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QUIZ

Analyst Fall 2004

Chemical Cleaning of an Industrial Boiler, an Overview

1. High heat-flux boiler tube surfaces mainly consist of:

   A. Anhydrite and Copper
   B. Calcite and Hematite
   C. Magnetite and Copper
   D. Brucite and Hydroxyapetite

2. Obtaining tube samples is the most reliable and effective means to determine the need for chemical cleaning. The tube sample should be at least:

A. 1 foot long
B. 2 feet long
C. 3 feet long
D. 4 feet long

3. Potassium Permanganate degreasing is used when:

A. The deposit contains primarily light oil and grease.
B. Scale contains considerable quantities of calcium sulfate.
C. Deposit consists primarily of mill scale and organic contamination.
D. Organic contamination is heavy and carbonized.

4. If copper concentration in the deposit is greater than _____%, a separate treatment is necessary to dissolve copper as much as possible before solvent cleaning.

A. 2%
B. 5%
C. 8%
D. 10%

5. Inhibited Hydrochloric Acid is the most widely used solvent because:

A. It can be used to clean Stainless Steel vessels
B. It is economical and easy to handle
C. It is effective with Copper greater than 30 %
D. It is effective cleaning oil and greases

6. Which chemical is effective with high- content copper scale, and is also compatible with steel alloy?

A. Hydrochloric Acid
B. Sulfuric Acid
C. Citric Acid
D. EDTA

7. Which chemical is generally considered expensive when comparing descaling solvents?

A. Citric Acid
B. EDTA
C. Hydrochloric Acid
D. Sulfuric Acid

8. Which chemical is considered more dangerous to handle than the others in the list?

A. Hydrochloric Acid
B. Sulfuric Acid
C. EDTA
D. Citric Acid

9. At what stage is pressure-water jetting most effective in removing loosened deposits?

   A. High pressure wash before Acid Cleaning
   B. High pressure wash after Acid Cleaning.
   C. High pressure wash before neutralization.
   D. High pressure wash without chemical additives.

10. Which of the following chemical combinations should be used after neutralization and passivation of a boiler when Citric Acid or EDTA has been used?

   A. Citric Acid, Ammonia and Nitrite
   B. Citric Acid, Ammonia and Nitrate
   C. Citric Acid, Nitrite and Caustic
   D. Nitrate, Phosphate and Carbonate

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Chemical Cleaning of an Industrial Boiler - An Overview

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Abstract

The internal surfaces of a boiler in contact with water and steam must be kept clean and free of deposits to assure efficient heat transfer in the generation of steam. This article discusses when and how to chemically clean a boiler. Several methods can be used to determine the need to chemically clean a boiler. The high heat flux boiler tube surfaces mainly consist of magnetite and copper. The article outlines cost effective cleansing solvents for chemically cleaning boilers with magnetite and copper deposits. The chemical cleaning must be thoroughly planned. Criterion for determining the success of a chemical procedure and the effectiveness of the solvent in dissolving the deposit is covered in detail.

Introduction

Water-side scale build-up in boilers is a progressive, inevitable process. Even with stringent control of feedwater and condensate chemistry, scale and deposition will occur. The main problems caused by boiler scales are:

- increase in tube wall temperature, hence, boiler tube ruptures
- decrease in overall boiler efficiency, hence, increase in energy cost and loss of reliability

The increase in tube wall temperature is a result of the low thermal conductivity of scales as compared to metal. The reduction in heat-transfer can lead to the design temperature of the tube wall being exceeded, which in turn may lead to failure of the tube by creep rupture.

Overall efficiency can be defined as the ratio of steam output to the fuel consumption ratio. Again, since scaling impedes heat transfer, more fuel is required to produce a given amount of steam, thus reducing overall efficiency and loss of energy.

