



# 硫离子电极操作指南

硫离子电极可简便快速地测量水溶液中和非水溶液中的硫离子浓度。因为硫离子电极也可以看做是一支银离子电极，所以凡是那些可与银离子反应生成沉淀的离子（如Cl<sup>-</sup>、Br<sup>-</sup>、I<sup>-</sup>、CN<sup>-</sup>、SCN<sup>-</sup>等），可以用硫离子电极作指示电极的电位滴定法测定。

## 电极性能

- 离子选择膜： 固态膜
- 测量范围： 10<sup>-6</sup> ~ 1 mol/l
- 温度范围： 0 ~ 50° C
- pH 范围： 12 ~ 14
- 电极内阻： < 5 MΩ
- 响应时间：
  - 由低浓度到高浓度检测时，若 S<sup>2-</sup> 离子浓度 < 10<sup>-4</sup> mol/l，响应时间约1分钟；若 S<sup>2-</sup> 离子浓度 > 10<sup>-4</sup> mol/l，响应时间少于30秒；
  - 由高浓度到低浓度检测时，一般需几分钟。不能含有阴离子表面活性剂，硫离子同时也对Ag<sup>+</sup>敏感，故测试溶液中Ag<sup>+</sup>的浓度应小于S<sup>2-</sup>的浓度。
- 干扰离子：

## 准备

### a. 硫电极活化

电极使用前，需0.01 mol/L S<sup>2-</sup> 离子溶液中浸泡活化半小时，再用去离子水反复清洗至空白电位稳定，并用纸轻轻吸干。

### b. 制备标准溶液

取250克Na<sub>2</sub>S · 9H<sub>2</sub>O加适量稀释液溶解后，稀释至1升制得S<sup>2-</sup>离子标准储备液，用碘量法标定其浓度，放冰箱中保存。用稀释液逐级稀释标准储备液来配制不同浓度的标准硫离子溶液。

### c. 参比电极

取下加液口的保护帽，轻轻旋开磨砂口交界隔膜，待外参比电解液全部流出后再旋紧交界隔膜（注意不能太紧）。从外参比电解质加液口充入1 mol/l NaOH 溶液，重新盖上保护帽。用手指捏紧电极，上下震动，去除液络部的气泡。

