ADELAIDE GALVANISING INDUSTRIES

# Adelaide Galvaninsing Pickle Solutions Testing Guide

Procedures for analysis of Galvanising Process Solutions

Edition 1: March 2026

# Safety Note

Prior to implementing the methods outlined in this guide, readers should ensure that they comply with the regulations covering the purchase, use and storage of chemicals in the workplace and have established appropriate safety systems. Failure to do so may result in injury or harm.

Readers should perform their own risk assessments that may require additional protective measures (equipment or facilities), or modified procedures to those suggested in the manual.

## Adelaide Galvanising Testing Schedule

| Bath to Test | Test | Frequency |
|---|---|---|
| Flux | Iron content<br>Zinc chloride content<br>Ammonium chloride content<br>Density/specific gravity or $^{0}$Baumé measurement<br>pH measurement | Weekly |
| Acid Baths | HCL Content, Iron Content | Weekly |
| Rinse | pH | Weekly |
| Caustic | Sodium Hydroxide Content and Ratio Determination | Monthly |

# Obtaining samples from baths

The process for obtaining samples from the Degreaser (caustic), Acid Baths and Preflux are the same. see below for the Method/Procedure.

## Equipment Required

- 250ml beaker/s
- 1m length of hose
- PPE (Gloves, Glasses and Overalls/Apron)

## Method/Procedure

1. Take the 1m length of hose identified for each solution (Caustic, Acid and Preflux).
2. Ensure you have on the correct PPE (Rubber Gloves, Glasses and Overalls/Apron)
3. Ensure you have a 250ml beaker to collect the samples from the solution to be tested.
4. Dip the length of hose into the solution to be tested as far down as you can without submerging your hand in the solution.
5. Cover the end of the hose not in the solution with your thumb (as shown in picture)
6. Withdraw the hose while still ensuring your thumb is covering the end.
7. Ensure the end of the hose dipped into the solution is inside/over the beaker.
8. Remove your thumb from the end and allow the solution to flow into the beaker.
9. Repeat the process until you have at least 100ml of solution to be tested in your beaker.

# Degreaser (Caustic)

Introductory Comments

This solution is used to remove oil and greases from the steel surface. The following information refers specifically to caustic soda based degreasing solutions which are the most commonly used in practice.

The speed at which steel articles are cleaned is dependent on temperature and sodium hydroxide content. The solution is usually heated between 70-90$^{0}$C and the sodium hydroxide content is usually maintained between 50-100g/L. Some commercially available degreasers may contain additional cleaning agents to improve the cleaning efficiency and extend the life of the bath.

For optimum performance, two properties need to be monitored and adjusted as necessary. These are;

1. **Free alkali -** This is the alkali available for cleaning. This is the sodium hydroxide content.
2. **Total Alkali -** This is the sum of the free alkali and the combined alkali. The combined alkali is the alkali which is combined with the soil and is not available for cleaning.

The ratio of free alkali to total alkali is used to give an indication of the cleaning capacity. It indicates how much free alkali is remaining to remove the soil as a proportion of the total alkali. For example, a high ratio of 0.9 shows that there is a large amount of alkali available for cleaning whereas a ratio of 0.5 shows that there is a low level of free alkali so the cleaning capacity is low.

As the bath ages, the proportion of by-products increases and thus the ratio of free alkali to total alkali decreases. The ratio should always be maintained above 0.5 by additions of fresh degreaser. When the amount of by-products build up to a level where the ratio cannot be maintained above 0.5 the solution may need to be partially or totally dumped.

Although zinc may be present in the degreaser from carry-over from work jigs, it is not usually present at high levels so is not considered a problem.

## Summary of Recommended Operating Conditions for the Degreaser

Temperature

Working Range: 70 - 90$^0$C

Sodium Hydroxide Content (NaOH)

Working Range: 50 - 100g/L

Ratio ("Free" to "Total Alkali")
Minimum: 0.5

Any proprietary additions to the degreaser must be maintained according to the suppliers' instructions.

