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## 中华人民共和国石油天然气行业标准

SY/T 5504.2—2013

中文/English

代替 SY/T 5504.2—2005

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### 油井水泥外加剂评价方法 第 2 部分: 降失水剂

Evaluation method for well cement additives—  
Part 2: Fluid loss additive

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## 目 次

前言 .....	II
1 范围 .....	1
2 规范性引用文件 .....	1
3 术语和定义 .....	1
4 要求 .....	1
5 试验方法 .....	2
6 评价报告 .....	5
附录 A（规范性附录） 油井水泥降失水剂评价报告 .....	6

## 前 言

SY/T 5504《油井水泥外加剂评价方法》分为8个部分：

- 第1部分：缓凝剂；
- 第2部分：降失水剂；
- 第3部分：减阻剂；
- 第4部分：促凝剂；
- 第5部分：防气窜剂；
- 第6部分：减轻剂；
- 第7部分：加重剂；
- 第8部分：膨胀剂。

本部分为SY/T 5504的第2部分。

本部分按照GB/T 1.1—2009《标准化工作导则 第1部分：标准的结构和编写》给出的规则起草。

本部分代替SY/T 5504.2—2005《油井水泥外加剂评价方法 第2部分：降失水剂》，与SY/T 5504.2—2005相比，主要变化如下：

- 增加了物理性能表（见4.1）；
- 修改了应用性能表，增加了稠化线形突变值（见4.2，2005年版的4.2.1）；
- 删除了抗盐水泥浆性能要求（见2005年版的4.2.2）；
- 删除了凝固点测定试验方法（见2005年版的5.3.4）；
- 修改了试验温度点的确定方法（见5.4.3.1，2005年版的5.4.3.1）；
- 修改了稠化时间试验的方法（见5.4.3.2，2005年版的5.4.3.2）；
- 修改了评价报告（见附录A，2005年版的第6章）。

请注意本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

本部分由石油钻井工程专业标准化委员会提出并归口。

本部分负责起草单位：中海油田服务股份有限公司。

本部分参加起草单位：中国石油化工股份有限公司石油工程技术研究院、中国石油集团海洋工程有限公司天津中油渤星工程科技有限公司。

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本部分代替了SY/T 5504.2—2005。

SY/T 5504.2—2005的历次版本发布情况为：

- SY/T 5960—1994。

本部分以中文和英文两种文字出版。当英文和中文两种版本有歧义时，以中文版本为准。

# 油井水泥外加剂评价方法

## 第2部分：降失水剂

### 1 范围

SY/T 5504 的本部分规定了油气井注水泥作业用油井水泥降失水剂的性能要求、评价方法及评价报告。

本部分适用于油气井注水泥作业用油井水泥降失水剂的评价。

### 2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本（包括所有的修改单）适用于本文件。

GB/T 4472 化工产品密度、相对密度的测定

GB/T 6678 化工产品采样总则

GB/T 6679 固体化工产品采样通则

GB/T 6680 液体化工产品采样通则

GB/T 8077 混凝土外加剂匀质性试验方法

GB 10238 油井水泥

GB/T 19139 油井水泥试验方法

SY/T 5377 钻井液参数测试仪器技术条件

SY/T 5504.1 油井水泥外加剂评价方法 第1部分：缓凝剂

### 3 术语和定义

SY/T 5504.1 界定的术语和定义适用于本文件。

### 4 要求

#### 4.1 物理性能

降失水剂的外观、密度、水分、细度的性能要求应符合表1的规定。

#### 4.2 应用性能

加有降失水剂的基准配方水泥浆（密度为  $1.90\text{g}/\text{cm}^3 \pm 0.01\text{g}/\text{cm}^3$ ），在其适用温度范围内的性能应符合表2的要求。

表1 油井水泥降失水剂物理性能要求

材料类别	检测项目	质量指标
固体类	外观	无受潮板结
	密度, g/cm <sup>3</sup>	基准密度±0.02
	水分, %	≤8
	细度 (0.315mm 筛余), %	<15
液体类	外观	均匀无霉变
	密度, g/cm <sup>3</sup>	基准密度±0.02

