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**KIM et al.**(10) **Pub. No.: US 2009/0139875 A1**(43) **Pub. Date: Jun. 4, 2009**(54) **ELECTROLYTE FOR ELECTRO-CHEMICAL  
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**C25F 3/02** (2006.01)(52) **U.S. Cl.** ..... **205/684**; 205/674; 205/685(57) **ABSTRACT**

Disclosed is an electrolyte for electrochemical machining of a metal product, which can reduce a defect of an electrochemical machining product, increase a lifespan of an electrolyte and an electrode, and improve efficiency of the electrochemical machining. The electrolyte includes an inorganic salt and at least one of a complexing agent and a reducing agent in a solvent.

## ELECTROLYTE FOR ELECTRO-CHEMICAL MACHINING OF METAL PRODUCT

### CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the priority of Korean Patent Application No. 2007-123791 filed on Nov. 30, 2007, in the Korean Intellectual Property Office, the disclosure of which is incorporated herein by reference.

### BACKGROUND OF THE INVENTION

#### [0002] 1. Field of the Invention

[0003] The present invention relates to an electrolyte for electrochemical machining of a metal product, and more particularly, to an electrolyte for electro-chemical machining of a metal product, which can reduce a defect of an electrochemical machining product, increase a lifespan of an electrolyte and an electrode, and improve efficiency of the electrochemical machining.

#### [0004] 2. Description of the Related Art

[0005] In general, electrochemical machining (ECM) is a machining method that uses an electro-chemical reaction of a conductive material. In the ECM, an electrode with a specific shape and a workpiece that is to be machined are dipped in an electrolyte. The workpiece serving as an anode is separated by a predetermined gap from the electrode which serves as a cathode. Then, a current is applied therebetween, so that a shape of the electrode is machined onto the workpiece.

[0006] The ECM is advantageous in that machining can be performed regardless of hardness of the workpiece, and a complicated shape such as a curved shape can be machined even on the inside of a hole. Using an ultrashort pulse in the ECM process has made it possible to machine micro-patterns such as micro-grooves for a fluid dynamic bearing.

[0007] In the ECM process, a metal component is eluted because of the metal workpiece serving as an anode. The eluted metal component may form metal hydroxide, precipitating in the form of sludge. Such a metal precipitate deposit on a surface of the workpiece, a surface of the electrode or on an electrolyte bath, causing defects in micro ECM.

[0008] A composition of the electrolyte contains just a single or composite inorganic salt as an electrolyte to conduct an electric current. Thus, in the ECM process, a metal component of the workpiece is eluted and precipitates in the form of hydroxide in the electrolyte. For example, if a main component of the workpiece is iron (Fe), a large amount of iron hydroxide such as  $\text{Fe}(\text{OH})_3$  or  $\text{Fe}(\text{OH})_2$  precipitates as ECM proceeds.

[0009] The precipitate in the form of sludge distorts a shape of the workpiece or interrupts power supply, resulting in defective machining. To remove the precipitate, a method of physically removing the precipitate by using a filter press or a centrifuge has been conventionally employed. However, this method also has limitations in that an electrolyte must be changed when the amount of precipitates increases above a predetermined level.

[0010] Therefore, there is a need for an electrolyte that can improve quality of a workpiece and efficiency of ECM by

reducing metal precipitates generated in an electrolyte in an ECM process for a metal product.

### SUMMARY OF THE INVENTION

[0011] An aspect of the present invention provides an electrolyte for electro-chemical machining of a metal product, which can reduce a defect of an electrochemical machining product, increase a lifespan of an electrolyte and an electrode, and improve efficiency of the electrochemical machining.

[0012] According to an aspect of the present invention, there is provided an electrolyte for electrochemical machining for a metal product, including: an inorganic salt in a solvent; and at least one of a complexing agent and a reducing agent. The metal may be one of iron (Fe), copper (Cu), nickel (Ni), aluminum (Al), tin (Sn), chrome (Cr), zinc (Zn) and an alloy thereof. The solvent may be pure water.