## Analysis Procedures for the Degreaser

On a routine basis, e.g., **once a month**, analysis needs to be made of free alkali (sodium hydroxide) and total alkali, from which the ratio can be determined. Then adjustment to the solution can be made accordingly to ensure operating conditions as above are maintained. The test procedures are outlined on the following pages which include;

1. Sodium hydroxide content, ie, the "free alkali" content
2. Ratio determination

## Sodium Hydroxide Content and Ratio Determination

Equipment Required

- 100ml measuring cylinder
- 50ml Burette
- 250ml conical flask
- adjustable pipettor (1ml increment)
- 100ml bottle with screw lid

Reagents Required

- 0.1N Hydrochloric acid (100ml)
- Phenolpthalein indicator (3 drops)
- Bromocresol Green indicator (3 drops)

## Method/Procedure

1. Take a sample of about 100mL from the degreasing bath in a plastic bottle and allow it to stand to cool to room temperature and to settle any sediment.
2. Using the clean pipettor, take 1mL of solution from the sample and transfer it into the clean conical flask, add about 100mL of water, and add a few drops of phenolphthalein indicator. The solution will turn pink.
3. Fill the clean burette to the zero mark with 0. IN hydrochloric acid and titrate slowly with this solution, while swirling the conical flask, until the pink colour vanishes.
4. Record the burette reading. This is the first endpoint (Titration, A).
5. To the above solution add a few drops of bromocresol green indicator. The solution will turn blue.
6. Continue titrating slowly with the standard 0. IN hydrochloric acid until the solution turns permanent yellow.
7. Record the burette reading. This is the second endpoint (Titration, B).

## Calculation

Sodium Hydroxide Content (NaOH)

If the bath contains caustic soda only, then its concentration is calculated as follows;

Where A is the first titration endpoint and B is the second titration endpoint the sodium hydroxide content is;

$$NaOH\ g/L = [A-(B-A)] \times 4.0$$

If the bath is a proprietary solution, this can only be used as a guide. For formulated baths the factor will not be 4.0 but one specific to the product, usually between 5.5 - 7.0.

Ratio Determination

Gives an indication of the cleaning capacity of the bath and is calculated as follows;

$$\text{Ratio} = \frac{\text{Titration A}}{\text{Titration B}}$$

Example

If the titration to the first endpoint is 22mL and the titration to the second endpoint is 24mL, then;

$$NaOH g/L = [22 - (24-22)] \times 4.0$$

$$= [22-2] \times 4.0$$

$$= 20 \times 4.0$$

$$= 80.0 g/L$$

$$\text{Ratio} = \frac{22}{24}$$

$$= 0.92$$

# Acid Pickle

## Introductory Comments

The acid pickle is used to remove rust, millscale and other iron oxides from the steel surface. The following information refers specifically to hydrochloric acid pickling as this is the most commonly used acid in galvanizing plants.

Hydrochloric acid is normally used at ambient temperature. The efficiency of the pickle depends on free acid concentration, iron content and acid circulation (agitation). Recommended normal practice is to start with approximately 15% (150g/L) acid concentration. Commercially supplied hydrochloric acid is typically 28-30% concentration, which means diluting this 1:1 with water.

**During use, two changes occur;**

The free acid content decreases

Normally when the hydrochloric acid concentration level falls to about 10% or 11% (100-11 Og/L) the pickle should be adjusted back to 15% (150g/L) using fresh acid.

The iron content increases

General practice is to discontinue the above acid make-up additions when the iron level in the bath reaches 120g/L. Above this level, the acid is saturated with iron and pickling efficiency is significantly reduced. Adding more acid will not improve the situation. Normal practice is that when 110-120g/L iron is reached, continue to use the acid until its strength drops to 50g/L. When this is reached the acid is no longer used for pickling steel but can be used for removing (stripping) zinc from rework items.

Agitation of steelwork in the pickling tank, or forced circulation of acid (pumping), will considerably improve the pickling rate due to removal of iron build-up in the pickle adjacent to the steel surface which would otherwise have a partial inhibiting effect on the pickling reaction.