## 标定及测量

—如果您使用离子计，可按下列步骤测定：

### a. 两点标定

取硫标准溶液用稀释液逐级稀释，选择两个浓度相差十倍的标准溶液。

根据离子计使用说明书将离子计设置在标定方式，设置浓度较小的标准溶液为第一个校正点，浓度较大的标准溶液为第二个校正点。

将硫电极和参比电极一起插入浓度较小的标准溶液中，进行第一点校正。

将两支电极插入浓度较大的标准溶液中，进行第二点校正。

离子计将自动显示电极斜率。

### b. 试样测量

用去离子水冲洗电极。

取试样15ml 于 50ml 塑料杯中，加10ml SAOB 溶液，将两支电极插入溶液中，测量试样浓度。

—如果您使用pH/mV计，可按下列步骤测定：

### a. 绘制工作曲线

根据pH/mV计使用说明书将pH/mV计设置在mV方式，然后将两支电极插入溶液中。由稀到浓测定不同浓度的标准硫离子溶液的mV值。

用上述测定的一系列mV值为y轴，以相应的标准溶液硫离子浓度为x轴，在半对数图纸上作图，即为工作曲线。

### b. 试样测量

每次测量后用去离子水冲洗电极。

取试样15ml 于 50ml 塑料杯中，加10ml SAOB 溶液，测其mV值，然后在工作曲线上读出该mV值对应的硫离子浓度值。

## 维护和保养

单晶片上若存在沉积物，可用湿纸巾轻轻擦去，注意不要划伤晶片表面，然后在0.01 mol/l S<sup>2-</sup>离子溶液中放置5~10分钟。

## 储藏

电极使用完毕后，应清洗到空白电位，用滤纸吸干保存。若需长期保存，最好用保护盖将探测表面盖好。

## 附件

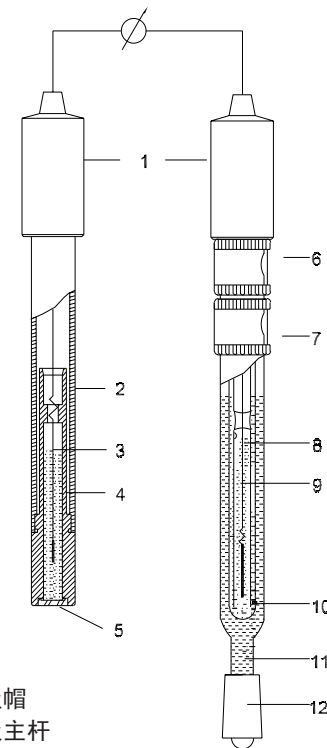
LE302 参比电极

（订货号：12107202或12107204）

稀释液：取40克NaOH和5克抗坏血酸溶于1升水中。

硫离子抗氧化缓冲剂 (SAOB): 取10克NaOH和1.25克抗坏血酸溶于100毫升水中。外盐桥电解质：1M NaOH

产品订货号：12107250 或 12107260



- 1 电极帽
- 2 电极主杆
- 3 内信号引出线
- 4 屏蔽层
- 5 离子敏感膜
- 6 内参比电解液充液口
- 7 外参比电解液充液口
- 8 内参比电解液
- 9 Ag/AgCl参比电极
- 10 多孔陶瓷隔膜
- 11 外参比电解液
- 12 磨砂口交界隔膜

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Printed by MTCS Marcom 2009/01 12107264B

## General information

Sulphide electrode is suitable for measurements of sulphide concentrations in aqueous and nonaqueous systems. Sulphide electrode may also be regarded as silver electrode; it can be the indicator electrode in potential titration for  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{CN}^-$ ,  $\text{SCN}^-$ , which can react with silver ion to form precipitation.

## Specifications

- Type of ion selective membrane:  
Solid state membrane
- Membrane impedance:  $< 5 \text{ M}\Omega$
- Measurement range:  
 $10^{-6} \text{ mol/l}$  up to approx.  $1 \text{ mol/l}$  sulphide
- Measurement temperature range:  $0\sim 50^\circ \text{ C}$
- Optimum pH range: 12 to 14
- Response time:  
when changing from lower to higher concentrations below  $10^{-4} \text{ mol/l S}^{2-}$  less than 30 seconds; when changing from higher to lower concentration several minutes.
- Interfering ion:  
Anionic surfactants (detergents) must be absent. The sulphide electrode is also sensitive to the  $\text{Ag}^+$  ion. The ratio ( $\text{Ag}^+/\text{S}^{2-}$ ) should thus not be larger than 1.

## Preparatory operations

### a. Activation

Before the sulphide electrode was used, it should be soaked in  $0.01 \text{ mol/l}$  sulphide solution for half an hour and rinsed repeatedly with deionized water till blank potential is steady. The sulphide electrode should be dabbed with paper tissue.

### b. Preparation of standard solution

Weight out  $250 \text{ g Na}_2\text{S}\cdot 9\text{H}_2\text{O}$  for a  $1 \text{ mol/l S}^{2-}$  standard sulphide stock solution and dissolve in diluent in a  $1000 \text{ ml}$  calibrated flask and make up to the mark. Calibrate the stock solution by iodimetry and store it in a refrigerator. Prepare solutions with different sulphide

concentrations by diluting the standard stock solution with diluent as appropriate.

### c. Reference electrode

Remove cap from electrolyte filling port. Slightly loosen the ground-joint diaphragm (by careful turning) so that outer reference electrolyte flows out. Tighten up ground joint again (but not too tight). Top up with  $1 \text{ mol/l NaOH}$  electrolyte to filling port for outer-mediator electrolyte and recover the cap. Clutch the electrode and shake up and down in order to reduce air bubbles.