表2 加有降失水剂的基准配方水泥浆性能要求

检测项目	技术指标
初始稠度, Bc	≤30
稠化线形实变值, Bc	≤10
过渡时间, min	≤40
失水量, mL	≤150
游离液, %	≤1.4
24h 抗压强度, MPa	≥14

## 5 试验方法

### 5.1 仪器与材料

#### 5.1.1 仪器

主要包括:

- 电子天平: 精度 0.01g。
- 恒温干燥箱: 温度范围 0℃ ~ 200℃。
- 干燥器: 内盛变色硅胶。
- 称量瓶:  $\phi 25\text{mm} \times 65\text{mm}$ 。
- 比重瓶: 25mL 或者 50mL。
- 分析天平: 称量范围 0g ~ 200g, 精度 0.1mg。
- 试验筛: 0.315mm 方孔筛。
- 恒速搅拌器、常压稠化仪、增压养护釜、抗压强度试验机、增压稠化仪: 应符合 GB 10238 的要求。
- 钻井液密度计: 应符合 SY/T 5377 的要求。
- 加压密度计、250mL 量筒: 应符合 GB/T 19139 的要求。

#### 5.1.2 材料

主要包括:

- 油井水泥: 符合 GB 10238 要求的高抗硫酸盐型 (HSR) G 级水泥;
- 硅粉: 0.095mm ~ 0.075mm (160 目 ~ 200 目) 筛余小于 4%, SiO<sub>2</sub> 含量不低于 98%;
- 水: 蒸馏水;

- d) 降失水剂：待测样品；
- e) 与降失水剂配伍的其他外加剂材料。

## 5.2 组批与采样

### 5.2.1 组批

- 5.2.1.1 生产商以一个生产批次进行组批。
- 5.2.1.2 油田用户以一次进货的产品量进行组批。

### 5.2.2 采样

- 5.2.2.1 按批为单位进行采样，每批的采样数按 GB/T 6678 确定。
- 5.2.2.2 固体降失水剂的采样按 GB/T 6679 进行。
- 5.2.2.3 液体降失水剂的采样按 GB/T 6680 进行。
- 5.2.2.4 固体降失水剂的采样总量不少于 1000g，液体降失水剂的采样总量不少于 2000mL。
- 5.2.2.5 将按批采到的样品进行充分混匀后，等量分装于两个清洁、干燥的瓶中，密封并贴上标签。标签上应注明供货商名称、产品名称、批号和采样日期。两瓶样品中，一瓶用于评价，另一瓶留作仲裁用。留为仲裁的样品保存期应与样品保质期一致。

## 5.3 物理性能试验方法

### 5.3.1 外观测定

自然光条件下目测。

### 5.3.2 密度测定

固体降失水剂按 GB/T 4472 中比重瓶法进行，液体降失水剂按 GB/T 4472 中密度计法进行。

### 5.3.3 水分测定

固体降失水剂的水分测定按 GB/T 8077 进行。

### 5.3.4 细度测定

固体降失水剂的细度测定按 GB/T 8077 进行。

## 5.4 水泥浆性能试验方法

### 5.4.1 水泥浆基准配方的来源

主要包括：

- 用户：供货商提供的配方或与用户材料配套形成的配方。
- 供货商：供货商自行配套形成或根据用户要求组成的配方。
- 第三方：委托方提供的配方。

### 5.4.2 水泥浆制备

- 5.4.2.1 按照基准配方制备  $1.90\text{g}/\text{cm}^3 \pm 0.01\text{g}/\text{cm}^3$  的水泥浆，制备方法按 GB/T 19139 进行。
- 5.4.2.2 水泥浆密度测定按 GB/T 19139 进行。
- 5.4.2.3 在  $110^\circ\text{C}$  (BHST) 以上试验时，应加入 35% (BWOC) 的硅粉。