Zinc contamination in operating pickling baths, for example from work jigs/rework, should be avoided if possible as it reduces significantly the rate of pickling of steel surfaces. For this reason, if possible, attempt to limit any zinc contamination to only one operating tank and one which has been in service for an extended period of time (ie. iron content is high).

## Summary of Recommended Operating Conditions for the Acid Pickle

Temperature

Working range ambient temperature approximately 20-30 degrees Celsius.

Hydrochloric Acid Content

New Solution: 150g/L (15%)

Optimum Operation: 100 - 150g/L (11-15%)

Iron Content (Fe)

Maximum: 120g/L

Pickle Agitation

Preferable to have some agitation

## Analysis Procedures for the Acid Pickle

Acid strength and iron content should be checked frequently (frequency is dependent on throughput and tank capacity). The analysis methods are outlined on the following pages which include;

1. Hydrochloric Acid Content
2. Iron content by Titration

## Hydrochloric Acid Content

Equipment Required

- 100ml measuring cylinder
- 50ml Burette
- 250ml conical flask
- adjustable pipettor (2ml increment)
- 100ml bottle with screw lid

Reagents Required

- 0.548N sodium bicarbonate
- 100mL bromocresol green indicator

Method/Procedure

1. Take a sample of about 100mL from the pickling bath in a plastic bottle and allow it to stand to cool to room temperature and settle any sediment.
2. Using the clean pipettor, take 2mL of solution from the sample and transfer to the clean conical flask, and add about 100mL of water (tap water is fine).
3. To this solution add a few drops of bromocresol green indicator. The solution will turn yellow.
4. Fill the clean burette to the zero mark with 0.548N sodium bicarbonate and titrate slowly with the above solution, while swirling the flask, until the solution turns permanent blue/green.
5. Record the burette reading. This is the endpoint (Titration, C).

## Calculation

Titration, C, gives a direct measure of the hydrochloric acid content in the bath as percent weight per volume,

i.e.. % w/v.

I-ICI % w/v= C

To convert this to grams/liter;

HCl g/L = c x 10

## Example

If the endpoint of the HCI titration is 12mL, then

I-ICI = 12% w/v

OR

I-ICI g/L = 12 × 10

= 120g/L

## Iron Content in the Acid Pickle (Titration Method)

Equipment Required

- 250ml measuring cylinder
- 100ml measuring cylinder
- 50ml burette
- 250ml conical flask
- adjustable pipettor (1ml increment)
- 100ml bottle with screw lid
- Filter paper

Reagents Required

- 0.02M potassium permanganate
- Zimmermann-Reinhardt reagent

Method/Procedure

1. Take a sample of about 100mL from the pickling bath in a plastic bottle and allow it to stand to cool to room temperature and settle any sediment.
2. Filter some of this sample into clean 25mL measuring cylinder to the 25mL mark exactly.
3. Pour this sample into clean 250mL measuring cylinder and make up to 250mL mark exactly with water. Stopper the measuring cylinder and invert several times.
4. Using the clean pipettor, dispense 10mL of this dilute solution into the clean conical flask and add 15mL of Zimmermann-Reinhardt reagent.
5. Fill the clean burette to the zero mark with 0.02M potassium permanganate and titrate slowly with the above solution, while swirling the conical flask, until the colour turns permanent pale pink.
6. Record the burette reading. This is the endpoint (Titration, D).

## Calculation

Where D is the titration endpoint, the iron content of the acid pickle is calculated as follows;

$$Fe\ g/L = D \times 5.6$$

## Example

If the endpoint from the iron titration is 10mL, then

$$Fe\ g/L = 10 \times 5.6$$

$$= 56\ g/L$$

# PREFLUX

## Introductory Comments

The purpose of the preflux is to provide a protective layer on the steel surface to prevent oxidation prior to galvanizing and to break down the zinc oxide layer on the molten zinc surface at the point of entry of the steel into the galvanizing bath. These conditions are critical to achieving a metallurgical reaction between the molten zinc and the steel surface.

