Report of the AOCS Subcommittee to Develop a Laboratory General Information Method for the Determination of Oil Retention in Bleaching Clay Filter Cake

First it was necessary to define the term "oil retention," then to design a method for determining it and finally to test the method for variability. The subcommittee recognized from experience that an adsorptive clay retains a *variety* of substances after bleaching, not merely oil alone, viz., (1) unoxidized oil; (2) oxidized oil; (3) soap; (4) organic color pigments (carotenoid pigments, chlorophyll, gossypol, etc.); and (5) organic matter other than the above (gums, resins, proteins, etc.).

Therefore the term "oil retention" might be a misnomer. However the subcommittee believed that from the refinery's standpoint "oil retention" should include items 1, 2, 3 and 4. The solvent "BAA" (equal parts by volume of benzene, acetone and alcohol) extracts all four of these provided the soap is first hydrolyzed to fatty acids with mineral acid. Consequently this solvent was chosen to use in an extraction method, the resulting extract from which would then be considered "oil retention."

"Total retention" includes item 5 in addition to the other four, i.e., it represents *total organic matter* retained. It can be determined merely by ashing the cake, taking into account the free moisture and volative matter (nonmoisture) content of the virgin clay.

The first samples examined by the subcommittee gave erratic results. Apparently this was due to nonuniformity of the filter cake, from inadequate mixing when the samples were prepared. The product is gummy and difficult to make uniform.

Consequently the next cake sample was prepared by first mixing the cake as thoroughly as possible with a paddle, putting it through a meat grinder and mixing it again. Both the filter cake and virgin clay samples were wrapped in plastic liners to prevent moisture loss.

The method is as follows.

General Information Method, Oil Retention

Definition

The method determines oil retention (unoxidized oil, oxidized oil, soap organic color pigments) in bleaching clay filter cake by extraction with a solvent.

Scope

Applicable to bleaching clay filter cake.

A. Apparatus

1. Beakers—250 ml capacity.
2. Steam bath.
3. Funnels, glass, short stem, 50 mm diameter.
4. Filter paper, Whatman No. 42, 90 mm.
5. Solvent extraction apparatus.
6. Extraction thimbles.
7. Wash bottles, 1 liter, with cork- or asbestos-covered neck.
8. Extraction flasks, 150 ml.
9. Dessicator.

B. Reagents

1. Hydrochloric acid, ACS grade, sp. gr. 1.19.
2. Methyl orange indicator solution (.1%), aqueous.
3. BAA solvent (equal parts by volume of benzene, acetone and alcohol, formula 30 or 3a.

C. Procedure

1. Weigh 5 ± 0.002 g filter cake samples into a 250 ml beaker.
2. Add ca. 100 ml distilled water and stir.
3. Add several drops of methyl orange indicator followed by concentrated hydrochloric acid, dropwise with stirring, until the solution is distinctly acid, then an additional 5 ml of hydrochloric acid.
4. Heat beaker on a steam bath over an open hole for 30 min, stirring occasionally.
5. Place a 12.5 cm Whatman filter paper (or equivalent) in a glass funnel and wet with distilled water.
6. Filter sample in beaker without suction. No oil should be present in the filtrate. (However, to assure no oil loss to the filtrate, the latter may be extracted.) Wash sample in the funnel twice with hot distilled water, then allow to drain until the paper is dry, or overnight.
7. Transfer filter paper and contents from funnel to an extraction thimble. Rinse funnel with BAA solvent into a tared extraction flask.
8. Extract 16 hr with a BAA solvent.
9. Remove extraction flask and evaporate off solvent on a steam bath, using a gentle current of dry air to carry off the vapors.
10. Continue heating on steam bath for another 15 min after no odor of solvent can be detected.
11. Cool flask in a dessicator and reweigh.

D. Calculations

$$\text{Oil retention, \%} = \frac{\text{wt combined extracts x 100}}{\text{wt. sample}}.$$

E. Precision

The standard deviation was 0.912 for a series of samples (see note 1).

F. Notes

The poor probable accuracy in the consensus of the committee, is due to the inherently nonuniform nature of the sample. It therefore represents the approximate unit of agreement that is attainable. This method is not intended to replace "house" methods now in use. It is presented for general information only.

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