Eventually, removal of scale from the boiler becomes essential if damage to the boiler is to be prevented. One way of removing scale is to chemically clean the boiler. Chemical cleaning is a multiple stage process that seeks to remove all the existing scale from the boiler internals, leaving a clean, passivated waterside system. One step in the process involves the use of inhibited acid to dissolve the scales. This acid stage is potentially damaging to the boiler and therefore, it requires careful

monitoring to prevent serious metal loss.
This article reviews the criteria for chemically cleaning industrial water-tube boilers of operating pressures up to 900 psi that are used for steam generation in plant operation. The article covers the major cleaning steps, as well as the choice of particular chemicals to use in some steps, with a focus on the removal of iron and copper scales.

The Chemistry of Boiler Scales

The primary constituent of boiler scales is magnetite (Fe_3O_4), which is formed as a result of the reaction of metallic iron with high-temperature steam. Other crystalline materials, some shown in Table 1, may form the scale. Copper is present due to corrosion of the copper alloy, aluminum bronze feedwater condensers and pre-heaters, often because of oxygen ingress into these systems. Copper is transported through the steam cycle where it forms on the boiler internals. Other constituents shown in Table 1 are transported through the steam cycle and deposited on boiler internals either from contaminants contained in the boiler feedwater system or from use of outdated phosphate-based water treatment chemicals. In addition to these crystalline inorganic compounds, there may be organic residuals present in the scale.

Table 1: Compounds Found in Boiler Scales

Compound	Formula
Anhydrite	$CaSO_4$
Aragonite	$CaCO_3$
Brucite	$Mg(OH)_2$
Copper	Cu
Calcite	$CaCO_3$
Hematite	Fe_2O_3
Hydroxyapetite	$Ca_{10}(OH)_2(PO_4)_6$
Magnetite	Fe_3O_4
Quartz	SiO_2
Thenardite	Na_2SO_4
Wollastonite	$\beta CaSiO_3$

Chemical cleaning proposals must take into consideration the different compounds present in the scale in order to formulate the optimum cleaning solutions. This recommendation should effectively remove scale without damaging the underlying metal.

Determining the Need to Chemically Clean Boilers

The need to chemically clean an operating boiler is usually determined from an inspection of a particular unit performed during planned inspections. An inspection should target specific areas in the boiler known to be problematic areas of excessive corrosion or scaling.

Other factors influencing the decision of whether or not to chemically clean include:

- overall efficiency
- hot spots - as evidenced by infrared inspection
- tube failure during normal operation

If the data gathered during the normal inspection process indicates a possible need to chemically clean a boiler, obtaining tube samples is the most reliable and effective means. Tube samples should be removed from locations where heavy scaling is suspected. The tube sample should be at least three feet long so the method of removal (cut-off wheels and cutting torches) does not contaminate the scale at the center of the sample with slag or filings.

The scale is then analyzed using several different techniques in order to determine its composition. The scale density is determined gravimetrically after dissolution of scale in inhibited HCl. Loss of weight on firing in a furnace will measure the percentage of hydrocarbons present, which then determines the need for alkaline degreasing. The need to clean is based on the scale density. Table 2 shows the range of scale density encountered, along with the appropriate action required.

Table 2: Scale Density Ranges and Required Action

Scale Density g/ft^2 or (mg/cm^2)	Recommended Action
< 23 (25)	No action required
23 - 70 (25 - 75)	Chemically clean within one year
70 - 93 (75 - 100)	Chemically clean within three months
> 93 (100)	Chemically clean before further operation

The Steps to Boiler Chemical Cleaning and Treatment Selection

Cleaning a boiler usually consists of a combination of the following stages:

- mechanical cleaning
- water flushing

- alkaline treatment
- solvent cleaning
- neutralization and passivation

Mechanical cleaning and water flushing can remove loose scale and other debris from the boiler. Alkaline treatment removes oils and hydrocarbons that might interfere with the dissolution of the scale by acid solvents. The solvent cleaning stage is the process in which inhibited acid is used to remove scale form the boiler. Once the scale is removed in the solvent stage, fresh active metal is exposed. The neutralization and passivation stages are designed to remove any last traces of iron oxides and to replace the active metal with a well-passivated surface layer.

Fouling composition, quantity and distribution will vary considerably between one boiler to another and even within the same boiler at various times during its life span. It is therefore essential in each case to select a specific treatment or series of treatments that will be most effective in achieving thorough and safe cleaning to the desired standards. This segment provides a guide to the selection of treatments.

