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Analysis of Morpholine in Water by UV Visible Spectrophotometer

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Abstract

Feed water is the aggregate of the total of condensate return and boiler make up water. The reasons to control pH are low pH or insufficient alkalinity can result in corrosive acidic attack, dissolved oxygen can cause pitting loss of protective oxide film $4\text{Fe}_3\text{O}_4 + \text{O}_2 \rightarrow 6\text{Fe}_2\text{O}_3$ & high pH or excess alkalinity can result in caustic gouging/cracking and foaming, with resultant carryover $\text{Fe}_3\text{O}_4 + 4\text{NaOH} = 2\text{NaFeO}_2 + \text{Na}_2\text{FeO}_2 + \text{H}_2\text{O}$. The corrosion rate of carbon steel at feed water temperatures approaches pH > 8.5. To maintain pH of feed water (>8.5) small amounts of Morpholine is dosed. It is completely miscible in water and stable in hot liquid and gaseous phases at all temp. Morpholine has the ability to maintain corrosion protection by neutralizing acid constituents such as a carbonic acid, throughout the entire boiler system.

Keywords- Morpholine, Alkalinity, Corrosion, Spectrophotometer etc.

Introduction & Principle-

Test method IS:12084 is available for analysis of purity of Morpholine (Minimum 99%) but no test method is available for analysis of Morpholine content at ppm level in water samples. **In the present work, a test method has been developed for analysis of Morpholine at ppm level (by UV spectrophotometer) in water samples.** The analysis for morpholine in boiler water is based upon morpholine's reaction to the sodium salt of 1,2 naphthoquinone sulphonic acid to

form a yellow coloured product with a minimum light transmittance at 480 nm. The intensity of the colour is determined by spectrophotometer and is directly proportional to the amount of morpholine in the range of 0 to 10 mg/L.

Reagent and chemical:

- Standard solution of morpholine.
10000 mg/L = Pipette 10 ml of 100 % morpholine into 1 litre volumetric flask and dilute with Millipore water.
100 mg/L = Pipette 10 ml of 10000 mg/L morpholine into 1 litre volumetric flask and dilute with Millipore water
- Iodine solution: 0.025 N.
- Sodium bicarbonate (10 %),
- Starch solution (1%).
- Indicator solution (sodium salt of 1, 2 naphthoquinone sulphonic acid).
Dissolved 0.3 gm in 10 ml of water. This solution must be prepared fresh every 24 hr when needed.

Apparatus & Glasswares:

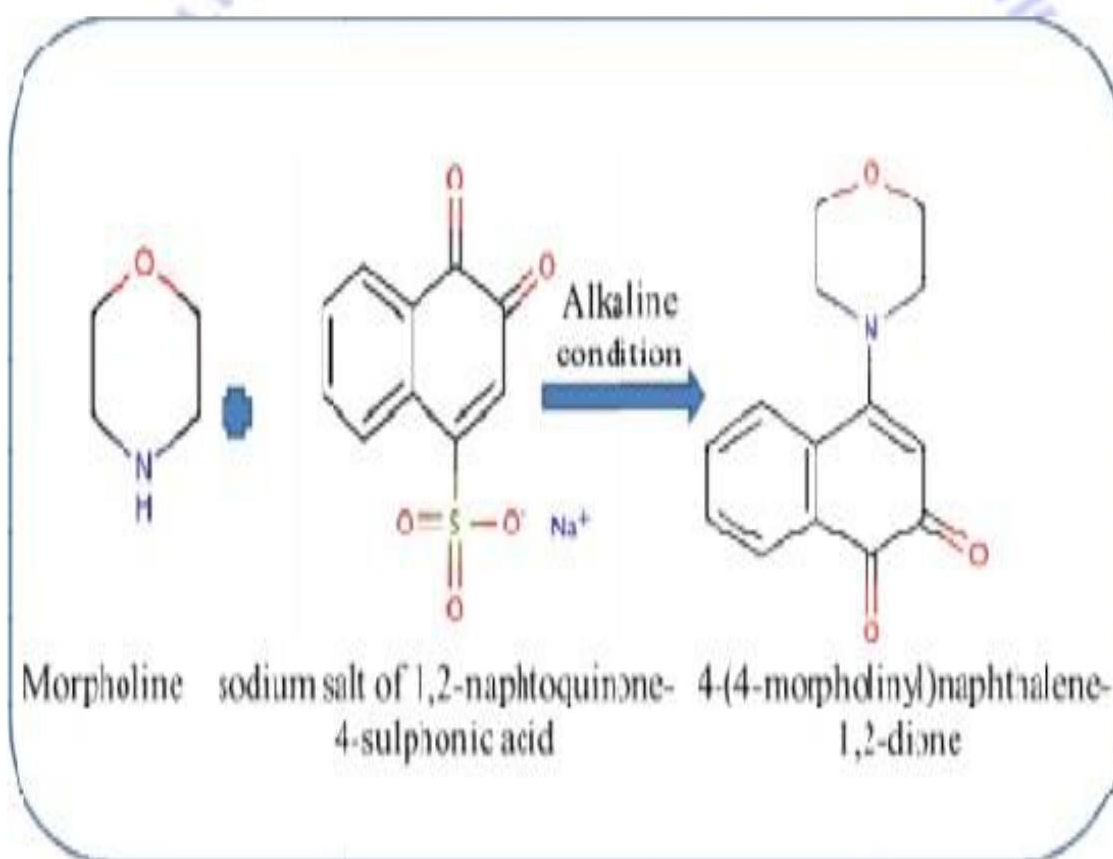
- UV Visible Spectrophotometer
- Volumetric flask 100 mL
- Pipette 1,5,10 mL
- Measuring cylinder 100 mL

