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**Evaluation procedures of environmental
protection for water-soluble oilfield chemicals**

水溶性油田化学剂环境保护技术评价方法

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Forward

This Standard shall be used together with SY/T 6787-2010 “*Technical requirements for environmental protection for water-soluble oilfield chemicals*”.

Annex A and annex B in this Standard are normative.

This Standard was proposed by and shall be under the jurisdiction of environmental protection working group of standardization technology committee of petroleum industry.

Drafting organizations of this Standard: CNPC Environmental Inspection Department, and HSE testing center of environmental technology research institute of china national petroleum corporation.

Main drafters of this Standard: Li Xiuzhen, Li Jufeng, Li Binlian, Du Weidong, Chen Qin, Chen Yilong, Li Yuzhong, and Dong Zhihe.

Evaluation procedure of environmental protection for water-soluble oilfield chemicals

1 Scope

This Standard specifies the environmental protection requirements and evaluation method of biological toxicity, biodegradability and heavy metal content in water-soluble oilfield chemicals that are used in oil and gas field.

This Standard applies to the testing and evaluation of biological toxicity, biodegradability and heavy metal content during the production, purchase and use of water-soluble oilfield chemicals that are used in oil and gas field.

2 Normative references

The articles contained in the following documents have become part of this Standard when they are quoted herein. For the dated documents so quoted, all the modifications (excluding corrections) or revisions made thereafter shall not be applicable to this Standard. For the undated documents so quoted, the latest editions shall be applicable to this Standard.

GB/T 6679-2003 General rules for sampling solid chemical products

GB/T 6680-2003 General rules for sampling liquid chemical products

GB 7466-1987 Water quality - Determination of total chromium

GB/T 7468-1987 Water quality - Determination of total mercury - Cold atomic absorption spectrophotometry

GB/T 7475-1987 Water quality; Determination of copper, zinc, lead and cadmium - Atomic absorption spectrometry

GB/T 7485-1987 Water quality - Determination of total arsenic - Silver diethyldithiocarbamate spectrophotometric method

GB 11914-1989 Water quality-Determination of the chemical oxygen demand - Dichromate method

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product standard.

- b) Preparation of samples: take one part of oilfield chemical reagent test solution and nine part of 30g/L sodium chloride solution according to volume ratio; stir it with 11000r/min±300r/min for 30 minutes; after standing for 60 minutes, calibrate the concentration of the sample solution to be 10^6 mg/L. Then sequentially dilute the solution into sample solutions with different concentrations (10^5 mg/L, 10^4 mg/L...10 mg/L and 1mg/L). When the diluted concentration of the measured sample is close to 50% of the relative luminosity, it is appropriate.

5.2.1.3 Recovery of lyophilized powder microbial inoculums: take an ampoules containing 0.5g of lyophilized powder out of the refrigerator at 2°C~5°C and 2.5g / 100mL sodium chloride solution. Add 1 mL of cooled 2.5g / 100mL sodium chloride solution into the open ampoules with lyophilized powder; mix them well. Then place the ampoules with lyophilized powder in a thermos containing ice cubes. After two minutes, lyophilized powder microbial inoculums will recover and illuminate; This is recovery bacteria liquid for later use.

5.2.1.4 Preheat and zero-setting of instrument: switch on the bioluminescence photometer; preheat it for 15 minutes; then set it to zero for use.

5.2.1.5 Inspect the quality of lyophilized powder: take a 2 mL test tube; add 2 mL of 30 g/L sodium chloride; add 10 μ L of recovery bacteria liquid. cover the cork. Shake it upside down for five times to achieve a uniform. Unplug the cork; put the test tube in test chamber with bioluminescence photometer to test. If it emits light and shows the number more than 600mV, this bottle of lyophilized powder can be used for test; otherwise, it shall be replaced.

5.2.2 Determination

5.2.2.1 Permutation of test tubes: arrange the test tubes according to the following order on test tube rack: place the sample test tubes at the front row (referred to as sample tube); place the control test tubes at the back row (referred to as CK tube). Determine the sample with the same concentration for three times.

5.2.2.2 Add 30g/L sodium chloride solution: use quantitative liquid bottle to add 2 mL of 30g/L sodium chloride solution in each CK tube.

5.2.2.3 Add sample solution: before test, use 1 mol/L NaOH and 1 mol/L HCl to adjust the pH value of sample solution to 6.5-8.5. Use 2 mL pipette to add 2 mL of sample solution into each sample tube.

5.2.2.4 Add recovery bacteria liquid: after the recovery bacteria liquid is stable (about half an hour), from left to right on test tube rack, in the sequence: sample tube - CK tube - sample tube - CK tube..., use adjustable micropipette to accurately absorb 10 μ L of recovery bacteria liquid; add it in each tube; cover the cork; shake it upside down for five times; then remove the cork; put it back to original place. The timing shall be accurate to second when adding recovery bacteria liquid, which refers to the start time of the reaction between sample solution and luminescent bacteria liquid. After 15 minutes, record the end

specified in product standard, use distilled water to prepare test solution. Take some amount of test solution; determine the COD_{cr} of this test solution in accordance with requirements of GB 11914-1989.

