



# Sample Preparation and Sampling Methods for the Gas Analysis of Steel

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## 1 Overview

The following provides information on the sample preparation and sampling methods for the determination of carbon, sulfur, oxygen nitrogen and hydrogen in steel. The sampling methods and preparations introduced here are typical. Other methods may be applied if they are proper and suitable.

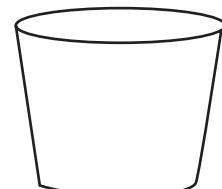
The main purpose of sampling is the selection of typical samples that represent a lot of corresponding products. The main purpose of sample preparation is the analysis of impurities that are not easily purged from the products. The samples must be free of any element that hinders the proper analysis of the samples.

It is very important to obtain accurate analysis results for quality control purposes. Sampling standards and sample preparation standards have been established in the manufacturing industry for this purpose.

## 2 Sampling of Molten Steel

There are a wide variety of samplers, among which the following four types are the most common. The use of these samplers depends on the element analysis method (i.e., wet chemical analysis, such as ICP, emission spectroscopy, X-ray fluorescence analysis, or C, S, N, O, or H analysis).

32 to 36 mm dia.



40 to 68 mm  
Height

28 to 34 mm dia.

**Figure 1:** Block-shaped sampling material. Casting or dipping material

13 to 15 mm  
thick



30 to 38 mm dia.

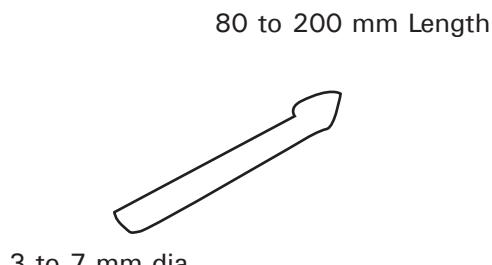
**Figure 2:** Disk-shaped sampling material. Casting or dipping material

4 mm  
thick



34 mm dia.  
12 mm  
thick

**Figure 3:** Lollipop-shaped sampling material. Casting or dipping material



**Figure 4:** Pin-shaped sampling material. Dipping method (vacuum sampling), Ladle out method (dropper suction)

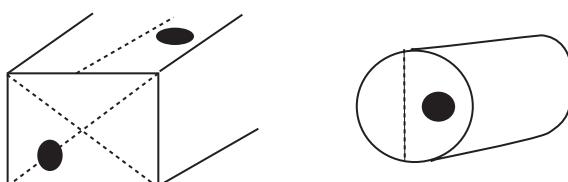
### 3 Preparation of Carbon and Sulfur Analysis Sample

If there is any scale, rust, de-carburized part, paint, metal coating, grease, oil, or dirt on the sample, remove it by cutting, polishing, acid cleaning, or degreasing.

#### 3.1 Collection from Steel Products or Pieces

##### 3.1.1. Steel Material with Large Cross-section

- (1) Collect a sample from the mid-point between the center of the cross-section and a corner with a drill that is 12 mm in diameter.
- (2) Collect a sample from the mid-point between the center axis and the side with a drill that is 12 mm in diameter.



**Figure 5:** Steel material with large cross-section

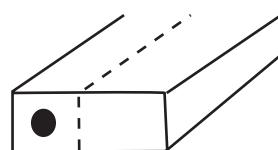
##### 3.1.2. Steel Material with Small Cross-section

- (1) Collect a sample by cutting the whole or half of the cross-sectional part with a shaper.



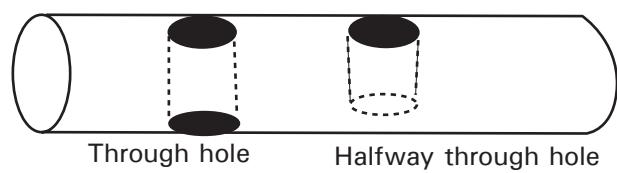
**Figure 6a:** Steel material with small cross-section

- (2) Collect a sample from the mid-point between the center and the side with a drill that is 6 mm in diameter.



**Figure 6b:** Steel material with small cross-section

- (3) Collect a sample from the middle part of the side by making a through or halfway-through hole with a drill that is 6 mm in diameter.



**Figure 6c:** Steel material with small cross-section



### 3.1.3. Thin Steel Plate

Collect a sample from the whole or half width of the plate in the direction that is perpendicular to the rolling direction with a nibbler, shaper, or drill that is 6 mm in diameter.