### 5.4.3 水泥浆性能试验

#### 5.4.3.1 代表性温度点的确定

代表性温度点依据该降失水剂样品的适用温度范围确定，选取下限温度（低温）和上限温度（高温）为两端温度点，均匀分割取点，相临温度点之差最大不超过 25℃。

#### 5.4.3.2 稠化时间试验

以基准配方，在全部的代表性温度点下，参考表 3 推荐的试验条件，按 GB/T 19139 进行稠化试验，读取基准配方的初始稠度、过渡时间、稠化线形突变值及稠化时间。

表 3 水泥浆试验条件和稠化时间适宜范围

试验温度 (BHCT) ℃	试验压力 MPa	升温时间 min	稠化时间适宜范围 min	强度试验温度 (BHST) ℃
40	19.1	28	90~210	61
50	25.9	32	100~225	71
60	32.8	37	110~240	82
70	39.6	41	120~255	92
80	46.5	45	130~270	102
90	53.3	49	140~285	113
100	60.2	53	150~300	123
110	67.0	57	160~315	133
120	73.9	61	170~330	144
130	80.7	65	180~345	154
140	87.6	69	190~360	164
150	94.4	73	200~375	175
160	101.3	77	210~390	185
170	108.1	81	220~405	196
180	115.0	86	230~420	206

#### 5.4.3.3 失水试验

以基准配方，在最高和最低的代表性温度点下，按 GB/T 19139 进行。

#### 5.4.3.4 游离液试验

以基准配方，在最高和最低的代表性温度点下，按 GB/T 19139 进行。

#### 5.4.3.5 抗压强度试验

以基准配方，在最高和最低的代表性温度点下，参考表 3 推荐的强度试验温度条件下，按 GB/T 19139 进行。

### 6 评价报告

油井水泥降失水剂的性能评价报告见附录 A。



附 录 A  
(规范性附录)

油井水泥降失水剂评价报告

样品名称: \_\_\_\_\_ 生产批号: \_\_\_\_\_ 生产厂商: \_\_\_\_\_  
 适用温度范围: \_\_\_\_\_ 委托单位: \_\_\_\_\_ 送(采)样日期: \_\_\_\_\_  
 送(采)样人: \_\_\_\_\_ 采样地点: \_\_\_\_\_ 评价单位: \_\_\_\_\_  
 执行标准: \_\_\_\_\_ 评价用水: \_\_\_\_\_ 水泥批号: \_\_\_\_\_  
 报告编号: \_\_\_\_\_

项目	指标	结果
固体类物理性能	外观	无受潮板结
	水分, %	≤8
	细度 (0.315mm 筛余), %	<15
	密度, g/cm <sup>3</sup>	基准密度 ± 0.02
液体类物理性能	外观	均匀无霉变
	密度, g/cm <sup>3</sup>	基准密度 ± 0.02
初始稠度, Bc	≤30	
过渡时间, min	≤40	
稠化线形突变值, Bc	≤10	
失水量, mL	≤150	
24h 抗压强度, MPa	≥14	
游离液, %	≤1.4	
备注	温 度: _____ °C 基准配方: _____ 稠化时间: _____ min	
结论		

实验人员:

审核:

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**Standard of Petroleum and  
Natural Gas Industries, P. R. C.**

**SY/T 5504. 2—2013**  
Replace SY/T 5504. 2—2005

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**Evaluation method for well cement additives—  
Part 2: Fluid loss additive**

**Date issued: 11—28—2013**

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## Contents

Foreword .....	9
1 Scope .....	11
2 Normative references .....	11
3 Terms and definitions .....	11
4 Requirements .....	11
5 Test methods .....	12
6 Evaluation report .....	14
Annex A (Normative) Evaluation report for well cement fluid loss additive .....	15

## Foreword

SY/T 5504 *Evaluation method for well cement additives* consists of eight parts:

- Part 1: *Retarder*;
- Part 2: *Fluid loss additive*;
- Part 3: *Dispersant*;
- Part 4: *Accelerator*;
- Part 5: *Anti-gas migration agent*;
- Part 6: *Light-weight agent*;
- Part 7: *Weighting agent*;
- Part 8: *Expanding agent*.

