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Reference Method for Measuring the Surface Tension of Chromium Electroplating, Chromium Anodizing and Reverse Etching Solutions with a Stalagmometer

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1. Introduction

Surface tension, with respect to liquids such as water or chromium electroplating chromium anodizing or reverse etching solutions, is a result of the stronger interactive molecular forces which exist between molecules in the interior of a liquid than the molecular force which exists on the molecules at the immediate surface of the liquid. This force imbalance is due to the lower number of molecules located immediately above the surface of the solution. This resultant force imbalance causes the molecules at the surface of the solution to be pulled towards the molecules in the liquid.

Surface tension is force acting over a length, which is measured in dynes/cm (dyn/cm) or milli Newtons/metre (mN/m). These units are equivalent.

Measurement of the surface tension of a chromium electroplating, chromium anodizing or reverse etching solution using a stalagmometer is based on the relationship between the weight of a drop of the solution (which creates a shear force on the solution) and the surface tension of the surface of the solution drop (which keeps the liquid together).

For a given volume of liquid, the surface tension, as measured with a stalagmometer, is proportional to the density of the solution and inversely proportional to the number of drops.

This Reference Method describes the procedure for determining the surface tension of chromium electroplating, chromium anodizing or reverse etching solutions, using a 2.5 mL or a 5 mL stalagmometer.

Note: prior to commencing any of the following steps, ensure that you have carefully reviewed, understood and implemented all of the safety information noted in section 10 of this document.

2. Definitions

2.1 Density – mass per unit volume (grams/cubic centimetre).

3. Reagents

3.1 Distilled Water – Water that has been distilled and passed through a 0.2 µm filter. Commercially available distilled water may be used. Water produced from Reverse Osmosis or ASTM Types I, II, III, or IV can also be used. Use fresh distilled water from stock for each use. Note:

Distilled water of the required purity may be obtained from drug stores or grocery stores.

3.2 Reagent grade concentrated nitric acid (supplied as a 70% solution) or concentrated sulphuric acid (supplied as a 98% solution).

3.2.1 To prepare 35% nitric acid: mix one part distilled water and one part concentrated nitric acid. CAUTION: Always add acid to water slowly with continual mixing.

3.2.2 To prepare 25% sulphuric acid: mix one part concentrated sulphuric acid with three parts distilled water. CAUTION: Extreme heat will be generated. Always add acid to water slowly with continual mixing.

3.3 Acetone or methanol (100 %).

4. Equipment

4.1 Stalagmometer, 2.5 mL, calibrated at 20 ± 1 drops of water at 25 °C. (A 5 mL stalagmometer can also be used, but is not recommended).

4.1.1 Cleaning new stalagmometers:

The cleanliness of a stalagmometer is essential for producing precise measurements of surface tension. Therefore, a new stalagmometer must be thoroughly cleaned before its first use. To clean a new stalagmometer, rinse the stalagmometer four times with distilled water using the following procedure:

(a) With a pipette bulb, pull up distilled water through the stalagmometer to above the top set of lines on the stalagmometer.

(b) Release the pipette bulb from the top and allow the water to drain.

(c) Blow-out any remaining water using the pipette bulb.

(d) Repeat steps (b) and (c) an additional three times.

(e) If beading occurs, clean the stalagmometer as per section 9.2.

4.1.2 Do not touch the flat part at the bottom of the stalagmometer once it has been cleaned.

4.2 Pipette bulb.

4.3 Sampling container.

4.4 Optional - A 100 mL or 200 mL wide bore pipette. (Note: The pipette must be long enough so that it can be placed at least 30 cm (12 in) below the surface of the solution, such that a sample of the solution from that depth can be drawn into the pipette).

4.5 Nitrile gloves.

4.6 Recommended additional equipment

4.6.1 Lab jack

4.6.2 Ring stand

4.6.3 Burette clamp

4.6.4 Hand tally counter

4.7 Waste receptacle: 500 mL or larger jar or beaker for collecting the solution after the surface tension measurement. (Note: After the surface tension measurement, the sample of solution can be returned to the tank). If not returned to the tank, then waste solutions are to be treated as hazardous waste.

