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代替 SY/T 5504—1996

中文/English

油井水泥外加剂评价方法 第1部分：缓凝剂

Evaluation method for well cement additives—Part 1: Retarder

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前　言

SY/T 5504《油井水泥外加剂评价方法》分为八个部分：

——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：充填剂。

本部分为SY/T 5504的第1部分。

本部分代替SY/T 5504—1996《油井水泥缓凝剂评价方法》。

本部分与SY/T 5504—1996相比，主要变化如下：

——修改了评价方式，由在单剂配制浆中评价改为在配套水泥浆体系中评价（1996年版的4.1.1，4.2.1；本版的4.2.1，5.4.1和5.4.2）；

——修改了试验温度，由原单一温度点试验改为：评价时在其系列温度点条件下进行，质检时在代表性温度点条件下进行（1996年版的4.1.2.1和4.2.2.1；本版的3.6，3.7与5.4.3.1）；

——修改了“稠化时间可调性”试验内容与判定方法（1996年版的4.1.2.1，4.2.2.1；本版的5.4.3.2）；

——删除了产品等级的评定内容与判定规则（1996年版的3.4和5.2）；

——删除了“168h抗压强度与48h强度之比”检验项目（1996年版的3.4，4.1.3和4.2.3）；

——增加了“稠化线形”检查项目（本版的4.2.1和5.4.3.2）；

——增加了不同循环温度条件下稠化和抗压强度的试验推荐条件（本版5.4.3.2中的表3）；

——增加了试验仪器及材料的要求（本版的5.1）；

——增加了质量稳定性试验方法及指标要求（本版的4.2.2和5.5）；

——增加了可选项目“温度敏感性”和“加量敏感系数”的评价内容（本版的4.2.1，5.4.3.6和5.4.3.7）；

——增加了材料外观、密度、水溶性、细度、凝固点及水分等理化性能试验方法及要求（本版的4.1，5.3和附录A）；

——增加了组批和采样方法（本版的5.2）。

本部分的附录B为规范性附录，附录A和附录C为资料性附录。

本部分由石油钻井工程专业标准化委员会提出并归口。

本部分起草单位：中海油田服务股份有限公司。

本部分主要起草人：沈伟、王蕾、王永松、杨振宇、方可炎、魏红梅、李维春。

本部分所代替标准的历次版本发布情况为：

——SY/T 5504—92，SY/T 5504—1996。

本部分以中文和英文两种文字出版。当英文与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1部分：缓凝剂

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缓凝剂的性能要求、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缓凝剂的评价。

2 规范性引用文件

下列文件中的条款通过SY/T 5504的本部分的引用而成为本部分的条款。凡是注日期的引用文件，其随后所有的修改单（不包括勘误的内容）或修订版均不适用于本部分，然而，鼓励根据本部分达成协议的各方研究是否可使用这些文件的最新版本。凡是不注日期的引用文件，其最新版本适用于本部分。

GB/T 510 石油产品凝点测定法

GB/T 4472 化工产品密度、相对密度测定通则

GB/T 6678—2003 化工产品采样总则

GB/T 6679—2003 固体化工产品采样通则

GB/T 6680—2003 液体化工产品采样通则

GB/T 8077—2000 混凝土外加剂匀质性试验方法

GB 10238—1998 油井水泥

GB/T 19139—2003 油井水泥试验方法

SY/T 5381 钻井液密度计技术条件

3 术语和定义

下列术语和定义适用于SY/T 5504的本部分。

3.1

40Bc～100Bc的时间 transition time from 40Bc to 100Bc

指水泥浆稠化试验过程中，稠度从首次到达40Bc开始计时，直至升到100Bc所经历的时间。

3.2

加量敏感系数 dosage sensitivity coefficient

表示缓凝剂加量变化对水泥浆稠化时间的影响程度的指标，用稠化时间增加率除以缓凝剂用量增加率得到。

3.3

温度敏感性 temperature sensitivity

温度变化对水泥浆稠化时间的影响程度，其指标用“温度升高5℃稠化时间变化率的绝对值”表示。

3.4

上限温度 upper temperature limit

缓凝剂的最高适用温度。

3.5

下限温度 lower temperature limit

缓凝剂的最低适用温度。

3.6

系列温度点 serial temperatures

对缓凝剂进行应用性能评价时所采用的系列试验温度。

3.7

代表性温度点 typical temperature

对缓凝剂进行质量检验时采用的试验温度。

3.8

质量稳定性 quality stability

不同生产批次之间产品质量的一致性程度。

3.9

参比样 reference sample

在质量稳定性试验时所用的缓凝剂基准材料。已通过合格检验、保存完好且尚处于保质期内的缓凝剂样品均可用作参比样。

3.10

适宜稠化时间范围 fitting thickening－time range

满足常规注水泥作业要求的合适的水泥浆稠化时间区域。

3.11

基准配方 basic recipe

在进行游离液、抗压强度、温度敏感性及质量稳定性试验时所采用的水泥浆配方。

4 要求

4.1 理化性能

外观、密度、水溶性、水分、细度、pH值及凝固点的性能要求参见附录A。

4.2 应用性能

4.2.1 加有缓凝剂的配套水泥浆（密度为1.90g/cm³±0.01g/cm³）在其适用温度范围内的性能应符合表1的要求。

表1 加有缓凝剂的水泥浆性能要求

检测项目	技术指标
初始稠度 Bc	≤30
40Bc～100Bc的时间 min	≤40
稠化线形	正常
游离液 %	≤1.4
24h抗压强度 MPa	≥14
稠化时间可调性	可调
加量敏感系数（可选项）	0.1～3.0
增加5℃稠化时间变化率（可选项） %	≤20

