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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS M 0202 : 1987** is replaced with **JIS M 0202 : 1999**.

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Testing methods for mine water and/or waste water

1 Scope This Japanese Industrial Standard specifies the testing method for mine water and/or waste water of the mines (including attached facilities, but excluding coal and petroleum mines), hereafter referred to as “mine water and/or waste water”.

- Remarks
- 1 The mine water refers to the water which wells out from underground excavated for the purpose of digging minerals.
 - 2 The waste water refers to the water which is discarded after having been used in the dressing plant, refinery and the like, and the water which flows out from the deposit of waste stones (including debris) and slag.
 - 3 This Standard shall be also applicable to the water discharged from the disposal facilities of mine water and waste water.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall apply.

JIS K 0016 *Iron standard solution*

JIS K 0094 *Sampling methods for industrial water and industrial wastewater*

JIS K 0101 *Testing methods for industrial water*

JIS K 0102 *Testing methods for industrial wastewater*

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS K 8005 *Reference materials for volumetric analysis*

JIS R 3503 *Glass apparatus for chemical analysis*

JIS R 3505 *Volumetric glassware*

JIS Z 8102 *Names of non-luminous object colours*

3 Common items The common items shall be as given in the following:

- a) **Quantitative ranges** The quantitative ranges given in respective testing methods shall be expressed by the mass (mg) in the final solution. However, as to the testing methods of the flame atomic absorption method, ICP atomic emission spectrometry, ion selective electrode method, ion chromatography and dissolved oxygen, these shall be expressed by the concentration (mg/L) in the final solution.

In the case where the quantitative ranges are different due to apparatus and measuring conditions, these matters have been appended.

- b) **Analytical accuracy** The analytical accuracy shall be indicated by the coefficient of variance⁽¹⁾ obtained in the repeated tests within the quantitative range of respective testing methods.

Note (1) Coefficient of variance (%) = $\frac{s}{\bar{x}} \times 100$

where, s : standard deviation

$\bar{x}$: mean value

- c) **Water** Water to be used in this Standard shall be that of A1 to A4 specified in **JIS K 0557**, unless otherwise specified in the item.

d) **Reagents**

- 1) The reagents which have been designated in **JIS K 0102** shall be used.

Further, if there are no standardized reagents, those of not impedimental to testings shall be used.

In the standardization of the standard liquids, the reference materials for volumetric analysis specified in **JIS K 8005** shall be used.

- 2) In the preparation of reagents, the concentration indicated in the parentheses after the name of solution means the approximate concentration. For example, sodium hydroxide solution (0.1 mol/L) indicates that it is the concentration of the sodium hydroxide solution of approximately 0.1 mol/L. In addition, the concentration indicated before the name of solution means the accurate concentration. However, in general, it shall be indicated by a non-fractional numerical value, and otherwise, a factor shall be obtained.
- 3) Water used for the preparation of reagents shall be of c), however, in the case where prescribed in respective items, it shall conform to that.
- 4) In the case where a standard solution is to be prepared by diluting the standard solution, 10 ml or over shall be taken by a whole pipette.
- 5) Naming of reagents should as closely as possible conform to the Nomenclature of Compounds established by the Chemical Society of Japan based on the IUPAC Nomenclature of Organic Chemistry and the IUPAC Nomenclature of Inorganic Chemistry.
- 6) As to the treatments such as the waste liquids of the reagents, cares shall be sufficiently taken in accordance with the related laws, ordinances and rules.

- e) **Glass apparatus and the like** The glass apparatus or glassware prescribed in **JIS R 3503** and **JIS R 3505**, unless otherwise specified, shall be used. However, in the case where particular apparatus is required, an example of it has been illustrated or explained in each item.

Remarks : In the case where arsenic, zinc and the like are to be tested, care shall be taken to the elution of these components from the borosilicate glass.

- f) **Measurement of absorbance (absorptiometry)** If there is no remarks about the absorption cell, that of 10 mm in optical path length shall be used.
- g) **Working curves (absorptiometry, atomic absorption method and ICP atomic emission spectrometry)** In preparing the working curves, divide the quantitative range given in the testing method into 4 to 6 stages, and take the standard solution so as to coincide with these. The working curves shall be prepared only within the quantitative range. In the absorptiometry, the working curves prepared in advance may be used.

In the atomic absorption method and ICP atomic emission spectrometry, the newly prepared working curves shall be used at the time of test, and when the same metal is tested on a large quantity of sample, the confirmation of absorbance or luminous intensity shall be carried out using a standard solution properly in the midst of the testing.