5. Sampling

5.1 Samples for surface tension analysis must be taken from solutions at the representative operating temperature of the facility. The solutions should also be agitated for at least 10 minutes before the sample is collected. Agitation of the solution can be done either by conventional mechanical or physical means. Allow any foam (which may be present on the surface of the solution) to dissipate, such that a clear area of solution is present. This allows for collection of a sample without any surface foam being collected in the bottle or pipette. During the sampling process and as the sample is being collected, foam may be created in the bottle or in the pipette, which is acceptable. Note: foam collected from the surface of the solution can interfere with the true surface tension reading of the solution.

5.2 Plating solution samples must be collected from at least 30 cm (12 inches) below the surface of the solution.

5.2.1 One method to do this is to place a 100 mL or 200 mL pipette below the surface of the solution (to avoid collecting any foam) and collect the sample into the pipette with a bulb. If the sample is collected from below the surface of the solution, then foam is not likely to be present.

5.2.2 Once the sample is collected from the tank, do not allow any sample to escape from the pipette and immediately transfer the sample from the pipette to a 200 ml or larger volume beaker.

5.2.3 As an alternative to collecting a sample with a pipette, a sample container may be used. With this sampling method, use safety gloves (of sufficient length to protect the wearer) and dip the sampling container under the surface, with the opening of the sample container facing down. Slowly turn the sampling container right side up and pull the container straight up once filled with sample.

5.3 Samples for surface tension must not **be preserved chemically or physically. If samples are to be sent off-site for surface tension analysis, do not pack them in ice. Ambient cooling of the samples during transit is allowable as long as the samples** are not cooled to below 10°C **for more than 24 hours.** Do not allow the samples to freeze.

6. Procedure for Measuring Surface Tension

6.1 The analysis for surface tension of the solutions is to be done at 25°C and at a rate of 20 drops per minute.

6.2 As part of the daily solution checks and before analyzing any samples for surface tension, check the drop count of the stalagmometer using distilled water. The stalagmometer must produce 20 ± 1 drops for the 2.5 mL stalagmometer. **If the number of drops is outside the 19 – 21 range, then clean the stalagmometer as per Section** 9**. If this does not produce acceptable results, discard the stalagmometer and obtain a new one.**

6.3 Collect a sample of plating solution from the plating tank as per Section 5**.**

6.4 When the temperature of the collected sample is at 25°C, place the flat end of the stalagmometer into the solution. Using the pipette bulb, draw up the solution to above the top set of lines on the stalagmometer. Place your finger on the top end of stalagmometer or hold the bulb in position while transferring the stalagmometer over a waste receptacle. (Wear nitrile gloves at all times). Release your finger or the bulb and drain the solution into the receptacle.

6.5 For each sample of solution for surface tension analysis, fill and empty the stalagmometer at least three times as per section 6.4 **with that sample before measuring. This ensures that the interior surface of the stalagmometer is thoroughly wetted with the solution.**

6.6 To measure the surface tension of the solution, fill the stalagmometer with the sample to above the top set of lines on the stalagmometer. If any foam should appear, then remove the foam by squeezing and releasing the pipette bulb so the solution raises and falls in the top portion of the stalagmometer.

6.7 Once the foam is removed, allow the solution to fall to the top set of lines **and record the point at which a drop is released.** This is the initial starting line**.**

6.8 Release the solution and count the number of drops that fall **from the stalagmometer as the liquid level falls to the equivalent line of the bottom set of lines on the stalagmometer. If a partial drop is forming at this point, use the drop count up to that point.** Do not count the partial drop as one drop.

6.9 Determine the density of the solution using one of the methods in section 7**.**

7. Measuring the Density of the Chromium Electroplating, Chromium Anodizing or Reverse Etching Solutions

The density of the chromium electroplating, chromium anodizing or reverse etching solution may be determined by one of two methods: 1) Direct measurement of the mass of a specific volume of solution; or 2) using a hydrometer (Baume stick). The density measurement is to be made at 25 °C.

