

ANALYTICAL PROCEDURE FOR DETERMINATION OF HN504.2™ ACID FREE ACTIVATOR CONCENTRATION

Method:

This method uses standards of known concentrations of activator to produce a graph showing percent transmittance of light verses concentration. The transmittance from the sample is interpolated from this graph to determine the concentration of activator. The procedure requires two basic tasks.

Completion of TASK 1 results in a graph showing transmittance values verses known concentrations of HN504™ Activator. Highest confidence in the results of the total analysis is achieved when the graph is produced whenever a new shipment of HN504.2™ Acid-Free Activator is received. However, it is possible to only perform this task quarterly and still obtain results that are adequate for monitoring and maintaining the operating bath.

Completion of TASK 2 results in transmittance value for the unknown concentration of HN504.2™ Acid Free Activator in the operating bath. The concentration is determined by extrapolating a value from the graph produced in TASK 1.

Procedure I: Establishing Your Standards

1. Prepare standards in separate 100-ml volumetric flasks:

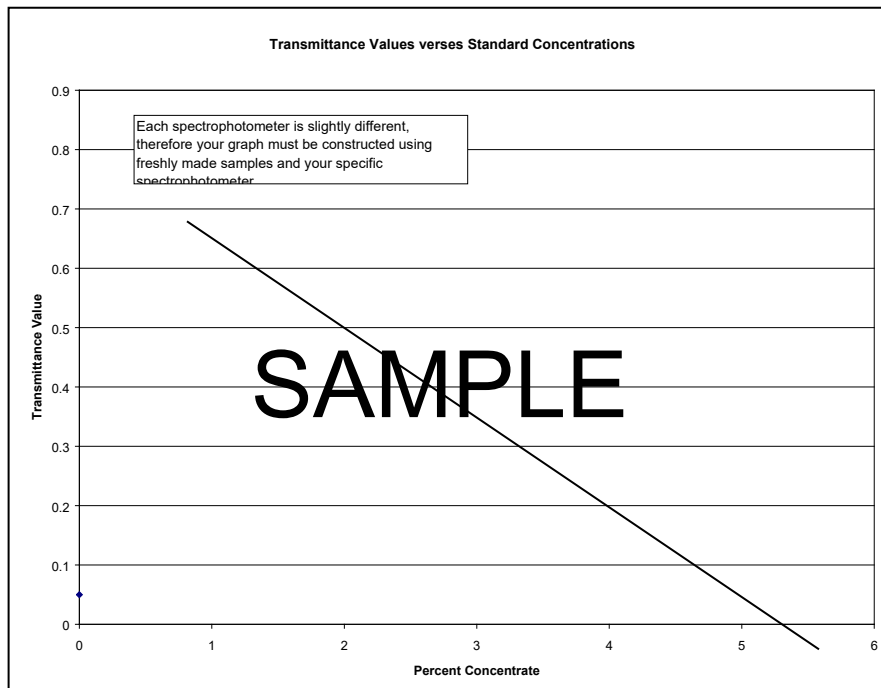
Standard #	HN504.2™ Concentrate	HN503™ Pre-Dip	Percent Concentrate
1	1 ml	99 ml	1%
2	2 ml	98 ml	2%
3	3 ml	97 ml	3%
4	4 ml	96 ml	4%
5	5 ml	95 ml	5%

Apparatus Required:

- 5 100 ml volumetric flasks
- 1 pipette with 1 ml graduation
- 1 test cell for calibration
- 5 test cells for transmittance determination

Procedure:

1. Using the 425 nanometer wave length, calibrate the spectrophotometer using a cell containing only HN503™. Set the resulting transmittance as 100%.
2. From each of the prepared standards pipette 1 ml into a separate clean and dry volumetric flask, then add fresh HN503™ Pre-Dip to fill the flask to the 100 ml mark. Mark each sample as it is prepared to avoid confusion. These will be the standards for producing the graph of absorbance verses concentration.
3. Determine the transmittance value of each standard (follow manufacturers reference guide for the spectrophotometer). Remember to use fresh HN503™ Pre-Dip as the blank for zeroing.
4. Plot the transmittance value versus the % HN504.2™ Acid Free Activator Concentrate. (Standard 1=1% through standard 5=5%). (Data points can be entered into a spreadsheet, then perform a linear regression to determine the equation of the best line through the data points.)
5. There are variations among spectrophotometers, so the following graph serves only as a representation.



Each % of HN504.2™ Acid Free Activator Concentrate contains approximately 59 mg of Palladium per liter of working bath.

Procedure II: Analyzing the Activator Bath

1. Pipette 1 milliliters of the working bath into a clean and dry 100-ml volumetric flask and fill to volume with fresh HN503™ Pre-Dip.
2. Determine the transmittance value of each standard (follow manufacturers reference guide for the spectrophotometer). Remember to use fresh HN503™ Pre-Dip as the blank for zeroing.
3. Compare the value obtained with the values on the predetermined graph or place the measured value into the equation generated by the linear regression of the standard data points.

Making Additions

Bath should be maintained at between 4% and 5% for optimum performance.. Add HN504.2™ to bring concentration into the optimum range.

Add Calculation:

$$\text{Milliliters HN504.2} = (5 - \% \text{ HN504.2}) \times 37.85 \times \text{tank volume (gallons)}$$

Note: Some spectrophotometers have customized analytical procedures for determination of mg/l of palladium. Using this method, maintain the bath between 236 mg/l and 295 mg/l of palladium.

Determination of Acid Normality

Reagents and Equipment

0.5 N Sodium Hydroxide

Phenolphthalein indicator solution

250 ml Erlenmeyer flask

10 ml pipet

50 ml buret

Procedure:

1. Pipet 10 ml of **HN-504** working solution into the 250 ml Erlenmeyer flask.
2. Add 3 to 4 drops of 50% H₂O₂ to break the complex.
3. Add 75 ml of de-ionized water and 1 to 2 drops of Phenolphthalein indicator solution.
4. While swirling the solution, titrate with 0.5 N sodium hydroxide to a purple endpoint.

5. CALCULATIONS:

Normality = (mls of base) x (Normality of base) x 0.1

The acid normality of a **HN-504** solution should be maintained between 0.30 and 0.60.

An addition 8 ml per liter of 35% hydrochloric acid will raise the normality 0.1 units.

Determination of Stannous Chloride

Reagents and Equipment

25 ml buret
10 ml pipet
250 ml Erlenmeyer Flasks
100 ml graduated cylinder
0.1 N Iodine Solution
Fresh Starch Indicator Solution
50% Hydrochloric Acid solution

Procedure

1. Pipet 10 mls of working solution into the 250 ml Erlenmeyer flask.
2. Add 25 mls of a 50% hydrochloric acid solution and 75 mls of de ionized water.
3. Add 5 mls of starch indicator solution.
4. Titrate the solution with 0.1 Normal Potassium Iodate-Iodide solution to a dark blue/black endpoint.

5. **CALCULATIONS:**

Stannous chloride (g/L) = (mls of Iodine Solution) x (Normality of Iodine Solution) x 9.8
Maintain Stannous Chloride between 18 and 24 grams per liter through **HN-504** additions.

pH Control

Maintain the pH of the working solution below 0.6 with additions of hydrochloric acid.

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