The primary criteria that must be satisfied are:

- The treatment must be safe to use and compatible with the materials of construction of the equipment to be cleaned.
- The foulant must show sufficient solubility in the treatment selected so when it is considered together with any insoluble matter that may reasonably be expected to detach fully during the process, it will achieve the desired degree of cleaning.

With these criteria satisfied, make the final selection with due reference to other constraints including cost, disposal problems and cleaning time available.

Chemical cleaning will normally involve one or more of the following steps:

- hot alkaline degreasing
- removal of copper
- acid solvent cleaning followed by neutralization and passivation treatment.

Laboratory evaluation of tube samples will determine the above sequence. High-pressure water jetting may follow to remove loosened scale. If high pressure water jetting is used, it must be followed by flash rust removal and passivation. Remote fiberscope inspection recorded on a videotape and tube removal for scale density analysis, before and after the chemical cleaning, will provide visual evidence on cleaning effectiveness.

Hot Alkaline Treatment Selection

If oil, grease, carbon or other organic compounds are present, they must be removed during chemical cleaning. Selection depends on the degree of contamination. Use hot alkaline treatment only when organic deposits interfere with solvent cleaning. If solubility of deposits is greater than 70 percent in solvent with or without addition of surfactants, then a separate alkaline boil out stage is not required.

Soda ash (Na_2CO_3) degreasing is a mild treatment used where contamination is primarily light oil and grease, with less than 5 percent organic contamination. Table 3 shows the control parameters for alkaline degreasing with soda ash.

Table 3: Control Parameters for Alkaline Degreasing with Soda Ash	
Chemical	**Concentration**
Sodium carbonate	0.5 to 1.0 % by weight
Sodium metasilicate	0.5 to 1.0 % by weight
Trisodium phosphate	0.5 to 1.0 % by weight
Surfactant	0.1 to 0.2 % by volume
Antifoam (if required)	0.05 to 0.1 % by volume
Temperature Limit	155 °C
Circulation	Normal boiler operational circulation
Residence Time	18 to 24 hours
Corrosion Rates	< 2 mpy

Caustic degreasing (NaOH) treatment is used for all new boilers, where mill scale is present or where organic contamination is 5 to 30 percent. Table 4 shows the control parameters for caustic degreasing.

Table 4: Control Parameters for Alkaline Degreasing with Caustic	
Chemical	**Concentration**
Sodium hydroxide	1.0 to 2.0 % by weight
Trisodium phosphate	0.5 to 1.0 % by weight
Surfactant	0.1 to 0.3 % by volume
Antifoam (if required)	0.05 to 0.1 % by volume
Temperature Limit	155 °C
Circulation	Normal boiler operational circulation
Residence Time	18 to 24 hours
Corrosion Rates	< 2 mpy

Potassium permanganate ($KMnO_4$) degreasing is used where organic contamination is heavy (> 30 percent) and carbonized. This treatment should only be employed where fouling type and quantity render it necessary, since costs, disposal problems and complication of subsequent solvent cleaning are greater than in the case of the alternatives. The control parameters alkaline degreasing with permanganate is shown in Table 5.

Table 5: Control Parameters for Alkaline Degreasing with Permanganate

Chemical	Concentration
Sodium hydroxide	1.0 to 2.0 % by weight
Potassium permanganate	1.0 to 3.0 % by weight
Temperature Limit	100 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	6 to 12 hours
Corrosion Rates	< 2 mpy

Where scales contain considerable quantities of calcium sulfate (i.e. 10 percent) a sulfate conversion treatment may be either beneficial or essential. This will tend to increase scale solubility during a subsequent solvent cleaning with an inorganic acid such as inhibited hydrochloric acid. The control parameters of sulfate conversion treatment are shown in Table 6.