7.1 Direct measurement

7.1.1 Equipment required:

7.1.1.1 Top-loading balance which is accurate to ± 0.1 gram;

7.1.1.2 10 mL or 100 mL class A volumetric flask or class A graduated cylinder;

7.1.1.3 Pipette

7.1.2 Procedure:

7.1.2.1 Tare a clean and dry 10 mL (or 100 mL) flask on the top-loading balance.

7.1.2.2 Pipette the solution into the flask such that the bottom of the meniscus is touching the fill line.

7.1.2.3 Record the weight of liquid in grams.

7.1.2.4 Divide the weight of liquid by 10 (or 100 if using a 100 mL flask) to give the solution density in g/mL.

7.2 Hydrometer

7.2.1 Equipment Required:

7.2.1.1 Hydrometer: Rated to read either a density from 1.00 to 1.30 (or greater) g/mL *or* ***a Baume reading of 0 to 35. (Note: The hydrometer must be able to measure for a density greater than the density of water.)***

7.2.1.2 Container: A container that is tall and wide enough to accept the hydrometer and be such that a reading can be taken from a near horizontal line. (Note: The container must be sufficiently wide to allow the hydrometer to float and not be subject to wall effects or interference from the container wall).

7.2.2 Procedure:

7.2.2.1 Pour the solution into the container, such that it will not overflow from the container when the hydrometer is immersed in the solution.

7.2.2.2 When the solution has cooled to 25°C, place the hydrometer in the solution and allow the hydrometer to level off or stabilize.

7.2.2.3 Take the density reading at the line on the hydrometer which is level with the solution.

7.2.2.4 The hydrometer reading is a direct reading of the density of the solution and no calculations are required if measured in g/mL. If using Baume density, the equation for determining specific gravity in g/mL is 145 / (145 – Baume reading). The table in Appendix A may also be used to cross-reference the number of drops with the Baume reading to find the surface tension in dynes/cm.

8. Calculations

8.1 For a 2.5 mL stalagmometer, use the following equation:

$$Surface\ Tension\ (Dynes/cm) = \frac{Density\ (g/ml) \times 1440}{Number\ of\ \ Drops}$$

8.2 For a 5 mL stalagmometer, use a constant of 2880 instead of 1440:

$$Surface\ Tension\ (Dynes/cm) = \frac{Density\ (g/ml) \times 2880}{Number\ of\ \ Drops}$$

8.3 Use the table in Appendix A for looking up the surface tension value for density (in g/mL or Baume) and the number of drops.

9. Cleaning Stalagmometers

9.1 Daily cleaning:

After completing the surface tension measurement of the solutions (at any time of the day that surface tension is to be measured), the stalagmometer must be properly rinsed with distilled water until no colour remains. Note: This may require several rinses.

After rinsing, place the stalagmometer horizontally in a tray of fresh distilled water and allow the stalagmometer to soak in the distilled water until next use.

9.2 If, after rinsing, beading of liquid occurs inside the stalagmometer, this is an indication that the stalagmometer is dirty. Beading can occur due to insufficient or improper rinsing of the stalagmometer; if the solution has been allowed to dry within the stalagmometer; or if oil or another

material has coated the inside. If this situation occurs, more aggressive measures using nitric or sulphuric acid must be used to clean the stalagmometer. Use the following procedure to clean the stalagmometer:

9.2.1 Place a piece of 0.5 cm (¼") (inside diameter) tubing on the top of the stalagmometer. Place a tubing clamp on the tubing between one and two centimetres from the stalagmometer.

9.2.2 Use the pipette bulb to draw up 50% nitric or 25% sulphuric acid (prepared as per Subsection 3.2) to above the upper line marking on the stalagmometer. CAUTION: These acids are corrosive and produce a lot of heat when mixing with water. Wear personal protective equipment at all times: goggles, acid-resistant gloves, chemical apron and lab coat.

9.2.3 Close the clamp and ensure that no liquid is released. Allow the acid to remain in the stalagmometer for one to two hours. More time may be required for very dirty equipment.

9.2.4 Drain the acid from the stalagmometer and rinse the stalagmometer at least three times with distilled water.

9.2.5 If oil is suspected to cause the beading, then, after soaking the stalagmometer in acid, rinse once with distilled water, then rinse once with full strength acetone or methanol, and finally rinse at least three times with distilled water.

10. Safety

10.1 Use heavy duty heat and chemical-resistant gloves for sampling the tanks.

10.2 Chemical-resistant nitrile gloves should be worn at all other times during this procedure.

10.3 Chromium electroplating, chromium anodizing and reverse etching solutions are carcinogenic and corrosive. Wear acid or chemical resistant gloves, eye protection and apron/coveralls when handling these solutions.

