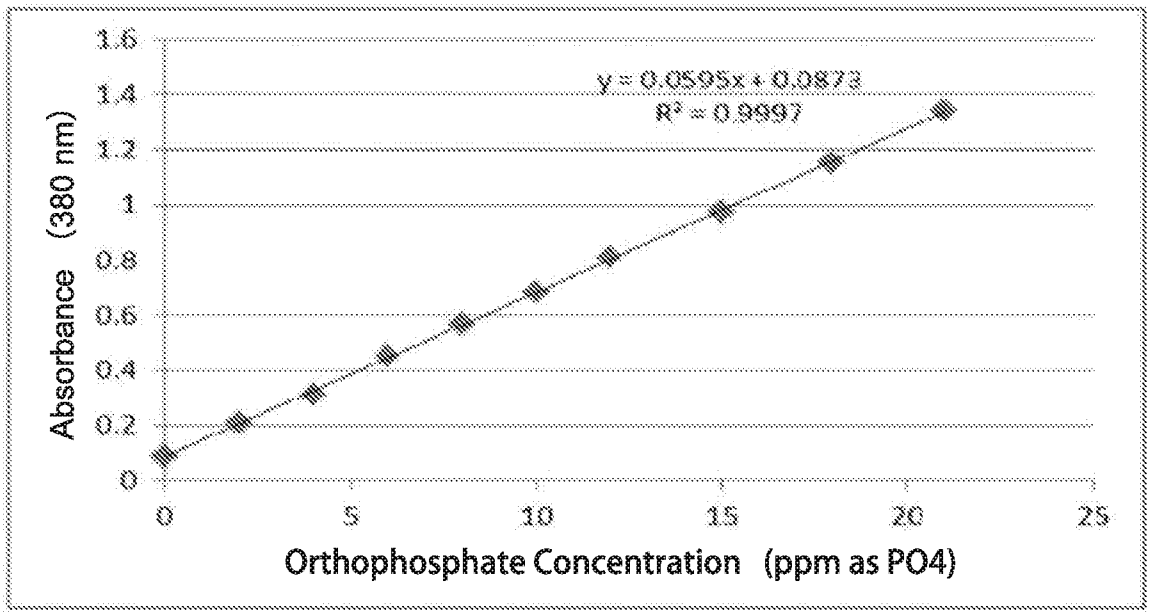
y = 0.0595x + 0.0873
R² = 0.9997
Absorbance (380 nm)
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
5
10
15
20
25
Orthophosphate Concentration (ppm as PO4)

Fig. 3

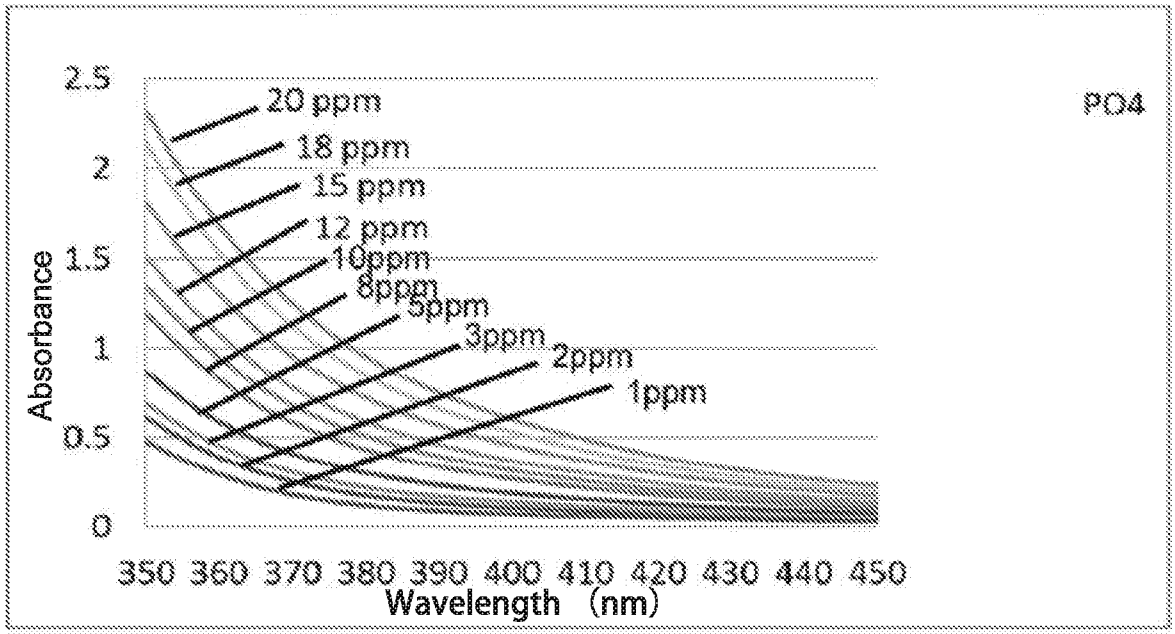
PO4
20 ppm
18 ppm
15 ppm
12 ppm
10ppm
8ppm
5ppm
3ppm
2ppm
1ppm
Absorbance
2.5
2
1.5
1
0.5
0
350 360 370 380 390 400 410 420 430 440 450
Wavelength (nm)

