Inorganic Application Note

Oxygen and Hydrogen Determination in Copper and Copper Alloys

Instrument

ROH600, TCH600

Sampling and Sample Preparation

Solid samples should be abraded with a clean file, rinsed in methanol, and dried in a stream of warm air.

Accessories

782-720S Crucibles, 761-739 Tin Pellet, 501-059 or 502-040 Tin Capsules, 617-997 Funnel (for Manual Top Load Procedure)

Calibration Samples

Copper or copper alloy reference materials with known O and H content (may be difficult to find); LECO 502-416, 502-457 One-Gram Steel Pins; NIST; or other suitable reference materials.

Method Parameters

Analysis Parameters	
Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis[1]
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled

[1]In earlier software programs this is the same as Auto Analysis. Auto Analysis is now used for instruments equipped with autosample loading capability; refer to the latest version of the operator's instruction manual for additional details.

Element Parameters	*Oxygen*	*Hydrogen*
Minimum Analysis Time	60 seconds	60 seconds
Significant Digits	5	6
Conversion Factor	1.000000	1.000000
Integration Delay	5 seconds	10 seconds
Comparator Level	1.000000%	1.000000%
Stop if Below	0.000000%	0.000000%

Furnace Parameters	
Furnace Control Mode	Current
Pre-Analyze Purge Time	—
Purge Time	10 seconds
Outgas Time	20 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	850 amps*
Outgas High Power	850 amps*
Outgas Ramp Rate	—
Analyze Low Power	765 amps*
Analyze High Power	765 amps*
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Temperature Sustain	None

**May vary depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*

ROH600/ TCH600

Procedure—Solid Samples

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g mass in Sample Login.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.
   d. Place one 761-739 Tin Pellet into a 782-720S Crucible.
   e. Place crucible on electrode pedestal.
   f. Press Loader Switch; the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2a through 2f a minimum of five times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~1 to 2 g of a calibration sample and enter mass in Sample Login.
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place sample into open port at top of loading head.
   d. Press Loader switch again, the loading head slide block will close and the lower electrode will open.
   e. Place one 761-739 Tin Pellet into a 782-720S Crucible.
   f. Place crucible on the electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 3a through 3g a minimum of five times for each calibration sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~1 to 2 g sample and enter mass in Sample Login.
   b. Proceed as directed in steps 3b through 3g.

Typical Results—Solid Samples

Sample	Mass g	O ppm	H ppm
Copper	1.8199	6.0	1.21
Wire	1.8352	6.1	1.25
	1.8397	5.9	1.24
	1.8129	5.7	1.29
	1.8096	5.3	1.34
	1.8808	6.0	1.39
	1.8132	6.4	1.49
	1.8170	5.3	1.22
	1.8293	5.6	1.28
	1.7830	5.3	1.31
	***X* =**	***5.8***	***1.30***
	***s* =**	***0.38***	***0.087***
OFHC	2.1448	3.4	0.75
Copper	2.0979	4.2	0.78
Rod	2.0956	3.7	0.72
	2.1093	4.5	0.87
	2.1532	3.4	0.77
	***X* =**	***3.8***	***0.78***
	***s* =**	***0.49***	***0.056***

Alternate Procedure—Powder/Chip Samples

If powder or chip samples are to be analyzed, they cannot be placed directly in the loading head. Options include weighing samples into capsules. Suitable tin capsules are available from LECO Corporation. There are issues related to blank, limited volume/sample weight and the increased time and manipulation to weigh samples in a capsule. In addition, there are two methods of manually loading a sample available.

Manual Analysis—The electrodes are opened after outgas and the sample is inserted into the crucible.

Manual Top Load—After outgas, loading head is opened and the sample is dropped into the crucible. For powder/chip samples, a LECO 617-997 Funnel can be inserted through loading head into crucible and the sample is transferred to crucible via the funnel.

The preferred alternative is to use the Manual Top Load function of the ROH600/TCH600. This option limits the outgassed crucible exposure to the atmosphere, reducing blank variability, subsequently improving precision.