- h) **Notes, remarks and tables** The notes, remarks and tables are attached with a consecutive numbers in each clause.

4 Sample The sample mentioned in this testing methods refers to water sampled for carrying out each kind of test, and it shall be representing the nature of water at the site where it has been sampled (hereafter referred to as "water at the site"). The sample shall be put in the sample container which has been cleansed in advance and further with water at the site, filling it up, and carried into the test room after tightly stoppering, and the test shall be carried out promptly.

- a) **Sampling** In the sampling, under sufficient understanding of the test object, considering the conditions of the mine water and/or waste water, namely the operation and seasonal/time functional matters, sufficient cares shall be taken, so as to collect the samples representing water at the site and being in accordance with the test objects. In the case where there is the possibility of variation in quality of water, the number of samplings shall be increased so as to recognize the actual state of the mine water and/or waste water correctly, as required.

- 1) **Sampling spots** The sampling spots shall be the places where the necessary conditions conforming to the test objects are to be satisfied.

Example 1 **Mine water** Pit mouth and the like

Example 2 **Supernatant water and penetrant water of waste stones, slag deposits and the like** Water opening of each type

Example 3 **Various waste waters of refinery** Water opening of each type

Example 4 **Processing facilities of mine water and waste water** Front and rear of a processing facilities or immediately before flowing into a public water region

- 2) **Sample vessels** The sample vessels used shall be the polyethylene bottles with a ground stopper or colorless borosilicate glass bottles with a ground stopper and the like.

Avoid the use of the sample vessels with rubber, cork stopper and the like, in order to prevent the mingling of other substances and loss of each component from the sample.

The sample vessels, which have been cleansed sufficiently in advance, shall be used after being washed 2 or 3 times with water at the site.

Remarks 1 Depending on the kinds of the mine water and waste water, if insoluble matters and the like in the sample adhere to the wall of vessel, when the vessel is washed with water at the site, the vessels washed with water and dried shall be used. Since the microcomponent is apt to be lost by adhering to the

vessel walls, and also, there is the possibility that it elutes when reusing this vessel, cares shall be taken in washing. In addition, cares shall be taken for the possibility of elution of zinc, arsenic and the like from the glass bottles.

- 3) **Sampling methods** The sampling shall be so conducted as to represent the properties of water at the site.

In sampling, the sampling vessels shall be used, and other water sampler shall be used for the special case. Depending on the water properties of the sample, the water sampler made of metal (bucket and other sampler made of metal) shall not be used. When the bucket made of metal can not be used, the bucket made of polyethylene, a water sampler with handle (dipper) and the like may be used.

Before using the water sampler, it shall be sufficiently washed with water at the site in the same manner as the sample vessel.

The ropes, strings and the like attached to the water sampler shall be sufficiently washed with water at the site so as to prevent the sample from being deteriorated.

As to these general operations, refer to 4 of JIS K 0094.

Remarks 2 The automatic water samplers consist of a system which collects a definite quantity of sample into separate vessels for a definite time or in accordance with an established condition, and a composite system which makes up a mixed sample by collecting the sample proportional to the flow rate continuously or intermittently into the same vessel, however, in using these systems, it is necessary to pay attention to the following points:

- a) Avoid the application to the measurements of the dissolved oxygen causing errors on measured values due to the suction collection of the sample, or the oil content or the like which is prohibited from transferring of the sample.
- b) At the time of the tests on easy deteriorating characteristics such as the biochemical oxygen demand (BOD) and chemical oxygen demand (COD), the sample vessels shall be preserved in a low temperature equipment.

- 4) **Handling of sample** The sample shall be carried to the test room keeping its quality, and shall be tested promptly with sufficient care so as to prevent the sample from changing in quality as far as possible. Although the handling of samples differs depending on the respective test items, samples shall be treated as classified in the following.

- 4.1) **Where metallic components are tested using total quantity component samples** When carrying out the test requiring the respective total quantity⁽¹⁾ of the component to be determined, the sample shall be left as sampled. However, if it is impossible to carry out the test immediately after the sampling, the samples to be used in the tests of copper, lead, zinc, cadmium, chromium, iron, manganese, arsenic, mercury, molybdenum, calcium, magnesium, and aluminium, immediately after sampling, add with the rate of 10 ml of nitric acid⁽²⁾ per L of sample water (in the case of strong

alkaline sample, add nitric acid so as to be about pH 1, and record the volume of nitric acid used) and shake. As to the sample to be used for the test of arsenic and it does not include organic matter, nitrate and nitrite, the hydrochloric acid (for arsenic analysis) shall be used instead of nitric acid. Further, the samples to be used in the tests of iron (II) and chromium (VI) shall be tested as soon as possible without carrying out the treatment for preservation.