This is Part 2 of SY/T 5504.

This part was drafted according to GB/T 1.1—2009 *Directives for standardization—Part 1: Structure and drafting of standards*.

This part replaces SY/T 5504.2—2005 *Evaluation method for well cement additives—Part 2: Fluid-loss additive*. Compared with SY/T 5504.2—2005, the main changes of this part are as follows:

- Physical performance table is added (4.1 in this part);
- Application performance table is revised. Linear sudden vibration value of thickening-time is added (4.2 in this part, 4.2.1 in SY/T 5504.2—2005);
- Property requirements of brine cement slurry are deleted (4.2.2 in SY/T 5504.2—2005);
- Freezing point test method is deleted (5.3.4 in SY/T 5504.2—2005);
- Determination method of test temperatures

is revised (5.4.3.1 in this part, 5.4.3.1 in SY/T 5504.2—2005);

- Thickening-time test method is revised (5.4.3.2 in this part, 5.4.3.2 in SY/T 5504.2—2005);
- Evaluation report is revised (Annex A in this part, Chapter 6 in SY/T 5504.2—2005).

Some contents in this document may be involved in patent. The publisher of this document assumes no responsibility of patent.

This part is proposed by and under the jurisdiction of China Petroleum Standardization Committee for Drilling Engineering.

This part was drafted responsibly by China Oilfield Services Limited.

This part was drafted secondly by SINOPEC Research Institute of Petroleum Engineering (SRIPE), Boxing Company of China National Petroleum Offshore Engineering Co. Ltd.

This part was mainly drafted by Qingshun Wang, Hu Zhao, Keman Feng, Yongsong Wang, Yuwei Luo, Shidong Ding, Jiandong Wang.

This part replaces SY/T 5504.2—2005.

The following previous editions of SY/T 5504.2—2005 are:

- SY/T 5960—1994.

This part is published in both Chinese and English. In the event of any discrepancy between the tests, the Chinese version shall prevail.

## Evaluation method for well cement additives— Part 2: Fluid loss additive

### 1 Scope

This part of SY/T 5504 specifies property requirements, evaluation methods and evaluation reports for well cement fluid loss additive used in cementing operations.

This part is applicable to evaluation of well cement fluid loss additive used in well cementing operations.

### 2 Normative references

The following referenced documents are essential to the application of this document. Only the dated editions of all the dated normative references are applicable to this document. The latest editions (including all revision forms) of all the undated normative references are applicable to this document.

GB/T 4472 *Determination of density and relative density for chemical products*

GB/T 6678 *General principles for sampling chemical products*

GB/T 6679 *General rules for sampling solid chemical products*

GB/T 6680 *General rules for sampling liquid chemical products*

GB/T 8077 *Methods for testing uniformity of concrete admixture*

GB 10238 *Oil well cement*

GB/T 19139 *Testing of well cements*

SY/T 5377 *Specifications for drilling fluid testing instruments*

SY/T 5504.1 *Evaluation method for well cement additives—Part 1: Retarder*

### 3 Terms and definitions

Terms and definitions defined in SY/T 5504.1 are applicable to this paper.

### 4 Requirements

#### 4.1 Physical properties

The appearance, density, moisture, fineness, of the fluid loss additive should meet the requirements of Table 1.

**Table 1 Physical property requirements**

Category	Item	Index
Solid	Appearance	No sweating and hardening
	Density, g/cm <sup>3</sup>	Datum density ± 0.02
	Moisture, %	≤ 8
	Fineness (residue on 0.315mm sieve), %	< 15
Liquid	Appearance	Uniform and no mildew
	Density, g/cm <sup>3</sup>	Datum density ± 0.02

#### 4.2 Application properties

The properties of basic recipe cement slurry

(density is 1.90 g/cm<sup>3</sup> ± 0.01 g/cm<sup>3</sup>) at typical temperature should meet the requirements of Table 2.

