

**TECHNICAL NOTE
AND
GENERAL GUIDE FOR
CORROSION COUPONS**

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GENERAL GUIDE FOR CORROSION COUPONS

SECTION 1 Introduction

SECTION 2 The Corrosion Coupon

SECTION 3 General theory

SECTION 4 Location of Corrosion Monitoring Points

SECTION 5 Proposed Corrosion Coupon Programmes

SECTION 6 Handling of Coupons

SECTION 7 Evaluation of Exposed Coupons

SECTION 8 Recording of Data

APPENDIX I Cleaning of Exposed Coupons

APPENDIX II Coupon Report Form

APPENDIX III Deposit Analysis procedures

APPENDIX IV Glossary of Terms

SECTION 1 - INTRODUCTION

Corrosion coupons are a simple tool, used in the quantitative estimation of corrosion rates occurring in the system being monitored.

Corrosion coupons also have the advantage of providing a visual indication of the type of corrosion which is occurring in the system which is being monitored.

We must point out at this time that the evidence obtained by a corrosion coupon must not be construed as an exact duplication of what is occurring in the system. The evidence is an approximation or an indication of the problem which may be occurring, and it's possible effects on the system.

The corrosion coupon, although it is an elementary tool, is still an accurate method of monitoring corrosion. Ideally, weight loss coupons are placed side by side with electrical resistance or linear polarisation type probes in order to define fluctuation in corrosion rates.

SECTION 2 - THE CORROSION COUPON

The Corrosion Coupon:

- a) is a piece of metal of predetermined shape, size and surface area.
- b) is a metal of very similar chemical composition to the process equipment.
- c) has a uniform reproducible surface finish
- d) has a distinguishing serial number
- e) has a predetermined weight measured to the nearest 0.0001 grams
- f) is stored in a protective container
- g) is exposed to the same corrosive medium as the process facilities
- h) is exposed for a fixed period of time
- i) gives an indication of the type of corrosion
- j) gives an indication of the average rate of corrosion

SECTION 3 - GENERAL THEORY

Coupon Corrosion Monitoring systems are designed for two coupon or coupon pair exposure. If a pair of coupons of the same material are exposed for the same length of time, in the same orientation, subject to the same environment, their weight loss and corrosion rate should be identical.

If all conditions are held constant except the period of exposure, the corrosion rate is not usually the same. The presence of corrosion products will alter the rate of corrosion depending on the type of attack.

For the above two reasons coupon pairs are used. In this way it is possible to achieve, ideally 30 day, 60 day, 90 day, etc. exposure periods plus regular 30 day checks to obtain immediate rates. Depending on the long term rates, one can establish if the corrosion products reduce or aggravate the corrosion rate. The short term rates can be used to help correlate with deviations in the long term trends in corrosion rates.

One must bear in mind, that coupons do not give an exact indication of what is actually happening, but indicate the general trends involved. In actual fact a coupon cannot be considered to be of exactly the same type of material and by physical shape and location, cannot be subject to exactly the same type of service condition.

With this knowledge, it is realised that the corrosion type and rate shown by corrosion coupons indicates the general trend of what is occurring in the system. By manipulating service conditions such as temperature, pressure, flow rates, inhibitor types, inhibitor injection rates, it is possible to alter the corrosion rate in the proper direction. The greater the reduction in corrosion rate indicated by the corrosion coupon, the greater (proportional) the reduction of the corrosion rate in the system.

The coupon, because of its physical shape and location is capable of detecting other problems, such as erosion and fouling. If such problems are detected, then by strategic placement of other monitoring devices, they can be more accurately measured and the required corrective action taken to alleviate the conditions.

SECTION 4 - LOCATION OF CORROSION MONITORING POINTS

The choice of monitoring points is governed by the type of system and the number of changes applied to the medium in that system. Ideally, the system should be monitored before and after any change in process variable. Examples are as follows:

1. Impressed change in temperature (exchangers)
2. Impressed change in pressure (PRC, FRC)
3. Removal of a chemical component (distillation)
4. Addition of any chemical (inhibitor, combining streams)
5. Areas of phase change (flash drum, condensers, etc.)

