Procedure for testing of EDTA solution for Cleaning of Boilers

1.0 The testing of EDTA for chemical cleaning of boilers involves three tests. These tests are conducted at vendors' works before dispatch to construction site as well as during actual chemical cleaning process during EDTA pickling. These tests are:

A. Determination of Total EDTA concentration in the test sample of ammoniated EDTA.
B. Testing pH value of test sample.
C. Testing specific gravity of test sample.

All three tests are described in this document.

2.1 Determination of Total EDTA concentration in the test sample.

2.1 **Principle:**
EDTA reacts with Zirconium ions under strong acid conditions to form a "chelate". The end point of the titration is indicated by a Xylenol orange indicator.

2.2 **Reagents and Apparatus Required:**

2.2.1 **Apparatus:**

- Volumetric flasks 100 ml capacity (2 nos.)
- Volumetric flasks 1000 ml capacity (1 nos.)
- Conical flasks 100 ml capacity. (2 nos.)
- Pipettes (5 ml – 1 nos.)
- Pipettes (10 ml – 1 nos.)
- Hot plate (Coil heater is not recommended)
- Burette (50 ml) with stand.
- Sample bottles (4 nos.)

2.2.2 **Safety Applications**

- Water Source for eye/ face protection wash.
- Safety apron/ gloves/ chemical shoes.

2.2.3 **Reagents:**

- Hydrochloric acid (concentrated, analytical grade). Prepare 1:1 HCl Solution from analytical grade (36%) HCl.
- Standard Zirconium oxychloride ($ZrOCl_2.8H_2O$) 0.1 M solution in 1 N HCl. (Take 1000 ml volumetric flask and 32.23 gram of $ZrOCl_2.8H_2O$ and add DM water to about 500 ml. To this, add 85 ml of concentrated HCl. Make up to 1000 ml mark with DM water and mix well)
- Ascorbic acid.
- Xylenol orange indicator.

2.3 **Testing Procedure:**

i. Pipette out 5 cc of the (40.0%, to be tested) ammoniated EDTA sample solution into a 100 ml volumetric flask and make up with DM water up to the mark.

ii. Pipette out 10 cc of the above diluted sample into a 100 ml conical flask.

iii. Add 5-6 ml of 1:1 HCl to the conical flask containing diluted solution of EDTA.

iv. Add a 0.5 gm of ascorbic acid.

v. Boil the solution on the hot plate for about 5 minutes. (The yellow color of the ferric ion, if any present in the solution should disappear due to reduction to ferrous iron.)

vi. Cool the solution and add 0.05 ml of 0.05 % xylenol orange indicator, the yellow colour reappears. The sample is titrated with 0.1M $ZrOCl_2$ until the color changes from yellow to red violet. The colour change should persist for at least 3 minutes.

2.4 **Calculations:**

i. The total EDTA w/v % = Volume of 0.1M $ZrOCl_2$ consumed x 5.84

ii. The total EDTA w/w % = (w/v) / Specific gravity

3.0 **Determination of pH.**

The determination of pH of the sample can be carried out by checking the test sample with pH meter or with the help of universal indicator.

The pH of the fresh EDTA sample can also be checked by universal indicator. For checking pH using universal indicator, take 10 ml solution and add 2-3 drops of universal indicator and compare the colour with colour strip given on the universal indicator bottle for different pHs.

4.0 **Determination of specific gravity.**

The specific gravity of the test sample is checked by either specific gravity meter (hydrometer) or specific gravity bottle.

4.1 **Specific gravity using Hydrometer:**

The hydrometer may be dipped in the solution and note the reading. The reading on the scale will indicate the specific gravity of the EDTA solution.

4.2 **Specific gravity using Specific Gravity Bottle.**

Take weight of the empty specific gravity bottle (capacity – 25 ml). Fill the specific gravity bottle with EDTA solution up to the mark and weigh. Find out the weight of the EDTA solution by subtracting the weight of the empty bottle.

Now fill the specific gravity bottle with water up to the mark and weigh. Find out the weight of the water by subtracting the weight of empty bottle

The ratio of two results determined above will give the specific gravity of the EDTA solution.

5.0 **Acceptance Criteria:**

i. pH – more than 9.5 (9.5 to 9.8)

ii. Specific Gravity – 1.17 to 1.18

iii. Percentage of active EDTA (w/v) – 47% to 48%
Percentage of active EDTA (w/w) – 40% or above.

6.0 **Grade of chemicals used for testing**: All chemicals for analysis purposes are required to be analytical grade only.