Agilent I9121 Ammonia Combination ISE
氨气敏复合电极

Operating Guide
用户手册

Agilent Technologies

Overview

The I9121 Ammonia Combination ISE can measure ammonia and ammonium concentration in aqueous solutions. It is used with the 3200I Ion Meter or similar meters.

WARNING **Use this probe according to the operating manual to avoid personal injury.**

WARNING **The probe solution can cause chemical burns or illness if it is taken orally or contacted by human skin. Use protective clothing or gloves to avoid contact. In case of contact, rinse contacted area with tap water or deionized water thoroughly.**

CAUTION The probe body material is glass. Handle with care to avoid damage to the instrument.

Specifications

Table 1 I9121 Ammonia Combination ISE specifications

Specification	**Value**
Concentration range	10^{-1} to 1×10^{-6} mol/L
Temperature range	0 to 50 °C
Probe impedance	$\leqslant$1500 MΩ
Reference type	Ag/AgCl
Probe diameter	16 mm
Probe length	180 mm
Cable interface	BNC
Cable length	1000 mm

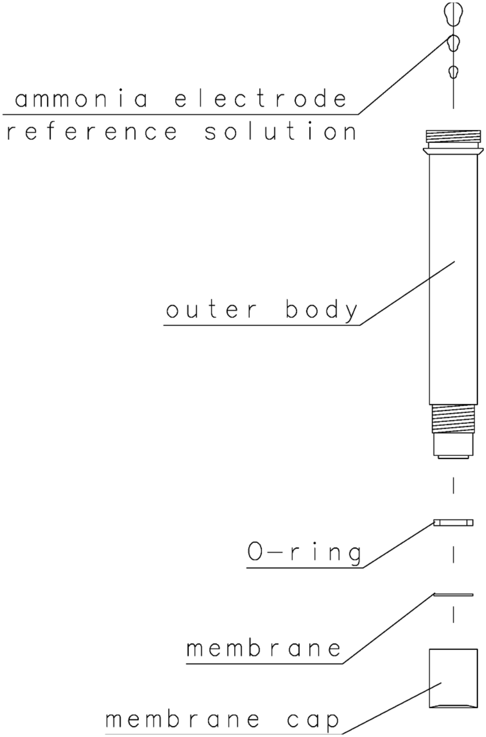

Figure 1 I9121 Ammonia Combination ISE outer body assembly

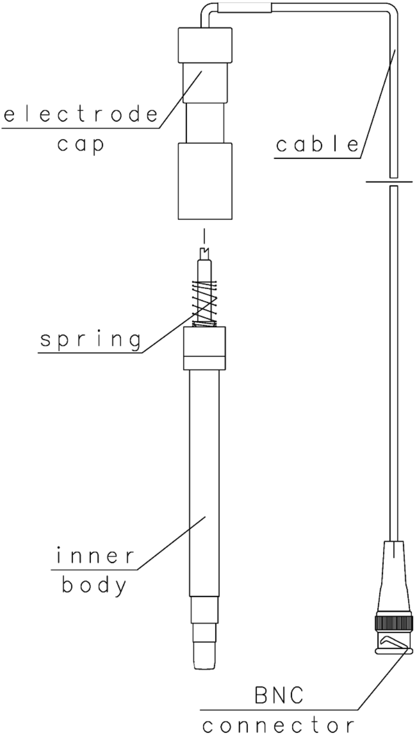

Figure 2 I9121 Ammonia Combination ISE assembly

Operation

Preparing the probe

1. Hold the probe upright and unscrew the probe cap (see Figure 2).
2. Remove the internal probe from the outer body. Rinse the internal probe and outer body with distilled or deionized water.
3. Remove the membrane cap off from the outer body (see Figure 1).
4. Remove the original composite membrane and O-ring from the membrane cap. Rinse the membrane cap with distilled water or deionized water.
5. Using tweezers, carefully place the membrane into the membrane cap. The membrane should evenly touch against the bottom of the membrane cap.
6. Set a new O-ring on the outer body and tightly screw the membrane cap onto the outer body. The composite membrane should be flat, and pressed against the bottom of the membrane cap without wrinkles.
7. Add 3.5 mL filling solution (5190-0544) of ammonia combination ISE into the outer body.
8. Tilt the probe slightly to insert it into the outer body. Be certain that the inner body is fully inserted to the bottom of the outer body and screw on the probe cap.
9. Pull the cable behind the probe cap backward slightly, and tighten the probe cap to the outer body. Allow the sensitive glass membrane at the tip of the internal probe to touch the composite membrane solely by the force of the spring.