[0013] The inorganic salt may be at least one of  $\text{NaNO}_3$ ,  $\text{NaCl}$ ,  $\text{NaClO}_4$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{KNO}_3$ ,  $\text{KCl}$ ,  $\text{KClO}_4$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{LiNO}_3$ ,  $\text{LiCl}$ ,  $\text{LiClO}_4$ , and  $\text{Li}_2\text{SO}_4$ . The inorganic salt may have a concentration ranging from 100 g/L to 500 g/L with respect to a volume of the entire electrolyte.

[0014] The complexing agent may be at least one of citric acid, acetic acid, oxalic acid, fulvic acid, succinic acid, tartaric acid, lactic acid, gluconic acid, maleic acid, malic acid and a salt thereof.

[0015] Particularly, the complexing agent may be acetic acid. In detail, the complexing agent may be at least one of ethylenediaminetetraacetic acid, hydroxyethylethylenediaminetriacetic acid, and nitrilotriacetic acid.

[0016] The complexing agent may have a concentration ranging from 0.5 g/L to 20 g/L with respect to a volume of the entire electrolyte.

[0017] The reducing agent may be at least one of L-ascorbic acid, D-isoascorbic acid and a salt thereof. The reducing agent may have a concentration ranging from 0.5 g/L to 20 g/L with respect to a volume of the entire electrolyte.

[0018] The electrolyte may further include a corrosion inhibitor, a surfactant, a viscosity modifier and a pH adjuster.

[0019] pH of the electrolyte may range from pH 2 to pH 7.

### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[0020] Preferred embodiments of the present invention will be described below in more detail. The present invention may, however, be embodied in different forms and should not be construed as limited to the embodiments set forth herein. Rather, these embodiments are provided so that this disclosure will be thorough and complete, and will fully convey the scope of the present invention to those skilled in the art.

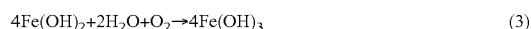
[0021] An electrolyte for electrochemical machining for a metal product according to an embodiment of the present invention contains an inorganic salt, and at least one of a complexing agent and a reducing agent in a solvent.

[0022] The electrolyte is prepared by dissolving the inorganic salt in water. The inorganic salt usable for the electrolyte according to the embodiment of the present invention may be at least one of  $\text{NaNO}_3$ ,  $\text{NaCl}$ ,  $\text{NaClO}_4$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{KNO}_3$ ,  $\text{KCl}$ ,  $\text{KClO}_4$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{LiNO}_3$ ,  $\text{LiCl}$ ,  $\text{LiClO}_4$ , and  $\text{Li}_2\text{SO}_4$ . A concentration of the inorganic salt adequate for an electrochemical machining (ECM) process may range from 100 g/L to 500 g/L with respect to a volume of the entire electrolyte.

**[0023]** Water may be used as a solvent for the electrolyte according to the current embodiment of the present invention. In this case, the electrolyte is an aqueous solution. The water may be pure water without a salt.

**[0024]** Any metal product may be used provided that it can be machined by the ECM process. Metal of the metal product may be one of iron (Fe), copper (Cu), nickel (Ni), aluminum (Al), tin (Sn), chrome (Cr), zinc (Zn) and an alloy thereof.

**[0025]** Hereinafter, metal precipitation in an ECM process of a metal product will now be described by using iron as an example. In the case of a product containing iron, iron is eluted in an electrolyte from a product surface during the ECM process. Iron hydrates in the electrolyte as expressed in the following Reaction formulas 1, 2 and 3:



**[0026]** As shown in Reaction formula 1, iron of the metal product is eluted as bivalent iron ions. As shown in Reaction formula 2, the bivalent iron ions in the electrolyte react with hydroxide ions in the electrolyte, thereby forming  $\text{Fe}(\text{OH})_2$ . Thereafter, the  $\text{Fe}(\text{OH})_2$  hydrates to form  $\text{Fe}(\text{OH})_3$ . The iron hydroxide precipitates in the form of sludge. Thus, to prevent the precipitation or remove the metal hydroxide, i.e., the iron hydroxide, having precipitated in the form of sludge, the electrolyte for electrochemical machining according to the embodiment of the present invention contains at least one of a complexing agent and a reducing agent.

**[0027]** The complexing agent forms a chemically stable chelate compound with metal. Thus, in the electrolyte, metal stably exists in an ion state, so that precipitation is prevented.