4.2.2 在相同的水泥批次、水质、配方及相同试验条件下，缓凝剂的质量稳定性应符合表2的要求。

表2 缓凝剂质量稳定性要求

检测项目	技术指标
稠化时间偏差率 %	≤20
40Bc～100Bc的时间之差的绝对值 min	≤10
注：在进行质量稳定性试验时，以代表性温度点作为试验温度。	

5 试验方法

5.1 仪器与材料

5.1.1 仪器

主要包括：

a）电子天平：精度0.01g。

b）恒温干燥箱：温度范围0℃～200℃。

c）干燥器：内盛变色硅胶。

d）称量瓶：25mm×65mm。

e）密度瓶：25mL或者50mL。

f）分析天平：称量范围0g～200g，精度0.1mg。

g）试验筛：0.315mm筛孔。

h）水泥浆恒速搅拌器：应符合GB 10238—1998附录A的要求。

i）常压稠化仪：应符合GB 10238—1998附录B的要求。

j）抗压强度试验机及强度养护设备：应符合GB 10238—1998附录C的要求。

k）增压稠化仪：应符合GB 10238—1998附录D的要求。

l）密度计：液体加压密度计或钻井液密度计，前者应符合GB/T 19139—2003第6章的要求，后者应符合SY/T 5381的要求。

m）250mL量筒：应符合GB 10238—1998附录B的要求。

5.1.2 材料

主要包括：

a）油井水泥：符合GB 10238—1998要求的高抗硫酸盐型（HSR）G级水泥。

b）硅粉：0.095mm～0.075mm（160目～200目）筛筛余小于4%，SiO_2含量不低于98%。

c）水：蒸馏水或生活饮用水。

d）缓凝剂：待测样品（质量稳定性检验时需要准备参比样）。

e）与缓凝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—2003中7.6确定。

5.2.2.2 固体缓凝剂的采样按GB/T 6679—2003中4.3.1进行。

5.2.2.3 液体缓凝剂取样按 GB/T 6680—2003 中 7.1 的规定进行全液位采样。

5.2.2.4 固体缓凝剂的采样总量不少于 500g，液体缓凝剂的采样总量不少于 1000mL。

5.2.2.5 将按批采到的样品进行充分混匀后，等量分装于三个清洁、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。三瓶样品中，一瓶用于评价，一瓶待评价合格后作参比样，一瓶留作仲裁用。留作参比样和仲裁的样品保存期为一年。

5.3 理化性能试验方法

5.3.1 外观测定

目测。

5.3.2 密度测定

按 GB/T 4472 中密度瓶法进行。

5.3.3 水溶性测定

对于湿混用固体缓凝剂，在 23℃ ± 1℃条件下配制成 5%的溶液，磁力搅拌器中挡转速下搅拌 10min 后，静置 10min，观察溶液底部有无明显的沉淀或分层现象。

5.3.4 水分测定

固体缓凝剂的水分测定按 GB/T 8077—2000 第 4 章进行。

5.3.5 细度测定

干混用固体缓凝剂的细度测定按 GB/T 8077—2000 第 6 章进行。

5.3.6 pH 值测定

液体缓凝剂的 pH 值使用精密 pH 试纸测定。

5.3.7 凝固点测定

液体缓凝剂的凝固点测定按 GB/T 510 进行。

5.4 水泥浆性能试验方法

5.4.1 水泥浆初始配方的来源

主要包括：

——用户检验：供货商提供的初始配方或与本油田材料配套形成的初始配方；

——供货商检验：供货商自行配套形成或根据用户要求组成的初始配方；

——第三方检验：委托方提供的初始配方。

5.4.2 水泥浆制备

5.4.2.1 按照初始配方制备 1.90g/cm^3 ± 0.01g/cm^3 的水泥浆，制备方法按 GB/T 19139—2003 第 5 章进行。

5.4.2.2 水泥浆密度测定按 GB/T 19139—2003 第 6 章进行。

5.4.2.3 在 110℃（BHST）以上温度试验时，应加入 35%（BWOC）的硅粉。

5.4.3 水泥浆试验

5.4.3.1 试验温度点的确定

对缓凝剂的评价应在其系列温度点条件下进行；对缓凝剂产品的质量检验应在其代表性温度点条件下进行。按照以下要求确定这些试验温度点：

a) 系列温度点包括在该缓凝剂的适用温度范围内所涉及的 60℃，80℃，100℃，120℃，150℃上限温度和下限温度，油田用户也可根据本油田的作业温度情况自行确定系列温度点。

b) 代表性温度点依据该产品的适用温度范围而定，若其适用范围偏向于 100℃以下，则以 80℃作为代表性温度点；若其适用温度范围偏向于 100℃以上，则以 120℃作为代表性温度点。油田用户也可根据本油田的作业温度情况自行确定该产品的代表性温度点。

c) 对于未知确切适用温度范围的缓凝剂产品或用户自组配套体系的外来缓凝剂产品的评价，应在其适用的大致温度范围内确定系列温度点（其中上下限温度通过摸索试验确定）。

示例1：如一缓凝剂适用温度大约为90℃～160℃，先进行100℃，120℃和150℃性能试验，然后按下列内容进行上下限温度摸索试验：

下限温度摸索试验：如100℃性能满足要求，则再降低10℃进行90℃试验，如不满足要求则升高10℃在110℃试验，依此类推，直至确定下限温度。

上限温度摸索试验：如150℃性能满足要求，则再升高10℃进行160℃试验，如不满足要求则降低10℃进行140℃试验，依此类推，直至确定上限温度。

5.4.3.2 稠化时间可调性试验

在确定的试验温度点条件下，稠化时间可调性试验按照下列内容进行：

a) 按初始配方配制水泥浆，在表3推荐的试验条件下按GB/T 19139—2003第9章进行稠化试验。

b) 改变缓凝剂加量，调出相邻点稠化时间间隔不小于30min，并且其值落在试验温度所对应的适宜稠化时间范围（见表3）内的3个配方。

表3 水泥浆试验条件和适宜稠化时间

稠化试验温度（BHCT）℃	稠化试验压力 MPa	升温时间 min	适宜稠化时间范围 min	适宜稠化时间范围中值 min	强度试验温度（BHST）℃
50	25.9	32	100～225	163	71
60	32.8	37	110～240	175	82
70	39.6	41	120～255	188	92
80	46.5	45	130～270	200	102
90	53.3	49	140～285	213	113
100	60.2	53	150～300	225	123
110	67.0	57	160～315	238	133
120	73.9	61	170～330	250	144
130	80.7	65	180～345	263	154
140	87.6	69	190～360	275	164
150	94.4	73	200～375	288	175
160	101.3	77	210～390	300	185
170	108.1	81	220～405	313	196
180	115.0	86	230～420	325	206