10.4 **Nitric and sulphuric acids are extremely corrosive and will cause burns to skin. When using these acids, use acid-resistant gloves, safety goggles, and an apron. Consult the supplier material safety data sheet (MSDS) for more information.**

10.5 **Work in a well ventilated area when using nitric acid due to the generation of acidic fumes when nitric acid is mixed with water.**

10.6 **Sulphuric acid solutions will become very hot when this acid is mixed with water. It is important to always add acid to water and never add water to acid as the heat of reaction can cause spattering and generation of steam.**

10.7 **Do not pipette any solution by mouth.**

Appendix A – Table of Surface Tension (Dynes/cm) Tabulated by Number of Drops and Density (in g/mL and Baume). To calculate the surface tension of your sample, find the corresponding value of the density (in g/mL) **or** the Baume value along the top two rows. On the left vertical axis, locate the drop count obtained during the surface tension analysis. The surface tension is where the Baume or density value column and drop count row intersect.

If the density or number of drops is outside of the values provided in the table use the equation in section 8 to calculate the surface tension.

	Baume	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34
	g/mL	1.00	1.01	1.01	1.02	1.03	1.04	1.04	1.05	1.06	1.07	1.07	1.08	1.09	1.10	1.11	1.12	1.12	1.13	1.14	1.15	1.16	1.17	1.18	1.19	1.20	1.21	1.22	1.23	1.24	1.25	1.26	1.27	1.28	1.29	1.31
# of Drops	15	96	97	97	98	99	99	100	101	102	102	103	104	105	105	106	107	108	109	110	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125
	16	90	91	91	92	93	93	94	95	95	96	97	97	98	99	100	100	101	102	103	104	104	105	106	107	108	109	110	111	112	113	113	114	115	117	118
	17	85	85	86	86	87	88	88	89	90	90	91	92	92	93	94	94	95	96	97	97	98	99	100	101	102	102	103	104	105	106	107	108	109	110	111
	18	80	81	81	82	82	83	83	84	85	85	86	87	87	88	89	89	90	91	91	92	93	94	94	95	96	97	97	98	99	100	101	102	103	104	105
	19	76	76	77	77	78	78	79	80	80	81	81	82	83	83	84	85	85	86	87	87	88	89	89	90	91	92	92	93	94	95	96	96	97	98	99
	20	72	73	73	74	74	75	75	76	76	77	77	78	78	79	80	80	81	82	82	83	84	84	85	86	86	87	88	88	89	90	91	92	92	93	94
	21	69	69	70	70	71	71	72	72	73	73	74	74	75	75	76	76	77	78	78	79	80	80	81	81	82	83	84	84	85	86	86	87	88	89	90
	22	65	66	66	67	67	68	68	69	69	70	70	71	71	72	72	73	74	74	75	75	76	77	77	78	78	79	80	80	81	82	83	83	84	85	86
	23	63	63	63	64	64	65	65	66	66	67	67	68	68	69	69	70	70	71	71	72	73	73	74	74	75	76	76	77	78	78	79	80	80	81	82
	24	60	60	61	61	62	62	63	63	64	64	64	65	65	66	66	67	67	68	69	69	70	70	71	71	72	73	73	74	74	75	76	76	77	78	78
	25	58	58	58	59	59	60	60	61	61	61	62	62	63	63	64	64	65	65	66	66	67	67	68	68	69	70	70	71	71	72	73	73	74	75	75
	26	55	56	56	57	57	57	58	58	59	59	59	60	60	61	61	62	62	63	63	64	64	65	65	66	66	67	67	68	69	69	70	70	71	72	72
	27	53	54	54	54	55	55	56	56	56	57	57	58	58	59	59	59	60	60	61	61	62	62	63	63	64	64	65	66	66	67	67	68	68	69	70
	28	51	52	52	53	53	53	54	54	54	55	55	56	56	56	57	57	58	58	59	59	60	60	61	61	62	62	63	63	64	64	65	65	66	67	67
	29	50	50	50	51	51	51	52	52	53	53	53	54	54	55	55	55	56	56	57	57	58	58	59	59	60	60	61	61	62	62	63	63	64	64	65
	30	48	48	49	49	49	50	50	50	51	51	52	52	52	53	53	54	54	54	55	55	56	56	57	57	58	58	58	59	59	60	61	61	62	62	63
	31	46	47	47	47	48	48	48	49	49	50	50	50	51	51	51	52	52	53	53	53	54	54	55	55	56	56	57	57	58	58	59	59	60	60	61
	32	45	45	46	46	46	47	47	47	48	48	48	49	49	49	50	50	51	51	51	52	52	53	53	53	54	54	55	55	56	56	57	57	58	58	59
	33	44	44	44	45	45	45	46	46	46	47	47	47	48	48	48	49	49	49	50	50	51	51	51	52	52	53	53	54	54	55	55	56	56	56	57
	34	42	43	43	43	44	44	44	45	45	45	45	46	46	47	47	47	48	48	48	49	49	50	50	50	51	51	52	52	52	53	53	54	54	55	55
	35	41	41	42	42	42	43	43	43	44	44	44	45	45	45	46	46	46	47	47	47	48	48	49	49	49	50	50	51	51	51	52	52	53	53	54
	36	40	40	41	41	41	41	42	42	42	43	43	43	44	44	44	45	45	45	46	46	46	47	47	48	48	48	49	49	50	50	50	51	51	52	52
	37	39	39	39	40	40	40	41	41	41	41	42	42	42	43	43	43	44	44	44	45	45	46	46	46	47	47	47	48	48	49	49	50	50	50	51
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	40	36	36	37	37	37	37	38	38	38	38	39	39	39	40	40	40	40	41	41	41	42	42	42	43	43	44	44	44	45	45	45	46	46	47	47
	41	35	35	36	36	36	36	37	37	37	37	38	38	38	39	39	39	39	40	40	40	41	41	41	42	42	42	43	43	44	44	44	45	45	45	46
	42	34	35	35	35	35	36	36	36	36	37	37	37	37	38	38	38	39	39	39	39	40	40	40	41	41	41	42	42	42	43	43	44	44	44	45

