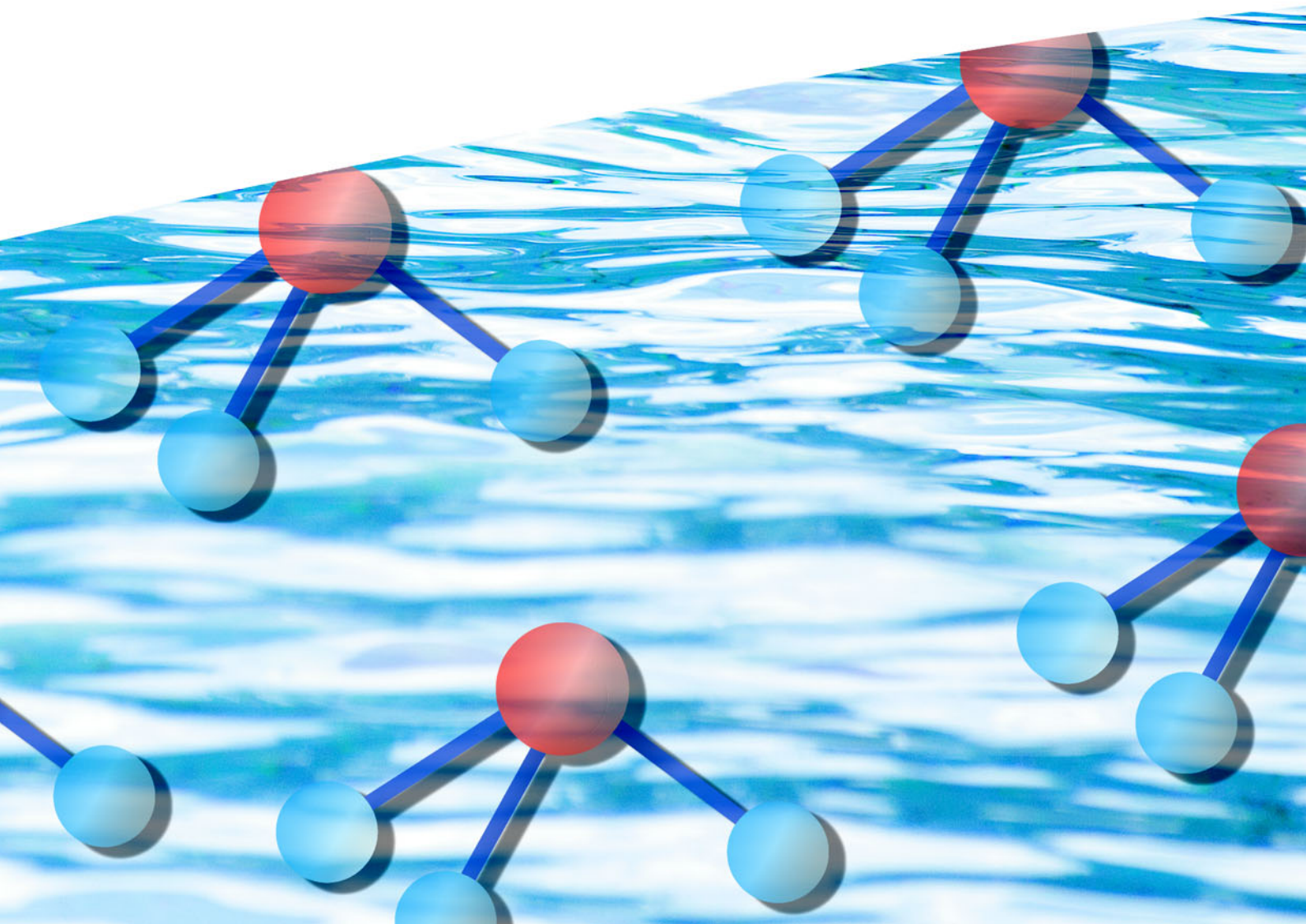


Information on measuring ammonia in water

Dr. Jürgen Schleicher



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Preface

Measurement of the concentration of ammonia in aqueous solutions is a requirement in many application areas, such as for coolant monitoring and laboratory measurement, to name but two examples. A fast and simple way of measuring ammonia can be achieved by using a membrane-covered, gas-sensitive sensor that operates on a potentiometric principle.