Table 2 Property requirements for basic recipe cement slurry

Item	Index
Initial consistency, Bc	$\leq 30$
Sudden change value of consistency, Bc	$\leq 10$
Transition time, min	$\leq 40$
Fluid loss, mL	$\leq 150$
Free fluid, %	$\leq 1.4$
Compressive strength for 24h, MPa	$\geq 14$

## 5 Test methods

### 5.1 Apparatus and materials

#### 5.1.1 Apparatus

Mainly including:

- Electronic balance; With the precision of 0.01g.
- Constant temperature drying oven; With the temperature range of 0°C ~ 200°C.
- Drier; Containing discolored silica gel.
- Weighing bottle;  $\phi 25\text{mm} \times 65\text{mm}$ .
- Density bottle; 25mL or 50mL.
- Analytical balance; With weighing range of 0g to 200g and the precision of 0.1mg.
- Sieve; With mesh size of 0.315mm.
- Slurry mixer, atmospheric consistometer, pressurized curing chamber, compressive strength tester, pressurized consistometer; Shall meet the stipulation of GB 10238.
- Mud balance; Shall meet the stipulation of SY/T 5377.
- Pressurized density balance, 250mL graduated cylinder; Shall meet the stipulation of GB/T 19139.

#### 5.1.2 Materials

Mainly including:

- Well cement; Class G well cement (HSR), shall meet the stipulation of GB 10238.
- Silica flour; The residue on 0.075mm to

0.095mm (mesh 200 to 160) sieve is less than 4% and the SiO<sub>2</sub> content is no less than 98%.

- Water; Distilled water.
- Fluid loss additive; Test sample.
- Other additives; Compatible with the fluid loss additive.

### 5.2 Batching and sampling

#### 5.2.1 Batching

**5.2.1.1** Take one production circle as a batch unit for manufacturer.

**5.2.1.2** Take one purchasing batch as a batch unit for user.

#### 5.2.2 Sampling

**5.2.2.1** Take a batch as a sampling unit, and determine the hits of each batch in accordance with GB/T 6678.

**5.2.2.2** Obtain solid sample in accordance with GB/T 6679.

**5.2.2.3** Obtain liquid sample in accordance with GB/T 6680.

**5.2.2.4** The amount of solid sample shall be no less than 1000g, and the amount of liquid sample shall be no less than 2000mL.

**5.2.2.5** All samples obtained from the same batch should be well mixed, evenly stored in two clean and dry bottles, and sealed. Each bottle shall be labeled and identified with the supplier's name, product name, batch number and date of sampling. One of the two bottles is for

evaluation, and the other is for arbitration. Samples for arbitration and reference should have the same guarantee period.

### 5.3 Test procedures for physical properties

#### 5.3.1 Appearance test

Performed by visual observation under the natural light.

#### 5.3.2 Density test

Performed in accordance with GB/T 4472.

#### 5.3.3 Moisture test

Performed in accordance with GB/T 8077.

#### 5.3.4 Fineness test

Performed in accordance with GB/T 8077.

### 5.4 Test procedures for cement slurry properties

#### 5.4.1 Source of basic cement slurry recipe

Source of basic cement slurry recipe mainly includes:

- User test: The recipe provided by the supplier or the recipe formed by the user with his own compatible materials.
- Supplier test: The recipe provided by the supplier or on the request of the user.
- Third party test: The recipe provided by

the client.

#### 5.4.2 Preparation of cement slurry

5.4.2.1 Prepare basic recipe slurry of  $1.90\text{g}/\text{cm}^3 \pm 0.01\text{g}/\text{cm}^3$  in accordance with GB/T 19139.

5.4.2.2 Density test should be performed in accordance with GB/T 19139.

5.4.2.3 35% (BWOC) silica flour should be added when the test temperature is at  $110^\circ\text{C}$  (BHST) or higher.