**[0028]** Any complexing agent may be used provided that it can form a complex compound with the metal and has no significant influence on the ECM process. The complexing agent may be at least one of citric acid, acetic acid, oxalic acid, fulvic acid, succinic acid, tartaric acid, lactic acid, gluconic acid, maleic acid, malic acid and a salt thereof.

**[0029]** Particularly, the complexing agent may be acetic acid, in detail, at least one of ethylenediaminetetraacetic acid, hydroxyethylethylenediaminetriacetic acid, and nitrilotriacetic acid.

**[0030]** A concentration of the complexing agent is varied according to a concentration of a metal ion in the electrolyte in the ECM operation. In general, the complexing agent may have a concentration of 0.5 g/L or higher so as to sufficiently function in the electrolyte. Also, the complexing agent may have a concentration of 20 g/L or lower to avoid significantly affecting physical properties of the electrolyte. That is, the complexing agent may have a concentration ranging from 0.5 g/L to 20 g/L with respect to a value of the entire electrolyte.

**[0031]** The electrolyte according to the embodiment of the present invention may contain a reducing agent. The reducing agent reduces metal hydroxide to form metal hydroxide having higher solubility, so that metal precipitates can maintain their dissolving state in the electrolyte.

**[0032]** This will now be described in more detail by using iron hydroxide as an example.  $\text{Fe}(\text{OH})_3$  forms within a wide PH range from pH 2, whereas  $\text{Fe}(\text{OH})_2$  forms within a pH range of pH 7 to pH 9. That is, generating  $\text{Fe}(\text{OH})_2$  in the electrolyte is more preferable than generating  $\text{Fe}(\text{OH})_3$  for-

mation in the electrolyte. Accordingly, precipitation can be prevented by reducing  $\text{Fe}(\text{OH})_3 \rightarrow \text{Fe}^{3+}$  to  $\text{Fe}(\text{OH})_2 \rightarrow \text{Fe}^{2+}$  having higher solubility.

**[0033]** Any reducing agent may be used for the present invention provided that it can reduce metal and has no significant influence on the ECM process. For example, the reducing agent may be at least one of L-ascorbic acid, D-isoascorbic acid, and a salt thereof.

**[0034]** In the ECM process, a concentration of the reducing agent is also varied according to a concentration of a metal ion. In general, the reducing agent may have a concentration of 0.5 g/L or higher so as to sufficiently function in the electrolyte. Also, the reducing agent may have a concentration of 20 g/L or lower to avoid significantly affecting physical properties of the electrolyte. That is, the reducing agent may have a concentration ranging from 0.5 g/L to 20 g/L with respect to a volume of the electrolyte.

**[0035]** The electrolyte for electrochemical machining for a metal product according to the current embodiment may further contain at least one of a corrosion inhibitor, a surfactant, a viscosity modifier and a pH adjuster in order to improve physical properties thereof. The corrosion inhibitor is an additive for inhibiting corrosion of the electrode. The surfactant, the viscosity modifier and the pH adjuster are additives for controlling physical properties to improve machining characteristics of the electrolyte.

**[0036]** The electrolyte for electrochemical machining for a metal product according to the embodiment of the present invention may have pH ranging from pH 2 to pH 7. As described above, in the case of iron hydroxide,  $\text{Fe}(\text{OH})_3$  having a higher oxidation number forms in pH 2 or higher, and  $\text{Fe}(\text{OH})_2$  reduced to have a lowered oxidation number forms within a pH range of pH 7 to pH 9. Since  $\text{Fe}(\text{OH})_2$  reduced by the reducing agent forms within a range of pH 7 to pH 9, precipitation of  $\text{Fe}(\text{OH})_2$  can be further prevented by controlling the pH range of the electrolyte within a range of pH 2 to pH 7.

#### Embodiments

**[0037]** In embodiments below, electrolytes for electrochemical machining for a metal product were prepared according to the embodiment of the present invention, metal ions (e.g., iron ions) were added to check an effect of removing metal precipitates in these electrolytes, and removal rates thereof were calculated.