Tin Capsule Procedure

1. Determine Blank.
   a. Enter 1.0000 g mass in Sample Login.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Place an empty tin capsule into open loading port at top of loading head.

      Note: Use same part number and lot number of capsules that will be used for the analysis of samples; leave capsule open.

   d. Press Loader Switch, the lower electrode will open.
   e. Place one 761-739 Tin Pellet into a 782-720S Crucible.
   f. Place crucible on electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 1a through 1g a minimum of five times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
2. Calibrate/Drift Correct.
   a. Weigh ~1 to 2 g of calibration sample into a tin capsule (leave capsule open) and enter mass in Sample Login.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Place capsule into open port at top of loading head.
   d. Press Loader Switch, the lower electrode will open.
   e. Place one 761-739 Tin Pellet into a 782-720S Crucible.
   f. Place crucible on electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 2a through 2g a minimum of five times for each calibration/drift sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
3. Analyze Samples.
   a. Weigh ~1 to 2 g sample into a tin capsule and enter mass in Sample Login (leave capsule open).
   b. Proceed as directed in steps 2b through 2g.

Typical Results Using Tin Capsule/Chip Samples

Sample	Mass g	O ppm	H ppm
Copper	1.0502	319	0.35
Chip	1.0890	311	0.26
	1.1200	307	0.32
	1.0563	313	0.39
	1.0610	310	0.36
	1.1026	311	0.34
	1.0283	312	0.31
	1.0858	311	0.31
	1.1236	309	0.32
	1.0808	310	0.34
	X =	***311***	***0.33***
	s =	***3***	***0.03***

Manual Top Load Procedure

1. Set Method Parameters as noted above with the following exceptions.
   a. Under Analysis Parameters set Analysis Type to Manual Top Load.
   b. Under Furnace Parameters set Pre-Analysis Purge Time to 50 seconds.
   c. Under Element Parameters set Minimum Analysis Time for oxygen to 45 seconds.
   d. Under Element Parameters set Comparator Level to 100% for oxygen.
2. Determine Blank.
   a. Enter 1.0000 g mass in Sample Login.
   b. Press Loader Switch on front of furnace, after a short delay the lower electrode will open.
   c. Place one 761-739 Tin Pellet into a 782-720S Crucible.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the outgas sequence will start automatically.
   f. When the outgas sequence is complete, an add sample message will appear in the lower left-hand corner of the instrument display. Press the Loader Switch and the loading head slide block will open.
   g. Place the 617-997 Funnel into the open loading head.
   h. Remove the funnel, press the Loader Switch, the loading head slide block will close and the analysis sequence will start and end automatically.
   i. Repeat steps 2a through 2g a minimum of five times.
   j. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~1 to 2 g of a calibration sample and enter mass in Sample Login.
   b. Press Loader Switch on front of furnace, after a short delay the lower electrode will open.
   c. Place one 761-739 Tin Pellet into a 782-720S Crucible.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the outgas sequence will start automatically.
   f. When the outgas sequence is complete, an add sample message will appear in the lower left-hand corner of the instrument display. Press the Loader Switch and the loading head slide block will open.
   g. Place the 617-997 Funnel into the open loading head and add sample taking care to make sure that all of the sample material is transferred into the crucible.
   h. Remove funnel and press the Loader Switch. The loading head slide block will close and the analysis sequence will start and end automatically.
   i. Repeat steps 3a through 3g a minimum of five times for each calibration/drift sample used.
   j. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~1 to 2 g of sample and enter mass in Sample Login.
   b. Proceed as directed in steps 3b through 3g.

Typical Results Manual Top Load Procedure—Chip Sample

Sample	Mass g	O ppm	H ppm
Copper	1.1382	314	0.23
Chip	1.0972	309	0.23
	1.1724	309	0.25
	1.0444	310	0.30
	1.1788	302	0.17
	1.0987	301	0.23
	1.0588	313	0.32
	1.1391	311	0.25
	1.0592	308	0.33
	1.1479	310	0.19
	X =	***309***	***0.25***
	s =	***4***	***0.05***

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