#### 5.4.3 Cement slurry test

##### 5.4.3.1 Determination of the typical temperatures

The typical temperature should be determined by the applicable temperature range of the retarder. It is evenly selected between the lower temperature limit and the upper temperature limit, and the temperature interval should be no more than  $25^\circ\text{C}$ .

##### 5.4.3.2 Thickening-time test

Referring to the conditions listed in Table 3, thickening-time test should be carried out at all typical temperatures in accordance with GB/T 19139, and then the initial consistency, transition time, sudden change value of consistency and thickening-time of the basic recipe should be read and recorded.

**Table 3 Conditions for cement slurry test and fitting thickening-time range**

Test temperature (BHCT) $^\circ\text{C}$	Test pressure MPa	Time to BHCT min	Fitting thickening - time range min	Temperature for strength test (BHST) $^\circ\text{C}$
40	19.1	28	90~210	61
50	25.9	32	100~225	71
60	32.8	37	110~240	82
70	39.6	41	120~255	92
80	46.5	45	130~270	102
90	53.3	49	140~285	113
100	60.2	53	150~300	123

Table 3 (Continue)

Test temperature (BHCT) ℃	Test pressure MPa	Time to BHCT min	Fitting thickening - time range min	Temperature for strength test (BHST) ℃
110	67.0	57	160~315	133
120	73.9	61	170~330	144
130	80.7	65	180~345	154
140	87.6	69	190~360	164
150	94.4	73	200~375	175
160	101.3	77	210~390	185
170	108.1	81	220~405	196
180	115.0	86	230~420	206

#### 5.4.3.3 Fluid loss test

Prepare cement slurry according to the basic recipe, and then carry out fluid loss test at the lowest typical temperature and the highest typical temperature in accordance with GB/T 19139.

#### 5.4.3.4 Free fluid test

Prepare cement slurry according to the basic recipe, and then carry out free fluid test at the lowest typical temperature and the highest typical temperature in accordance with GB/T 19139.

#### 5.4.3.5 Compressive strength test

Prepare cement slurry according to the basic recipe, and then carry out compressive strength test at the lowest typical temperature and the highest typical temperature referring to the conditions listed in table 3, and in accordance with GB/T 19139.

### 6 Evaluation report

See Annex A for evaluation report of physical properties for well cement fluid loss additive.



**Annex A**  
**(Normative)**

**Evaluation report for well cement fluid loss additive**

Product name: \_\_\_\_\_

Batch number: \_\_\_\_\_

Supplier: \_\_\_\_\_

Applicable temperature range: \_\_\_\_\_

Client: \_\_\_\_\_

Date of sampling: \_\_\_\_\_

Person of sampling: \_\_\_\_\_

Sampling place: \_\_\_\_\_

Evaluation unit: \_\_\_\_\_

Executive standard: \_\_\_\_\_

Water for evaluation: \_\_\_\_\_

Batch number of cement: \_\_\_\_\_

Report number: \_\_\_\_\_

Item		Index	Result
Physical properties of solid	Appearance	No sweating and hardening	
	Moisture, %	≤8	
	Fineness (residue on 0.315mm sieve), %	<15	
	Density, g/cm <sup>3</sup>	Datum density ± 0.02	
Physical properties of liquid	Appearance	Uniform no mildew	
	Density, g/cm <sup>3</sup>	Datum density ± 0.02	
Initial consistency, Be		≤30	
Transition time, min		≤40	
Sudden change value of consistency, Be		≤10	
Fluid loss, mL		≤150	
Compressive strength for 24h, MPa		≥14	
Free fluid, %		≤1.4	
Note		Temperature: ____℃ (Basic recipe: _____ Thickening time: _____ min)	
Conclusion			

Experimenter: \_\_\_\_\_

Auditor: \_\_\_\_\_

Approver: \_\_\_\_\_

Date: \_\_\_\_\_