In addition, monitoring should be carried out in areas where multiphase flow exists. If gas and liquids exist in the same stream, one can never be sure that the mixture is homogeneous, therefore, three critical zones can exist:

1. Gas phase (gas or gas entrained liquids)
2. Gas-liquid phase interface
3. (a) Liquid phase (liquid or liquid with entrained gas) +
(b) Liquid phase (two immiscible liquids with entrained gas)

A multiphase system makes it necessary to monitor in each phase and/or across the phase interface to obtain more meaningful data. The phase interface is a very critical area particularly if a finite level is maintained.

The choice of locations for corrosion monitoring points is governed by the type of system, process conditions and past experience with that system.

SECTION 5 - PROPOSED CORROSION COUPON PROGRAMME

PROGRAMME A

This programme uses a 30 day and multiples of 30 day periods as an initial check on a system. This type of programme is normally used on a system which shows low corrosion rates with a long term deceleration trend within a 60 to 150 day period.

PROGRAMME B

this programme uses a 15 day and multiples of the 15 day period. this type of programme can be reverted to and used in a system which shows high corrosion rates with erratic long term trends. The programme can also be used to monitor corrosion rates in a system where control procedures are being applied on a test basis or are being altered in an attempt to reduce corrosion rates or types.

CONVERSION

These types of programmes allow for conversion from A to B or B to A without any serious loss of data continuity. See following pages for programme layout.

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I = Install Coupon
P = Pull Coupon

SECTION 6 - HANDLING OF COUPONS

The critical aspects of coupon handling are those which:

- a) do not alter the reproducible surface and weight of a new coupon before exposure.

- b) do not alter the deposit, corroded surface and weight of an exposed coupon before analysis can be completed.
- c) do not alter the interpretation of data.

The most common of the problems encountered in the handling of corrosion coupons are listed below:

- i) recording errors in coupon number.
- ii) recording errors in coupon weight.
- iii) recording errors in coupon installation and removal date.
- iv) recording errors in coupon exposure location.
- v) improper storage of new coupons allowing for atmospheric corrosion and contamination.
- vi) improper manual handling which allows for contamination of coupon surface by:
 - a) foreign agents (grease, oil, dirt, moisture, etc.)
 - b) human body oils which tend to act as inhibitors.
 - c) human perspiration which tends to act as activating agents.
- vii) mechanical damages of new coupons which can:
 - a) result in weight loss by surface abrasion.
 - b) result in stress zones which are more susceptible to corrosion action.
- viii) improper mounting which allows for:
 - a) loss of coupons if bolts are not properly secured.
 - b) galvanic corrosion if insulating washers are lost.
 - c) mechanical damage and loss of corrosion products if bolts are loose and coupons vibrate excessively.

HANDLING OF COUPONS

Continued

- ix) improper alignment when in service which allows for excessive erosion and corrosion product removal.
- x) improper installation which results in mechanical damage to coupon and mounting hardware. This results in the problems indicated in vii) and viii).

xi) improper removal which results in mechanical damage to exposed coupon will give:

- a) an error in weight loss.
- b) incorrect analysis of corrosion deposit because of losses.
- c) incorrect assessment of damage because of surface distortion.

xii) improper cleaning procedures which result in incorrect weight loss because of:

- a) incomplete removal of corrosion product.
- b) removal of metal by excessive cleaning.
- c) incomplete drying of cleaned coupon.

Although many of the points listed above appear very elementary, they are critical to the collection of meaningful and accurate data.

SECTION 7 - EVALUATION OF EXPOSED COUPON

Since one of the main advantages of corrosion coupons is the estimation of surface effects, special attention must be paid to the appearance of the coupon before and after cleaning. Some of the terms used in the following procedures and descriptions may seem obscure at the present time, but their significance will become clearer as the personnel become familiar with coupon evaluation. A glossary of terms used can be found in Appendix IV.

In the following portion of this section are detailed descriptions of the information required at various stages of coupon processing.

