Application Note 109

Attaining Envelope Density Reproducibility and Accuracy with Your GeoPyc

This application note contains guidelines for attaining reproducible and accurate envelope density measurements with your GeoPyc®.

Reproducibility

Achieving high reproducibility in any analytical measurement often requires performing tests in an identical manner using a single instrument, fixed instrument parameters, and the same quantity of test material. This is particularly true with the GeoPyc technique because it is very sensitive to procedural variations and deviations in test parameters. Reproducibility of results of approximately ±1.0% can be expected when parameters are controlled to the fullest extent possible. A description of these parameters and the criteria that must be observed to achieve this level are described below.

Envelope density is calculated from specimen mass and envelope volume, that is, volume including both open and closed pores. This volume is measured using a non-intruding, free-flowing, dry powder medium called Dry*Flo*®, which is confined in a cylindrical sample chamber having one of five diameters from 12.7 mm (0.5 in.) to 50.8 mm (2.0 in.). The volume of the specimen is determined by subtracting the volume of consolidated Dry*Flo* (blank run) in a sample chamber from the volume of the same consolidated Dry*Flo* in the same chamber with the specimen included (test run). The medium bed is agitated through rotation and vibration, and the consolidation force is gradually increased to the same set value in both phases of a test.

1. The first criterion for a GeoPyc analysis is that the Dry*Flo* consolidate identically in the blank and test runs. Repeated testing of the medium alone has shown that, almost without exception, it actually consolidates with a reproducibility of ± 0.34% or better in all size sample chambers for bed depths of one-half to twice the chamber diameter. Somewhat better reproducibility of ± 0.25% is typically achieved when the bed depth is restricted to approximately the chamber diameter. In any event, between one-third and one-quarter of the minimal overall error of ± 1.0% is due to the nonideal behavior of Dry*Flo*.

   Guideline 1. Start an analysis with a Dry*Flo* bed depth a little less than the chamber diameter.

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2. Sample quantity plays the most significant role in reproducibility. Obviously, the specimen extracted from a larger quantity of material must be of sufficient quantity to be representative of the whole. The quantity of sample determines the minimum sample chamber size required for analysis. A chamber should be selected in which the sample constitutes a minimum of 20% of the total sample-plus-Dry*Flo* volume when consolidated. A larger percentage of sample is preferable; however, keep in mind that the sample must always be surrounded sufficiently by Dry*Flo*.

   Every envelope density result is derived from the difference in two volumes, the consolidated Dry*Flo* and the consolidated Dry*Flo* with sample. That difference should be as large as possible simply for mathematical significance.

   For example, in one series of tests on a typical granular product where the product volume relative to the bed volume was varied from 6.9 to 41.7%, there was almost a 9.0% variability in envelope density. At the highest percentage, the sample quantity may have been sufficient for bridging of sample pieces to interfere with medium consolidation. At the lower percentage, small errors in consolidation were magnified in the difference value. However, the envelope volume within a ±1.3% error band was registered when the sample volume ranged from 30 to 35%. The current program for the GeoPyc automatically calculates the sample volume percentage. This percentage is a useful guide to optimum performance and should always be considered when assessing the validity of results.

   Guideline 2. Select sample chamber dimensions, Dry*Flo* volume, and specimen quantity to yield a sample volume percentage of at least 20%.

3. The error band was reduced to ± 0.95% when another series of tests was run with the material used in the above guideline and both Dry*Flo* and sample weights were held constant to the third decimal place. The reported sample-to-bed volume varied only between 32.1 and 33.4% in this case. Such control is not practical or even feasible in many instances, but this technique should be considered when possible.

   Guideline 3. Maintain constant all parameters susceptible to control for optimum reproducibility.

4. Both the blank and test steps of an envelope density determination consist of an equal number of preparation and analysis cycles.

   Preparation cycles are unrecorded, repetitious, agitation and consolidation attempts intended to orient the Dry*Flo* grains and the specimen into a uniformly mixed bed. Analysis cycles follow the preparation cycles and yield statistical information on consolidated volumes. The bed is expected to become more and more consolidated during the preparation cycles, but little or no consistent increase or decrease in value should be evident in the analysis cycles. Diminishing information can be gleaned once the cycles exceed a certain number. The results presented above were primarily obtained with 10 preparation and 5 analysis cycles. Some specimens require more, but fewer are adequate in other cases; 10 preparation and 5 analysis cycles are good starting numbers.

   Guideline 4. Choose the number of preparation and analysis cycles such that little or no consistent increase or decrease in value is revealed by the recorded data.

Accuracy

Make sure you follow the guidelines for reproducibility described earlier in this application note. Those guidelines must be followed, in conjunction with the guidelines listed below, to produce accurate envelope density measurements.

1. Sample shape influences GeoPyc results, but the effect cannot be rigorously quantified because shape itself is subject to infinite variation. The GeoPyc handles this problem by calibration. Two calibration values for each sample chamber, referred to as conversion factors, are noted in the operator's manual included with the GeoPyc.

   The first conversion factor (calculated factor) is simply derived from geometry and mechanical couplings and relates the plunger movement to chamber volume as if there were no sample shape influence. The second factor (adjusted factor) is modified to include an average shape influence experimentally determined from many different shapes. Neither is likely to apply precisely to any particular specimen. True calibration for shape can be achieved only when the predetermined envelope density of a representative specimen of the material in question is used.

   The representative specimen preferably is one from an evaluation procedure that was being followed before GeoPyc introduction. GeoPyc results can be expected then to track prior records. A completely nonporous specimen of the same shape as the material in question affords a degree of calibration but, because it is nonporous, cannot have the same surface texture and cannot be as satisfactory. Because in the final analysis the GeoPyc operates best as a comparison device, there is no real substitute for a truly representative specimen for calibration. A GeoPyc user should set aside enough of the selected calibration material to be able to recheck the calibration from time to time.

   Guideline 1. Select for calibration a quantity of the material in question and determine its envelope density by the prior test procedure or some other method.

2. Calibration itself will only be reproducible to the degree the guidelines given earlier for reproducibility are followed. Accordingly, the weight of the representative sample, the quantity of Dry*Flo*, and the sample chamber size should be selected on the basis of the amount of sample to be used later. Also, all calibration tests should be made with the same consolidation force and the same number of preparation and test cycles to be used in analyses.

   Guideline 2. Conduct calibration tests using parameters identical to those to be used in analyses.

3. Finally, a number of calibration tests should be made and the median selected as the conversion factor.

   Guideline 3. Use the median value from a number of calibration tests as the conversion factor for the material to be analyzed.