Calibration

If the meter is an ion meter:

1. Choose more than two NH_4Cl calibration solutions. Calibrate the probe in these solutions in the order from low to high concentration.
2. Add appropriate amount of alkalizer (such as AR grade pure NaOH), dropwise into the calibration solution until its pH value is controlled in the range of 11-12.
3. Exchange the calibration solution during calibrating, rinse the measuring tip thoroughly and absorb away the water on the measuring tip.

If the meter is an mV meter (such as pH meter):

1. Calibrate the probe in more than two NH_4Cl calibration solutions in order from low to the high concentration.
2. Add appropriate amount of alkalizer (such as AR grade pure NaOH) dropwise into the calibration solution until its pH value is controlled in the range of 11-12.
3. Record mV readings in each calibration solution.
4. Plot mV-pNH_4 linear diagram or calculate the linear equation for the probe (pNH_4 is the negative logarithm of ammonium concentration).
5. Exchange the calibration solution during calibrating, and rinse the measuring tip thoroughly.
6. Absorb away the water on the measuring tip.
7. After calibration repeat probe preparation.

Measurement

1. Add appropriate amount of alkalizer, such as AR grade NaOH, to the sample solution. Soak the measuring tip in sample solution. Generally the pH value of the sample solution is controlled in the range of 11-12.
2. Record the readings of the meter when the readings become stable.
3. If the meter is an ion meter, read the concentration value of sample solution directly. If the meter is an mV meter (such as a pH meter), input the mV reading into the mV-p NH_4 linear diagram or the linear equation for the probe to calculate the pNH_4 value of the sample solution.
4. Rinse the measuring tip with deionized water. Absorb the water on the measuring tip.

Operating hints

- Before using the probe, refer to the appropriate measurement standards and select the correct measuring method.
- For measurement of samples with low concentration, use distilled water or deionized water that does not contain ammonium for making standard solution.
- Soak the internal probe in ammonia combination ISE filling solution for 24 hours before measurement.
- Once assembled, make sure the composite membrane is flat and sealed, with no leaks when the filling solution is added into the outer body.
- Keep the calibration solution and sample solution at a uniform temperature and flow rate by swinging the probe gently or turning on the magnetic stirrer.
- Keep the probe upright to prevent loss of filling solution. Replenish filling solution immediately in the event of a spill.
- Maintain constant temperature of solution, environment, and probe to obtain maximum accuracy.
- During calibration or measurement, make sure there are no bubbles in the surface of the composite membrane.
- Use the auxiliary device for the Ammonia Gas Sensing Probe, for accurate measurement of ammonia combination ISE.

- Exchange composite membrane and ammonia combination ISE filling solution if the probe response is too slow.
- If the temperature of the sample solution and the ammonium ion concentration of solution are very low, the response time will be prolonged.
- Dilute samples with high concentration (>500 ppm) before measurement.
- Rinse the composite membrane free of any substance, such as precipitation, that could clog the membrane. Do not wipe the composite membrane directly or you will damage the composite membrane.
- Take probe reading as soon as it becomes stable. Use a sealed container to decrease the evaporation of ammonia.
- After measurement, soak the measuring tip in 0.01 mol/L sulfuric acid solution or rinse it in distilled water or deionized water to remove excess ammonia diffused into the filling solution.

Maintenance

Exchange of reference filling solution

1. Siphon the probe filling solution and add fresh solution (5190-0545) until the level is 5 mm lower than the filling hole.
2. Repeat several times.

Cleaning of inorganics

Soak the measuring tip in 0.1 mol/L HCl or EDTA solution for 15 minutes.

Cleaning of organics

Soak the measuring tip in absolute ethyl alcohol, or other solvent that can dissolve organics for 15 minutes.

Cleaning of grease

Soak the measuring tip in warm, weakly alkaline detergent for 15 minutes.

Cleaning of protein precipitation

Soak the measuring tip in 0.1 mol/L HCl solution that contains 1% pepsin for 15 minutes.

Troubleshooting

Meters

Refer to the operating manual of the meter. Check all relevant parts, such as electrode, calibration solution, and samples.

Probe

1. Check the mV value after the calibration solution concentration has been changed ten times. The change of mV value should be greater than 52 mV. If not, clean the probe as described in the maintenance procedures.
2. Use effective calibration solution. Make sure that the distilled or deionized water used to prepare the calibration solution meets all requirements. Make sure the calibration solution is not contaminated or beyond its shelf life.

Method

Refer to relative standards to ensure the measuring methods are correct.

For any other problems during probe use, contact your Agilent Technologies customer service representative.