BEFORE CLEANING

The initial analysis of an exposed coupon involves an accurate description of its condition as soon as it is pulled out of service. The following information is required:

1. The presence or absence of a deposit.
2. If there is no deposit, record "No Deposit".
3. If there is a deposit, indicate:
 - (a) Its colour.
 - (b) Distribution - all over, patchy, where located.
 - (c) Texture - smooth, rough, mottled, dry, oily.
 - (d) Physical properties - hard, soft, solid, porous.
 - (e) Adherence - firmly attached, loose.
 - (f) Thickness - estimate thickness and if it is uniform.
 - (g) Magnetic property if any should be indicated. Test with a magnet.

After a description of the deposit is recorded, a chemical analysis for the presence of the following should be performed:

- (a) Sulphide ion
- (b) Carbonate ion
- (c) Ferrous ion
- (d) Ferric ion
- (e) Hydrocarbon

See Appendix iii for deposit analysis procedures.

CLEANING

There are many cleaning procedures in use at the present time. It is essential that the procedure chosen will:

1. Not remove any metal.
2. Remove all adherent corrosion product.

3. Provide reproducible results.

See appendix I for examples of cleaning procedures and equipment requirements.

AFTER CLEANING

A cleaned coupon will give a visual example of the type of attack which can be expected in that particular system.

The following information can be obtained from careful inspection of a properly cleaned coupon:

1. Type of attack of damage.

- a) etching
- b) pitting
- c) blistering
- d) delamination
- e) cracking
- f) crevice corrosion
- g) mechanical damage
- h) cavitation
- i) erosion

2. Pattern of attack.

- a) general
- b) scattered
- c) localised

3. Location of attack.

- a) overall
- b) centre face
- c) edge
- d) under holder

AFTER CLEANING

Continued.....

4. Degree of attack.

- a) mild
- b) moderate
- c) severe

In addition to the above general terms, detailed specific information should also be given for each specific type of attack. The method of detailing for each type of attack is as follows:

i) Etching -

- is it considered fine or coarse
- has it resulted in a marked degree of surface loss.
- how much loss (measure)
- is the loss uniform for the whole coupon.

ii) Pitting -

- are the pits numerous, indicate number.
- what is the diameter of the pits (measure)
- what shape are the pits.
- what type of surface at bottom of pit.

iii) Blistering -

- are the pits numerous, indicate number
- what is the diameter of the blisters.
- how high have they risen from surface.
- have they ruptured or not.
- how thick is the blister material.

iv) Delamination -

- at what location from surface.
- extent of delamination.
- gap at greatest point of delamination.

v) Cracking -

- location of crack.
- length of cracks.
- number of cracks.
- depth of cracks, if possible.

AFTER CLEANING Continued.....

vi) Crevice Corrosion -

- location of crevice
- orientation of crevice
- length of crevice
- width of crevice
- depth of crevice.

vii) Mechanical Damage -

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- type of damage - scars, scrapes, bends.
- extent (measure parameters).
- try to establish the cause.

viii) Cavitation -

- insure you differentiate from pitting (each cavity is hemispherical unless a second cavity has formed within the first).
- location.
- bulk of coupon lost.
- this action is mechanical in nature and is rarely observed in coupons unless orientation is incorrect..

ix) Erosion -

- this phenomena occurs on the leading edge of the coupon. It is marked by a general thinning of the leading edge, and can progress to form a knife edge and rounding of the leading corner. If the axis of the coupon was not parallel to direction of flow, this pattern of attack is modified.
- report amount of thinning of edge.
- report amount of rounding of corner.
- report average reduction of coupon width.

When making an evaluation, the duration of exposure should be kept in mind. For example, a coupon noticeably thinned would be described as severe for a one month exposure, but moderate for a six month exposure.

If a coupon is completely perforated or has a portion corroded away, this must be mentioned.

The purpose of this section is to indicate that the more accurate the observation and detailing of the damage, the more accurate the ability to predict what is possibly occurring in the system. This also indicated the need for detailed and accurate coupon analysis report. A sample report form is attached in Appendix II

SECTION 8 - RECORDING OF DATA

The analysis of any type of problem requires the accurate tabulation of data, and the proper storage of that data. This point cannot be over-emphasised.

When evaluating all the required parameters regarding a corrosion coupon, the recorded results should be concise, accurate and in sufficient detail to allow second party evaluation without a loss of information.