**[0038]** First, to prepare an electrolyte, sodium nitride ( $\text{NaNO}_3$ ) as an inorganic salt was dissolved in pure water serving as a solvent, thereby preparing a  $\text{NaNO}_3$  aqueous solution of about 150 g/L. A predetermined amount of ferric nitride ( $\text{Fe}(\text{NO}_3)_3 \cdot 12\text{H}_2\text{O}$ ) was added to the solution. Thus, the electrolyte contained iron ions at a predetermined concentration. The electrolyte was controlled at neutral pH by using a sodium hydroxide ( $\text{NaOH}$ ) solution of about 0.1M. As a result, ochre yellow precipitates were observed with the naked eye.

**[0039]** It was observed whether the precipitates were removed, adding a predetermined amount of a complexing agent and a reducing agent in the electrolyte. Also, concentration changes of the iron ions were measured using an inductively coupled plasma-atomic emission spectrometry (ICP-AES). In the experiment, EDTA, NTA, citric acid and HEDTA were used as the complexing agent, and L-ascorbic acid and D-isoascorbic acid were used as the reducing agent.

**[0040]** The precipitate removal rate was calculated by a method of comparing a concentration of iron dissolving in the

electrolyte with a concentration of iron added to an initial reference electrolyte. That is, centrifugal separation was performed on the reference electrolyte containing initial precipitates. Since the most of iron precipitates in the form of iron hydroxide, almost no iron was detected in an electrolyte supernatant separated from the precipitates when an iron concentration of the supernatant separated from the precipitates was measured.

**[0041]** In contrast, when the complexing agent or the reducing agent for precipitate removal was added to the reference electrolyte, iron components in the precipitates dissolve in the electrolyte. Thus, the precipitates were removed and the iron concentration of the electrolyte increased. The precipitate removal rate in the electrolyte was measured by comparing the concentration of iron added to the initial electrolyte with the concentration of iron in the electrolyte after addition of an additive such as the complexing agent or the reducing agent. The precipitate removal rates according to the kinds and amounts of the added complexing agents and the reducing agents are shown in Tables 1 through 3 below.

TABLE 1

| Embodiments  | Complexing agent | g/L | Fe concentration (mg/L) |                 | Precipitate removal rate (%) |
|--------------|------------------|-----|-------------------------|-----------------|------------------------------|
|              |                  |     | Added amount            | Measured amount |                              |
| Embodiment1  | EDTA             | 1   | 808                     | 156             | 19                           |
| Embodiment2  | EDTA             | 3   | 808                     | 594             | 74                           |
| Embodiment3  | EDTA             | 5   | 808                     | 748             | 93                           |
| Embodiment4  | EDTA             | 7   | 808                     | 762             | 94                           |
| Embodiment5  | HEDTA            | 1   | 808                     | 126             | 16                           |
| Embodiment6  | HEDTA            | 3   | 808                     | 460             | 57                           |
| Embodiment7  | HEDTA            | 5   | 808                     | 701             | 87                           |
| Embodiment8  | HEDTA            | 7   | 808                     | 732             | 91                           |
| Embodiment9  | NTA              | 1   | 808                     | 170             | 21                           |
| Embodiment10 | NTA              | 3   | 808                     | 711             | 88                           |
| Embodiment11 | NTA              | 5   | 808                     | 754             | 93                           |
| Embodiment12 | NTA              | 7   | 808                     | 754             | 93                           |
| Embodiment13 | Citric acid      | 1   | 808                     | 196             | 24                           |
| Embodiment14 | Citric acid      | 3   | 808                     | 662             | 82                           |
| Embodiment15 | Citric acid      | 5   | 808                     | 772             | 95                           |
| Embodiment16 | Citric acid      | 7   | 808                     | 753             | 93                           |

**[0042]** In the embodiments 1 through 16, EDTA, HEDTA, NTA and citric acid are respectively contained as complexing agents in respective electrolytes at a concentration ranging from 1 g/L to 7 g/L.

**[0043]** It can be seen from Table 1 that, when the electrolyte contains the complexing agent as in the embodiments 1 through 16, iron is removed and a higher precipitate removal rate is obtained as the concentration of the complexing agent increases.