Storage

Short-term

Soak the internal probe in pH 4.01 calibration solution.

Long-term

1. Remove the probe cap and the membrane cap in sequence. Drain the filling solution in the ammonia combination ISE. Rinse with distilled water or deionized water.
2. Place the internal probe in the outer body and store the probe in dry condition.

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概述

本电极用于测量水中的氨和铵离子浓度。本电极需要与 3200I 仪器或类似仪器一起使用。

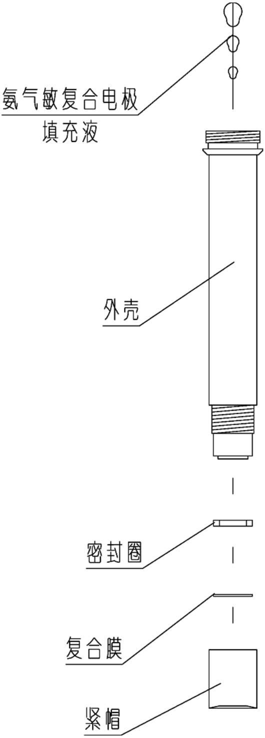
安全提示

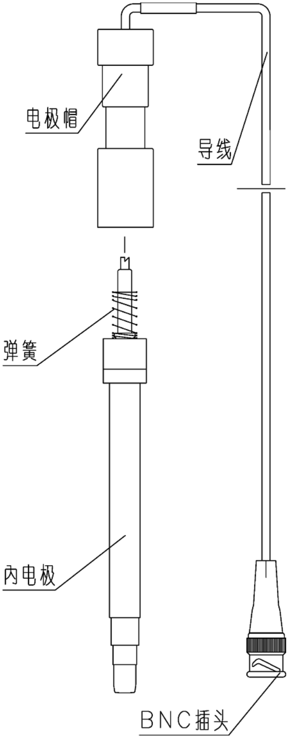
- 按使用说明使用本产品。
- 电极内电解质溶液、附带的化学试剂和建议使用的清洗溶液，不能口服，不能接触皮肤或各种器官等，如意外接触，用干净水清洗接触部位。在没有成年人监管时，不要让十八岁以下未成年人接触或使用本产品。注意：本产品有易碎玻璃零件，测试过程中需要使用强碱。

技术参数

浓度范围	10^{-1}– 10^{-6} mol/L
适用温度范围	0–50 ℃
电极内阻	⩽ 1500 MΩ
参比系统	Ag/AgCl
电极直径	16 mm
电极总长度	180 mm
导线接口	BNC
导线长度	1000 mm

电极插图





使用步骤

电极准备 (参考电极安装图)

1 将电极竖直，拧开电极帽。

2 小心将内电极从外壳内移出，用蒸馏水或去离子水清洗内电极和外壳。

3 将紧帽从外壳上旋开。

4 移去紧帽中原有的复合膜和密封圈，用蒸馏水或去离子水清洗紧帽。

5 用镊子小心夹取新的复合膜 （此膜为多层叠合结构的复合膜，不得拆分）缓慢放入电极紧帽内，使膜平整贴靠电极紧帽底部。

6 将新的密封圈套在外壳上，再将紧帽拧上拧紧，要求复合膜平整无凹凸、无折皱且紧贴于紧帽底部。

7 在外壳内注入 3.5 毫升氨气敏复合电极填充液 (p/n 5190-0544)。

8 将内电极稍倾斜地插入电极外壳至底部，然后将电极帽拧上。

9 向后轻拉电极帽后的电极导线将电极帽与外壳旋紧，缓慢松开电极导线，使内电极头部的敏感玻璃面完全依靠电极内部的弹簧压力与复合膜接触。

10 查阅标准 （国家标准，行业标准等）或相关文献资料，确定电极使用方法。电极的常规使用方法按照以下步骤。

11 电极校正

    a 如果仪器是离子计，则按照仪器说明，选择两种以上不同浓度的 NH_4Cl 校正溶液，由稀至浓对电极进行校正。期间电极更换校正溶液时，需仔细冲洗电极测量端并吸干表面水迹。校正溶液需滴加适量碱化剂 （如分析纯 NaOH）使其 pH 值控制在 11–12 pH 范围内。

    b 如果仪器是 mV 计 （例如 pH 仪器），选择两种以上不同浓度的 NH_4Cl 校正溶液，由稀至浓对电极进行校正 , 记录各校正溶液的 mV 值，然后绘制 “mV-pNH_4” 电极线性曲线图或电极的线性校正方程 （其中 pNH_4 为铵离子浓度值的负对数值）。期间电极更换校正溶液时，需仔细冲洗电极测量端并吸干表面水迹。校正溶液需滴加适量碱化剂 （如分析纯 NaOH）使其 pH 值控制在 11-12 pH 范围内。

