

Sulfur and Carbon Determination in Cement, Clay, Fly Ash, Limestone, Soil, and Ore

LECO Corporation; Saint Joseph, Michigan USA

Instrument: SC832 Series

Introduction

Both carbon and sulfur measurement in cement, and in the raw materials utilized in the production of cement such as limestone and fly ash materials, play significant roles in characterizing the materials and ensuring the quality of both the final and in-process products.

Carbon and sulfur content in soils play important roles in characterizing the quality of arable soils.

Determining the amount of carbon and sulfur present in metal-bearing ores is a necessary step in the metal recovery process. This information is used by mine operations to control metallurgical process kinetics, and to support environmental monitoring of the process waste.

Sample Preparation

A representative, uniform sample is required. Samples should be ground to pass through No.100 (150 micron) sieve.

NOTE: Carbon and sulfur results for ore and soil are typically reported on a dry basis. Samples are normally dried at 105 °C for 1 hour; alternately, moisture can be determined on a separate portion of the sample and results corrected to a dry basis. Some Reference Materials require drying; see the Certificate of Analysis for drying instructions if applicable.

Accessories

528-203 Ceramic Boat, 502-321 COM-CAT™*

*Required for SC832 and SC832DR models.

Calibration Samples

LECO, NIST, or other suitable cement, fly ash, ore, or limestone reference materials.

Instrument Model

The SC832 Series has three instrument models that include the base SC832, the dual range sulfur SC832 (SC832DR) and the high-temperature dual range sulfur SC832 (SC832HT). All of the SC832 models are applicable to this application and will offer different advantages, with some impacting the instruments method parameters, precision of results, required reagents, and resulting costs associated with the application.

The base SC832 model has a single sulfur IR cell and a furnace maximum temperature of 1450 °C. The single IR cell covers the range required for this application and simplifies the instrument setup and calibration. The maximum temperature of 1450 °C for the furnace is utilized for the application and also requires the addition of the combustion accelerator COM-CAT to the sample.

The SC832DR model has two sulfur IR cells providing the widest sulfur range and generally improved precision in samples with lower sulfur compositions. The maximum temperature of 1450 °C for the furnace

is utilized for the application and also requires the addition of the combustion accelerator COM-CAT to the sample.

The SC832HT model also has two sulfur IR cells providing the widest sulfur range and generally improved precision in samples with lower sulfur compositions. In addition, the SC832HT has the ability to support a furnace temperature of 1550 °C for this application. The higher furnace temperature of 1550 °C does not require the addition of the combustion accelerator COM-CAT to the sample resulting in a lower cost-per-analysis and a simplification of the sample preparation.



Method Parameters*

	SC832/SC832DR	SC832HT
Furnace Temperature	1450 °C	1550 °C
Lance On Delay	20 seconds	20 seconds
Manual Analysis Model	Single Sample	Single Sample
Nominal Blank Mass	1.0000 g	1.0000 g

Element Parameters

	Sulfur	Carbon
Wait for Baseline Stability	Yes	Yes
Starting Baseline	2 sec	2 sec
Use Comparator	Yes	Yes
Comparator	1.00	0.30
Minimum Integration Time	120 sec	60 sec
Maximum Integration Time	360 sec	360 sec
Range Select	Auto	
Range Lower Limit	800	
Range Upper Limit	950	

Automatically Started Analysis

Auto Detect Data		
Missed Time	3 sec	3 sec
Low Cell Autostart Level	0.01 V	0.01 V
High Cell Autostart Level	0.01 V	

Manually Started Analysis

Integration Delay	0 sec	0 sec
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*Refer to SC832 Operator's Instruction Manual for Method Parameter definitions.