But for successful measurement, several factors must be observed when handling and using of ammonia sensors. This brochure is intended to provide practical help for the users in these matters. A special emphasis is placed on the two application situations mentioned above.

Furthermore, the construction and mode of operation of the sensor is also briefly described.

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Contents

1 What is ammonia?

Ammonia (NH_3) is a poisonous and pungent-smelling colorless gas that makes the eyes water. Ammonia is readily soluble in water. The aqueous solution has an alkaline reaction and is known as ammonia water or tincture of ammonia. In conjunction with acid, ammonia forms salts that disassociate in water, to form ammonium ions (NH_4^+) and the corresponding anions. In nature, ammonia is produced by the decomposition of animal or vegetable material that contains nitrogen. The substantial latent heat of vaporization of ammonia is utilized in refrigeration machinery, where ammonia can replace the halogenated refrigerants that are based on fluorine-hydrocarbons which damage the ozone layer.

2 Typical areas of application for ammonia sensors

The JUMO ammonia sensor (Figure 1, Page 7) can be used to measure ammonia (NH_3) in aqueous solutions. Ammonia in an aqueous solution is in a pH-dependent equilibrium with ammonium ions. If ammonium ions are converted into ammonia by the addition of an alkali, then the sensor will detect the resulting ammonia gas. The ammonium ions themselves will not be detected.

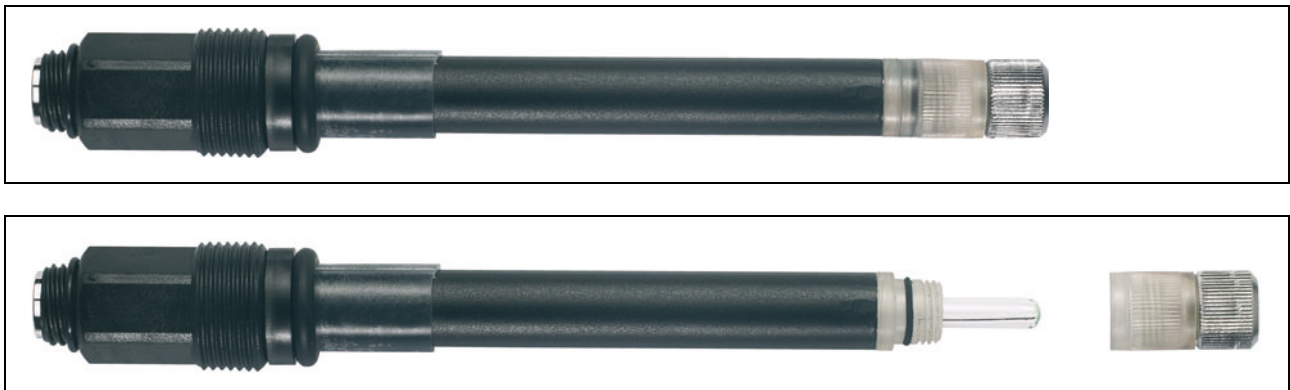


Fig. 1: Ammonia sensor

Some typical applications of ammonia sensors that may be mentioned are:

- Monitoring for leaks in cooling plant, e. g. large cold stores for storing food, ice rinks, refrigeration installations in supermarkets.
- Ammonia measurement:
 - in freshwater, salt water and seawater,
 - in boiler feed water,
 - in fish farms,
 - in electroplating baths,
 - in the wastewater of gas scrubbers,
 - in wastewater monitoring,
 - in the food sector, e. g. for wine and beer,
 - in laboratories.

The measurement can be made directly in the liquid medium. This means that it is not necessary to carry out time-consuming and complicated sample preparation. Colored or cloudy samples can also be measured. If the pH value is less than about 7.5, then some alkali must be added before the measurement, to shift the chemical equilibrium of the ammonium ions towards the ammonia state.

