



TITRATION GUIDE

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INTRODUCTION

The control of your surface treatment tanks/baths is vital to ensure the highest possible quality and also to obtain the best economical use of the chemicals which you use.

The easiest and fastest way to get an accurate control point is by carrying out a **TITRATION** on the bath solution.

A titration is a method that will allow you to determine the concentration of a solution and therefore the precise quantity of a product in the working solution.

The method will involve taking a sample of the working solution and adding to it an **INDICATOR** then adding a second solution of known strength. The indicator will change colour when the reaction between the working solution and the second solution is complete.

PreTreat Ltd. issue technical data sheets (TDS) which give the exact details of the titration method. If the method is followed then the working solution can be controlled according to the running specification(s) that have been established for your particular process.

The following guide is intended to make it easier for you to be able to carry out the titrations you require to control your tanks correctly.

EQUIPMENT

The following list of equipment is required to carry out titrations, although the individual TDS will detail exactly what is required.

DO NOT worry if you don't have the exact equipment as shown below, you will have an equivalent which will still do the job.

SAFETY: The chemicals used can be harmful / corrosive, so eye protection must be worn.



Plastic syringe for measuring the working solution.



Glass conical flask for carrying out the titration reaction. Used when the solution needs to be heated.



Plastic beaker used to carry out the titration reaction. (Not suitable for heating)



Indicator storage & dropping bottle.



Self-zeroing burette. This holds the second solution used in the titration reaction.



Safety glasses. The chemicals used in titrations can be corrosive.

Titration Procedure

The technical data sheet supplied for the Pre-Treatment Technologies Ltd product in use will detail the method and reagents needed to carry out the titration relevant to that product.

The following is a typical method:

Pointage Titration

Measure 10 cm³ of the bath into a flask and add 4 – 6 drops of Phenolphthalein Indicator solution. Titrate with 0.1M Sodium Hydroxide until the colour changes to a permanent pink.

In this method the burette will be filled with the 0.1M sodium hydroxide solution. It is vital that the burette is zeroed before use and that it does not go beyond its capacity (usually 20cm³ or 25cm³), once it reaches its capacity it will need to be re-zeroed and the titration carried on with. The result will be the total volume of the 0.1M sodium hydroxide added in cm³.

It is important that the amount of solution added from the burette is accurately measured.



**In this example the reading is
21.3cm³.**



The pink colour is the end-point.

The result will give you a figure which will then be compared to the running specification. If it is within the spec then no further action is needed.

If it is lower than the spec then extra product will need to be added to the process tank in accordance with the TDS replenishment. This will usually be “add X litres of product per 1000 lts of bath”. It is good practice to re-check the titration after any addition and make a note of the amount added.

The table below shows how the results can be recorded. It will help if the actual working volume of the bath is known; this will help calculate the amount of product needed. (The overall tank volume is not the same as the working volume)

Date of test	Titration result (mls) (spec 8.0 – 10.0mls)	Action required
01/03/14	9.6	None (bath is in spec)
08/03/14	7.5	Bath below spec-add 1lt/1000lts
08/03/14	8.2 (re-test)	None (addition got bath in spec)

If the result is higher than the spec then no addition is required but the bath may be too concentrated and could result in coatings of a lesser quality. It may need just using to bring down the concentration or water added.

The titrations that are carried out can be broadly covered in the following sections:





- 1) An acid based working bath using alkali titrating solution,
- 2) An alkaline based working bath using acid titrating solution,
- 3) Chromic acid based working bath using sodium thiosulphate solution.


For each titration there will be different amounts of bath solution used, different strengths of titrating solution used.

IT IS ESSENTIAL THAT THE TECHNICAL DATA SHEET IS READ AND THE TITRATION CARRIED OUT AS DESCRIBED.