- 4.2) **Where metallic components are tested using dissolved component samples** When the test requiring the dissolved component only, the sample shall be filtrated by the filter paper type 5 C at the site⁽³⁾ (the first 50 ml of the filtrate is discarded), and its filtrate shall be put in the sample vessel. However, in the case where the test is unable to carry out immediately after the sampling, its filtrate shall be treated as in 4.1.
- 4.3) **Where other components are to be tested** As to the test items requiring the particular treatments such as the chemical oxygen demand (COD), biochemical oxygen demand (BOD), dissolved oxygen, hexane extracted material, cyanide, sulfide ion, they shall be in accordance with the treating method mentioned on each item.

Notes (1) The total quantity mentioned herein is the sum quantity of the insoluble matter and soluble matter.

(2) Considering the properties of the sample, the method of pre-treatment, methods of determination, etc., instead of nitric acid, hydrochloric acid may also be added.

(3) Even in the case where the filtrate still remains turbid due to colloid and the like, despite being filtrated, it may be recognized as has been filtrated.

Remarks 3 The addition of nitric acid or hydrochloric acid to the sample is for the sake of minimizing the errors due to respective components adsorbed to the vessel walls or to the change of composition, etc.

- 5) **Test items and sampling quantity** The sampling quantity shall differs depending on the number of items to be tested and the combination of the temperature of test component and storage treatment of samples. In the case where the tests are not carried out immediately and the samples are to be preserved, it is preferable well to determine the number of sample vessels and sampling quantity arranging the samples together by common preserving treatment of test items. An example⁽⁴⁾ shall be given in Table 4.1.

Table 4.1 An example of principal test items and sampling quantity

Test items	Sampling quantity L
Suspended matters, COD _{Mn} , COD _{Cr} , BOD, others	Approx. 2 to 3
Metallic components	Approx. 1
Cyan compounds, sulfide ion	Approx. 1

Note (4) The sampling quantity is indicated in the case where, among the test items, the samples of the items in common with the preserving treatment are extracted together.

6) **Measurements, observations and mentioned items at the time of sampling** The measurements, observations, and mentioned items at the time of sampling shall be in accordance with the following:

- 6.1) Name of sample and sample number
- 6.2) Name of sampling place
- 6.3) Sampling method
- 6.4) Date and time of sampling
- 6.5) Weather at the time of sampling
- 6.6) Name of sampler
- 6.7) Reference matters
 - 6.7.1) Conditions at sampling place (matters considered to be influential to the quality of water of the sample)
 - 6.7.2) Weather of the day of sampling and of the previous day
 - 6.7.3) Atmospheric temperature and water temperature at the time of sampling
 - 6.7.4) Appearance of sample (colour, turbidity, etc. of sample)
 - 6.7.5) When the sample includes the suspended matters and precipitates, approximate quantity and colours thereof.
 - 6.7.6) Presence of odour of the sample
 - 6.7.7) pH of sample (A simple measuring method may be used.)
 - 6.7.8) Other matters to be of value for reference

5 Measurement of flow rate The measurement of the flow rate shall be in accordance with 8 of JIS K 0094.

6 Decomposition of sample Since the mine water and/or waste water contain various types of inorganic and organic substances, the decomposing operation suitable to the sample and test item is required. Decomposition shall be conducted in accordance with the method designated in each test item, however, the common operations, in the case where the metallic components other than mercury, arsenic, iron (II) and chromium (IV) are to be tested, are specified as given in the following.

a) **Boiling under hydrochloric or nitric acidity** This method shall apply to the sample of extremely small in organic substances and/or suspending substances.

- 1) Take 100 ml of sample⁽¹⁾ (or an arbitrary definite quantity of 20 to 250 ml) into a beaker, add 5 ml of hydrochloric acid or 5 ml of nitric acid, and heat to boil for about 10 min.
- 2) After leaving it to cool, transfer it into the volumetric flask of an appropriate capacity as occasion calls, and add water to the marked line.

Note ⁽¹⁾ When testing the dissolved component, the dissolved component sample which has been filtered in accordance with 4 a) 4.2) is used.

b) **Decomposition by hydrochloric acid or nitric acid** This method shall be applied to the sample less in organic substances and containing hydroxides, oxides, sulfides, phosphates, etc. as the suspending substances.