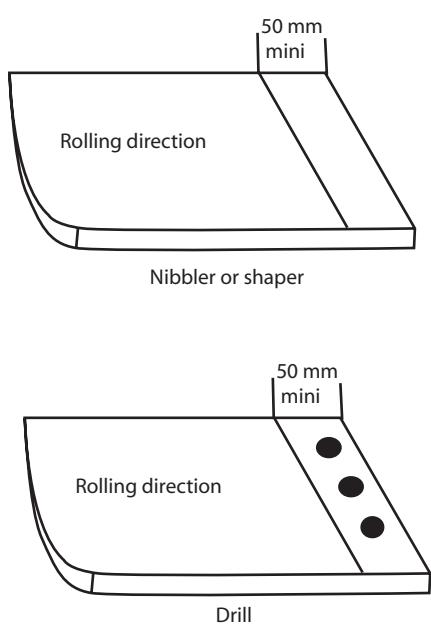


Figure 7: Thin steel plate

## 3.2 Collection from Cast Steel

3.2.1. Cut the block-shaped sampling material at a position 15 to 20 mm up from the bottom of the steel piece, remove the black scale, and collect a sample from the whole cross-section with a shaper, lathe, or drill. If the diameter of the drill point is approximately 6 mm in diameter, collect a sample from a quarter of the cross-section.

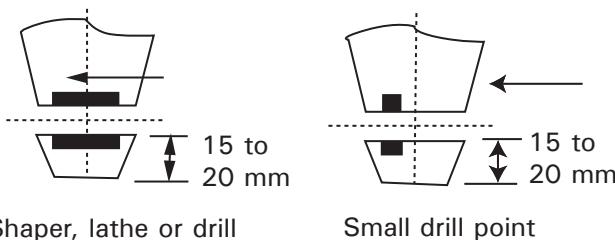


Figure 8: Collection from cast steel, step 1

3.2.2. Remove the black scale on the whole bottom of the block-shaped sampling material and collect a sample with a shaper, lathe, or drill. If the diameter of the drill point is approximately 6 mm in diameter, collect a sample from a quarter of the cross-section.

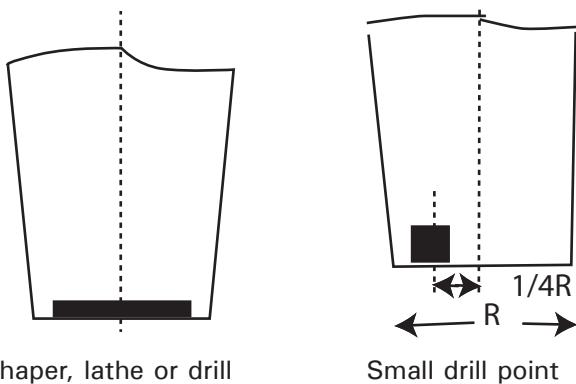


Figure 9: Collection from cast steel, step 2

3.2.3. Remove the black scale on the surface of the disk-shaped sampling material and collect a sample from the whole surface with a drill, shaper, or milling cutter.

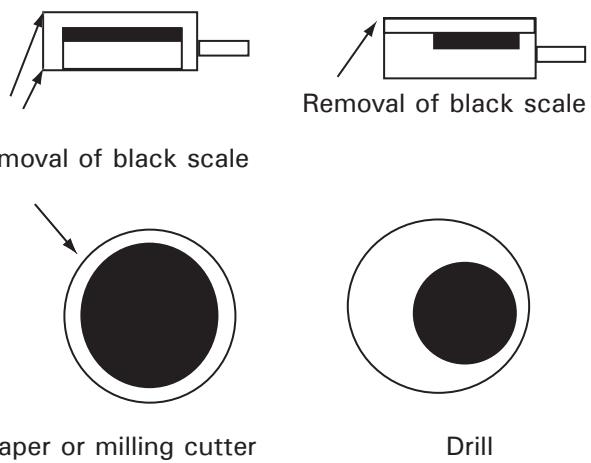


Figure 10: Collection from cast steel, step 3



3.2.4. Remove the black scale of the lollipop-shaped sampling material and collect a sample with a punching machine. Punching on lollipop-shaped sampling material

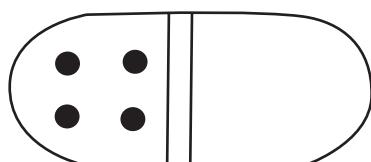
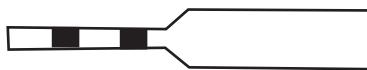


Figure 11: Collection from cast steel, step 4

### 3.3 Precautions

3.3.1. When collecting samples with a drill, shaper, milling cutter, or lathe, make sure that the surface of the samples will not be oxidized. Furthermore, do not use grease, oil, or any other lubricant or apply cooling water while cutting the samples.

3.3.2. When collecting samples from steel wires or foils, clean the surface of the steel wires or foils with ethyl alcohol or ethyl ether, and then cut out the samples with scissors.

3.3.3. If the sample pieces are soiled with grease or oil, clean the sample pieces with ethyl alcohol or ethyl ether.

3.3.4. All samples must be kept in clean glass bottles which are to be sealed tightly.

## 4 Preparation for Nitrogen Analysis

### 4.1 Collection from Steel

#### 4.1.1. Cuttings Samples

Collect cuttings samples from thick steel plates with a drill, shaper, milling cutter, or lathe.



Figure 12: Cuttings samples

#### 4.1.2. Block Samples

Collect block samples from thin or thick steel plates with a punching machine or cutting machine.