c) 对落在适宜稠化时间范围内的所有稠化曲线进行分析，如在稠化30min时间点至整个稠化曲线的4/5时间点之间无鼓包和走台阶线形（特指高于峰后基线稠度至少5Bc并且持续3min以上的鼓包，或者稠度超过30Bc的台阶），则判定稠化线形正常；否则继续分析，如存在至少有两个案例，则判定稠化曲线异常；如确认是个例，则重复该配方稠化试验，若试验结果中仍出现鼓包或走台阶线形，则仍判定稠化曲线异常，反之则判定稠化曲线正常。

注：峰后基线稠度指鼓包之后的最低稠度。

d) 读取落在适宜稠化时间范围内所有稠化曲线上的初始稠度，记录其中的最大值。

e) 读取落在适宜稠化时间范围内所有稠化曲线上40Bc～100Bc的时间，记录其中的最大值。

f) 对该试验温度下的所有稠化实验数据进行分析，如满足稠化时间随缓凝剂加量增加而增加的条件，则判定缓凝剂在该试验温度下稠化时间可调，反之则判为不可调。

示例2：某缓凝剂在110℃条件下稠化试验数据见例表1，其中有2号、3号及4号配方的稠化时间落在适宜范围内，且各自间隔不小于30min。综合分析所有试验数据，稠化时间随缓凝剂加量的增加而增加，稠化线形正常，因此判定该缓凝剂在110℃条件下稠化时间可调。

例表1

编　号	缓凝剂加量 %	水泥浆稠化时间 min
1	0.1	100
2	0.3	200
3	0.4	250
4	0.6	310
5	0.9	460

示例3：某缓凝剂在70℃条件下稠化试验数据见例表2，尽管其中有2号、3号及5号三个配方的稠化时间落在适宜稠化时间范围，相互间隔30min以上。如果仅对这三个数据进行分析是有规律的，但分析3号和4号数据，缓凝剂加量增加稠化时间反而降低，因此判定该缓凝剂在70℃试验条件下不可调。

例表2

编　号	缓凝剂加量 %	水泥浆稠化时间 min
1	0.2	80
2	0.4	150
3	0.6	200
4	0.7	160
5	0.9	240

5.4.3.3　基准配方的确定

从5.4.3.2的实验数据中选出水泥浆稠化时间落在适宜稠化时间范围中值±30min内的配方，将其作为基准配方用于下列游离液、抗压强度、温度敏感性及质量稳定性试验。

5.4.3.4　游离液试验

按GB/T 19139—2003第15章进行。

5.4.3.5　抗压强度试验

在表3推荐的强度试验温度条件下，按GB/T 19139—2003第7章进行。

5.4.3.6　温度敏感性试验（可选项）

在试验温度增加5℃条件下进行稠化试验，读取稠化时间。

按式（1）计算试验温度增加5℃条件下的稠化时间变化率：

$$TT_{CR}=\frac{|TT_{T+5}-TT_T|}{TT_T}\times 100\% \qquad \cdots\cdots (1)$$

式中：

TT_{CR}——增加5℃条件下稠化时间变化率；

TT_{T+5}——试验温度（T+5℃）条件下水泥浆的稠化时间，min；

TT_T——试验温度T条件下水泥浆的稠化时间，min。

5.4.3.7 加量敏感系数的确定（可选项）

从5.4.3.2试验得到的数据中选取稠化时间最接近适宜稠化时间范围上下限、且均加有缓凝剂的两组水泥浆配方数据，按下列内容进行计算：

a）按式（2）计算稠化时间增加率：

$$TT_{IR}=\frac{TT_2-TT_1}{(TT_1+TT_2)/2}\times 100\% \quad \cdots\cdots (2)$$

式中：

TT_{IR}——稠化时间增加率；

TT_1——配方1（接近下限点）的稠化时间，min；

TT_2——配方2（接近上限点）的稠化时间，min。

b）按式（3）计算缓凝剂用量增加率：

$$D_{IR}=\frac{D_2-D_1}{(D_1+D_2)/2}\times 100\% \quad \cdots\cdots (3)$$

式中：

D_{IR}——缓凝剂用量增加率；

D_1——配方1中的缓凝剂用量（BWOC）；

D_2——配方2中的缓凝剂用量（BWOC）。

c）按式（4）计算缓凝剂加量敏感系数：

$$DSC=\frac{TT_{IR}}{D_{IR}} \quad \cdots\cdots (4)$$

式中：

DSC——缓凝剂的加量敏感系数。

示例4：在100℃试验温度条件下调整某体系水泥浆配方中的缓凝剂的加量，调出两个配方所对应的稠化时间分别为：

1%缓凝剂（BWOC）——210min；

1.5%缓凝剂（BWOC）——290min。

计算得稠化时间增加率：$TT_{IR}=100\%\times(290-210)\div[(290+210)/2]=32\%$；

计算得缓凝剂用量增加率：$D_{IR}=100\%\times(1.5\%-1.0\%)\div[(1.5\%+1.0\%)/2]=40\%$；

计算得缓凝剂加量敏感系数：$DSC=TT_{IR}/D_{IR}=32\%\div40\%=0.8$。

5.5 质量稳定性试验方法

5.5.1 稠化对比试验

用缓凝剂参比样替代缓凝剂试样，按照基准配方配制水泥浆，在相同水泥批次、相同水质及相同试验条件下进行水泥浆稠化试验，读取稠化时间及40Bc～100Bc的时间，并与5.4.3.2中加有缓凝剂试样的同配方水泥浆的稠化实验数据进行比较。

