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Short Communication

Recipe for Ferric Salts of Ethylenediaminetetraacetic Acid

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ABRAM A. STEINER AND HELEN VAN WINDEN
Centre for Plant Physiological Research, P.O. Box 52, Wageningen, The Netherlands

The supply of iron to plant roots was one of the main problems in water culture until about 1950. The main source of iron was originally ferrous sulfate, already used by Sachs (9). It had to be supplied almost daily to the nutrient solution as it precipitates gradually. Some improvement was obtained with organic compounds, mainly ferric citrate, introduced by Gile and Carrero (4). The favorable effect of natural humates on plants in water culture was noticed by Bottomley (1). Olsen (8) interpreted this as a natural formation of iron humate complexes, resulting in experiments with iron humate. Horner *et al.* (6) developed a synthetic humate, capable of holding iron in solution. Unfortunately, this humate is difficult to prepare, besides which all humates are of indeterminate composition. Iron humates and other sources such as iron lignin sulfonate (2) and iron metaphosphate (3) gave satisfactory results for certain plants, but none of these compounds has been generally accepted.

After a publication by Jacobson (7), ferric salts of ethylenediaminetetraacetic acid soon met with general acceptance in water culture. One addition of FeEDTA proved to be sufficient for many weeks. Thanks to Jacobson, FeEDTA became the main source of iron for the greater part of water culture.

Jacobson's original recipe is as follows:

It is convenient to prepare the complex by dissolving 26.1 gm ethylene diamine tetra-acetic acid in 268 ml of 1.0 N KOH, then adding 24.9 gm $FeSO_4 \cdot 7\,H_2O$ and diluting to one litre. After aerating overnight to produce the stable ferric complex, the pH should be about 5.5. One ml of this solution provides 5 ppm to one litre nutrient solution.

When this recipe is followed, the pH is 2.4 to 2.6 directly after preparation, and 3.5 to 3.6 after 12 hr of intensive aeration, instead of the required pH about 5.5. It was evident that not all the ferrous iron changed into chelated ferric form. A good criterion for a complete reaction is the color of the solution, which should be dark yellow-brown (No. 156, Senf color chart).

Jacobson's recipe has been repeated in many other publications, including Hewitt's standard work on water culture methods (5). In a personal communication Hewitt concluded that no unfavorable effect is produced from using an acid EDTA preparation provided the nutrient solution in which it will be used has a pH of at least 5.5; all the iron will still change into the chelated form. This would actually happen if iron were the only heavy metal in the nutrient solution. However, some copper, zinc, and manganese will also be present. Under certain circumstances these metals will be preferred for incorporation into the chelate (10).

To make up the FeEDTA solution to pH about 5.5, 276 ml of 1 N KOH are needed instead of 268 ml as given in the original recipe. The pH will then be about 5.5 after 12 hr of aeration.

During the preparation of the FeEDTA solution, another more practical difficulty arose: that of dissolving the ferrous sulfate. It gave a precipitate of $Fe(OH)_3$. To overcome this, the ferrous sulfate must be dissolved in water containing 4 ml of 1 N H_2SO_4 before being added to the solution containing EDTA and KOH. Because of the addition of sulfuric acid, the amount of required KOH has to be increased from 276 to 280 ml.

With the use of FeEDTA, some other elements besides iron and EDTA are introduced, sulfur and potassium. This makes the preparation unsuitable for plant physiological experiments on potassium and sulfate deficiency. For this reason, the recipe had to be changed. It has been worked out for chloride instead of sulfate, and sodium or ammonium instead of potassium in all possible combinations. All calculations are based on the international atomic weights of the elements. The amounts of the various compounds to be used for 1 liter of iron solution are given in Table I.

With the exception of the NH_4-containing chelates, the complete recipes, based on the amounts given in Table I, are as follows:

A. Dissolve 26.2 g of EDTA in about 500 ml of hot water (about 70 C) containing 279.8 or 283.6 ml of 1 N KOH or 281.6 or 284.4 ml of 1 N NaOH (see Table I).

B. Dissolve 24.9 g of $FeSO_4 \cdot 7H_2O$ or 17.8 g of $FeCl_2 \cdot 4H_2O$ in about 300 ml of hot water (about 70 C), containing 4 ml of 1 N H_2SO_4 or HCl (see Table I).

C. Mix A and B and add water to about 950 ml, aerate vigorously for 12 hr, then make to 1000 ml with water.

To prepare the NH_4-containing chelates, another procedure is necessary:

I. Dissolve 26.2 g of EDTA in about 500 ml of water (about 50 C) containing 179.0 ml of 1 N NH_4OH.

II. The same as B above.

III. Mix I and II and add water to about 850 ml, aerate vigorously for 12 hr.

Table I. *Quantities of Reagents Required for 1 Liter of Iron Solution Containing 5000 mg Fe*

Solution	EDTA	$FeSO_4 \cdot 7H_2O$	$FeCl_2 \cdot 4H_2O$	1 N H_2SO_4	1 N HCl	1 N KOH	1 N NaOH	1 N NH_4OH
	g	*g*	*g*	*ml*	*ml*	*ml*	*ml*	*ml*
K + SO_4	26.2	24.9	...	4	...	279.8	...	...
K + Cl	26.2	...	17.8	...	4	283.6	...	...
Na + SO_4	26.2	24.9	...	4	...	...	281.6	...
Na + Cl	26.2	...	17.8	...	4	...	284.4	...
NH_4 + SO_4	26.2	24.9	...	4	...	...	...	276.0
NH_4 + Cl	26.2	...	17.8	...	4	...	...	276.0

IV. Add 97.0 ml of 1 N NH_4OH, make to 1000 ml with water, and aerate vigorously for 1 hr.

The pH of all solutions will be about 5.5. All solutions will contain 5000 mg of Fe per liter or 268.6 meq Fe^{3+} (89.5 mmoles of Fe) and 183.1 meq SO_4^{2-} or Cl^- per liter. The amounts of K^+, Na^+, and NH_4^+ in meq per liter can be found in Table I.

Because of the high price of the acid form of EDTA, the disodium salt of EDTA will sometimes be preferred. This, of course, can only be used for solutions which need not be free of sodium. For this purpose the recipe given above must be changed as follows:

1. Dissolve 33.3 g of disodium EDTA in about 500 ml of warm water (about 30 C), containing 100.4 ml of 1 N NaOH if $FeSO_4$ is used, or 103.0 ml of 1 N NaOH if $FeCl_2$ is used.

2. The same as B and C above.

The amount of Na^+ will be 279.4 or 282.1 meq, the amount of SO_4^{2-} or Cl^-, 183.1 meq, all per liter in the SO_4 or Cl solutions, respectively. The iron content will be 5000 mg.

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