The following information refers specifically to preflux solutions consisting of zinc ammonium chloride (ZAC) "triple salt" dissolved in water. Zinc ammonium chloride "triple salt" is the most commonly used form of preflux and has the formula ZnC12.3NH4Cl. The components are zinc chloride (ZnC12) and ammonium chloride (NH4Cl) in the ratio 46% : 54% by weight, respectively.

<u>Important parameters influencing flux performance and which must be monitored are:</u>

<u>Zinc ammonium chloride (ZAC) concentration</u>

A concentration of 20 - 30% by weight is normally used. Measurement of the preflux density will give an approximate indication of the ZAC concentration. Usually, density is measured as either Specific Gravity (S.G.) or $^{0}$Baumé (Bé). It should again be stressed that density measurements provide only an approximation of ZAC concentration in that significant departures from the optimum ratio (refer below) can introduce some error.

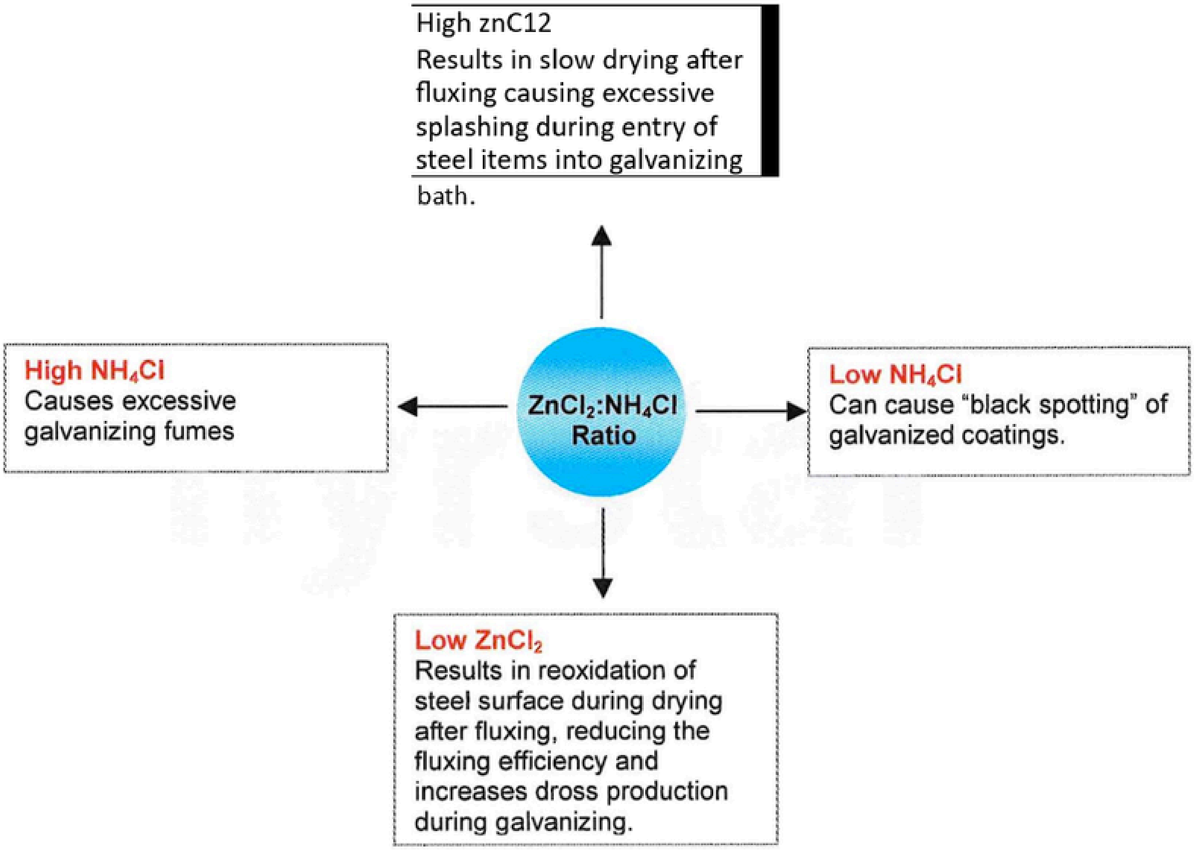
During use, the concentration of ZAC in the preflux will be reduced.