Table 6: Control Parameters of Sulfate Conversion Treatment

Chemical	Concentration
Sodium carbonate	1.0 to 5.0 % by weight
Surfactant	0.1 to 0.2 % by volume
Temperature Limit	95 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	12 to 24 hours
Corrosion Rates	< 2 mpy

If copper concentration in the scale deposit is greater than 10 percent, a separate treatment is necessary to dissolve copper as much as possible before solvent cleaning. Make an estimate of copper to be removed from the scale analysis and use any one of the following two alkaline treatments to reduce the copper level below 10 percent. Other alkaline treatments with ammonium carbonate and sodium nitrite are also acceptable for the removal of copper greater than 10 percent. Any copper remaining in the boiler will be removed during the acid cleaning stage. Table 7 shows the control parameters of copper removal with ammonium bicarbonate, air or oxygen.

Table 7: Control Parameters of Copper Removal with Ammonium Bicarbonate, Air or Oxygen

Chemical	Concentration
Ammonium bicarbonate	1.6 kg/kg of copper to be removed
Aqua ammonia	2.4 liter/kg of copper to removed, and adjust pH to 9.5
Air or Oxygen	1.3 to 1.5 cubic meters per minute
Temperature	50 °C to 60 °C
Residence Time	2 to 4 hours
Corrosion Rates	< 2 mpy

Solvent/Acid Cleaning Selection

Hydrochloric Acid - Inhibited hydrochloric acid is a most widely used solvent since it produces good solubility with a wide variety of scales, is economical and is easy to handle. It shows good corrosion characteristics when adequately inhibited and the process is controlled within the accepted limits. The process is flexible and can be tailored to complex copper by the addition of thiourea to enhance silica removal by the addition of ammonium bifluoride or to remove organics by addition of surfactants. It is not compatible with stainless steels.

Where higher concentration of copper (greater than 10 percent) is present, a separate copper removal treatment is required prior to using the hydrochloric acid procedure. Table 8 indicates hydrochloric acid cleaning stage control parameters.

Table 8: Hydrochloric Acid Cleaning Stage Control Parameters

Chemical	Concentration
Hydrochloric acid	3.5 to 7.5 % by weight
Inhibitor	0.2 to 0.3 % by volume or as recommended by manufacturer
Surfactant	0.0 to 0.2 % by volume
Ammonium bifluoride	0.0 to 1.0 % by weight

Thiourea	0.0 to 1.5 % by weight (at 5 kg/kg of copper to be removed)
Oxalic acid	1.0 % by weight
Temperature	70 °C to 82°C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	8 to 18 hours
Corrosion Rates	< 600 mpy
Total dissolved Iron	10,000 mg/L maximum

Citric Acid - Citric acid is compatible with alloy steels, requires low chloride solvents and presents good handling, safety and corrosion characteristics in comparison to hydrochloric acid. It is less aggressive in its attack of some iron oxide scales and therefore usually requires higher temperatures or longer contacts times. It has a very limited effect on calcium salts present in boiler scales. In general, it is more expensive than hydrochloric acid treatment.

The normal reasons for its selection are:

- Presence of austenitic materials of construction.
- Extremely effective copper removal from high copper-content scales.
- Reduces cleaning time by eliminating the need to drain, flush and refill the boiler between stages because iron removal, copper removal, neutralization and passivation can be achieved using a single solution.

The citric acid cleaning stage control parameters are shown in Table 9.

Table 9: Citric Acid Cleaning Stage Control Parameters	
Chemical	**Concentration**
Iron Removal Phase	
Citric acid	2.5 to 5 % by weight
Inhibitor	0.2 to 0.3 % by volume or as recommended by manufacturer
Ammonia	To pH 3.5 to 4.0
Copper Removal and Passivation Phase	
Ammonia	To pH 9.5
Ammonium bicarbonate	1.0 % by weight

Sodium nitrite	0.5 % by weight
Temperature Limit: Iron removal phase Copper removal and passivation phase	79 °C to 93 °C 45 °C to 50 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	
Iron removal phase	4 to 8 hours
Copper removal and passivation phase	4 to 8 hours
Total dissolved Iron	10,000 mg/L maximum
Circulation Rate	1200 liters/minute to 4500 liters/minute
Corrosion Rates	< 660 mpy

EDTA - Ethylenediaminetetraacetic Acid (EDTA) salts are generally expensive in comparison with citric and hydrochloric acid. Higher temperatures are required to achieve satisfactory cleaning. Corrosion rates are low under properly controlled conditions, and iron oxide removal, copper removal, neutralization and passivation can be achieved sequentially with the single solution. The EDTA is circulated by normal boiler operation and air blowing. The provision of temporary circulating pumps, connections and pipe work can be largely eliminated. Shown in Table 10 are the control parameters of EDTA cleaning stage.