- 1) Take 100 ml of sample⁽¹⁾ (or a definite quantity of 50 to 500 ml) into a beaker, add 5 ml of hydrochloric acid or 5 ml of nitric acid, and heat to concentrate until the liquid quantity becomes approximately 15 ml.
- 2) When the insoluble matter has remained, it shall be washed well with water after having been filtrated with filter paper of type 5 B.
- 3) After leaving it to cool, put the filtrate and washings into the volumetric flask of appropriate capacity, and add water to the marked line.

Remarks : In the case of the sample favorable to decomposition by the mixed acid of hydrochloric acid and nitric acid, after the treatment up to 1) has been carried out, add 5 ml of nitric acid when the hydrochloric acid has been used in 1) and add 5 ml of hydrochloric acid when the nitric acid has been used, cover with a watch glass and heat again, remove the watch glass when a vigorous reaction has terminated, heat further to expel nitrogen oxide, and concentrate to approximately 5 ml. In the case of deficiency in acidity in this operation, add a proper quantity of hydrochloric acid or nitric acid, and heat to dissolve by the same operation. When the insoluble matter has remained, add 15 ml of warm water and carry out the operations of 2) and 3).

c) **Decomposition by nitric acid and sulfuric acid** This method shall be applicable to the various kinds of samples. However, it is not suitable for the samples containing lead, barium, calcium, etc.

- 1) Take 100 ml of the sample⁽¹⁾ (or a definite quantity of 50 to 100 ml) into a beaker, add 10 ml of nitric acid and 10 ml of sulfuric acid (1+1), and heat to concentrate.
- 2) When the liquid quantity has become approximately 10 ml, cover the vessel with a watch glass. Continue heating until the white smoke of sulfuric acid generates, and organic matters, sulfides, etc. are decomposed.
- 3) When the organic matters, sulfides, etc. have remained undecomposed, repeat the operation 2) adding further 5 ml of nitric acid, and decompose by heating. When the decomposition has finished, take off the watch glass, and heat to condensate until nearly dry-hardened⁽²⁾.

4) After leaving it to cool, add 2 ml of hydrochloric acid (1 + 1), 2 ml of nitric acid (1 + 1) and 20 to 30 ml of water, and heat gently to dissolve the soluble salts. In the case where insoluble matters have remained, after having been filtered with filter paper type 5 B, wash sufficiently with water.

5) After leaving it to cool, put the filtrate and washings into the volumetric flask of an appropriate capacity, and add water to the marked line.

Note (2) Since the residual quantity of sulfuric acid is unable to be kept at a defined value, in the case where the flame atomic absorption method and ICP atomic emission spectrometry, in which the aqueous solution itself is sprayed, are applied, this method is not preferable.

d) **Decomposition by hydrofluoric acid, nitric acid and sulfuric acid** This method shall apply to the sample containing a large quantity of clayey materials and silicates.

1) Take 100 ml of sample⁽¹⁾ (or a definite quantity of 20 to 250 ml) into a quartz glass or porcelain evaporating dish, and concentrate the liquid quantity until it becomes 10 to 20 ml.

2) Transfer the contents into a platinum dish using water, scrape off the adhering matters of vessel walls by a glass stick with rubber, and transfer into the platinum dish with water.

3) Add 5 ml of hydrofluoric acid, 5 ml of nitric acid and 2 ml of sulfuric acid (1+1), heat to evaporate and allow to generate the white smoke of sulfuric acid for approximately 5 min⁽²⁾.

4) After leaving it to cool, add 5 ml of hydrochloric acid (1 + 1) and 20 to 30 ml of water, and dissolve the soluble salts by heating quietly. In the case where the insoluble matters have remained, after having been filtered with the filter paper type 5 B, wash sufficiently with water.

5) After leaving it to cool, put the filtrate and washings into the whole flask of appropriate capacity, and add water to the marked line.

e) **Decomposition by incineration** This method shall be applied to the sample which is easily incinerated without vaporizing the objective components at 450 to 500 °C. For the sample containing large quantity of ammonium chloride, magnesium chloride, calcium chloride, etc., when lead, iron and the like are the objective components, it shall not be appropriate since these are apt to be lost due to evaporation.

1) Take 100 ml of sample⁽¹⁾ (or a definite quantity of 20 to 250 ml) into a quartz glass or porcelain evaporating dish, and subject to evaporation to dryness.

2) Put the evaporation dish into an electric furnace, increase the temperature gradually, and incinerate⁽³⁾ by ignition to 450 to 500 °C.

3) After leaving it to cool, add 5 ml of hydrochloric acid (1 + 1) and 20 to 30 ml of water, and dissolve the soluble salts by heating quietly. In the case where the insoluble matters have remained, after filtration using the filter paper type 5 B, wash sufficiently with water.