Procedure—SC832/SC832DR

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by following the procedure outlined in step 5, using three replicates.
3. Determine instrument blank.
 - a. Login a minimum of three blanks.
 - b. Add ~1 g of 502-321 COM-CAT into a 528-203 Ceramic Boat.
 - c. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
 - d. Initiate the analysis by pressing the Analyze button.
 - e. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
 - f. Repeat steps 3b through 3e a minimum of three times.
 - g. Set the Blank according to the procedure outlined in the operator's instruction manual.
4. Instrument calibration/drift correction.
 - a. Login a minimum of three Standard reps for each calibration/drift reference material to be used for calibration/drift.
 - b. Weigh ~0.1 to 0.25 g of a calibration/drift reference material into the ceramic boat and enter the mass and reference material identification into the standard login.
 - c. Add 1 g of 502-321 COM-CAT into a 528-203 Ceramic Boat and thoroughly mix with the sample.
 - d. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
 - e. Initiate the analysis by pressing the Analyze button.
 - f. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
 - g. Repeat steps 4b through 4f a minimum of three times.
 - h. Calibrate/drift correct by following the procedure in the operator's instruction manual.
5. Sample Analysis
 - a. Login a Sample with a desired number of reps.
 - b. Weigh ~0.1 to 0.25 g of sample into the ceramic boat and enter the mass and sample identification into the sample login.
 - c. Add 1 g of 502-321 COM-CAT into a 528-203 Ceramic Boat and thoroughly mix with the sample.
 - d. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
 - e. Initiate the analysis by pressing the Analyze button.
 - f. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
 - g. Repeat steps 5b through 5f as necessary.

Procedure—SC832HT

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by following the procedure outlined in step 5, using three replicates.
3. Determine instrument blank.
 - a. Login a minimum of three blanks.
 - b. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
 - c. Initiate the analysis by pressing the Analyze button.
 - d. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
 - e. Repeat steps 3b through 3d a minimum of three times.
 - f. Set the Blank according to the procedure outlined in the operator's instruction manual.
4. Instrument calibration/drift correction.
 - a. Login a minimum of three Standard reps for each calibration/drift reference material to be used for calibration/drift.
 - b. Weigh ~0.1 to 0.25 g of a calibration/drift reference material into the ceramic boat and enter the mass and reference material identification into the standard login.
 - c. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
 - d. Initiate the analysis by pressing the Analyze button.
 - e. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
 - f. Repeat steps 4b through 4e a minimum of three times.
 - g. Calibrate/drift correct by following the procedure in the operator's instruction manual.
5. Sample Analysis
 - a. Login a Sample with a desired number of reps.
 - b. Weigh ~0.1 to 0.25 g of sample into the ceramic boat and enter the mass and sample identification into the sample login.
 - c. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
 - d. Initiate the analysis by pressing the Analyze button.
 - e. For manual systems, when prompted by the software, load the sample into the furnace and press the Analyze button.
 - f. Repeat steps 5b through 5e as necessary.