5.5.2 对比数据的处理

5.5.2.1 按式（5）计算分别加有试样与加有参比样的两组水泥浆的稠化时间偏差率：

$$TT_{DR}=\frac{|TT_S-TT_R|}{TT_R}\times 100\% \quad \cdots\cdots (5)$$

式中：

TT_{DR}——稠化时间偏差率；

TT_S——加有缓凝剂试样的水泥浆的稠化时间，min；

TT_R——加有缓凝剂参比样的水泥浆的稠化时间，min。

5.5.2.2 计算分别加有试样与加有参比样的两组水泥浆的40Bc～100Bc的时间之差的绝对值。

6 评价报告

油井水泥缓凝剂的应用性能评价报告格式见附录B，理化性能评价报告格式参见附录A，质检报告格式参见附录C。

附 录 A
（资料性附录）
油井水泥缓凝剂理化性能评价报告格式

样品名称：_____ 生产批号：_____ 供货商：_____
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：_____ 报告编号：_____

检验项目	质量指标	试验结果
外观	（液体材料）均匀无霉变 （固体材料）无受潮板结	
密度（20℃±1℃） g/cm³	规定范围	
水溶性	良好	
水分（固体材料） %	≤8	
细度（0.315mm筛筛余） %	<15	
pH值	规定范围	
凝固点 ℃	规定范围	

主检人： 审核： 批准： 评价日期：

附 录 B
（规范性附录）
油井水泥缓凝剂应用性能评价报告格式

样品名称：_____　生产批号：_____　供货商：_____
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：_____

试验温度与基准配方	评价项目	质量指标	试验结果
上限温度：_____℃ 基准配方： _____ _____ （稠化时间：_____min）	初始稠度 Bc	≤30	
	40Bc～100Bc的时间 min	≤40	
	稠化线形	正常	
	游离液 %	≤1.4	
	24h抗压强度 MPa	≥14	
	稠化时间可调性	可调	
	加量敏感系数（可选项）	0.1～3.0	
	增加5℃稠化时间变化率（可选项） %	≤20	
下限温度：_____℃ 基准配方： _____ _____ （稠化时间：_____min）	初始稠度 Bc	≤30	
	40Bc～100Bc的时间 min	≤40	
	稠化线形	正常	
	游离液 %	≤1.4	
	24h抗压强度 MPa	≥14	
	稠化时间可调性	可调	
	加量敏感系数（可选项）	0.1～3.0	
	增加5℃稠化时间变化率（可选项） %	≤20	

表（续）

试验温度与基准配方	评价项目	质量指标	试验结果
试验温度：_____℃ 基准配方： _____ _____ （稠化时间：_____min）	初始稠度 Bc	≤30	
	40Bc～100Bc的时间 min	≤40	
	稠化线形	正常	
	游离液 %	≤1.4	
	24h抗压强度 MPa	≥14	
	稠化时间可调性	可调	
	加量敏感系数（可选项）	0.1～3.0	
	增加5℃稠化时间变化率（可选项） %	≤20	
试验温度：_____℃ 基准配方： _____ _____ （稠化时间：_____min）	初始稠度 Bc	≤30	
	40Bc～100Bc的时间 min	≤40	
	稠化线形	正常	
	游离液 %	≤1.4	
	24h抗压强度 MPa	≥14	
	稠化时间可调性	可调	
	加量敏感系数（可选项）	0.1～3.0	
	增加5℃稠化时间变化率（可选项） %	≤20	
试验温度：_____℃ 基准配方： _____ _____	初始稠度 Bc	≤30	
	40Bc～100Bc的时间 min	≤40	
	稠化线形	正常	
	游离液 %	≤1.4	
	24h抗压强度 MPa	≥14	

表（续）

试验温度与基准配方	评价项目	质量指标	试验结果
（稠化时间：_____min）	稠化时间可调性	可调	
	加量敏感系数（可选项）	0.1～3.0	
	增加5℃稠化时间变化率（可选项）%	≤20	
评价结论			

主检人：　　审核：　　批准：　　评价日期：

附 录 C
（资料性附录）
油井水泥缓凝剂质量检验报告格式

产品名称：_____　　生产批号：_____　　供货商：_____

适用温度范围：_____　　委托单位：_____　　送（采）样日期：_____

送（采）样人：_____　　采样地点：_____　　检验单位：_____

执行标准：_____　　检验用水：_____　　水泥批号：_____

报告编号：_____

类　型	检验项目	质量指标	试验结果
理化性能	外观	（液体材料）均匀无霉变 （固体材料）无受潮板结	
	细度（0.315mm 筛筛余） %	＜15	
	水分（固体材料） %	≤8	
_____℃水泥浆性能 基准配方： _____ _____ （稠化时间：_____min）	稠化时间可调性	可调	
	初始稠度 Bc	≤30	
	40Bc～100Bc 的时间 min	≤40	
	稠化线形	正常	
	游离液 mL	≤2.5	
	24h 抗压强度 MPa	≥14	
质量稳定性 （与批号××××比较）	稠化时间偏差率 %	≤20	
	40Bc～100Bc 的时间之差的绝对值 min	≤10	
检验结论			

主检人：　　审核：　　批准：　　检验日期：

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The People's Republic of China
Standard of Petroleum and Natural Gas Industry

SY/T 5504.1—2005
Replace SY/T 5504—1996

Evaluation method for well cement additives—Part 1: Retarder

Issue Date: 07—26—2005 Implementation Date: 11—01—2005

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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: *Gas migration control additive*;

—*Part* 6: *Lighting agent*;

—*Part* 7: *Weighting agent*;

—*Part* 8: *Filling additive*.

This is Part 1 of SY/T 5504.

This part replaces SY/T 5504—1996 *Evaluation method for well cement retarder*.