Fig. 4

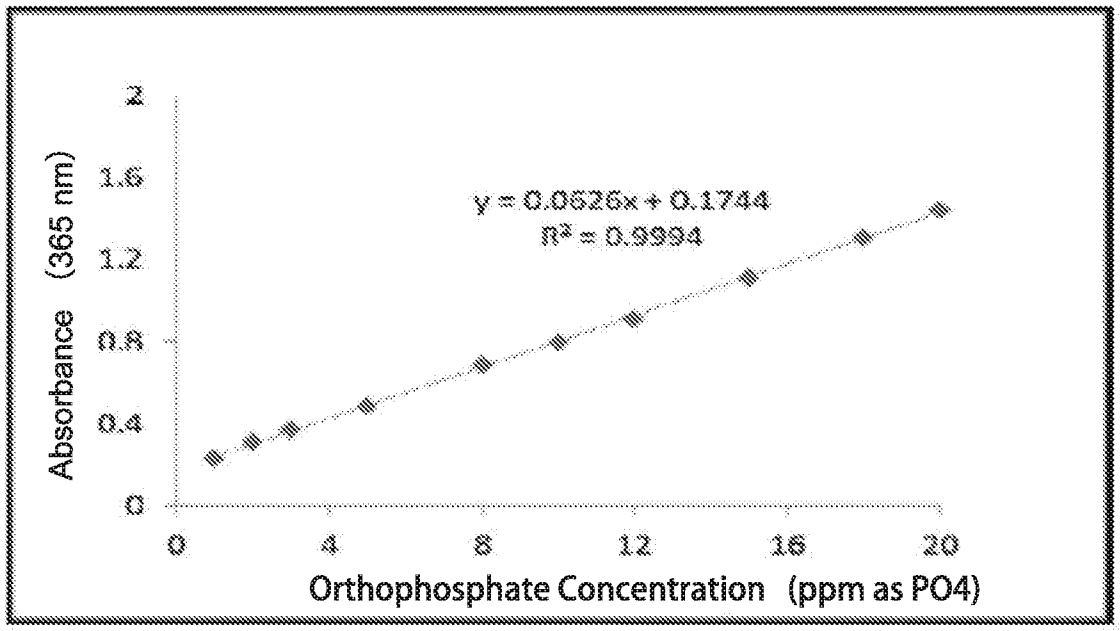
Absorbance (365 nm)
2
1.6
1.2
0.8
0.4
0
y = 0.0626x + 0.1744
R² = 0.9994
0
4
8
12
16
20
Orthophosphate Concentration (ppm as PO4)

Fig. 5

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2016/018031

A. CLASSIFICATION OF SUBJECT MATTER
IPC (2016.01) G01N 33/18, G01N 21/78

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 (2016.01) 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 practicable, search terms used)
Databases consulted: Esp@cenet, Google Patents, Google Scholar, FamPat database
Search terms used: ORTHOPHOSPHATE , MOLYBDATE , VANADATE , COLOR , SOLID ACID , INDUSTRIAL WATER

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Gee A. et al. "Determination of Phosphate by Differential Spectrophotometry" (1953) Analytical Chemistry, Vol. 25, No. 9, Pages 1320-1324, DOI: 10.1021/ac60081a006 (September 1953). 30 Sep 1953 (1953/09/30) Pages 1320,1322	1-19
A	HACH company , "MATERIAL SAFETY DATA SHEET , MSDS NO. M00297" , (2006) [online], [retrieved on 2016- 05-09]. Retrieved from the Internet <URL: http://www.camlab.co.uk/originalimages/sitefiles/MSDS/Hach/HH_2767245.pdf >. 31 Dec 2006 (2006/12/31) Pages 1-3	1-19
A	KR 20130115526 A UNIV. OF SEOUL INDUSTRY CO. FOUNDATION 22 Oct 2013 (2013/10/22) Abstract	1-19
A	Davies J. L. et al. "Comparison of the Stannous Chloride and Vanadate Methods for Estimation of Serum Inorganic Phosphorus by Use of the "SMA 12/60"" (1973) Clinical Chemistry, Vol. 19, No.4 , Pages 411-414 (April 1973). 30 Apr 1973 (1973/04/30) Page 411	1-19

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 May 2016	22 May 2016
Name and mailing address of the ISA: Israel Patent Office Technology Park, Bldg.5, Malcha, Jerusalem, 9695101, Israel Facsimile No. 972-2-5651616	Authorized officer GUTMAN Ariel Telephone No. 972-2-5657816

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2016/018031

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Quinlan K. P. et al." Spectrophotometric Determination of Phosphorus as Molybdo-vanadophosphoric Acid" (1955) Analytical Chemistry, Vol. 27, No. 10, Pages 1626-1629 DOI: 10.1021/ac60106a039 (Oct. 1955). 31 Oct 1955 (1955/10/31) Page 1627	1-19
A	Michelsen O. B." Photometric Determination of Phosphorus as Molybdovanado-phosphoric Acid" (1957) Analytical Chemistry, Vol. 29, No. 1, Pages 60-62, DOI: 10.1021/ac60121a017 (Jan. 1957). 31 Jan 1957 (1957/01/31) Page 60	1-19
P,A	US 2015198540 A1 ECOLAB USA INC. 16 Jul 2015 (2015/07/16) Abstract ; Para. [0057]	1-19

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/US2016/018031

Patent document cited search report	Publication date	Patent family member(s)	Publication Date
KR 20130115526 A	22 Oct 2013	KR 20130115526 A	22 Oct 2013
		KR 101340766 B1	11 Dec 2013
US 2015198540 A1	16 Jul 2015	US 2015198540 A1	16 Jul 2015
		WO 2015105722 A1	16 Jul 2015