TABLE 2

| Embodiments  | Reducing agent     | g/L | Fe concentration (mg/L) |                 | Precipitate removal rate (%) |
|--------------|--------------------|-----|-------------------------|-----------------|------------------------------|
|              |                    |     | Added amount            | Measured amount |                              |
| Embodiment17 | L-ascorbic acid    | 1   | 808                     | 364             | 45                           |
| Embodiment18 | L-ascorbic acid    | 3   | 808                     | 682             | 84                           |
| Embodiment19 | L-ascorbic acid    | 5   | 808                     | 690             | 85                           |
| Embodiment20 | L-ascorbic acid    | 7   | 808                     | 766             | 95                           |
| Embodiment21 | D-isoascorbic acid | 1   | 808                     | 155             | 19                           |
| Embodiment22 | D-isoascorbic acid | 3   | 808                     | 630             | 78                           |
| Embodiment23 | D-isoascorbic acid | 5   | 808                     | 764             | 95                           |
| Embodiment24 | D-isoascorbic acid | 7   | 808                     | 776             | 96                           |

**[0044]** In the embodiments 17 through 24, L-ascorbic acid and D-isoascorbic acid are contained as reducing agents in respective electrolytes at a concentration ranging from 1 g/L to 7 g/L.

**[0045]** It can be seen from Table 2 that when the electrolyte contains the reducing agent as in the embodiments 17 through 24, iron is removed and a higher precipitate removal rate is obtained as the concentration of the reducing agent increases.

TABLE 3

| Embodiments   | Complexing agent | Reducing agent  | g/L | Fe Concentration |                 | Precipitate removal rate (%) |
|---------------|------------------|-----------------|-----|------------------|-----------------|------------------------------|
|               |                  |                 |     | Added amount     | Measured amount |                              |
| Embodiment 25 | NTA              | L-ascorbic acid | 1   | 882              | 789             | 89                           |
|               |                  | L-ascorbic acid | 1   |                  |                 |                              |
| Embodiment 26 | NTA              | L-ascorbic acid | 1   | 882              | 818             | 93                           |
|               |                  | L-ascorbic acid | 3   |                  |                 |                              |
| Embodiment 27 | NTA              | L-ascorbic acid | 1   | 882              | 816             | 93                           |
|               |                  | L-ascorbic acid | 5   |                  |                 |                              |
| Embodiment 28 | NTA              | L-ascorbic acid | 3   | 882              | 835             | 95                           |
|               |                  | L-ascorbic acid | 1   |                  |                 |                              |
| Embodiment 29 | NTA              | L-ascorbic acid | 3   | 882              | 853             | 97                           |
|               |                  | L-ascorbic acid | 3   |                  |                 |                              |

TABLE 3-continued

| Embodiments   | Complexing agent | Reducing agent  | Fe Concentration |              | Precipitate removal rate (%) |
|---------------|------------------|-----------------|------------------|--------------|------------------------------|
|               |                  |                 | g/L              | Added amount |                              |
| Embodiment 30 | NTA              |                 | 3                | 882          | 98                           |
|               |                  | L-ascorbic acid | 5                |              |                              |
| Embodiment 31 | NTA              |                 | 5                | 882          | 94                           |
|               |                  | L-ascorbic acid | 1                | 826          |                              |
| Embodiment 32 | NTA              |                 | 5                | 882          | 95                           |
|               |                  | L-ascorbic acid | 3                | 839          |                              |
| Embodiment 33 | NTA              |                 | 5                | 882          | 94                           |
|               |                  | L-ascorbic acid | 5                | 832          |                              |

**[0046]** In the embodiments 25 through 33, electrolytes contain NTA as a complexing agent and L-ascorbic acid as a reducing agent at various concentrations.

**[0047]** Referring to Table 3, when both complexing and reducing agents are added in the electrolyte as in the embodiments 25 through 33, the precipitate removal rate of 89% or higher is calculated. Particularly, in the embodiment 25, a high precipitate removal rate of 89% is obtained although NTA and L-ascorbic acid each are added at relatively low concentrations of 1 g/L.

**[0048]** Referring to Tables 1 and 2, iron precipitates are removed when the electrolyte contains the complexing agent as in the embodiments 1 through 16 and when the electrolyte contains the reducing agent as in the embodiments through 17 through 24. Also, the precipitate removal rate increases as the concentration of each of the complexing and reducing agents increases.