Compared with SY/T 5504—1996, the main changes of this part are as follows:

—The evaluation method of retarder is chang-ed from using single agent slurry to using compatible system slurry (4.1.1 and 4.2.1 in SY/T 5504—1996; 4.2.1, 5.4.1 and 5.4.2 in this part);

—Test temperature for retarder is revised (4.1.2.1 and 4.2.2.1 in SY/T 5504—1996; 3.6, 3.7 and 5.4.3.1 in this part);

—Test and judgment methods of *thickening-time adjustability* are revised (4.1.2.1 and 4.2.2.1 in SY/T 5504—1996; 5.4.3.2 in this part);

—Evaluation for product grade and associated judgment rules are deleted (3.4 and 5.2 in SY/T 5504—1996);

—Test item, *compressive strength ratio of 168h to 48h*, is deleted (3.4, 4.1.3 and 4.2.3 in SY/T 5504—1996);

—Test item, *shape of thickening-time curve*, and its requirements are added (4.2.1 and 5.4.3.2 in this part);

—Recommended conditions of thickening-time and compressive strength tests are added (Table 3 of 5.4.3.2 in this part);

—Requirements of test apparatus and materials are added (5.1 in this part);

—Test methods and requirements of quality stability are added (4.2.2 and 5.5 in this part);

—Two optional evaluation items, *temperature sensitivity* and *dosage sensitivity coefficient*, are added (4.2.1, 5.4.3.6 and 5.4.3.7 in this part);

—Requirements and test methods of physical and chemical properties including material appearance, density, water-solubility, fineness, freezing point and water content are added (4.1, 5.3 and Annex A in this part);

—Methods of batching and sampling are added (5.2 in this part).

Annex B in this part is normative; Annexes A and C in this part are informative.

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

This part was drafted responsibly by China Oilfield Services Limited, CNOOC.

This part was mainly drafted by Shen Wei, Wang Lei, Wang Yongsong, Yang Zhenyu, Fang Keyan, Wei Hongmei and Li Weichun.

This part replaces the following previous editions:

—SY/T 5504—92, SY/T 5504—1996.

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

Evaluation method for well cement additives
Part 1: Retarder

1 Scope

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

This part is applicable to evaluation for well cement retarder used in well cementing operations.

2 Normative references

The following normative documents contain provisions which, through references in this part of SY/T 5504, constitute provisions of this part. For dated references, subsequent amendments to, or revisions of, any of these publications (exclude errata) do not apply. However, parties to agreements based on this part are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

GB/T 510 *Petroleum products—determination of solidification point*

GB/T 4472 *General rule for determination of density and relative density for chemical products*

GB/T 6678—2003 *General principles for sampling chemical products*

GB/T 6679—2003 *General rules for sampling of solid chemical products*

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

GB/T 8077—2000 *Methods for testing uniformity of concrete admixture*

GB 10238—1998 *Oil well cement*

GB/T 19139—2003 *Procedure for testing well cements*

SY/T 5381 *Technical condition of mud balance*

3 Terms and definitions

The following terms and definitions are applicable to this part of SY/T 5504.

3.1

transition time from 40 Bc to 100 Bc

The elapsed time of cement slurry consistency changing from 40 Bc for the first time to 100 Bc during the thickening-time test.

3.2

dosage sensitivity coefficient

The index expresses impact degree of retarder dosage on the thickening-time, and it can be obtained by calculating the ratio of increasing rate of thickening-time to the increasing rate of retarder dosage.

3.3

temperature sensitivity

The impact degree on thickening-time due to the changes of temperature. It is expressed by the changing rate of thickening-time at 5℃ increment.

3.4

upper temperature limit

The highest temperature applicable for retarder.

3.5

lower temperature limit

The lowest temperature applicable for retarder.

3.6

serial temperatures

A series of testing temperatures adopted in the property evaluation for retarder.

3.7

typical temperature

The testing temperature adopted in the quality inspection for retarder.

3.8

quality stability

The coincidence degree of product quality between different batches.

3.9

reference sample

The retarder sample adopted as standard material in quality stability test, which is from the samples that had passed examination and were well preserved.

3.10

fitting thickening-time range

The range of thickening-time suitable for conventional cementing operations at BHCT.

3.11

basic recipe

The recipe of cement slurry, used for the testing of free fluid, compressive strength, temperature sensitivity and quality stability.

4 Requirements

4.1 Physical and chemical properties

The requirements for appearance, density, water-solubility, water content, fineness, pH value and freezing point of the retarder are given in Annex A as reference.

4.2 Application properties

4.2.1 The properties of compatible cement slurry (density is $1.90g/cm^3 \pm 0.01g/cm^3$) added with the retarder should meet the requirements in Table 1 within the applicable temperature range.

Table 1 Property requirements for cement slurry added with the retarder

Inspection item	Technical index
Initial consistency Bc	≤30
Transition time from 40 Bc to 100 Bc min	≤40
Shape of thickening-time curve	Normal
Free fluid %	≤1.4
24h compressive strength MPa	≥14
Thickening-time adjustability	Adjustable
Dosage sensitivity coefficient (optional)	0.1~3.0
Deviation rate of thickening-time at 5℃ increment (optional) %	≤20

4.2.2 For the same batch cement, water quality, recipe and test conditions, the quality stability of the retarder should meet the requirements in Table 2.

Table 2 Requirements of quality stability for the retarder

Inspection item	Technical index
Deviation rate of thickening-time %	≤20
Absolute value of deviation of transition time from 40 Bc to 100 Bc min	≤10
Note: Quality stability test is performed at the typical temperature.	

5 Test methods

5.1 Apparatus and materials

5.1.1 Apparatus

Mainly include:

a) Electronic balance: With the precision of 0.01g.

b) Constant temperature drying oven: With the temperature range of 0℃ to 200℃.

c) Desiccator: Containing discolored silica gel.

d) Weighing bottle: 25mm×65mm.

e) Density bottle: 25mL or 50mL.

f) Analytical balance: With weighing range of 0g to 200g and the precision of 0.1mg.

g) Sieve: With mesh size of 0.315mm.

h) Slurry mixer: Shall meet the stipulation of Annex A in GB 10238—1998.

i) Atmospheric consistometer: Shall meet the stipulation of Annex B in GB 10238—1998.

j) Compressive strength curing and testing devices: Shall meet the stipulation of Annex C in GB 10238—1998.

k) Pressurized consistometer: Shall meet the stipulation of Annex D in GB 10238—1998.

l) Fluid density balance: Pressurized fluid density balance or mud balance. The former shall meet the stipulation of Clause 6 of GB/T 19139—2003; the latter shall meet the stipulation of SY/T 5381.

m) 250mL graduated cylinder: Shall meet the stipulation of Annex B in GB 10238—1998.