<u>Zinc chloride to ammonium chloride ratio</u>

The ratio in the preflux solution should be maintained in the proportions similar to that in the solid crystal form, ie. 46%ZnC12 : 54%NH4Cl. During operation, the ratio can change and accordingly, adjustments may be required from time to time to restore the ratio. Relatively small departures from the optimum ratio do not have any detrimental consequences, however, if the ratio imbalance becomes excessive some adverse effects can be experienced (refer to diagram on the following page).

In order to determine the ratio, measurement must be made of the individual components of the ZAC solution, ie. Zinc content, ammonia content and chloride content.

## Effect of ZnC12 to NH4Cl Ratio Imbalance



Iron content in the preflux

During operation of the preflux, the solution becomes contaminated with iron. The major sources of iron are:

- Reaction of the preflux with the surface of the steel articles.
- Carry-over of iron salts on the surface of the steel articles from the acid pickle (water rinsing after the acid pickle removes most but not all of the iron salts).

This iron contamination in the preflux will be transferred on the steelwork into the galvanizing zinc bath, resulting in increased dross production. It is therefore very important in that the preflux be routinely measured and that steps be taken to reduce this contamination to a minimum. It is generally accepted that the iron content should not exceed 10g/L and through good practice many galvanizers are able to achieve less than 5g/L.

It is important to note that iron in the preflux exists in two forms:

i. Insoluble iron hydroxide. This form is responsible for the orange-brown colouration of the preflux solution after it has been in use for a period of time. Most of this settles as a sludge in the bottom of the tank and therefore does not present a major problem in the galvanizing process. This sludge must be removed periodically otherwise it will be carried over into the galvanizing bath.

ii. Soluble iron chloride. Typically, in the preflux solution, the soluble iron content far exceeds the insoluble form and it is the soluble iron which is therefore of greatest concern. Besides causing increased dross production during galvanizing, it also reduces the effectiveness of the preflux to chemically clean the steel surface.

Optimum operation of the preflux tank requires, firstly, that water rinsing after pickling is carried out as effectively as possible to minimise iron salt carry over and, secondly, that as much of the soluble iron in the preflux solution is converted to insoluble iron hydroxide. To achieve this conversion, the following procedures are commonly used;

Control of the preflux pH between 4.0 - 5.5. If preflux pH falls below 4.0, insoluble iron hydroxide will tend to reconvert to soluble iron. Solutions with pH values less than 4.0 can be best adjusted to the optimum range (pH 4.0 - 5.5) by addition of ammonium hydroxide solution (NH4OH).

Oxidation of the soluble iron content by either continuous air sparging of the preflux solution or by hydrogen peroxide treatment.

pH of the preflux solution

As indicated above pH should preferably be maintained in the range 4.0 - 5.5. The importance of the 4.0 minimum has been discussed above. Above the 5.5 maximum, zinc chloride will precipitate from solution as zinc hydroxide and is therefore not available for prefluxing the steel. Usually, preflux solutions stabilize under normal operating conditions below pH 5.5; however if the pH should exceed 5.5 the preflux solution can be adjusted back to the optimum range by addition of hydrochloric acid.

## Summary of Recommended Operating Conditions for the Preflux

Temperature

Working Range: 60 - 80$^0$C (to facilitate drying of the steel prior to galvanizing)

Zinc Ammonium Chloride Concentration (ZAC)

Optimum Concentration: 200g/L — 300g/L

Zinc Chloride to Ammonium Chloride Ratio

Optimum Ratio: 46%ZnC12 : 54%NH4Cl

Iron Content (Fe)

Maximum: 10g/L

Preferable: <5g/L

Density measured at room temperature

Working range: 1.100 - 1.150 Specific Gravity (S.G.) or 13.2 -18.9 Baume (Be)

pH

Working range: 4.0 - 5.5

## Analysis Procedures for the Preflux

A full analysis of the preflux is recommended on a monthly basis. Measurement of density, pH and iron content should be done more frequently (e.g. Weekly) and adjustments made as appropriate. The analysis procedures are outlined in the following pages and include:

1. Iron content
2. Zinc chloride content
3. Ammonnium chloride content
4. Density/specific gravity or $^{0}$ Baumé measurement
5. pH measurement

## Iron Content in the Preflux

Equipment Required

- 250ml measuring cylinder
- 100ml measuring cylinder
- 50ml burette
- 250ml conical flask
- Plastic funnel
- adjustable pipettor (1ml increment)
- 100ml bottle with screw lid
- Filter paper

Reagents Required

- 0.02M potassium permanganate
- Zimmermann-Reinhardt reagent

## Method/Procedure

1. 1. Take a sample of about 100mL from the preflux bath in a plastic bottle and allow it to stand to cool to room temperature and settle any sediment.
2. 2. Filter some of this sample into clean 25mL measuring cylinder to the 25mL mark exactly.
3. 3. Pour this sample into clean 250mL measuring cylinder and make up to 250mL mark exactly with water. Stopper the measuring cylinder and invert several times.
4. 4. Using the clean pipettor, dispense 10mL of this dilute solution into the clean conical flask and add 15mL of Zimmermann-Reinhardt reagent.
5. 5. Fill the clean burette to the zero mark with 0.02M potassium permanganate and titrate slowly with the above solution, while swirling the conical flask, until the colour turns permanent pale pink.
6. Record the burette reading. This is the endpoint (titration E)

## Calculation

Where E is the titration endpoint, the iron content of the preflux is calculated as follows;

$$Fe\ g/L = E \times 5.6$$

Example

If the endpoint from the iron titration is 1.7mL, then

$$Fe\ g/L = 1.7 \times 5.6$$

$$= 9.5\ g/L$$

## Zinc Chloride Content in the Preflux

Equipment Required

- 1L volumetric flask
- 100ml measuring cylinder
- 150ml beaker
- 50ml burette
- 250ml conical flask
- Plastic funnel
- adjustable pipettor (10ml increment)
- 100ml bottle with screw lid
- Filter paper

Reagents Required

- 0.01M EDTA Solution
- 30% hydrogen peroxide
- Buffer solution (ph 10)
- Erichrome black indicator

## Method/Procedure

***(CAUTION: Perform this determination in a well-ventilated room or fume cupboard because of ammonia fumes)***

1. Take a sample of about 100mL from the preflux bath in a plastic bottle and allow it to stand to cool to room temperature and settle any sediment.
2. Using the clean pipettor, take 10mL of solution from the sample and add to the clean volumetric flask, and add water to the IL mark exactly.
3. To this solution add about 2mL of the hydrochloric acid, stopper the volumetric flask and invert several times.
4. Using the clean pipettor, measure a 10mL sample from this dilute solution into the clean beaker, add a few drops of hydrogen peroxide and about 10mL of the buffer solution. (NOTE: The solution will have an orange-brown iron precipitate floating in it. This is normal and there is no need for concern).
5. The solution will need to be filtered through a No.41 paper into the clean conical flask to remove the iron. After the solution has filtered through, rinse with water through the filter paper a few times into the flask.
6. To the filtered solution add a few drops of Erio T indicator and swirl the flask until the indicator mixes. The solution will turn dark red.
7. Fill the clean burette to the zero mark with 0.01M EDTA solution and titrate slowly with the above solution, while swirling the conical flask, until the colour turns permanent dark blue/green.
8. Record the burette reading. This is the endpoint (titration F)

## Calculation

Where F is the titration endpoint, the zinc content of the preflux is calculated as follows;