Attached in this section and in Appendix II is a sample data form. If the problems in a particular system require a modified form, this should be done. However, the basic form and its requirements should not be reduced.

The calculations and corrosion rate classifications are also included in this section.

TYPICAL COUPON REPORT FORM

Area: _____	Coupon No: _____
_____	_____
Location: _____	Coupon Weight: _____
_____	Coupon Factor: _____
Date Installed: _____	Coupon material: _____
_____	Coupon Size: _____
	Installed By: _____

Date Removed:	Removed By:
Type of Environment and Service:	Exposure Interval: Days

Analysis:

Before Cleaning: _____

Deposit Analysis: Sulphide Carbonate Ferrous
 Ferric Oil Other

After Cleaning: _____

Calculations:

	<u>Initial Wt.</u>	<u>Final Wt.</u>	<u>Wt. Loss</u>	<u>Corrosion Rate (mpy)</u>
Coupon				
Blank				

Corrected Corrosion Rate: _____ mpy

Pitting Rate: _____
 _____ Uncorrected Pitting Rate: _____ mpy

Special Remarks: _____

Analysis and calculation conform to standards.

Analysis and Calculations by

Signed:

CALCULATIONS

Pitting Rate

$$\text{Pitting Rate} = \frac{365 \times d}{T}$$

d = pit depth in mils (of deepest pit)

T = exposure time in days

Corrosion Rate

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$$\text{MPY} = \frac{365 \times W \times 1000}{T \times D \times A}$$

365 = days/year

W = weight loss in grams

1000 = mils/inch

T = exposure time in days

D = density in $\frac{\text{gm}}{\text{in}^3}$
 = 128.59 gm/in³ (density of 1018 steel)

A = exposed surface in in²

MPY = mils per year

AVERAGE CORROSION RATE CLASSIFICATION

Classification	Av. Corr. Rate		Av. Pitting Rate	
	mpy	µm/9	mpy	µm/a
Low	1.0	25.00	12.00	305.00
Moderate	1.0-4.9	25-125	12-24	305-610
Severe	5.0-10.0	126-254	25-96	635-2438
Very Severe	10 +	254+	96+	2438+

mpy = Mils-per-year (0.901") (one thousandth of an inch per year.)

µm/a = micrometers per annum (0.000001M) (equal to 0.04mpy)

APPENDIX 1

CLEANING OF EXPOSED COUPONS

The basic purpose of cleaning is to remove all corrosion products without removing any metal from the coupon and yet give reproducible results.

The method of cleaning should be checked against a blank. A standard unused coupon should undergo the same cleaning procedure as the exposed coupons. Any weight loss suffered by the blank coupon should be converted to a corrosion rate expressed in mpy and be subtracted from the corrosion rate of an exposed coupon to give a corrected corrosion rate in mpy for the exposed coupon.

$$\begin{aligned} & \text{CORROSION RATE (mpy of coupon)} - \text{CORROSION RATE} \\ & \quad \text{(mpy of blank)} \\ & = \text{CORRECTED CORROSION RATE (mpy of coupon)} \end{aligned}$$

The methods of cleaning are listed below in order of preference:

1. Ultrasonic Cleaning:

- a) Degrease coupons in benzene, toluene or carbon tetrachloride.
- b) Clean for 5 minutes in the ultrasonic cleaner containing a 10% solution of Hydrochloric Acid or Sulphuric Acid inhibited with 2%, or organic acid inhibitor such as Rodine 220, Polyrad 1110A or Konotol 400.
- c) Rinse with distilled water.
- d) Repeat b) and c) if necessary (if deposit is extremely tenacious brushing with a fine scouring powder may be required).
- e) Rinse in acetone.
- f) Place in a dessicator to cool and dry until ready for weighing.
- g) Weight to the nearest 0.0001 grams.

This method is fast, easy and produces low loss of bare metals. however, this method requires special equipment such as Branson B-220H Ultrasonic Cleaner. Some difficulties have been experienced with extremely tenacious deposits.