5.1.2 Materials

Mainly include:

a) Well cement: Class G well cement (HSR), shall meet the stipulation of GB 10238—1998.

b) Silica flour: The residue on 0.075mm to 0.095mm (200 mesh to 160 mesh) sieve is less than 4% and the SiO_2 content is no less than 98%.

c) Water: Distilled water or tap water.

d) Retarder: Test sample (and reference sample for quality inspection).

e) Other additives: Compatible with the retarder.

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 7.6 in GB/T 6678—2003.

5.2.2.2 Obtain solid sample in accordance with 4.3.1 in GB/T 6679—2003.

5.2.2.3 Obtain liquid sample in accordance with 7.1 in GB/T 6680—2003.

5.2.2.4 The amount of solid sample should be no less than 500g; the amount of liquid sample should be no less than 1000mL.

5.2.2.5 All samples obtained from the same batch should be well mixed, evenly stored in three 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 three bottles is for evaluation; one is as the reference sample after passing examination; and the other is for arbitration. Samples for reference and arbitration should be preserved for one year.

5.3 Test procedures for physical and chemical properties

5.3.1 Appearance test

Appearance test is performed by visual observation.

5.3.2 Density test

Density test should be performed by density bottle method in GB/T 4472.

5.3.3 Water-solubility test

Water-solubility test for the solid retarder used by wet mixing should be performed at 23℃ ± 1℃ as follows: prepare solid retarder solution at 5% concentration, and agitate for 10min with magnetic mixer at medium speed, then deposit it quietly for 10min and inspect if the sedimentation or bedding appears in the bottom part of the solution.

5.3.4 Water content test

Water content test for solid retarder should be performed in accordance with Clause 4 of GB/T 8077—2000.

5.3.5 Fineness test

Fineness test for solid retarder used by dry mixing should be performed in accordance with Clause 6 of GB/T 8077—2000.

5.3.6 pH value test

pH value test for liquid retarder should be performed by using pH test paper of high precision.

5.3.7 Freezing point test

Freezing point test for liquid retarder should be performed in accordance with GB/T 510.

5.4 Test procedures for cement slurry properties

5.4.1 Source of original cement slurry recipe

Mainly includes:

—User test: The recipe provided by the supplier or the recipe composed 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 cement slurry of 1.90g/cm^3 ± 0.01g/cm^3 in density according to original recipe, and in accordance with Clause 5 of GB/T 19139—2003.

5.4.2.2 Density test for cement slurry should be performed according to Clause 6 of GB/T 19139—2003.

5.4.2.3 35% (BWOC) silica flour should be added into the cement slurry when the test temperature is at 110℃ (BHST) or higher.

Table 3 Conditions for slurry test and fitting thickening-time

Thickening-time test temperature (BHCT) ℃	Thickening-time test pressure MPa	Time to BHCT min	Fitting thickening-time range min	The median of fitting thickening-time range min	Temperature for strength test (BHST) ℃
50	25.9	32	100～225	163	71
60	32.8	37	110～240	175	82
70	39.6	41	120～255	188	92
80	46.5	45	130～270	200	102
90	53.3	49	140～285	213	113
100	60.2	53	150～300	225	123
110	67.0	57	160～315	238	133
120	73.9	61	170～330	250	144
130	80.7	65	180～345	263	154
140	87.6	69	190～360	275	164
150	94.4	73	200～375	288	175
160	101.3	77	210～390	300	185
170	108.1	81	220～405	313	196
180	115.0	86	230～420	325	206

5.4.3 Cement slurry test

5.4.3.1 Determination of test temperatures

Evaluation for the retarder should be performed at the serial temperatures; quality inspection for the retarder should be performed at the typical temperature. Determine those test temperatures according to the following requirements:

a) The serial temperatures should be determined on the basis of its applicable temperature range. They include 60℃, 80℃, 100℃, 120℃, 150℃, upper and lower temperature limits involved in the applicable temperature range of the retarder and can be determined on the basis of the operation temperature range in the local oilfield by the user, as well.

b) The typical temperature should be determined on the basis of the applicable temperature range of the retarder: 80℃ is taken as the typical temperature if the applicable range tends to be below 100℃; 120℃ as the typical temperature if the applicable range tends to be over 100℃. It can be determined on the basis of the operation temperature range in the local oilfield by the user, as well.

c) For the evaluation of new retarder of which applicable temperature range is unidentified, or for the first evaluation of foreign retarder as a part of cement slurry recipe composed by the user, the serial temperatures can be determined in the approximate applicable temperature range (the upper and lower limits can be determined by identifying test).

Example 1: For a retarder with approximate fitting temperature range from 90℃ to 160℃, the test is conducted at 100℃, 120℃ and 150℃ firstly, then the identifying test for lower/upper temperature limits is carried out as follows:

Identifying test for lower temperature limit: If the result of test at 100℃ is satisfactory, the test is conducted at 90℃, i.e., by 10℃ lower; if not satisfactory, test conducted at 110℃, i.e., by 10℃ higher, and so forth until the lower limit of temperature is determined.

Identifying test for Upper temperature limit: If the result of test at 150℃ is satisfactory, the test is performed at 160℃, i.e., by 10℃ higher; if not satisfactory, test performed at 140℃, i.e., by 10℃ lower, and so forth until the upper limit of temperature is determined.

5.4.3.2 Test for thickening-time adjustability

The adjustability test of thicken-time at determinate temperature is performed as follows:

a) Prepare cement slurry according to the original recipe, then carry out thickening-time test under the conditions as recommended in Table 3 in accordance with Clause 9 of GB/T 19139—2003.

b) Adjust the retarder dosage to obtain three recipes with the thickening-times falling in the fitting thickening-time range (see Table 3) corresponding to the test temperatures and their spacing between adjacent points no less than 30min.

c) Analyze all the thickening-time curves falling in the fitting thickening-time range. If no bulgy and step-shaped part is found within the time spacing between 30min and 80% time point of thickening-time (especially refer to a bulge at least 5 Bc higher than the posterior baseline consistency with a duration longer than 3min, or a step with consistency over 30 Bc), the thickening-time curve is judged as normal; otherwise, the analysis has to be continued. If two or more such cases exist, the curve is judged as abnormal. If only one such case exists, repeat the test, if such case appears again, the curve is judged as abnormal; otherwise, as normal.