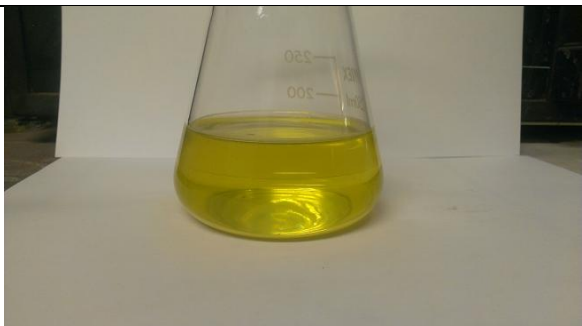
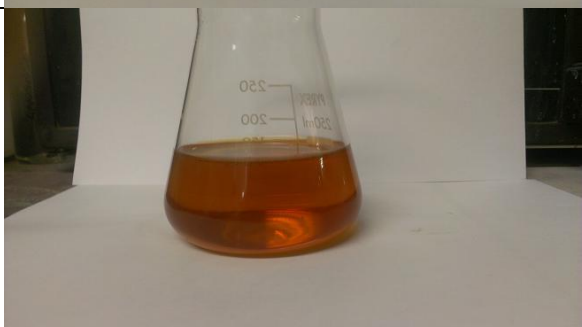
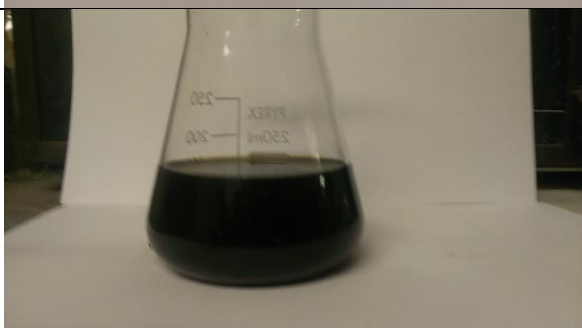
Determining the end point of the titration can be quite tricky; it involves a colour change and ideally should be carried out in good light conditions.

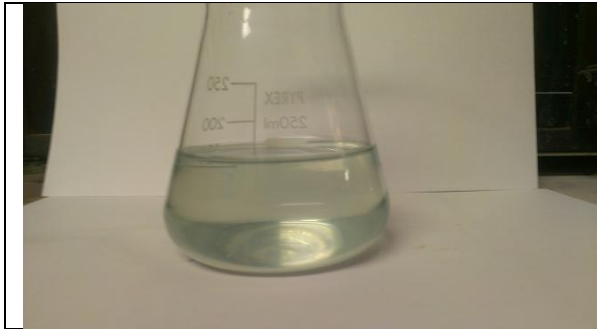
The section below gives the colour changes for the indicators that are used most commonly.

	<p>PHENOLPHTHALEIN</p> <p>The colour change is: COLOURLESS to PINK</p>
	<p>BROMOPHENOL BLUE</p> <p>The colour change is: YELLOW to BLUE</p>
	<p>INDICATOR 11-13</p> <p>The colour change is: YELLOW TO VIOLET</p>
	<p>METHYL ORANGE</p> <p>The colour change is: YELLOW to RED</p>

	<p style="text-align: center;">BROMOCRESOL GREEN</p> <p style="text-align: center;">The colour change is:</p> <p style="text-align: center;">YELLOW to BLUE</p>
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The titration to measure the Chrome (vi) level is detailed on TDS's such as Alocrom 1200 (Chromcote 7420), Alocrom 407/47(Chromcote 7407), WALTERISATION[®] 7460 etc.

	<p>A chrome (vi) solution before any additions or titration. Note the yellow colour, this is normal for Cr(vi) solutions.</p>
	<p>A chrome (vi) solution after the addition of potassium iodide and acid. Note the dark colour, this is normal.</p>
	<p>A chrome (vi) solution after the addition of the starch indicator. Note the black colouration, this is normal.</p>



A chrome (vi) solution after the titration with 0.1M sodium thiosulphate.
Note the very pale green colouration, this is normal.

A typical description of a Cr (vi) titration is given below so it can be related to the pictures above.

Titration: Measure 5 cm³ of the bath into a flask and dilute to about 100 cm³ with water. Add approximately 1 gram of Potassium Iodide and approximately 10 cm³ of concentrated Hydrochloric Acid and agitate until dissolved. Titrate with 0.1M Sodium Thiosulphate to a pale straw colour, then add approximately .5 grams of Iodine Indicator and continue the titration until the blue-black colour just disappears.

Use of Results

Once a titration has been carried out and the result recorded, it may be necessary to then use a factor to either multiply or divide the titration result by.

For example, the WALTERISATION[®] 7460 concentration is given by:

Titration in ml of 0.1M Sodium Thiosulphate x 1.613 = % Surtec 650

(The 1.613 is the factor)

In this example, the WALTERISATION[®] 7460 bath can be controlled either on the titration result alone and/or by the % concentration. Either will do but you must be consistent!