2. Chemical Cleaning:

- a) Degrease coupon in benzene, toluene or carbon tetrachloride.
- b) Clean for 5 minutes in a 20% solution of Hydrochloric Acid or Sulphuric Acid inhibited with an organic acid inhibitor such as Rodine 220, Polyrad 11109A or Konotol 400.
- c) Rinse with distilled water.
- d) Scrub with a brush and fine scouring powder, detergent and water.

- e) Rinse with distilled water.
- f) Repeat steps b). c). d). and e) of necessary.
- g) Rinse with acetone.
- h) Store in a dessicator to cool and dry until ready for weighing.
- i) Weigh to the nearest 0.0001 grams.

This method is inexpensive and fairly easy. Care must be taken to avoid excessive metal loss.

3. Electrolytic Cleaning:

- a) Degrease coupon in benzene, toluene or carbon tetrachloride.
- b) Scrub with a brush to remove loose corrosion product.
- c) Expose coupon as cathode for 3 minutes to 5% Sulphuric Acid inhibited with 2% organic acid inhibitor such as Rodine 220, Polyrad 1110A or Konotol 400 at 75°C using lead or carbon as the anode. Current density required is 1.3 amperes/inch². (or 20amp/decimeter²).
- d) Rinse with distilled water.
- e) Repeat steps c) and d) if required.
- f) Rinse with acetone.
- g) Store in dessicator to cool and dry until ready for weighing.
- h) Weigh to the nearest 0.0001 grams.

3. Electrolytic Cleaning: (continued.....)

This method cleans thoroughly with very little metal loss. However, the following objections may be raised:

- 1) Poor cathode contact will result in corrosion of coupon.
- 2) Decomposition of organic inhibitor by electric current requires frequent changing of solution.
- 3) Although lead anode gives the best cleaning, lead can be plated on the coupon giving low apparent metal loss.

4. Sodium Hydroxide - Zinc Cleaning:

- a) Clean for 8 minutes in a boiling solution of 20% NaOH and 20% zinc dust.
- b) Rinse with distilled water.
- c) Scour with brush, fine scouring powder and detergent.
- d) Repeat steps a), b), and c) if necessary.
- e) Rinse with distilled water.
- f) Rinse with acetone.
- g) Store in dessicator to cool and dry until ready for weighing.
- h) Weigh to the nearest 0.0001 grams.

This method involves inherent hazards to personnel and is not recommended.

5. Mechanical Cleaning:

- a) Clean coupon using stiff bristle brush, scouring powder and detergent until all deposits are removed.
- b) Rinse with distilled water.
- c) Rinse with acetone.
- d) Store in a dessicator to cool and dry until ready for weighing.
- e) Weigh to the nearest 0.0001 grams.

This method is acceptable for loose, soft, non-adherent deposits. If the deposit is tenacious and hard, excessive metal loss is experienced with resulting errors.

Equipment Required

1. Organic Solvent - benzene, toluene or carbon tetrachloride.
2. Concentrated Hydrochloric Acid.
3. Concentrated Sulphuric Acid.
4. Organic Acid Inhibitor - Rodine 220, Polyrad 1110A, Konotol 400 or equivalent.

5. Stiff bristled brush.
6. Detergent.
7. Scouring Powder.
8. Acetone.
9. Analytical balance (weigh to nearest 0.0001 grams).
10. Distilled water.
11. Dessicator.
12. Pit Gauge - Starret Model 643 or equivalent.
13. Magnet.

Optional

1. Ultrasonic Cleaner - Branson B-220H or equivalent.
2. Direct Current Power Source.
3. Sodium Hydroxide.
4. Zinc Dust.
5. Hot Plate.

APPENDIX II

COUPON REPORT FORM

Area:	Coupon No:
_____	_____
_____	Coupon Weight:
Location:	Coupon Factor:
_____	Coupon material:
_____	Coupon Size:
Date Installed:	Installed By:
Date Removed:	Removed By:
_____	Exposure
Type of Environment and Service:	Interval: Days
_____	_____

Analysis:

Before Cleaning:

Deposit Analysis: Sulphide Carbonate Ferrous

Ferric Oil Other

After Cleaning:

Calculations:

	<u>Initial Wt.</u>	<u>Final Wt.</u>	<u>Wt. Loss</u>	<u>Corrosion Rate (mpy)</u>
Coupon Blank				

Corrected Corrosion Rate: _____ mpy

Pitting Rate:

Uncorrected Pitting Rate: _____ mpy

Special Remarks:

Analysis and calculation conform to standards.