Note: The posterior baseline consistency in thickening-time curve refers to the minimum consistency after the bulge occurs.

d) Read the initial consistencies on all the thickening-time curves within the fitting thickening-time range and record the maximum one.

e) Read the transition time from 40 Bc to 100 Bc on all the thickening-time curves within the fitting thickening-time range and record the maximum one.

f) Analyze all the thickening-time test data obtained from experiments at the test tempera-

tures. If the thickening-time extends along with the increasing of the retarder dosage, the retarder at the test temperature is judged as adjustable in the thickening-time; otherwise, as unadjustable.

Example 2: Example table 1 presents the thickening-time test data for a retarder at 110℃, the thickening-times of slurry prepared according to the recipes No. 2, 3 & 4 are within the fitting range and with their spacing over 30min between adjacent points. The result of comprehensive analysis of all the test data shows that the thickening-time extended along with the inreasing of the retarder dosage and the shape of thickening-time curve is normal, therefore, the retarder is judged as adjustable in the thickening-time at 110℃.

Example table 1

Number	Retarder dosage %	Thickening-time min
1	0.1	100
2	0.3	200
3	0.4	250
4	0.6	300
5	0.9	460

Example 3: Example table 2 presents the thickening-time test data for a retarder at 70℃. The thickening-times of slurry prepared according to the recipes No. 2, 3 & 5 are within the fitting thicken-time range and with the spacing over 30min between adjacent points, so it seems to be rational only by analysing these three data, but the result of analysing No. 3 & 4 data shows that the thickening-time shortened along with the increasing of the retarder dosage, therefore, the retarder is judged as unadjustable in the thickening-time at 70℃.

Example table 2

Number	Retarder dosage %	Thickening-time min
1	0.2	80
2	0.4	150
3	0.6	200
4	0.7	160
5	0.9	240

5.4.3.3 Determination of basic recipe

The cement recipe, on which thickening-time within ±30min of the median of fitting thickening-time range is based, is selected from the thickening-time test data in 5.4.3.2 and regarded as the basic recipe for testing free fluid, compressive strength, temperature sensitivity and quality stability.

5.4.3.4 Free fluid test

Free fluid test for cement slurry should be performed in accordance with Clause 15 of GB/T 19139—2003.

5.4.3.5 Compressive strength test

Compressive strength test for cement slurry at strength test temperature as recommended in Table 3 should be performed in accordance with Clause 7 of GB/T 19139—2003.

5.4.3.6 Temperature sensitivity test (optional)

Carry out the thickening-time test for cement slurry by increasing 5℃, and read the result.

Calculate the changing rate of thickening-time at 5℃ increment according to the following equation:

$$TT_{CR} = \frac{|TT_{T+5} - TT_T|}{TT_T} \times 100\% \quad \cdots\cdots (1)$$

Where:

TT_{CR} is the changing rate of thickening-time at 5℃ increment;

TT_{T+5} is the thickening-time of cement slurry at 5℃ increment, min;

TT_T is the thickening-time of cement slurry at test temperature, min.

5.4.3.7 Determination of dosage sensitivity coefficient (optional)

Two sets of test data of the cement slurry with the thickening-time mostly approaching the upper/lower limit of the fitting thickening-time range and both added with retarder, are selected from the experiment in 5.4.3.2, then dosage sensitivity coefficient is calculated as follows:

a) Calculate the increasing rate of thickening-time according to the following equation:

$$TT_{IR} = \frac{TT_2 - TT_1}{(TT_1 + TT_2)/2} \times 100\% \quad \cdots\cdots (2)$$

Where:

TT_{IR} is the increasing rate of thickening-time;

TT_1 is the thickening-time of recipe 1 (near lower limit), min;

TT_2 is the thickening-time of recipe 2 (near upper limit), min.

b) Calculate the increasing rate of retarder dosage according to the following equation:

$$D_{IR} = \frac{D_2 - D_1}{(D_1 + D_2)/2} \times 100\% \quad \cdots\cdots (3)$$

Where:

D_{IR} is the increasing rate of retarder dosage;

D_1 is the retarder dosage in recipe 1 (BWOC);

D_2 is the retarder dosage in recipe 2 (BWOC).

c) Calculate the dosage sensitivity coefficient of retarder according to the following equation:

$$DSC = \frac{TT_{IR}}{D_{IR}} \quad \cdots\cdots\cdots (4)$$

Where:

DSC is the dosage sensitivity coefficient of retarder.

Example 4: Adjust the dosage of retarder in a cement slurry recipe and carry out the thickening-time test at 100℃. The thickening-time turns out as follows for two recipes respectively:

1%retarder (BWOC) —210min;

1.5%retarder (BWOC) —290min;

The increasing rate of thickening-time is calculated as:

$TT_{IR} = 100\% \times (290 - 210) \div [(290 + 210)/2] = 32\%$

The increasing rate of retarder dosage is calculated as:

$D_{IR} = 100\% \times (1.5\% - 1.0\%) \div [(1.5\% + 1.0\%)/2] = 40\%$

The dosage sensitivity coefficient of retarder is calculated as:

$$DSC = TT_{IR}/D_{IR} = 32\% \div 40\% = 0.8$$

5.5 Test procedure for quality stability

5.5.1 Thickening comparison test

Replace the test sample with the reference sample and prepare cement slurry with the same cement and water according to the same basic recipe, then run a thickening-time test under the same temperature and pressure. Read the thickening-time and the transition time from 40 Bc to 100 Bc and compare them with the thickening data of the slurry added with test sample (see 5.4.3.2).

