

JIS

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JAPANESE INDUSTRIAL STANDARD

Methods for Determination of
Lead in Steel

JIS G 1229-1980



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Translated

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Methods for Determination of Lead in Steel G 1229-1980

1. Scope

This Japanese Industrial Standard specifies the methods for determination of lead in steel.

2. General Items

General items common to determining methods shall be in accordance with JIS G 1201.

3. Classification of Methods

The method for determination of lead in steel shall be in accordance with one of the following:

(1) Lead molybdate gravimetric method

This method applies to the sample of 0.05 % and over in lead content.

(2) Dithizone extraction absorptiometry

This method applies to the sample of 0.01 % and over, up to 0.5 % in lead content.

(3) Iron separation dithizone extraction absorptiometry

This method applies to the sample of 0.0001 % and over, up to 0.01 % in lead content.

Applicable Standards:

JIS G 1201-General Rules for Chemical Analysis of Iron and Steel

JIS R 3503-Glass Apparatus for Chemical Analysis

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4. Lead Molybdate Gravimetric Method

4.1 Summary The sample is decomposed with hydrochloric acid and excessive acid is removed. Thereafter, lead sulfide is precipitated by hydrogen sulfide, filtered and this precipitate is decomposed with nitric acid.

Lead molybdate is precipitated by ammonium molybdate, filtered and its mass is measured by igniting that.

4.2 Applicable Fields When the sample contains a large amount of silicon, tungsten, niobium, tantalum, zirconium and chromium and is difficult to be decomposed with hydrochloric acid, the preparation of sample solution shall be in accordance with other operation.

4.3 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1+1)
- (3) Nitric acid
- (4) Nitric acid (1+1)
- (5) Sulfuric acid (1+20)
- (6) Aqueous ammonia
- (7) Hydrogen sulfide The hydrogen sulfide originated from bomb or hydrogen sulfide generator is used.
- (8) Hydrogen sulfide saturated hydrochloric acid After violently passing hydrogen sulfide through 1 l hydrochloric acid (1+99) for about 10 min, it is used.
- (9) Ammonium chloride

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- (10) Ammonium nitrate solution (5 w/v %)

- (11) Lead-saturated sulfuric acid The acid prepared by a method wherein 300 ml of sulfuric acid is added gradually into 1800 ml of water, this hot solution being added with 1 g of lead acetate, sufficiently mixed with stirring, stood for 24 h or more, and thereafter filtered with a filter paper (Class 5 C).

- (12) Ammonium molybdate solution The solution prepared by a method wherein after dissolving 10 g of ammonium molybdate (4 hydrate) with 200 ml water during heating it, it is filtered with a filter paper (Class 5 B). About 0.055 g of lead is precipitated by 1 ml of this solution.

- (13) Tartaric acid

- (14) Ammonium acetate solution Ammonium acetate saturated water solution at the ordinary temperature is diluted 2 times with water, added with 1 to 2 drops of Methyl Red-alcohol solution (0.1 w/v %) and neutralized by acetic acid or aqueous ammonia. The ammonium acetate solution is prepared by adding 30 ml of acetic acid per 1 l of this solution.

- (15) Ammonium acetate cleaning fluid The fluid prepared by diluting ammonium acetate solution [4.3 (14)] 10 times with water.

- (16) Ethyl alcohol (95 v/v % min.)

4.4 Apparatus The following apparatus shall generally be used.

Hydrogen Sulfide Generator The hydrogen sulfide generator of a suitable size is selected among Kipp's gas generators given in Attached Fig. 35 of JIS R 3503 according to the using amount and let generate hydrogen sulfide with iron sulfide and hydrochloric acid (1+1).

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4.5 Weighing-Out Amount of Sample The sample shall generally be weighed out in accordance with Table 1.

Table 1

Lead content %	Weighing-out amount of sample g	Using amount of hydrochloric acid (1+1) ml
0.05 and over, up to 0.1	10.0	100
0.1 and over	5.0	50

4.6 Operation4.6.1 Preparation of Sample Solution(1) Sample Easy to Be Decomposed with Hydrochloric Acid

(1.1) Transfer the weighed-out sample into a beaker (500 ml), cover it with a watch glass, add hydrochloric acid (1+1) according to Table 1 and decompose it by heating. After salts begin to be deposited by continuing to heat it, remove the watch glass and evaporate it to dryness as far as possible to the degree that the bottom of beaker does not turn brown due to burn and seizing. After cooling, dissolve salts by adding about 400 ml of hot water, add 10 g of ammonium chloride ⁽¹⁾, precipitate lead as sulfide by violently passing hydrogen sulfide [4.3 (7)] for 10 min, filter it with a filter paper (Class 5 A) and wash it several times with hydrogen sulfide saturated hydrochloric acid [4.3 (8)]. Discard the filtrate and washings.

(1.2) Receive a beaker (300 ml) under a funnel, decompose lead sulfide by pouring 10 ml of hot nitric acid (1+1) onto the precipitate on filter paper and further wash it several times with hot water. Combine the filtrate with the washings, dilute it to about 100 ml by adding water and add 2 g of tartaric acid thereto. After dissolving tartaric acid by mixing with stirring, neutralize it with aqueous ammonia and further add 5 ml of aqueous ammonia in excess.