APPENDIX III

DEPOSIT ANALYSIS PROCEDURES

These are the recommended spot-tests required to estimate the mechanism of attack and establish the approximate qualitative composition of the corrosion product.

A. Sulphide and Carbonate

- 1) Add a few drops of hydrochloric acid to a sample of deposit.
- 2) If effervescence occurs, expose a piece of damp lead acetate paper to the vapours.
- 3) If the lead-acetate paper turns black, sulphide is present.
- 4) If the paper remains white, carbonate is present.

B. Ferrous Ion

- 1) Dissolve a small amount of deposit in 5% Hydrochloric Acid.
- 2) Add 1 drop of Potassium Ferricyanide ($K_3Fe(CN)_6$) solution.
- 3) A blue coloration and precipitate indicates presence of ferrous ion.

C. Ferric Ion

- 1) Dissolve a small amount of deposit in 5% Hydrochloric Acid.
- 2) Add 1 drop of Ammonium Thiocyanate (NH_4SCN) solution.
- 3) a deep red colour indicates the presence of Ferric Ion.

D. Hydrocarbons

Check solubility of deposit in carbon tetrachloride, benzene or toluene.

APPENDIX IV

GLOSSARY

Blister (Blistering) - process in which a thin layer of metal is separated from the coupon in the shape of a portion of a hemisphere. The blister may be intact or ruptured.

Carbonate - refers to the ion CO_3^{2-}

Cavitation - the process in which a liquid vaporises, and by this rapid expansion tears fragments of metal from the surface.

Corrosion - the process by which metal is lost to the solution which it contains. This process can be one or a combination of chemical, electrochemical and physical action.

Cracking - the process of fracturing in which the break is not complete.

Delamination - the process by which a solid metal is separated into layers. This is commonly seen in metal objects which have been produced by rolling, and by that process have inherent parallel zones of stress.

Deposit - that which is laid down on the coupon either by the corrosion process or by precipitation from the solution to which it is exposed.

Duration - that period of time something is subject to a special set of circumstances.

Electrolytic - pertaining to electrical current flow through a conductive medium.

Erosion - process by which solids are removed due to the flow of gas, liquids, solids or any combination thereof.

Etching - the process by which a surface is uniformly removed with minimal roughening, usually associated with acidic attack.

Exposure - having been subjected to a set of conditions.

Inhibitor - (organic acid inhibitor) an organic material which has the ability to protect a metal from acidic corrosion attack but does not hinder acid reactions with compounds of that metal.

Interface - that plane at which two different materials or phases of a material meet.

Lead Acetate Paper - paper which has been soaked in a lead acetate solution and then dried.

Localised - restricted to one area or locality.

Mil - one-one thousandth of an inch or 0.001 inch.

MPY - mils per year.

Orientation - position relative to a given reference point. In this case the reference point is the coupon itself.

Perforated - to have a hole all the way through.

Pitting - partial penetration of an object at a location which had an initial surface shape which was approximately circular.

Plateau Pitting - during the penetration action sequentially smaller areas are preferentially attacked giving an apparent step like wall to the pit.

Porous - to have holes which are interconnected and be permeable to liquids.

Scar - a mechanical deformity having length, width and depth produced by a fixed shape under varying pressure.

Scattered - to occur at irregular intervals on a plane surface.

Scrape - a deformity which reduces a surface area by irregular length, width and depth produced by mechanical action.

Scrub - to clean with mechanical action, usually referring to cleaning with a brush.

Spot-test - a qualitative test performed on site.

Sulphide - referring to S ion

Tabulation - to record data in a table or predetermined form.

Tenacious - to adhere to or stick to with great strength.

Tubercles - knob-like mounds of corrosion product. May also appear like mounds upon mounds or grow lump by lump in one general direction, usually vertical to the surface in question.

Ultrasonic - very high frequency sound. Ultrasonic cleaning utilizes very high frequency sound to break up corrosion products or precipitates.

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