5.5.2 Processing of comparison data

5.5.2.1 Calculate the deviation rate of thickening-time between two kinds of cement slurry added with test sample and reference sample respectively according to the following equation:

$$TT_{DR} = \frac{|TT_S - TT_R|}{TT_R} \times 100\% \quad \cdots\cdots (5)$$

Where:

TT_{DR} is the deviation rate of thickening-time;

TT_S is the thickening-time of slurry added with test sample, min;

TT_R is the thickening-time of slurry added with reference sample, min;

5.5.2.2 Calculate the absolute value of deviation of transition time from 40 Bc to 100 Bc between two kinds of cement slurry added with test sample and reference sample respectively.

6 Evaluation report

See Annex B for evaluation report of application properties for well cement retarder; see Annex A for evaluation report of physical and chemical properties for well cement retarder and Annex C for quality inspection report for well cement retarder (as reference).

Annex A
(informative)
Evaluation report form of physical and chemical properties for well cement retarder

Product name: _____ Batch number: _____

Supplier: _____ Applicable temperature range: _____

Client: _____ Date of sampling: _____

Person of sampling: _____ Sampling place: _____

Evaluation unit: _____ Executive standard: _____

Report number: _____

Evaluation item	Quality index	Test result
Appearance	Uniform, not deteriorating (liquid) Not damping or agglomerating (solid)	
Density (20℃ ± 1℃) g/cm³	Within prescribed limits	
Water-solubility	Good	
Water content (solid) %	≤8	
Fineness (residue on 0.315mm sieve) %	<15	
pH value	Within prescribed limits	
Freezing point ℃	Within prescribed limits	

Chief inspector: Auditor: Approver: Evaluation date:

Annex B
(normative)
Evaluation report form of application properties for well cement retarder

Product name: _____ Batch number: _____
Supplier: _____ Applicable temperature range: _____
Client: _____ Date of sampling: _____
Person of sampling: _____ Sampling place: _____
Evaluation unit: _____ Executive standard: _____
Water quality: _____ Cement batch number: _____
Report number: _____

Test temperature and basic recipe	Evaluation item	Quality index	Test result
Upper temperature limit: _____℃ Basic recipe: _____ _____ (Thickening-time: _____min)	Initial consistency Bc	≤30	
	Transition time from 40 Bc to 100 Bc min	≤40	
	Shape of thickening-time curve	Normal	
	Free fluid %	≤1.4	
	24h compressive strength MPa	≥14	
	Thickening-time adjustability	Adjustable	
	Dosage sensitivity coefficient (optional)	0.1~3.0	
	Deviation rate of thickening-time at 5℃ increment (optional) %	≤20	
Low temperature limit: _____℃ Basic recipe: _____ _____ (Thickening-time: _____min)	Initial consistency Bc	≤30	
	Transition time from 40 Bc to 100 Bc min	≤40	
	Shape of thickening-time curve	Normal	
	Free fluid %	≤1.4	
	24h compressive strength MPa	≥14	
	Thickening-time adjustability	Adjustable	
	Dosage sensitivity coefficient (optional)	0.1~3.0	
	Deviation rate of thickening-time at 5℃ increment (optional) %	≤20	

continued

Test temperature and basic recipe	Evaluation item	Quality index	Test result
Test temperature: _____℃ Basic recipe: _____ _____ (Thickening-time: _____min)	Initial consistency Bc	≤30	
	Transition time from 40 Bc to 100 Bc min	≤40	
	Shape of thickening-time curve	Normal	
	Free fluid %	≤1.4	
	24h compressive strength MPa	≥14	
	Thickening-time adjustability	Adjustable	
	Dosage sensitivity coefficient (optional)	0.1~3.0	
	Deviation rate of thickening-time at 5℃ increment (optional) %	≤20	
Test temperature: _____℃ Basic recipe: _____ _____ (Thickening-time: _____min)	Initial consistency Bc	≤30	
	Transition time from 40 Bc to 100 Bc min	≤40	
	Shape of thickening-time curve	Normal	
	Free fluid %	≤1.4	
	24h compressive strength MPa	≥14	
	Thickening-time adjustability	Adjustable	
	Dosage sensitivity coefficient (optional)	0.1~3.0	
	Deviation rate of thickening-time at 5℃ increment (optional) %	≤20	
Test temperature: _____℃ Basic recipe: _____ _____ (Thickening-time: _____min)	Initial consistency Bc	≤30	
	Transition time from 40 Bc to 100 Bc min	≤40	
	Shape of thickening-time curve	Normal	
	Free fluid %	≤1.4	
	24h compressive strength MPa	≥14	
	Thickening-time adjustability	Adjustable	
	Dosage sensitivity coefficient (optional)	0.1~3.0	
	Deviation rate of thickening-time at 5℃ increment (optional) %	≤20	
Conclusion			

Chief inspector: Auditor: Approver: Evaluation date:

Annex C
(informative)
Quality inspection report form for well cement retarder

Product name: _____ Batch number: _____
Supplier: _____ Applicable temperature range: _____
Client: _____ Date of sampling: _____
Person of sampling: _____ Sampling place: _____
Inspection unit: _____ Executive standard: _____
Water quality: _____ Cement batch number: _____
Report number: _____

Type	Inspection items	Quality index	Test result
Physical and chemical properties	Appearance	Uniform, not deteriorating (liquid) Not damping or agglomerating (solid)	
	Fineness (residue on 0.315mm sieve) %	<15	
	Water content (solid) %	≤8	
_____℃ Slurry properties Basic recipe: _____ _____ (Thickening-time: _____min)	Thickening-time adjustability	Adjustable	
	Initial consistency Bc	≤30	
	Transition time from 40 Bc to 100 Bc min	≤40	
	Shape of thickening-time curve	Normal	
	Free fluid mL	≤2.5	
	24h compressive strength MPa	≥14	
Quality stability (compare to batch××××)	Deviation rate of thickening-time %	≤20	
	Absolute value of deviation of transition times from 40 Bc to 100 Bc min	≤10	
Conclusion			

Chief inspector: Auditor: Approver: Evaluation date: