TOTAL SULFUR

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.

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). The sample is introduced into a 1050°C combustion furnace and oxygen is introduced 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.

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 and place the boat containing the sample into the combustion tube. Seal the end of the tube and move the boat into the furnace
3. Immediately reset the coulometer and inject oxygen into the system. When the coulometer reading is stable, (approximately five minutes), record the micrograms S from the coulometer display.
4. A blank run with only V_2O_5 should be performed to ascertain the instrument blank and a known sample should be run occasionally to confirm proper operation of the system.

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:

$$\%\ S = \frac{(\text{ug S Coulometer reading}) - (\text{ug S Blank})}{\text{ug Sample}} \times 100$$

Or, alternatively, ppm S is calculated as follows:

$$\text{ppm S} = \frac{(\text{ug S Coulometer reading}) - (\text{ug S blank})}{\text{g Sample}}$$

TABLE I.

TYPICAL RESULTS USING COULOMETRICS SULFUR COULOMETER WITH A VARIETY OF SAMPLES

SAMPLE	WT. RANGE (mg)	THEORY % S	FOUND % S	# OF RUNS	RANGE FROM AVG. %S	STANDARD DEVIATION
Dibenzyl disulfide	2-50	26.03	26.06	8	0.18	0.10
Coal-Std. (4.03% S)	20-50	4.03	4.02	5	0.04	0.02
$BaSO_4$	5-10	13.74	13.52	2	0.06	0.08
Na_2SO_4 (98.5% Pure)	20-25	25.05	24.98	2	0.03	0.04
Sulfamic Acid	5-20	33.02	32.90	2	0.01	0.01

TABLE II.

SPECIFICATIONS

Titration Rate	-	2400 ug S per minute
Accuracy	-	0.15% +/-2 digits for standard range
Sensitivity	-	0.1 ug S
Calibration	-	Factory Set
Typical Analysis Time	-	5 minutes
Range	-	ppm to 100%