Procedure for Calibration:

- Take 1,2,3,4,5 ml from 100 mg/L stock solution of Morpholine in 100 ml Volumetric flask
- Add 40 ml distilled water in each flask.
- **For sample** take 50 ml sample.
- Add 1 ml NaHCO_3 in each flask and mix well.
- Add 1 ml starch solution in each flask and mix well.
- With swirling, titrate the contents of each flask with Iodine solution to permanent blue end point & transfer the titrate content of each flask to a graduated cylinder, dilute to 60

ml and return to its flask.

- Add 4 drops of indicator solutions to each flask swirl. Place the flask into water bath (55°C to 65°C). Loosen the stopper of flask and with a thermometer in blank, heat the sample to 40-43°C.
- Remove flask from the bath and let them stand for 3-4 minutes & make up to 100 ml with Millipore water.
- Read the absorbance value on spectrophotometer at 480 nm of standards and sample using blank.
- Draw calibration curve and with its help read conc. of morpholine in the sample.

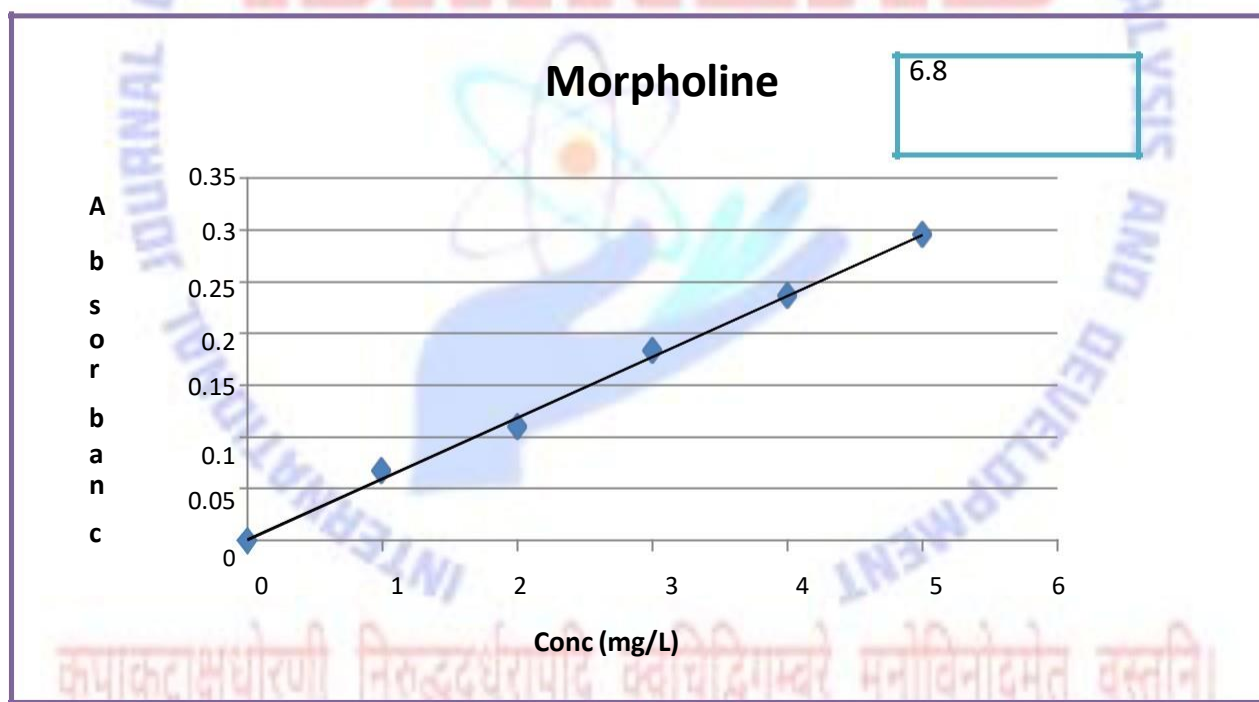


Calibration of Morpholine in Water Sample:

1 mg/L to 5 mg/L Morpholine doses in Millipore water sample to make calibration curve.

