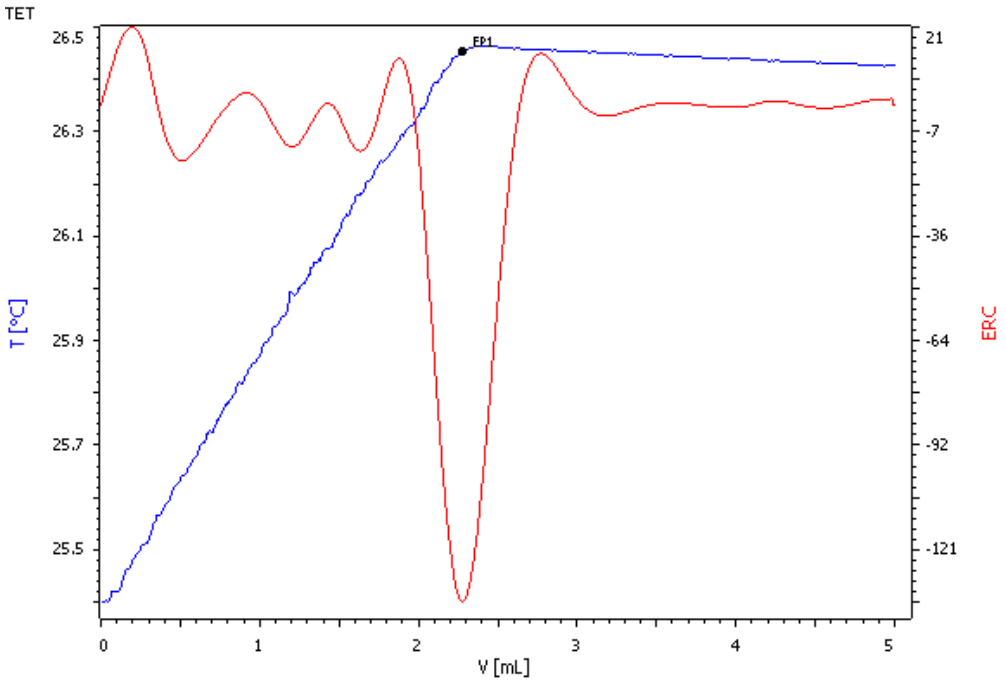
Titration Application Note H-143

Sulfuric acid and tartaric acid in tartaric sulfuric anodizing bath

Rapid, sequential determination using a thermometric sensor (thermometric titration)



Tartaric Sulfuric Anodizing (TSA) is an established technique for corrosion protection in the aerospace industry. It is an alternative to the environmentally harmful chromic anodizing process. As such, a method to monitor the levels of sulfuric acid and tartaric acid in TSA plating baths is required. Potentiometric titration methods have been developed, and are widely used across the industry. Their disadvantage is that two titrations with different electrodes and solvents are required.

In this Application Note, an alternative method is presented, where the concentration of both acids is determined in sequence using a thermometric sensor. Compared to potentiometric titration, thermometric titration is faster and more convenient (no sensor maintenance required). On a fully automated system, the determination of both parameters takes about 7 minutes.

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Method description

Sample

Tartaric sulfuric anodizing bath

Sample preparation

No sample preparation is necessary.

Configuration

859 Titrotherm	2.859.0010
814 USB Sample Processor (1T/1P)	2.814.0010
772 Pump Unit – aspirate	2.772.0120
800 Dosino, 3x	2.800.0010
802 Stirrer	2.802.0020
Dosing unit 10 mL, 2x	6.3032.210
Dosing unit ETFE 5 mL	6.1575.150
Sample rack 22 x 120 mL	6.2041.470
Stirring propeller (intensive)	6.1909.060
Titration head for 859 Titrotherm with 120 mL PP beakers	6.9914.159
Sample beaker 120 mL	6.1459.300
Thermoprobe HF	6.9011.040

Solutions

Titrant 1 Barium chloride	c($BaCl_2$) = 1 mol/L 244.28 g $BaCl_2$ is weighed into a 1000 mL volumetric flask and dissolved in deionized water. The flask is then filled up to the mark with deionized water.
Titrant 2 Sodium hydroxide	c(NaOH) = 2 mol/L 80 g NaOH is weighed into a 1000 mL volumetric flask and dissolved in deionized water. The flask is then filled up to the mark with deionized water.
Potassium fluoride	β(KF) = 300 g/L 300 g KF is weighed into a 1000 mL volumetric flask and dissolved in deionized water. The flask is then filled up to the mark with deionized water.

Analysis of samples

2 mL sample is pipetted into the titration vessel. 40 mL deionized water is added using the pumps. The solution is then titrated with c($BaCl_2$) = 1 mol/L until after the exothermic end point to determine sulfuric acid content. To the titrated solution 5 mL β(KF) = 300 g/L is added and the solution is titrated with c(NaOH) = 2 mol/L until after the exothermic end point to determine the total acid content. The tartaric acid content is calculated by subtracting the sulfuric acid content from the total acid content.

After the titration the solution is aspirated and the sensor and buret tips are automatically rinsed with deionized water.

Parameters

Titration	Sulfuric acid	Total acid
Mode	TET	TET
Start volume	0 mL	0 mL
Pause	20 s	20 s
Stirrer	-15	-15
Dosing rate	2 mL/min	2 mL/min
Filter factor	50	70
Damping until	0.3 mL	0.3 mL
Stop volume	7 mL	7 mL
Evaluation start	0.3 mL	0.3 mL
Reaction type	Exothermic	Exothermic
EP criterion	-30	-60

Result

Sample no. (n = 5)	Sulfuric acid / (g/L)	Tartaric acid / (g/L)
Bath 1	42.9 ± 0.6	78.2 ± 0.7
Bath 2	44.5 ± 0.7	81.0 ± 1.0
Bath 3	43.9 ± 0.8	81.0 ± 1.7

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Version 1, published in February 2019

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