## Calibration and measurement

If you are using **ionic meter**, proceed as follows to measure sulphide ion concentration:

### a. 2 point calibration

Prepare two standard solutions by diluting the standard stock solution with diluent as appropriate. One standard has 10 times difference in concentration from another one. The standard solutions can be prepared with any concentration unit.

According to the menu setting process of your ionic meter, set the lower concentration standard as the first calibration point, and the higher one as the second calibration point.

Place the sulphide electrode and reference electrode together in the lower standard solution, and start the first point calibration.

Place two electrodes in the higher standard solution, and start the second point calibration. Ionic meter will determine the slope automatically.

### b. Measurement Sample

Rinse two electrodes with deionized water.

To  $15 \text{ ml}$  sample solution add  $10 \text{ ml SAOB}$  solution and stir. Place two electrodes in the mixed solution and start measurement.

If you are using **pH/mV meter**, proceed as follows to measure sulphide ion concentration:

### a. Plotting calibration diagram

According to pH/mV meter instruction manual, select mV mode. Place the sulphide electrode and

reference electrode in a series of sulphide solutions one after another and measure their mV from lower to higher concentrations.

Plot measured mV values on a calibration diagram against the logarithm of the sulphide concentration of the corresponding solution (use logarithm paper).

### b. Measurement Sample

Rinse two electrodes with deionized water after each measurement.

To  $15 \text{ ml}$  sample solutions add  $10 \text{ ml SAOB}$  solution and stir. According to above method measure the mV of sample solution. Use the calibration diagram to read off the sulphide concentration of the sample solutions from the measured mV values.

It is advisable when carrying out lower sulphide concentration determination to apply a standard addition method.

## Maintenance

Deposits on the crystal membrane can be removed by carefully wiping with a damp cellulose tissue. Take care to avoid damages (scratches) to the crystal surface. Afterwards, conditioning in dilute sulphide solution (approx.  $0.01 \text{ mol/l}$ ) during 5 to 10 minutes is necessary.

## Storage

After the sulphide electrode was used, it should be rinsed to blank potential and then wiped off. If not used for a long time, it may be stored dry in the protective tube.

## Accessories

LE302 Ref. electrode  
(Order No. 12107202 or 12107204)

Diluent:

Prepare as follows: weigh out  $40 \text{ g NaOH}$  and  $5 \text{ g ascorbic acid}$ , then dissolve in deionized water and make up to 1 liter.

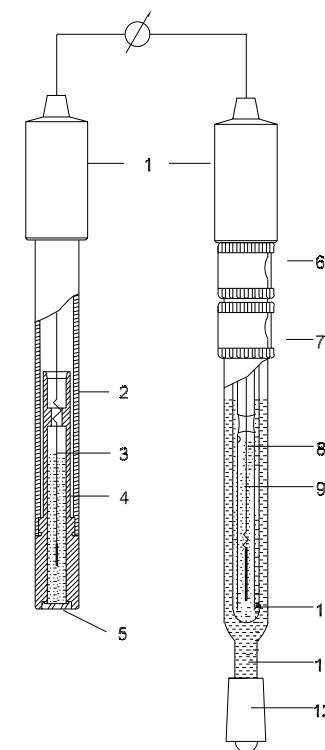
Sulphide Antioxidant Buffer (SAOB):

Prepare as follows: weigh out  $10 \text{ g NaOH}$  and

$1.25 \text{ g ascorbic acid}$ , then dissolve in deionized water and make up to  $100 \text{ ml}$ .

Intermediate electrolyte for the reference electrode:  $1 \text{ M NaOH}$

**Order No.: 12107250 or 12107260**



- 1 Electrode cap
- 2 Plastic electrode body
- 3 Internal lead out
- 4 Electric screw
- 5 Ion-selective membrane
- 6 Filling port for inner electrolyte
- 7 Filling port for outer electrolyte
- 8 Internal reference electrolyte
- 9 Ag/AgCl reference element
- 10 Porous ceramic diaphragm
- 11 Outer reference electrolyte
- 12 Ground-joint diaphragm