6.2 Determining method of biochemical oxygen demand BOD

According to the determined result of COD_{cr} , determine BOD of this test solution in accordance with requirements of HJ/T 86-2002.

6.3 Biodegradability assessment method

Determine the index of biodegradability assessment method according to the ratio BOD / COD_{cr} .

6.4 Test report format

Test report format is shown in annex B.

7 Heavy metal assessment method

7.1 Experiment preparation

7.1.1 Instruments

7.1.1.1 Atomic fluorescence spectrometer.

7.1.1.2 Plasma emission spectrometer.

7.1.1.3 Atomic absorption spectrophotometer.

7.1.1.4 Temperature-control heating plate.

7.1.1.5 Muffle furnace.

7.1.1.6 Glass beads.

7.1.1.7 Quartz crucible.

7.1.2 Reagents

Unless otherwise specified, all reagents shall be analytical reagents that comply with national standard; test water shall be newly prepared distilled water.

7.1.2.1 Nitric acid (HNO_3): $\rho_{20} = 1.42g/mL$, guarantee reagent.

7.1.2.2 Hydrochloric acid (HCl): $\rho_{20} = 1.19g/mL$, guarantee reagent.

7.1.2.3 Perchloric acid ($HClO_4$): $\rho_{20} = 1.68g/mL$, guarantee reagent.

use 1% oxalic acid solution to fix its volume to be 25mL. Shake it well; store it for future use. In the process of digesting samples, there shall have corresponding standard sample and reagent blank; the whole process of digestion shall be carried out in ventilated chamber.

7.2.3 Digestion method of heavy metals - arsenic

Weigh water-soluble oilfield chemical samples (0.1g-0.2g of solid samples and about 1 g of liquid sample); place it in a PTFE tube. Add 5 mL of 10:1 nitric acid - perchloric acid into water-moistened solid sample or into liquid sample; shake it well. Cover it with watch glass; reflux it on electric hot plate at 100°C for one hour. Then rise the temperature to be 120°C to digest it until there is white smoke. After white smoke is completed, the sample solution shall be clear or has slight-color, then it is deemed completely digested; otherwise, continue to add acid to digest it. Take the digested sample out; cool to room temperature. After adding 10 mL of aqua regia to digest, then add 1 mL of potassium permanganate solution; place it for 30 minutes after shaking well; then use 1% oxalic acid solution to fix its volume to be 20mL. shake it well; store it for future use. In the process of digesting samples, there shall have corresponding standard sample and reagent blank; the whole process of digestion shall be carried out in ventilated chamber.

7.3 Heavy metal assessment method to oilfield chemicals

7.3.1 Assessment method to heavy metals - lead, cadmium and chromium

For water-soluble oilfield chemical samples that have been digested according to 7.2.1, determine the contents of heavy metals lead, cadmium and chromium in water-soluble oilfield chemicals in accordance with GB/T7475-1987, GB7466-1987 or determination method of lead (eight) ICP-AES method, determination method of cadmium (seven) ICP-AES method, and determination method of total chromium (two) ICP-AES method in "Water and Wastewater Monitoring and Analysis Methods (4th edition)".

7.3.2 Assessment method to heavy metal - mercury

For water-soluble oilfield chemicals samples that have been digested according to 7.2.2, determine the content of mercury in water-soluble oilfield chemicals in accordance with GB/T 7468-1987 or the determination method of mercury (four) - Atomic Fluorescence Spectrometry in "Water and Wastewater Monitoring and Analysis Methods (4th edition)".

7.3.3 Assessment method to heavy metal - arsenic

For water-soluble oilfield chemicals samples that have been digested according to 7.2.3, determine the content of arsenic in water-soluble oilfield chemicals in accordance with GB/T 7485-1987 or the determination method of arsenic (five) - atomic fluorescence spectrometry in "Water and Wastewater Monitoring and Analysis Methods (4th edition)".

7.4 Test report format

Test report format is shown in annex B.

8 Original record

The original record shall be complete; it shall include sampling person (sending person of samples), time and place of the experiment, test personnel, sampling log, sample flow record, sample name and number, sample detection items and detection basis, storage method of samples, quality control records, instrument calibration and maintenance records, test report and so on.

9 Test report

The test report shall include sample name, sample number, entrusting organization and address, entrusting person and contact number, entrusting time (sampling time), quantity of samples, the state of samples, storage method of samples, sampling person, sampling location, name and number of detection standard, name and number of instrument and equipment, and detection result. And determine the test result of water-soluble oilfield chemicals in accordance with SY/T 6787-2010. The test report format is shown in annex B.

Bibliography

- (1) Editorial Committee. Water and Wastewater Monitoring and Analysis Methods (4th edition). Beijing: China Environmental Science Press, 2002: 308~311, 341, 346, 364 and 384.

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