

SOP—Determination of Requirement Density

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1. Objective:

The purpose of this Standard Operating Procedure (SOP) is to give guidelines on how to determine the density of a sample that will be used as the requirement density. This will be the requirement density of record for the specimens examined by Micro CT and EDS measurements. This density will then be set as the formulation requirement for radiography measurements. This SOP is referred to in TP 48—Preparation of Hydrogen Peroxide/Icing Sugar Specimens for X-ray Measurements by J. G. Reynolds and H. E. Martz.

2. Definitions:

Sample—this is the basic chemical material that is made (synthesized or mixed) according to a Formulation

Formulation—the experimental procedure used to make the sample; this is a separate document that is attached and usually different for each type of sample

Specimen—this is what is examined by x-ray; the sample is put in the appropriate container and called the specimen

Container—the holder of the sample that, when full of sample, is called the specimen

3. Background:

Determining accurate densities of specimens used in the IDD program are essential. The density has been determined to be the requirement to indicate whether the sample has been prepared properly and/or whether that specimen is stable over time. This stability can be due to intrinsic instability of the sample or due to the sample reacting with the container that is used to make the specimen.

The requirement density has been taken from the 2009 preparation of the specific formulation. In TP 48, which uses SOP—Determining Volume of a Container and SOP—Measurement of Density of a Specimen to determine densities, there is a possibility that the samples and specimens prepared do not meet this 2009 requirement density. There are two reasons for this potential situation—the sample was not prepared properly, or the requirement density is incorrect. Because there is no way to go back and assess the 2009 value experimentally, this SOP gives a method to re-determine the requirement density.

In TP 48, the formulator is given three chances to meet the requirement density. Meeting the requirement is to have the density within $\pm 5\%$ of the requirement value. After three attempts, if this value cannot be reached, then TP 48 directs the formulator to this test plan, meaning the 2009 requirement density is in correct. The reason for this are many and beyond the scope of this SOP. However, for many materials, re-determining this value is a simple, straightforward process.

4. Redetermination of density requirement.

This is done if the preparation of the first specimen and the repeats do not every reach the density requirement. It is done to establish a new density requirement.

- i. Prepare sample according to formulation using a neutral sample container, such as a passivated Nalgene bottle
- ii. From TP 50, pick a method for determination of density (using the most accurate method possible)
- iii. Determine the density
- iv. Repeat this process 5 times
 - v. Determine the average and the spread of the data. If the spread of the data less than or equal to $\pm 5\%$, this is the new requirement density for the sample
 - vi. If the spread is larger than $\pm 5\%$ of the average, then the sample is probably not stable, so work should be stopped
 - vii. Make official note of the new target density and record it as a modification of the Formulation document.

5. Flow chart

