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TITANIUM DIOXIDE

DETERMINATION OF OIL ABSORPTION (OA)

METHOD: T4400.010.06.WP

TITANIUM DIOXIDE
(Ti-Pure®)

Determination of Oil Absorption

I. Principle

The oil absorption of a sample of titanium dioxide pigment is measured by determining the minimum amount of linseed oil which when worked into the pigment will change the mass from a comparatively dry state to one where all the particles cling together as a plastic mass and is measured as lbs. oil per 100 lbs. of TiO₂. The calculation uses two standards of known oil absorption.

II. Applicability

This test may be used to measure the oil absorption (OA) of any grade of titanium dioxide pigment.

III. Limitations

The most important part of the test is to work the oil into the TiO₂ pigment with a spatula and this must be the same from sample to sample.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

Not applicable.

B. Precision

1. Single Operator

The average analysis (\bar{X}), standard deviation (s), and 95% confidence limits (95% CL) established for the single operator precision of the method were as follows:

	\bar{X}	s	95% CL
OA	15.28	0.29	± 0.68

The above data were calculated from eight replicate analyses of the same sample by the same technician in one day.

2. Multiple Operator

The average analysis (\bar{X}), standard deviation (s) and 95% confidence limits (95% CL) established for the multiple operator precision of the method were as follows:

	\bar{X}	s	95% CL
OA	27.36	0.68	± 1.35

The above data were calculated from 32 replicate analyses of one sample performed by 8 technicians, two tests on each of two days.

C. Accuracy

No data are available; however, the method has proved satisfactory.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Smooth tempered glass plate, 18 inches square minimum x 1/4 inch, anchored to the table. Edges and corners should be ground smooth. Obtain locally.
2. A two place top loading balance, scoop, etc. Accuracy good to 0.01 g. (Top-loading digital balance e.g. Mettler PM is preferred). A shield around the balance is required.

Bal-guard Model TL-143-17-MET, available from Instruments for Industry and Research (I2R), 108 Franklin Avenue, Cheltenham, PA 19012.

Balance pan or scoop, aluminum or polypropylene 20 to 30 mL Capacity, for weighing pigment.

3. Two steel spatulas, 4" x 1/2" steel blade. Dexter 2494 INK KNIFE, available from Russell Harrington Cutlery, 44 Green River Street, Southbridge, MA.
4. Micro Burette, Class A, 2 mL capacity, graduated in .01 mL, with bulb reservoir for oil. Kimbel buret, automatic, #17132f, Fisher Scientific.
5. A piece of rubber, approximately 1/4" thick under the glass OA plate.
6. Rectangular base laboratory ring stand. Fisher Scientific, Catalog No. 14-670-B.
7. Clamps and clamp holders. Regular Hastaloy support clamps. Fisher Scientific, Catalog No. 05-754.
8. Thermometer clamps. To mount buret. Fisher Scientific, Catalog No. 05-809.

VI. Reagents (Reagent grade except as noted)

1. Linseed oil.

VII. Special Safety Considerations

A. Product Hazards

There are no unusual product hazards. However, titanium dioxide is regulated as an air contaminant and care should be exercised to minimize product dusting. Wipe up all spills with a damp paper towel. For more information see MSDS.

B. Procedure Hazards

Safety glasses should be worn.

VIII. Procedure

A. Operating Conditions

Not applicable.

B. Calibration

The low and high OA standards are run along with a reference standard for Statistical Process Control by the following procedure and used in the calculation in the section below. See the "Standard Sample Management" procedure for the standards and standard values. The low standard is usually about 10, while the high standard is in the 35-40 range.

C. Sampling

Not applicable.

D. Sample Analysis

1. Weigh 3.00 ± 0.01 g of the low standard or any other sample expected to be below 22 in OA. Weigh 1.50 ± 0.01 g of the high standard, or any other sample expected to have OA of 22 or above.
2. Zero the buret before starting every sample. Add sufficient oil from the buret to the glass plate to give approximately one-half of the expected volume and spread the oil with a 4-in. spatula. Then spread the pigment over the oil and work it into the pigment over a 1-2 minute period. Rubbing can be done in a standing or sitting position, but this position must be maintained throughout the entire series. The 4-in. spatula should be held in such a manner that a firm even pressure can be exerted on the pigment throughout the test. This is accomplished by holding the index finger part way down the blade. The stroke is generally a down and outward push away from the body along the long axis of the spatula. In this method the pigment will become spread out on the plate. It is scraped up into a pile with the spatula edge and again rubbed out.
3. Repeat the above procedure using $3/4$, and then $7/8$ of the expected volume of oil until the character of the mixture indicates the proximity of the endpoint. Then final oil may be added by drops or $1/2$ drops until the endpoint is reached. The oil is always added to the plate and the pigment oil mixture is rubbed into the oil in the manner described above. A standard rubbing time of 8 minutes has been specified. This means that the endpoint should be reached in approximately 8 minutes from the start of rubbing. It is necessary to incorporate the bulk

of the oil in the first 5-6 minutes. This leaves time to thoroughly incorporate the last bit of oil before the endpoint is reached. The endpoint is reached when the pigment is just moist enough to cling to one side of the spatula, but does not stick to the glass plate. If the pigment tends to smear or stick to the plate, the endpoint has been passed and the test must be repeated. The tester will, with experience, develop his/her technique to a point where he/she will be able to split a drop of oil to reach the endpoint.

4. After allowing the oil level in the buret to stabilize, record the volume of oil for all standards and samples to the nearest hundredth of a mL.

E. Calculations

1. The calculation may be made mathematically according to equations below, or by using an appropriately programmed computer or calculator.
2. Calculation

Let HS = oil absorption of high standard
LS = oil absorption of low standard
x = vol. of oil for high std. (mL)
y = vol. of oil for low std. (mL)
z = vol. of oil for the sample (mL)

For a low OA sample where 3.00 gram sample used:

$$\text{OA sample} = \text{LS} + \{(z - y) \times (\text{HS} - \text{LS})\} / (2x - y)$$

For a test where 1.50 gram of sample is taken, the above equation should be modified to read:

$$\text{OA sample} = \text{LS} + \{(2z - y) \times (\text{HS} - \text{LS})\} / (2x - y)$$

The OA value is reported to the first decimal place.

Example of expected high OA

HS = 38.0 high std. OA Value
LS = 11.0 low std. OA Value
x = 0.78 mL oil measured -high std., 1.5 g
y = 0.40 mL oil measured -low std., 3.0 g
z = 0.60 mL oil measured -sample, 1.5 g

$$OA = 11 + \{[(2 \times 0.60) - 0.40] \times (38.0 - 11.0)\} / [(2 \times 0.78) - 0.40]$$

$$OA = 29.6$$

IX. Quality Control

Three standards are run per technician on each shift. The high and low standards are used to establish the regression line. The value of the reference standard is determined and is used for Statistical Process Control.

X. Comments

Operator fatigue can influence the OA measurement. Therefore, samples should not be run when operator becomes fatigued.

XI. References

1. This procedure is similar to ASTM method D 281-31, reapproved 1970.

XII. Appendix

None

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