Sr No	Morpholine Conc in sample (mg/L)	Absorbance of Sample	Factor
1	0	0	0
2	1	0.0674	14.837
3	2	0.1094	18.332
4	3	0.1824	16.447
5	4	0.2364	16.92
6	5	0.2946	16.972

Calculation-



Morpholine (mg/L)

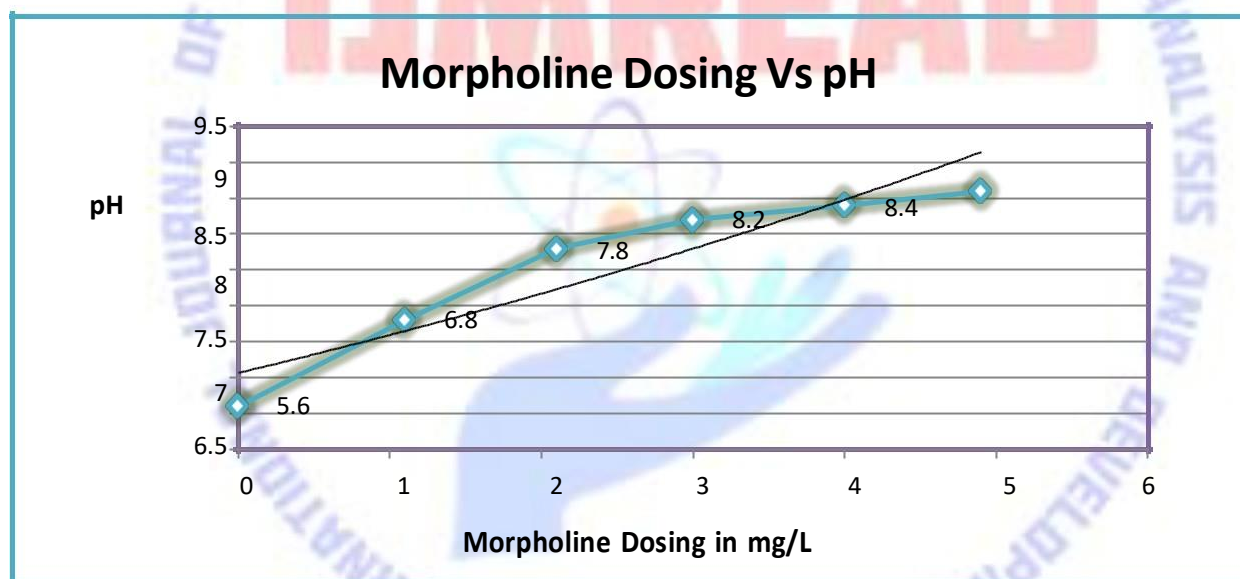
$$= \frac{h_i \left(\frac{1}{50} \right) * (100)}{me(50)}$$

Test Results- The test results of Morpholine in water samples are as follows:

<i>Sl. No.</i>	<i>Sample</i>	<i>Plant</i>	<i>Date</i>	<i>Time</i>	<i>Morpholine (mg/L)</i>	<i>pH</i>
1	Boiler feed pump	CPP	08.08.2018	7:00	3.2	8.5
2	Boiler feed pump	CPP	09.08.2018	7:00	3.0	8.5
3	Boiler feed pump	CPP	11.08.2018	7:00	2.4	8.6
4	Boiler feed pump	CPP	13.08.2018	7:00	2.4	8.7
5	Boiler feed pump	CPP	15.08.2018	7:00	2.6	8.5
6	Boiler feed pump	CPP	17.08.2018	7:00	2.8	8.5
7	Deaerator Feed Pump	CPP	08.08.2018	7:00	2.9	8.6
8	Deaerator Feed Pump	CPP	09.08.2018	7:00	2.8	8.5
9	Deaerator Feed Pump	CPP	11.08.2018	7:00	2.4	8.5
10	Deaerator Feed Pump	CPP	13.08.2018	7:00	2.6	8.6
11	Deaerator Feed Pump	CPP	15.08.2018	7:00	2.6	8.5
12	Deaerator Feed Pump	CPP	17.08.2018	7:00	2.8	8.4
13	Polished Condensate	CPP	08.08.2018	7:00	3.0	7.8
14	Polished Condensate	CPP	09.08.2018	7:00	4.0	8.4
15	Polished Condensate.	CPP	11.08.2018	7:00	4.0	8.3
16	Polished Condensate	CPP	13.08.2018	7:00	4.0	8.2
17	Polished Condensate	CPP	15.08.2018	7:00	2.2	7.7
18	Polished Condensate	CPP	17.08.2018	7:00	3.4	7.8

Validation of method and recovery Check:

Sr No	Sample ID	Date	Time	Morpholine Dosing (mg/L)	pH
1	Millipore Water	08.08.2018	07:00 hr	0	5.6
2	Millipore Water	08.08.2018	07:00 hr	1.1	6.8
3	Millipore Water	08.08.2018	07:00 hr	2.1	7.8
4	Millipore Water	08.08.2018	07:00 hr	3	8.2
5	Millipore Water	08.08.2018	07:00 hr	4	8.4
6	Millipore Water	08.08.2018	07:00 hr	4.9	8.6

**Conclusion-**

By the newly developed method results are repeatable and verified by recovery of Morpholine in water. Results by newly developed method are validated by increase in pH as concentration of Morpholine increase from 1-2 ppm in water but no significant alter in pH if morpholine doses > 2 ppm in water. In-house Developed test method can be used for the analysis of morpholine feed water at ppm level.

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