电极测量

1 样品中加入适量碱化剂(如分析纯NaOH),将电极测量端浸于被测溶液中，一般情况下使被测溶液 pH 值控制

在 11–12 pH 范围内。将电极测量端浸于碱化后的样品中，稳定后读数。

2 读数处理

  a 如果仪器是离子计，则直接读取被测溶液的氨离子浓度值。

  b 如果仪器是 mV 计（例如 pH 计），则将仪器显示的 mV 读数代入绘制的 “mV- pNH_4” 电极线性曲线图或线性校正方程，计算得知被测溶液的 pNH_4 值。

3 将电极测量端用去离子水冲洗，吸干电极测量端表面水迹 (不可用力擦拭)。

储存方法

短期储存

将内电极浸在 pH 4.01 的缓冲溶液中保存。

长期储存

a 将电极帽和紧帽依次旋开，去除氨气敏复合电极填充液并用蒸馏水或去离子水洗净。

b 将内电极装入外壳内，干放保存。

注意事项

1 电极使用前，应查阅相关的文献或参照标准进行操作，选择正确的测量方法。

2 对于低浓度测量，确保配制校正溶液使用的蒸馏水或去离子水中不含铵离子。

3 内电极在测试前必须在氨气敏复合电极填充液中浸泡，对测量高精要求较高时，建议浸泡 24 小时。

4 复合膜安装后应平整、严密、牢固、加入内溶液后不得渗漏。

5 校正溶液和样品溶液的搅拌速度应保持一致。

6 电极使用时不能平放或倒放，以防止氨气敏复合电极填充液流失。如有流失，应及时补充。

7 电极校正或测量时，溶液的温度控制 （环境温度、电极温度、溶液温度）必须尽量一致，否则影响测量精度。

8 电极校正或测量时，复合膜表面不能有气泡附着。

9 基于氨气敏复合电极使用的复杂性，客户需要精确测量时建议客户使用辅助测量装置。

10 如果电极响应太慢，尝试更换复合膜和氨气敏复合电极填充液。

11 被测溶液温度较低或被测溶液的铵离子浓度低时，电极的响应时间会相应延长。

12 建议高浓度 （大于 500 ppm）测量时稀释样品。

13 测量时应及时清除阻塞复合膜的物质 （如沉淀等）。但不得直接擦拭复合膜，以免损害复合膜。

14 电极浸入碱化后的溶液，由于氨气的挥发，应在电极充分响应的前提下及时读取读数。密封容器可以减少氨气的挥发。

15 测量完毕后，应尽量快速将电极测量端浸入 0.01 mol/L 稀硫酸溶液或在蒸馏水或去离子水中清洗电极，以去除过多进入氨气敏复合电极填充液的氨。

电极维护

更换氨气敏复合电极填充液

1 将电极的氨气敏复合电极填充液吸空。

2 从加液孔注入新鲜的氨气敏复合电极填充液（p/n 5190-0545）至距离加液孔 5 mm 左右处。

3 再次吸空氨气敏复合电极填充液，从加液孔注入新鲜的氨气敏复合电极填充液至距离加液孔 5mm 左右处。

电极清洗和修复

无机物清洗：

将电极测量端浸于 0.1 mol/L 的 HCl 或 EDTA 溶液中 15 min。

有机物清洗：

将电极测量端浸于无水乙醇 （或能够溶解该有机物的溶剂）中 15 min。

油脂类清洗：

将电极测量端浸于温热的弱碱性洗涤剂中 15 min

蛋白质沉淀的清洗：

将电极测量端浸于含 1 % 胃蛋白酶的 0.1 mol/L 盐酸溶液中 15 min。

疑难解答

电极使用中发现异常情况，请按下列步骤查找原因

仪器

参看仪器说明书的相关部分

查看仪器、电极、校正溶液、样品等相关部分之间的衔接。

电极

电极校正时，校正溶液浓度值每相差 10 倍，则 mV 值应相差 52mV 以上，否则需要对电极进行维护操作。

校正溶液

校正溶液来源有效。配制校正溶液用的蒸馏水或去离子水应符合要求。校正溶液应在有效期内，不会污染或变质。

方法

查阅相关资料或文献，以确认测试方法 （样品处理，测试步骤等）是否正确。

如在电极使用过程中有其他疑问，请联系售后服务部门。如需购买，请与安捷伦经销商联系或者登陆安捷伦官方网站。

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