Advice & Counsel



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Test Finished Parts for Hex Chrome

Dear Advice & Counsel,

My company is a job shop that services the automotive industry. Recently, I have been getting letters from some of my clients asking me to verify that the parts we are processing for them are free of hexavalent chromium. Just responding with a letter telling them that we believe the parts are free of hexavalent chromium is apparently not good enough for these manufacturers. How do I "prove" it?

Signed, Free At Last

Dear Mr. Free,

As you are probably aware, these requests originate from the European ban(s) on hexavalent chromium on manufacturers automotive and electronic goods. You can "prove it" by testing your finished parts for hexavalent chromium content. Normally, ASTM would be your source for a procedure, but until ASTM produces a broadly accepted procedure, you are left with using one of several methods developed by the manufacturers of the products. Since the procedure is not identical, you need to contact your customer and confirm that the procedure you intend to use will be acceptable. The following are descriptions of procedures developed by GM Delphi Safety and Interior Systems, and ISO. We will assume the test parts have chromate conversion coatings. Painted parts (topcoat plus primer) are more complicated to test. Be sure to obtain a copy of the procedure and follow it carefully, as each procedure has slightly different requirements. We are summarizing below to illustrate what is involved.

GM:

Summary of Method

The coated test sample is leached in boiling water for five minutes. The hexavalent chromium is reacted with diphenylcarbizide to develop a pink color. The intensity of the color is measured using a colorimeter.

Test Sample

You need enough test sample(s) to yield 50(+/-5) cm² of surface area. All of this surface area must have been processed in the finishing operation (do not include masked off areas, for example). The test sample(s) can not have any sealer/organic topcoat.

Procedure

(Note: It is assumed you are aware of the dangers and hazards of handling the chemicals involved).

- In a glass beaker, boil 50 mL of DI water. Immerse the 50cm² of parts and boil for exactly five minutes.
- 2. Remove the test part(s).
- 3. Cool the boiled water to room temperature.
- 4. If the water is cloudy or has solids, filter the water through a 0.5 micron membrane filter (or use any filter that will deliver a clear liquid).
- 5. Transfer the water to a 50 mL volumetric flask.
- 6. Add 1.5 mL of 25% volume, sulfuric acid and mix.
- 7. Dilute to 50 mL with DI water.
- 8. Remove 25 mL of the liquid in the volumetric flask and transfer to a glass beaker. Transfer the remaining liquid into another beaker of the same size and style.
- Add one mL of diphenylcarbizide solution (0.5 g diphenylcarbizide dissolved in 50 mL of acetone then add 50 mL DI water, store refrigerated) to only one of the two beakers.
- 10. Compare the color of the liquid in the two beakers. If the liquid in the beaker

treated with diphenylcarbizide contains hexavalent chromium, it will turn pink.

- 11. The pink color may not be easy to see. You may nee to use the liquid that was not treated with diphenylcarbizide as a blank in a spectrophotometer or colorimeter (use 540 nm wavelength). After zero-ing the blank, read the absorbance of the liquid treated with diphenylcarbizide.
- 12. Use a "Standard" solution to confirm the procedure. Dilute 0.5 mL of a hexavalent chromium standard (purchased from any chemical supply house such as Fisher Scientific) to 50 mL with water. This liquid will reflect the maximum allowable hexavalent chromium concentration (0.01g/cm²). If your treated portion is pinker than this standard, you have failed the test.

Delphi:

The Delphi specification requires chromated parts are tested after at least 24 hours and before 30 days has elapsed since production of the coating. Parts that have been conversion coated with hexavalentfree processes can be tested immediately, but no later than 30 days after production.

The Delphi procedure requires that the test be conducted in triplicate (results are averaged, and variation is also reported).

In this procedure, parts are boiled in 45 mL of water, for 5–7 minutes (no more than 7). A method blank using the same amount of water and no samples is treated along with the sample.

Parts are rinsed above the beaker upon removal. The liquid is filtered if necessary, then acidified and diluted to volume as described above in the GM procedure. However, the Delphi procedure requires

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every two years. As Steven Hayward notes: "While we need several more years of data to resolve questions about many chemicals, the early results suggest that fears of human exposure to chemicals are exaggerated and unwarranted."¹ I hope he's correct and that the alarmists don't jump on every minuscule concentration that's found, without looking at concentration levels.

References

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that the parts be re-extracted a second time to prove that all of the hex-chrome was extracted.

The leached water is acidified and treated in a similar manner as described in the GM procedure. However, the color developed is not visually compared. It is measured using a spectrophotometer calibrated with standard solutions.

ISO 3613:2000(E):

The ISO specification is similar to the GM specification, except that:

- a. Leached parts are rinsed above the beaker.
- b. 3 times as much acid is added to the test sample prior to color development.

- c. The leach water is transferred to a 250 mL volumetric flask.
- d. 3 mL of the diphenylcarbizide solution is added for color development.
- e. 2 minutes after addition of the diphenylcarbizide, 25 mL of a buffer solution (55g di-hydrogen orthophosphate monohydrate in 100 mL water) is added before diluting the liquid to 250 mL.
- <u>f. A calibrated spectrophotometer is</u> <u>used to quantitate the hexavalent</u> <u>chromium</u>

It would be nice if we all could agree on a procedure. In the meantime, the above illustrates the need for you to obtain guidance from your customer as to which procedure is to be used.



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