**[0049]** Thus, it can be seen that eluted metal ions such as iron ions can be removed at a high rate when one of the complexing agent and the reducing agent is contained in the electrolyte for electro-chemical machining for a metal product. Particularly, as seen from the experimental results, more than 90% of the precipitates can be removed when the complexing agent or the reducing agent is contained at a concentration of 5 g/L or higher.

**[0050]** According to the embodiments 25 through 33, both complexing and reducing agents are added in the electrolyte. In this case, equivalent precipitate removal rates can be obtained even if smaller amounts of complexing and reducing agents are used as compared to when one of the complexing agent and the reducing agent is used as in the embodiments 1 through 24.

**[0051]** Iron (Fe) is used in the current embodiments. However, the present invention is not limited thereto, and may be applied to a product formed of metal such as Cu, Ni, Al, Sn, Cr, Zn or an alloy thereof. Even in this case where the product is formed of another metal besides iron, metal precipitates can be removed similarly with regard to the influence of the complexing and reducing agents on metal.

**[0052]** In the electrolyte for electrochemical machining for a metal product according to the embodiments of the present invention, generation of metal participates can be prevented in an ECM process. Accordingly, precipitates can be prevented from depositing on an electrode or a workpiece in the

ECM process, thereby preventing process defects of the ECM process and improving efficiency of the ECM process.

**[0053]** Also, a lifespan of the electrolyte for electrochemical machining increases, contributing reduction of a cost for the ECM process. Since an additional process for metal-precipitate removal is not required, a process time can be saved and thus productivity can be improved.

**[0054]** While the present invention has been shown and described in connection with the exemplary embodiments, it will be apparent to those skilled in the art that modifications and variations can be made without departing from the spirit and scope of the invention as defined by the appended claims.

What is claimed is:

1. An electrolyte for electrochemical machining for a metal product, comprising:
  - an inorganic salt in a solvent; and
  - at least one of a complexing agent and a reducing agent.
2. The electrolyte of claim 1, wherein the metal product comprises one selected from the group consisting of iron (Fe), copper (Cu), nickel (Ni), aluminum (Al), tin (Sn), chrome (Cr), zinc (Zn) and an alloy thereof.
3. The electrolyte of claim 1, wherein the solvent is pure water.
4. The electrolyte of claim 1, wherein the inorganic salt is at least one selected from the group consisting of NaNO<sub>3</sub>, NaCl, NaClO<sub>4</sub>, Na<sub>2</sub>SO<sub>4</sub>, KNO<sub>3</sub>, KCl, KClO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub>, LiNO<sub>3</sub>, LiCl, LiClO<sub>4</sub>, and Li<sub>2</sub>SO<sub>4</sub>.
5. The electrolyte of claim 1, wherein the inorganic salt has a concentration ranging from 100 g/L to 500 g/L with respect to a volume of the entire electrolyte.
6. The electrolyte of claim 1, wherein the complexing agent is at least one selected from the group consisting of citric acid, acetic acid, oxalic acid, fulvic acid, succinic acid, tartaric acid, lactic acid, gluconic acid, maleic acid, malic acid and a salt thereof.
7. The electrolyte of claim 1, wherein the complexing agent is at least one selected from the group consisting of ethylenediaminetetraacetic acid, hydroxyethylethylenediaminetriacetic acid, and nitrilotriacetic acid.
8. The electrolyte of claim 1, wherein the complexing agent has a concentration ranging from 0.5 g/L to 20 g/L with respect to a volume of the entire electrolyte.

**9.** The electrolyte of claim **1**, wherein the reducing agent is at least one selected from the group consisting of L-ascorbic acid, D-isoascorbic acid and a salt thereof.

**10.** The electrolyte of claim **1**, wherein the reducing agent has a concentration ranging from 0.5 g/L to 20 g/L with respect to a volume of the entire electrolyte.

**11.** The electrolyte of claim **1**, further comprising a corrosion inhibitor, a surfactant, a viscosity modifier and a pH adjuster.

**12.** The electrolyte of claim **1**, wherein pH of the electrolyte ranges from pH 2 to pH 7.

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