Table 10: Control Parameters of EDTA Cleaning Stage

Chemical	Concentration
Iron Removal Phase	
EDTA	3 to 10.0 % by weight
Inhibitor	0.2 to 0.3 % by volume or as recommended by manufacturer
Ammonia	Add enough to obtain pH 9.2
Copper Removal and Passivation Phase	
Sodium Nitrite	0.5 % by weight
Temperature Limit: Iron removal phase Copper removal and passivation phase	121 °C to 149 °C 60 °C to 71 °C

Circulation Limit	Natural circulation
Residence Time	12 to 18 hours
Corrosion Rates	< 200 mpy

Sulfuric Acid - Sulfuric acid is an effective solvent for iron oxides and iron sulfides and is generally lower in cost than hydrochloric acid. It is also compatible with stainless steels. However, it is considerably more dangerous to handle. Sulfuric acid in its concentrated form is aggressive to organic material and contact with the skin or eyes is extremely dangerous. Sulfuric acid is not recommended where scales contain significant calcium due to the formation of insoluble calcium sulfate. Table 11 shows the control parameters of sulfuric acid cleaning stage.

Table 11: Control Parameters of Sulfuric Acid Cleaning Stage

Chemical	Concentration
Sulfuric acid	4.0 to 8.0 % by weight
Inhibitor	Inhibitor 0.2 to 0.3 % by volume or as recommended by manufacturer
Surfactant	0.0 to 0.2 % by volume
Temperature Limit	60 °C to 82 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	4 to 12 hours
Corrosion Rates	< 600 mpy
Total dissolved Iron	10,000 mg/L maximum

Sulfamic Acid - Sulfamic acid has the advantage of being a crystalline solid, which is easy to store, handle and mix. It is frequently sold with an inhibitor and a color indicator of effective acid strength added. It is compatible with stainless steels and is a moderately aggressive solvent for iron oxide and calcium carbonate. Due to its relatively high cost, it is mainly used on low volume equipment. The control parameters of the sulfamic acid cleaning is shown Table 12.

Table 12: Control Parameters of Sulfamic Acid Cleaning Stage

Chemical	Concentration
Sulfamic acid	5.0 to 10.0 % by weight
Inhibitor	0.1 to 0.2 % by volume or as

Inhibito	recommended by manufacturer
Surfactant	0.0 to 0.2 % by volume
Temperature Limit	55 °C to 65 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	4 to 12 hours
Corrosion Rates	< 600 mpy
Total dissolved Iron	10,000 mg/L maximum

High Pressure Water Jetting (HPWJ)
HPWJ is very effective to remove loosened deposits. The use of HPWJ is recommended after acid cleaning and neutralization. After the HPWJ, always remove flash rust and passivate the boiler before start up. Table 13 shows the control parameters of high pressure water jetting.

Table 13: Control Parameters of High Pressure Water Jetting	
Equipment	**Specification**
Pump Capacities	750 kW, 68.94 MPa (10,000 psig) 1500 kW, 137.88 MPa (20,000 psig)
Water Volume	30 liters/minute to 50 liters/minute
Nozzle orifice diameter	0.8 to 2.4 mm
Number of Nozzles & Orientation	4 each, facing 45 ° rearwards and 90 ° to the direction of lance advancement
Maximum distance between nozzle tip and surface to be cleaned	25 mm
Flexible Hose diameter	19 mm minimum
Water for jetting	Cold steam condensate
Additives	**Concentration**
Polymer	0.3 % by volume
Surfactant	0.1 to 0.2 % by volume

Neutralization and Passivation
It is essential that the equipment be thoroughly neutralized following acidization of

It is essential that the equipment be thoroughly neutralized following acidization of a boiler. This is achieved by neutralization alone, usually with 0.5 percent sodium carbonate, or during the passivation treatment, where pH values of seven or greater are required by the process.

