

Determination of Unulfonated Matter in Linear Alkyl Benzene Sulfonic Acids using an InfraCal IR Filtometer

Linear alkyl benzene sulfonic acids are one of the most common precursors for the surfactant industry. They are typically neutralized and added to liquid dish formulations or converted to a powder for laundry detergents. The acids are generally made by reacting sulfur trioxide (SO_3) with linear alkyl benzene (LAB). The acid is digested and stabilized with a small water addition. The composition is typically:

Active – 96%
Sulfuric acid – 1.5%
Water – 0.5%
Unulfonated matter – 2%

The composition (and acid color) will vary depending on the molar ratio of SO_3 to LAB as well as the digestion conditions. The unulfonated matter (UM) conventionally refers to the total organic nonionics after neutralization.

The UM is conventionally analyzed by petroleum ether extraction of the neutralized sulfonic acid. The method is fairly time consuming and involves large quantities of petroleum ether. The nonionics are a mixture of unreacted LAB, paraffins and sulfones. The composition will vary with reaction and digestion conditions, but are generally consistent within a given sulfonic acid class. All three of these components are readily extractable and all possess a hydrocarbon chain.

A Wilks Enterprise InfraCal IR Filtometer with a 3.4 micrometer fixed filter/detector and equipped with a horizontal ATR cubic zirconia crystal provides an easy, fast and cost-effective method for analyzing the UM by measuring the amount of C-H (hydrocarbon) absorption in the extract. The amount of C-H absorption is proportional to the percent concentration of UM.



Figure 1. Petroleum ether extraction



Figure 2. Extract deposited on ATR Crystal

The methodology developed for UM determination involves a small-scale petroleum ether extraction (8 dram vial) followed by depositing an aliquot (50 μl) of the extract onto an ATR crystal (Figures 1 and 2). A delay time is introduced before measurement to allow the solvent to evaporate. The instrument measures a hydrocarbon absorbance proportional to the amount of organics deposited (as long as the film thickness is below the penetration depth of the IR beam).

The general calibration procedure is as follows (specifics should be worked out on a case by case basis):

- 1) Obtain 5-6 samples of sulfonic acid covering as large of a range in UM as possible. Ideally, the range should span 1-1.5% centered on the typical UM value. Spike samples with LAB if needed.
- 2) Analyze the samples according to the primary methodology (typically a gravimetric petroleum ether extraction).
- 3) Perform the extraction and deposit an aliquot of the organic layer onto the crystal. Allow an appropriate amount of time for the solvent to evaporate and perform the IR analysis.
- 4) Plot UM content vs. instrument response (per sample weight) as the calibration curve (Figure 3).

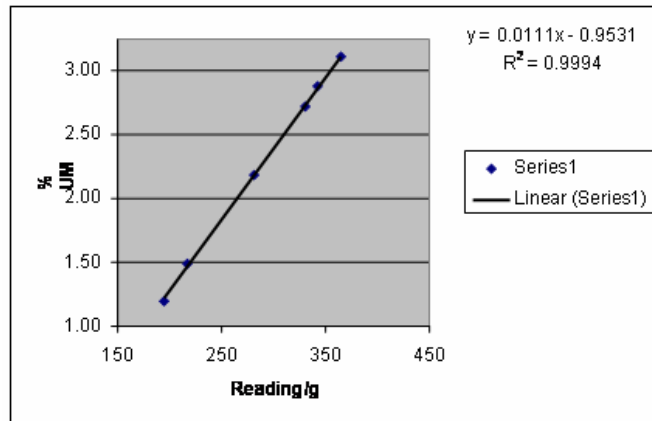


Figure 3: Typical calibration curve for LAB UM analysis using an InfraCal IR Filtometer

It is important to stay close to the sample size chosen since the reading/gram does depend on sample size. This is most likely a partitioning phenomenon due to the surfactant present. The other option is to establish the relationship to sample size and correct for it.

This procedure takes a fraction of the time of the conventional method and uses a fraction of the amount of petroleum ether.

Ordering Information

405-1009 **Infracal IR Platform Analyzer, Model HATR-T2** Complete with HATR-T2 Sample Stage with built-in Cubic Zirconia ATR crystal, Power Supply, Dust Cover and Instruction Manual

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