

DETERMINATION OF SULFUR CONTENT
ON GLASS FIBER FILTERS

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Total Sulfur content on 4.25 cm. or smaller glass fiber filters can be determined on the Exeter Analytical 440 Elemental Analyzer with single-sample automation (SAI) or 64 sample automation (HA-64). This procedure is suitable for oceanographic samples and environmental air samples where total sulfur per filter is expected to be less than 500 micrograms.

EQUIPMENT

Sulfur Operating Kit HA P/N 0240-0702H
or
Sulfur Operating Kit SAI P/N 0240-0702S
HA Filter Loading Kit P/N 125-00020
Ceramic Sleeves, Sulfur, HA P/N 6703-0582 (included in kit P/N 0240-0702H)
Glass Fiber Filters, Whatman GF/C, 4.25 cm.

PROCEDURE

The Sulfur Operating Kit is installed according to the instructions in the 440 manual. Helium pressure is adjusted at the regulator to provide a fill time of 40-60 seconds.

Using the HA Filter Loading Kit, one-half of a 4.25 cm. glass fiber filter is pressed into a ceramic sleeve. The process is repeated for the second half of the sample filter.

Filter Blanks (double-drop and single-drop) are prepared with 400-600 micrograms of acetanilide folded into one-half of the filter blank. The weight of acetanilide is not entered for a blank run.

The following calibration routine is recommended:

SAMPLE WHEEL POSITION	DESCRIPTION	REMARKS
1	Conditioner	1/2 Filter with 600-800 ug. S-Benzyl Thiuronium Chloride
2	Conditioner	1/2 Filter with 600-800 ug. S-Benzyl Thiuronium Chloride
3	Blank	1/2 Filter with 400-600 us. Acetanilide
4	Conditioner	1/2 Filter with 600-800 ug. S-Benzyl Thiuronium Chloride
5	STD 5	1/2 Filter with 600-800 ug. S-Benzyl Thiuronium Chloride

SAMPLE WHEEL POSITION	DESCRIPTION	REMARKS
6	STD 5	1/2 Filter with 600-800 ug. S-Benzyl Thiuronium Chloride
7	+Blank	1/2 Filter with 400-600 ug. Acetanilide
8	+Blank	1/2 Filter (double-drop w/position no. 7)

The plus sign (+) designation before the sample ID designates a double-drop operation of the 440. The autosampler will drop two ceramic capsules onto the ladle (comprising one complete filter) and run them as one. For sample filters, if 100 is entered for the sample weight, the weight of sulfur in micrograms will be reported on the printout for the complete filter.

The use of acetanilide with the filter blanks provides a correction for secondary gas effects resulting from carbon in the samples.. This is a detector-related phenomenon and does not indicate combustion interferences resulting from carbon in the sample.

For optimum results, the micrograms of carbon included in the Filter Blank should coincide with the average amount of carbon present on the sample filters.

It is recommended that the ceramic sleeves (P/N 6703-0582) be ignited at 800-900°C for two hours prior to use and stored in a clean glass container. Glass fiber filters used in the calibration procedure should be conditioned in the same manner as those used for sampling.