The selection of passivation treatment is sometimes governed by the selection of the solvent. Where citric acid or EDTA processes have been used, these are normally extended to effect neutralization and passivation by a suitable pH adjustment and the addition of an oxidizing agent. This is accomplished with citric acid, ammonia and nitrite or with carbonate/nitrite. If ambient temperature is all that can be achieved, the nitrite/phosphate treatment will provide some protection to the metal surfaces. If internal surfaces have been allowed to rust following acidization, this rusting is removed by citric acid. Ammonia and sodium nitrite are added later to achieve a high degree of passivation. The control parameters of neutralization and passivation using carbonate, phosphate and nitrite and citric acid, ammonia and sodium nitrite are shown in Tables 14, 15 and 16 respectively.

Table 14: Control Parameters of the Carbonate Neutralization

Chemical	Concentration
Sodium carbonate	0.5 to 1.5 % by weight
Sodium nitrite	0.5 % by weight
Temperature Limit	88 °C to 93 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	8 to 12 hours
Corrosion Rates	< 2 mpy

Table 15: Control Parameters of the Phosphates and Nitrite Neutralization

Chemical	Concentration
Sodium nitrite	0.5 % by weight
Monosodium phosphate	0.25 % by weight
Disodium phosphate	0.25 % by weight
Sodium hydroxide	Adjust pH to 7
Temperature Limit	50 °C to 65 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute

Residence Time	8 to 12 hours
Corrosion Rates	< 2 mpy

Table 16: Control Parameters of the Citric Acid, Ammonia, Sodium Nitrite Neutralization

Chemical	Concentration
Citric acid	2.5 % by weight
Ammonia	Adjust 4.0 during flash rust removal and adjust pH to 9.5 during passivation
Sodium nitrite	0.5 % by weight
Inhibitor	0.2 to 0.3 % by volume or as recommended by manufacturer
Temperature Limit	Flash rust removal 65 °C to 90 °C
Passivation stage	45 °C to 50 °C
Circulation Rate	1200 liters/minute to 4500 liters/minute
Residence Time	8 to 18 hours
Corrosion Rates	< 600 mpy during flash rust removal < 2 mpy during passivation

Chemical Cleaning Evaluation

Inspection of the boiler after the chemical cleaning is crucial to determine if the procedure has been successfully completed. Visual and video boroscope inspections determine the effectiveness of the cleaning. There should be no visible traces of water and loose or adherent scale inside the boiler drums and tubes. Remove the corrosion coupons and the polarization probes, visually examine them, determine their weight loss and calculate the loss of metal thickness (usually < 25 microns) due to chemical cleaning.

Scale Density after cleaning - Cut a tube sample after chemical cleaning and determine the density of any deposit. Use Table 17 for evaluating the effectiveness of chemical cleaning.

Table 17: Evaluating Effectiveness of Chemical Cleaning	
Deposit Present, g/ft^2; (mg/cm^2)	**Performance Guideline**
0.93; (1.0) or less	Best
Between 0.93 - 1.86; (1 - 2)	Better
Between 1.86 - 2.79; (2 - 3)	Good
Between 2.79 - 4.65; (3 - 5)	Acceptable
> 4.65; (5)	Flash rust removal 65 °C to 90 °C
Passivation stage	Not Acceptable

Conclusion

It is essential to clean a boiler periodically for efficient operation, corrosion control, reliability and prevention of tube failures. Cleaning is accomplished by a combination of steps. For some boilers it may not be necessary to use all the cleaning steps, since the degree of contamination will vary from one boiler to another. The exact procedure to be used depends upon the scale density and its analysis, tube bulging or failure, water treatment analysis, inspection and history or the unit itself. The formulations recommended do not override the boiler manufacturer's chemical cleaning recommendations especially where warranty rights are involved.

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