Zn g/L = F x 6.538

To convert this to Zinc Chloride ZnC12,

ZnC12 g/L = Zn g/L x 2.08

Example

If the endpoint from the Zinc titration is 10mL, then

Zn g/L = 10 × 6.538

= 65.4 g/L

To convert this to Zinc Chloride ZnC12,

ZnC12 g/L = 65.4 × 2.08

=136g/L

## Ammonium Chloride Content in the Preflux

Required

- 250 mL measuring cylinder
- 100 mL measuring cylinder
- 150 mL beaker
- 50 mL burette
- 250 mL conical flask
- Adjustable pipettor (set to 1 mL)
- 100 mL plastic bottle with screw top lid
- Distillation apparatus (see Appendix 1, pages 61 to 63)

Reagents Required

- 1.0 N sulphuric acid
- 1.0 N sodium hydroxide
- 40% sodium hydroxide
- 100 mL methyl red indicator

Method

***(Caution: Perform distillation behind a Perspex screen or in a fume cupboard due to the risk of explosion from an acid base reaction.)***

1. Set up the distillation apparatus as per the diagram.
2. Add exactly 50.0 mL of 0.1 N sulphuric acid to a clean 150 mL beaker (collection vessel), then add a few drops of methyl red indicator. The solution will turn dark pink.
3. Place the collection vessel under the condenser tip of the distillation apparatus, ensuring the tip is immersed in the acid solution.
4. Take approximately 100 mL of sample from the preflux bath in a plastic bottle. Allow it to cool to room temperature and let any sediment settle.
5. Using a clean pipettor, add 1 mL of the sample to a clean distillation flask containing about 150 mL of water.
6. Add 50 mL of 40% sodium hydroxide to this solution. Immediately seal the flask with a glass stopper, as ammonia release begins straight away.
7. Turn on the water at a moderate flow rate so it runs continuously through the condenser during distillation.
8. Heat the distillation flask with a Bunsen burner until boiling begins. Then reduce the heat, continue boiling for 10 minutes, and collect the distillate in the collection vessel.
9. Once distillation is complete (approximately 10 minutes):
   i. Remove the collection vessel from under the condenser tip
   ii. Turn off the Bunsen burner and the water

***(Caution: This step must be followed in the exact order to prevent the distillate from being drawn back into the distillation flask, which could cause an explosion due to an acid base reaction.)***

1. Transfer the contents of the collection vessel into a clean conical flask.
2. Fill a clean burette to the zero mark with 0.1 N sodium hydroxide. Titrate the solution slowly while swirling the conical flask until a permanent yellow colour is observed.
3. Record the burette reading. This is the endpoint (Titration G).

## Calculation

Where G is the titration endpoint, the ammonia content of the preflux is calculated as follows:

$NH_4^+$ g/L = (50.0 − G) × 1.8

To convert this to ammonium chloride ($NH_4Cl$):

$NH_4Cl$ g/L = $NH_4^+$ × 2.96

Example

If the endpoint from the ammonia titration is 20 mL:

$NH_4^+$ g/L = (50.0 − 20) × 1.8
= 30 × 1.8
= 54 g/L

To convert this to ammonium chloride ($NH_4Cl$):

$NH_4Cl$ g/L = 54 × 2.96
= 160 g/L

## Density Measurement of the Preflux

The density of the flux solution at room temperature provides a quick but approximate indication of ZAC concentration. The following page shows a graph of ZAC concentration versus density, expressed as specific gravity (SG), at room temperature. A table converting density (g/mL) to degrees Baumé (°Bé) is also provided on page 35.

Equipment Required

- Hydrometer for specific gravity (density range 1000 to 1200, with 0.005 subdivisions)
- 100 mL measuring cylinder

Method

1. Take a 100 mL sample from the preflux bath and allow it to cool to room temperature. Let any sediment settle.
2. Pour approximately 60 to 70 mL of the clear sample into a clean measuring cylinder.
3. Place the hydrometer into the measuring cylinder and gently spin it to eliminate surface tension effects.
4. Read the specific gravity (SG) at the fluid level.
5. Record the specific gravity of the preflux sample.

