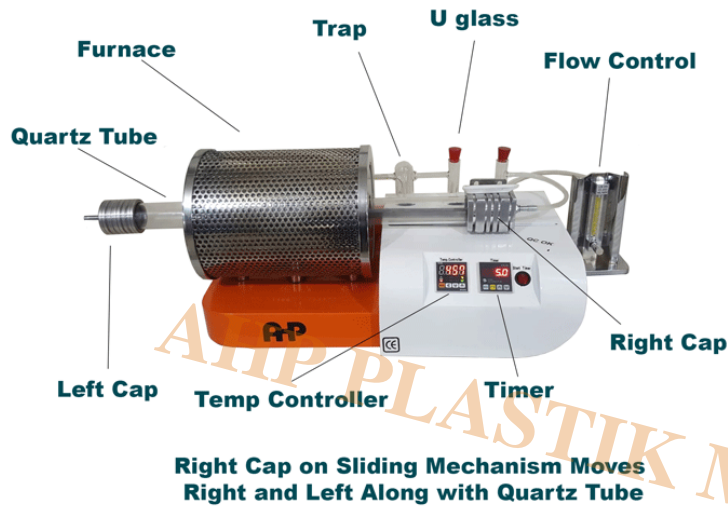


Key Tech Notes about Carbon Content Testing as per ISO 6964

Description



5 Procedure

5.1 Test conditions

Carry out the weightings at standard laboratory temperature (23 ± 2 C)

5.2 Test portion

Take three test portions as follows.

Weigh, to the nearest 0,000 1 g, approximately 1 g of the material taken from the consignment or from the wall of the pipe or from the fitting, reduced to small fragments.

5.3 Determination

The determination on each test portion shall be carried out as described in 5.3.1 to 5.3.5.

5.3.1 Place the test portion in the sample boat (4.1) and place the boat in the inlet of the combustion tube of the electric tube furnace (4.2) which has been previously heated to 550 ± 50 C.

Fix the nozzle to the tube inlet and then connect it to the outlet of the nitrogen stream after, if necessary, the nitrogen has passed through the purification system; circulate the nitrogen in the apparatus at a rate of 200 cm³/min for approximately 5 min.

5.3.2 Move the sample boat towards the center of the furnace, adjust the nitrogen flow rate to 100 cm³/min and leave to pyrolysis for approximately 45 min.

5.3.3 At the end of this period, return the sample boat to the cold section of the furnace and leave it there for 10 min while maintaining the flow of nitrogen.

5.3.4 Remove the sample boat from the furnace, allow it to cool in the desiccator (4.4) and weigh under the same conditions as prior to the pyrolysis. Record the mass to the nearest 0.0001 g.

5.3.6 Place the sample boat in the H_2SO_4 muffle furnace (4.3) at a temperature of 900 ± 50 C. and calcine until all traces of carbon black have disappeared. Allow to cool in the desiccator and weigh.

6 Calculation and expression of results

Calculate the carbon black content, expressed as a percentage by mass, from the formula

$$\frac{m_2 - m_3}{m_1} \times 100$$

where

m₁ is the mass, in grams, of the test portion;

m₂ is the mass, in grams, of the sample boat plus the test portion after pyrolysis at 500 C;

m₃ is the mass, in grams, of the sample boat after calcination at 900 C, with ash where appropriate.

Calculate the arithmetic mean of the carbon black contents determined on the three test portions and report the result to two significant figures.

NOTE – Where appropriate (see note to clause 21, calculate the ash yield, expressed as a percentage of the original mass, from the formula

$$\frac{m_3 - m}{m_1} \times 100$$

where

m is the mass, in grams, of the sample boat;

m, and m₃ have the Same meanings as above.

Calculate the arithmetic mean of the ash yields determined on the three test portions and report the result to two significant figures.

Main parameters that drastically effects the test results:

- 1- N₂ gas need to be pure and dry, having an oxygen content less than 20 ppm
- 2- Before weighting empty sample boat put it in the oven or furnace to be sure it is free of moisture
- 3- When putting sample boat into quartz tube, place it near to right cap inside quartz tube then close right cap, open N₂ purge, wait to be sure quartz tube is completely free of air, then slide cap along with quartz tube(while cap is closed) to left side so that sample boat slowly moves toward center of furnace.
- 4- When pyrolysis is finished and you want to take sample boat out of quartz tube, don't open right cap, slide quartz tube (while cap is closed) to right side so that sample boat comes out of center of furnace and let is cool down while it is inside quartz tube and N₂ purge is connected.
- 5- After sample boat cooled down inside quartz tube (while N₂ purge was connected) you can slowly take it out and let it cool for weighting.
- 6- Cooling need to be done in dry place like desiccator with silica-gel.
- 7- Be sure there is no leak from two end caps from outside to inside. When N₂ purge is going inside quartz tube it should be completely free from air.
- 8- Cleaning of the sample boat is very important. it should be free from any residue of black on it when you want to start the test. For cleaning black residue from sample boat, you can put it inside furnace at 550 or 600 C for 10 minute. then you will see that any residue of black on it will be vanished. Then put it inside desicator for cooling and then weight it for starting of the test.
- 9- It is very important that in starting of the test sample boats need to be inside of quartz tube, caps

closed but you should not take samples inside furnace. you should keep sample boats outside of hot area of furnace while N2 is connected. after some time that air is purged out, you can slide the sample boats to inside of hot zone of furnace.

10- It is very important when test is finished and you want to take samples out of hot zone of furnace. take sample boats to cold area while caps are closed. keep them in cold zone to cool down then open the end caps.

[Carbon Black Content Test Furnace \(CBC Tester\)](#)

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[ASTM D 1603 Carbon Black Content in Olefin Plastics / Testing Equipment](#)

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Category

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2. Standards

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