Typical Results

	SC832			SC832DR			SC832HT		
	Mass (g)	% Carbon	% Sulfur	Mass (g)	% Carbon	% Sulfur	Mass (g)	% Carbon	% Sulfur
Portland Cement	0.0952	0.22	0.829	0.1014	0.20	0.850	0.1028	0.19	0.856
NIST SRM 1886a	0.1032	0.20	0.820	0.1066	0.18	0.848	0.1063	0.20	0.846
0.834% Sulfur	0.0955	0.21	0.832	0.0984	0.18	0.851	0.1011	0.19	0.851
±0.032%	0.1090	0.20	0.834	0.1012	0.18	0.852	0.0956	0.19	0.846
Note: Carbon content not certified	0.1103	0.21	0.831	0.1093	0.23	0.848	0.0967	0.20	0.844
	Avg =	0.21	0.829	Avg =	0.19	0.850	Avg =	0.19	0.849
	s =	0.008	0.006	s =	0.02	0.002	s =	0.004	0.005
Coal Fly Ash	0.2513	0.42	0.156	0.2486	0.41	0.150	0.2479	0.41	0.151
NIST SRM 2690	0.2534	0.41	0.154	0.2476	0.40	0.149	0.2432	0.41	0.150
0.15%Sulfur	0.2487	0.41	0.157	0.2602	0.41	0.149	0.2437	0.41	0.151
± 0.01 %	0.2529	0.41	0.157	0.2481	0.41	0.149	0.2544	0.40	0.150
Note: Carbon content not certified	0.2496	0.42	0.156	0.2570	0.41	0.150	0.2548	0.41	0.151
	Avg =	0.41	0.156	Avg =	0.41	0.149	Avg =	0.41	0.151
	s =	0.004	0.001	s =	0.004	0.001	s =	0.003	0.001
Ore Tailings LECO	0.2539	2.63	2.45	0.2603	2.64	2.45	0.2585	2.63	2.45
502-320 Lot 1006	0.2578	2.62	2.43	0.2536	2.64	2.44	0.2431	2.63	2.43
2.63%Carbon	0.2559	2.64	2.45	0.2594	2.63	2.45	0.2532	2.62	2.43
±0.04 %	0.2462	2.62	2.44	0.2522	2.65	2.45	0.2559	2.63	2.44
2.44%Sulfur	0.2488	2.64	2.43	0.2568	2.64	2.42	0.2528	2.64	2.44
±0.09 %	Avg =	2.63	2.44	Avg =	2.64	2.44	Avg =	2.63	2.44
	s =	0.010	0.011	s =	0.008	0.012	s =	0.005	0.007
Soil LECO	0.2585	12.12	0.142	0.2568	12.04	0.134	0.2499	12.14	0.140
502-309 Lot 1012	0.2525	12.21	0.143	0.2552	12.05	0.135	0.2587	11.65	0.130
11.98%Carbon	0.2489	12.32	0.144	0.2496	11.79	0.132	0.2587	12.16	0.139
±0.44 %	0.2488	12.22	0.143	0.2550	12.05	0.135	0.2473	12.00	0.137
0.136%Sulfur	0.2518	12.01	0.142	0.2527	12.08	0.135	0.2515	11.88	0.136
±0.009 %	Avg =	12.17	0.143	Avg =	12.00	0.134	Avg =	11.97	0.136
	s =	0.115	0.001	s =	0.121	0.001	s =	0.208	0.004
Soil LECO	0.2552	22.31	0.321	0.2508	22.62	0.316	0.2480	22.30	0.327
502-814 Lot 1002	0.2503	22.22	0.321	0.2448	22.61	0.316	0.2487	22.48	0.326
22.6%Carbon	0.2564	22.34	0.322	0.2455	22.62	0.316	0.2403	22.34	0.324
±0.3 %	0.2526	22.28	0.322	0.2522	22.80	0.318	0.2407	22.24	0.323
0.323%Sulfur	0.2539	22.35	0.322	0.2527	22.80	0.317	0.2400	22.32	0.325
±0.012 %	Avg =	22.30	0.321	Avg =	22.68	0.316	Avg =	22.34	0.325
	s =	0.050	0.001	s =	0.090	0.001	s =	0.091	0.001
Soil LECO	0.2503	2.04	0.029	0.2556	2.06	0.282	0.2544	2.05	0.028
502-062 Lot 1016	0.2558	2.04	0.028	0.2566	2.00	0.026	0.2434	2.08	0.029
2.02%Carbon	0.2432	2.05	0.032	0.2547	2.02	0.028	0.2560	2.06	0.032
±0.06 %	0.2567	2.03	0.031	0.2438	2.07	0.028	0.2529	2.08	0.027
0.029%Sulfur	0.2503	2.06	0.032	0.2551	2.02	0.028	0.2608	2.10	0.031
±0.004 %	Avg =	2.05	0.030	Avg =	2.04	0.028	Avg =	2.07	0.029
	s =	0.011	0.002	s =	0.027	0.001	s =	0.019	0.002
Ore LECO	0.2588	1.54	1.10	0.2531	1.52	1.07	0.2419	1.54	1.06
502-319 Lot 1017	0.2499	1.55	1.10	0.2579	1.53	1.07	0.2554	1.52	1.08
1.53% Carbon	0.2571	1.54	1.08	0.2506	1.54	1.07	0.2469	1.56	1.08
±0.06 %	0.2571	1.55	1.08	0.2572	1.54	1.06	0.2515	1.53	1.07
1.10% Sulfur	0.2593	1.51	1.09	0.2518	1.54	1.07	0.2555	1.54	1.08
±0.03%	Avg =	1.54	1.09	Avg =	1.53	1.07	Avg =	1.54	1.07
	s =	0.015	0.011	s =	0.007	0.003	s =	0.013	0.011



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