Note: For example, if the SG reading from the hydrometer is 1170, the true representation of SG is 1.170.

## Conversion of Density to Baumé

A formula can be used to convert density in g/mL to degrees Baumé (°Bé) and vice versa, as shown below.

Where density is known and degrees Baumé is required:

°Bé = 145 – (145 ÷ d)
 where d = density in g/mL

Example

If the density of the solution is 1.100 g/mL:

°Bé = 145 – (145 ÷ 1.100)
 = 13.2 °Bé

Conversely, where degrees Baumé is known and density is required:

d = 145 ÷ (145 – °Bé)
 where d = density in g/mL

Example

If the solution measures 13.2 °Bé:

d = 145 ÷ (145 − 13.2)
 = 1.100 g/mL

The following table lists the conversion of density in g/mL to degrees Baumé at room temperature.

| Density (g/mL) | °Baumé |
|---|---|
| 1.000 | 0.0 |
| 1.050 | 6.9 |
| 1.100 | 13.2 |
| 1.150 | 18.9 |
| 1.200 | 24.2 |
| 1.250 | 29.0 |
| 1.300 | 33.5 |

## pH Measurement of the Preflux

The pH level of the preflux should be checked regularly using either a portable pH meter or pH indicator test strips. The method is outlined below.

Equipment Required

a) Portable pH meter

or

b) Box of pH indicator papers
(The papers should cover a range of pH 0 to pH 7 and be graduated in 0.1 pH intervals)

Method

Using a pH meter:

1. Calibrate the pH meter before use, following the manufacturer’s instructions.
2. Take approximately 20 mL of preflux sample and allow it to cool to room temperature.
3. Place the probe of the pH meter into the sample and read the pH from the digital display.
4. Rinse the probe with water and wipe it clean between samples.

Alternatively, using pH indicator strips:

1. Take approximately 20 mL of preflux sample and allow it to cool to room temperature.
2. Dip a test strip into the sample for a few seconds.
3. Remove the strip and compare the colour to the reference chart provided on the packaging to determine the pH.

# Preparation of Reagents and Indicators

## Reagents

Buffer solution (pH-10) – Weigh out 70g ammonium chloride, then dissolve in 568mL of ammonium hydroxide and make up to the 1L mark exactly in a volumetric flask with distilled water.

0.01M EDTA solution – Dissolve 3.72g of A.R. disodium dihydrogen ethylenediaminetetra-acetate dihydrate (EDTA) in distilled water and make up to the 1L mark exactly in a volumetric flask with distilled water.

0.548N Sodium Bicarbonate – Dissolve 46g of sodium bicarbonate in distilled water and make up to the 1L mark exactly in a volumetric flask with distilled water.

40% Sodium Hydroxide – to 1L of water, cautiously add 40g of sodium hydroxide pellets and stir until dissolved.

Zimmermann-Reinhardt Reagent – Dissolve 70g of manganese sulphate in 500mL of water, cautiously add 125mL (98%) sulphuric acid and 125mL (85%) phosphoric acid, make up to 1L mark exactly with water.

CAUTION: 40% sodium hydroxide solution and Zimmermann-Reinhardt reagent. (Preparation of these solutions must be performed in a fume cupboard or behind a Perspex screen for safety reasons as an explosion can result).

## Indicators

Bromocresol Green – generally available as “off the shelf” solution.

Dissolve 0.1g of bromocresol green in 100mL of distilled water.

Eriochrome Black T (Erio T)

Dissolve 1g of eriochrome black T dyestuff in 75mL of triethanolamine and 25mL of ethanol.

Methyl Red – generally available as “off the shelf” solution.

Dissolve 0.1g of methyl red, water-soluble sodium salt, in 100mL of distilled water.

Phenolphthalein

Dissolve 0.5g of solid phenolphthalein in 50mL of absolute alcohol, then add 50mL of distilled water. (Filter if necessary).