Application Note 17

TOTAL SULFUR MEASUREMENTS

Figure 1: Model CM320 Total Sulfur Analyzer

The UIC, Inc. Total Sulfur Analyzer rapidly and directly determines sulfur from a wide variety of sources including organics, coal, geologic materials, inorganics and natural products. The CM320 consists of a dual zone, high temperature furnace and a sulfur coulometer. The CM320 easily handles solid or liquid samples with concentrations from ppm levels to 100% without user calibration. UIC's analyzers are rugged, accurate and adaptable to most applications. They are used extensively in industrial, research and educational laboratories worldwide.

PRINCIPLES OF OPERATION

The coulometer cell is filled with a solution which initially contains a slight excess of free iodine. When SO_2 enters the cell, iodine is consumed. The amperometric-sensing circuit detects the deficiency of iodine in the solution and causes iodine to be electrically generated at a rate proportional to the sensed deficiency. When all of the SO_2 has been titrated, the iodine is restored to its initial concentration. The total current used to generate the iodine is integrated by the coulometer and digitally displayed in user-selectable units, usually micrograms S.

Samples are weighed into a ceramic combustion boat and covered with vanadium pentoxide (V_2O_5) or tungsten trioxide (WO_3). The sample is introduced into a 1050°C combustion furnace and oxygen is introduced periodically into the nitrogen carrier gas. The combustion products are passed over a combustion catalyst to ensure complete decomposition and are also passed over copper to quantitatively convert all sulfur to SO_2. The SO_2 is swept into the coulometer cell, where it is quantitatively absorbed and coulometrically titrated, as described above.

Conversion of sulfur to SO_2 is not matrix-dependent as in many other high temperature sulfur analyzers. Complete conversion of sulfur to SO_2 and coulometric measurement eliminates the need for standardization and/or sample calibration.

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PROCEDURE

1. Weigh a sample containing from 0.1 to 10 mg S. (The range of S given is the optimum range of S for detection. Larger amounts can be determined but will extend the analysis time. Conversely, smaller amounts may affect accuracy due to sample handling.)
2. Cover the sample with S-free V_2O_5 or WO_3 and place the boat containing the sample into the combustion tube. Seal the end of the tube and wait for the system to purge (1-2 min).
3. After purging, press Begin Analysis on the Coulometer and move the boat into the furnace. Coulometer will inject oxygen into the system periodically, depending on the amount of SO_2 titrated.
4. A blank run with only V_2O_5 or WO_3 should be performed to ascertain the instrument blank and a known sample should be run occasionally to confirm proper operation of the system.

Names, weights, volumes or areas of up to 50 samples can be entered, to be used by the CM5017S in calculating the final result. Analytical progress is digitally displayed in user-selectable units. A detailed report is printed while each sample is running that includes the final result. The results can also be stored on USB Flash Drive for further data handling.

MAINTENANCE

The coulometer cell solution should be changed after approximately 250 mg of S has been titrated. The copper tube filling must be regenerated or replaced when it becomes oxidized to within 2 cm of the end of the filling. In-house laboratory tests have indicated several hundred analyses before the need to regenerate.

RESULTS

The % S in the sample is calculated as follows:

$$\%S = \frac{(\mu g\ S\ Coulometer\ reading) - (\mu g\ S\ Blank)}{\mu g\ Sample} \cdot 100$$

Or, alternatively, ppm S is calculated as follows:

$$ppm\ S = \frac{(\mu g\ S\ Coulometer\ reading) - (\mu g\ S\ Blank)}{g\ Sample}$$

TABLE I-Typical results using sulfur coulometer with a variety of samples

SAMPLE	WT. RANGE, mg	THEORY, %S	FOUND, %S	# OF RUNS	RANGE FROM AVERAGE %S	STANDARD DEVIATION
Dibenzyl disulfide	2-50	26.03	26.06	8	0.18	0.1
Coal-Standard (4.03% S)	20-50	4.03	4.02	5	0.04	0.02
BaSO4	5-10	13.74	13.52	2	0.06	0.08
Na2SO4 (98.5% Pure)	20-25	25.05	24.98	2	0.03	0.04
Sulfamic Acid	5-20	33.02	32.9	2	0.01	0.01

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TABLE II - Specifications

Titration Rate	-	2400 µg S per minute
Accuracy	-	0.15% +/-2 digits for standard range
Sensitivity	-	0.1 µg S
Calibration	-	Factory Set
Typical Analysis Time	-	10 minutes
Range	-	ppm to 100%

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