Figure 13: Block samples

#### 4.1.3. Core-shaped Samples

Collect core-shaped samples from steel wires or rods with a bolt clipper or fine cutter.



Figure 14: Core-shaped samples

#### 4.1.4. Cutout Samples

Collect cutout samples from thin steel plates with a nibbler.



Figure 15: Cut-out samples

### 4.2 Collection from Cast Steel

#### 4.2.1. Block-shaped Sampling Material

Collect "cuttings" samples or block samples.

#### 4.2.2. Disk-shaped Sampling Material

Collect cut-dust samples or block samples.

#### 4.2.3. Lollipop-shaped Sampling Material

Collect block samples.

#### 4.2.4. Pin-shaped Sampling Material

Collect core-shaped samples.



### 4.3 Collection of Cutout Samples

4.3.1. Fine grains of steel cause grain size segregation and hold a large quantity of nitrogen as a result.

### 4.4 Nitrogen Trace Analysis

4.4.1. Polish the surface of the block or core-shaped sample with a file or silicon carbide sandpaper. Then collect a sample from the new surface with a punching machine, cutter, or bolt clipper.

4.4.2. Clean the sample with a detergent such as ethyl alcohol and keep the sample in the detergent.

## 5 Preparation for Oxygen Analysis

### 5.1 Collection from Steel

5.1.1. Block Samples: Collect block samples from thin or thick steel plates with a punching machine or cutting machine.

5.1.2. Core-shaped Samples: Collect core-shaped samples from steel wires or rods with a bolt clipper or fine cutter.

### 5.2 Collection from Cast Steel

5.2.1. Block-shaped Sampling Material

Collect block samples.

5.2.2. Disk-shaped Sampling Material

Collect block samples.

5.2.3. Lollipop-shaped Sampling Material

Collect block samples.

5.2.4. Pin-shaped Sampling Material

Collect core-shaped samples.

### 5.3 Collection of Cutout Samples

5.3.1. Slowly polish the surface of the sample with a file or silicon carbide sandpaper until a new surface appears so that the surface will not be heated and will be dry so that the surface will not be oxidized.

5.3.2. Clean the sample with a solvent such as acetone and keep the sample in the solvent.

### 5.4 Oxygen Trace Analysis

#### 5.4.1. Machine Polishing

In addition to items in 5.1 Collection from Steel Products or Pieces to 5.3 Collection of Cutout Samples, make sure of the following.

1. Clean the sample with a solvent such as ethyl alcohol and keep the sample in the detergent.
2. The size of the surface area of the sample affects the analysis results. Therefore, the weight tolerance must be  $\pm 0.1$  g. Each sample must be the same in shape and dimensions so that there is no difference in surface area between them.

#### 5.4.2. Electrolytic Polishing

1. Electrolytic polishing is applied to block or core-shaped samples.

2. After polishing the sample, clean the sample with water and then with a solvent such as ethyl alcohol to eliminate any water. Keep the sample in the detergent.

#### Example of Electrolytic Polishing Conditions

Electrolyte:

Acetic acid and 60% perchloric acid in a volume ratio of 1 (up to 14) to 1.

1% TMAC + 10% acetylacetone and methyl alcohol solvent

(No water washing is required)

Cathode: 18-8 stainless or platinum

Anode: Sample

Electrolytic voltage: 10 V

Electrolytic current: 0.5 A

Electrolysis time: 5 min.



## 6 Preparation of Samples for Hydrogen Analysis

### 6.1 Collection from Cast Steel Material

- 6.1.1. Collect samples from the pin-shaped sampling material, put the samples into the water tank within 30 seconds, crack the samples, and cool to room temperature.
- 6.1.2. After the samples have cooled, further cool the samples with dry ice or liquid nitrogen. Transfer the samples for storage.
- 6.1.3. Take out the samples, wipe off the condensation on the samples, and slowly polish the samples with a file or sandpaper to remove the black scales so that the sample will not be heated.
- 6.1.4. Cut the sample with an appropriate tool, such as a bolt clipper.
- 6.1.5. Wash the sample with a solvent such as acetone, dry the sample, and keep the sample in a dessicator.

### 6.2 Sampling Precautions

- 6.2.1. Sampling materials must be clean.
- 6.2.2. No samples must be oxidized.
- 6.2.3. Samples must be free of slag.
- 6.2.4. Oxygen must not be leaked. Put aluminum, titanium, or metal silicon as a deoxidizing agent into the sampling chamber, provided that the deoxidizing agent will not affect the analysis of the samples.
- 6.2.5. Samples taken must be cooled constantly at high speed.
- 6.2.6. The sampling chamber must be made of a suitable material such as metal, quartz, or fireproof materials.

Table 1: Preparation of samples for Hydrogen analysis

	Block	Disk	Lollipop	Pin
Wet chemical analysis	**			
Emission spectroscopy	*	**	*	
Luminescent X-ray analysis	*	**	*	
C and S analysis	*		**	
N analysis	*		**	
O analysis				**
H analysis				**

\*\* Very favorable

\* Favorable



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