	Baume	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34
	g/mL	1.00	1.01	1.01	1.02	1.03	1.04	1.04	1.05	1.06	1.07	1.07	1.08	1.09	1.10	1.11	1.12	1.12	1.13	1.14	1.15	1.16	1.17	1.18	1.19	1.20	1.21	1.22	1.23	1.24	1.25	1.26	1.27	1.28	1.29	1.31
	43	33	34	34	34	34	35	35	35	35	36	36	36	37	37	37	37	38	38	38	39	39	39	39	40	40	40	41	41	42	42	42	43	43	43	44
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	50	29	29	29	29	30	30	30	30	30	31	31	31	31	32	32	32	32	33	33	33	33	34	34	34	35	35	35	35	36	36	36	37	37	37	38
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	53	27	27	28	28	28	28	28	29	29	29	29	29	30	30	30	30	31	31	31	31	32	32	32	32	33	33	33	33	34	34	34	35	35	35	35
	54	27	27	27	27	27	28	28	28	28	28	29	29	29	29	30	30	30	30	30	31	31	31	31	32	32	32	32	33	33	33	34	34	34	35	35
	55	26	26	27	27	27	27	27	28	28	28	28	28	29	29	29	29	29	30	30	30	30	31	31	31	31	32	32	32	32	33	33	33	34	34	34
# of Drops	56	26	26	26	26	26	27	27	27	27	27	28	28	28	28	28	29	29	29	29	30	30	30	30	31	31	31	31	32	32	32	32	33	33	33	34
	57	25	25	26	26	26	26	26	27	27	27	27	27	28	28	28	28	28	29	29	29	29	30	30	30	30	31	31	31	31	32	32	32	32	33	33
	58	25	25	25	25	26	26	26	26	26	26	27	27	27	27	27	28	28	28	28	29	29	29	29	30	30	30	30	31	31	31	31	32	32	32	32
	59	24	25	25	25	25	25	25	26	26	26	26	26	27	27	27	27	27	28	28	28	28	29	29	29	29	29	30	30	30	31	31	31	31	32	32
	60	24	24	24	25	25	25	25	25	25	26	26	26	26	26	27	27	27	27	27	28	28	28	28	29	29	29	29	29	30	30	30	31	31	31	31
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	63	23	23	23	23	24	24	24	24	24	24	25	25	25	25	25	25	26	26	26	26	27	27	27	27	27	28	28	28	28	29	29	29	29	30	30
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Additional information can be obtained at:

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