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If the sample contains substances that could damage the membrane, such as oils, fats or tensides, then the ammonia can still be measured, using the technique known as “head space”. In this case, damage to the sensor is ruled out, because there is no direct contact between the sensor and the sample. Measurement is performed in a gas-tight space above the substance being measured. This gas-tight space above the substance being measured is in equilibrium with the liquid as far as ammonia is concerned.

The simple sample preparation makes determination of the ammonia concentration a fast and economical operation that can be applied with high precision over a wide range of concentration.

Of course, ammonia concentration can also be measured in other substances, such as fertilizers or soil samples, if an appropriate sample preparation is carried out first, i. e. an aqueous solution is prepared with an ammonia concentration within the measuring range of the sensor and the sample is adjusted to a suitable pH value.

3 Construction and mode of operation

The basic construction of the ammonia sensor is shown in Figure 2, Page 8: The ammonia sensor consists of a glass pH electrode and a reference electrode which are surrounded by a common electrolyte that is separated from the liquid being measured by a hydrophobic, gas-permeable membrane.

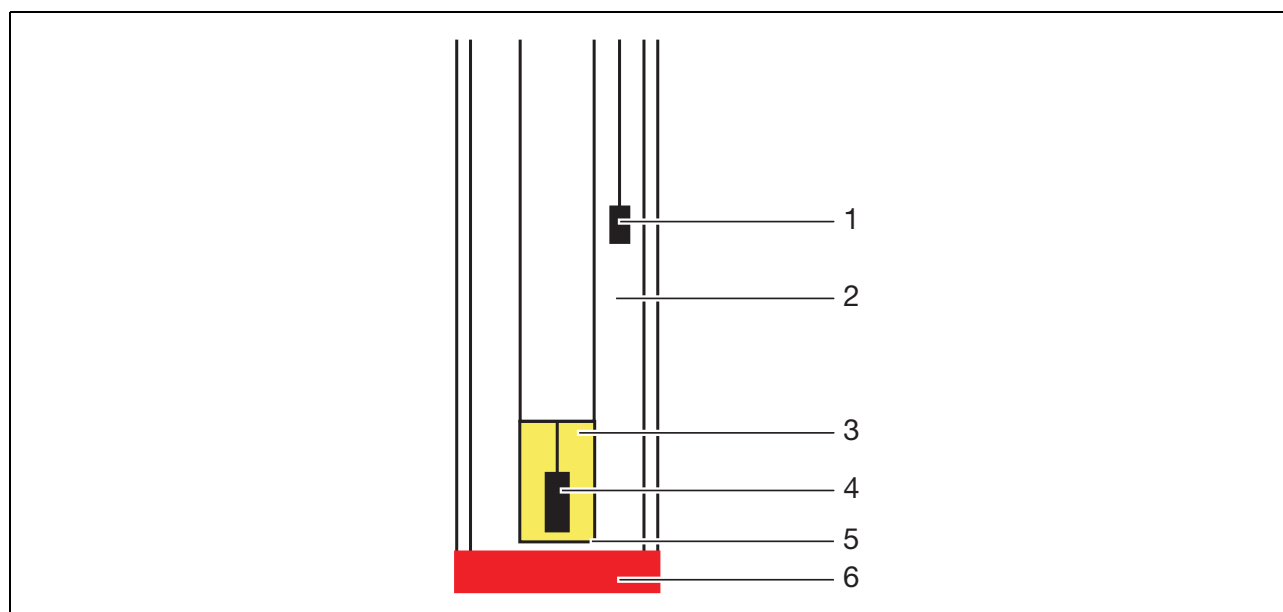


Fig. 2: Basic construction of the ammonia sensor

- | | | | |
|---|--|---|-------------------------------|
| 1 | Conduction of the reference system (Ag/AgCl) | 2 | Electrolyte |
| 3 | Internal buffer | 4 | Internal conduction (Ag/AgCl) |
| 5 | Glass membrane | 6 | Gas-permeable PTFE membrane |

A thin layer of electrolyte is placed between the hydrophobic membrane and the glass pH electrode. Its pH value increases when NH_3 gas makes its way through the membrane. The chemical processes in the electrolyte layer are described by the following reaction equation.



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This means that NH_3 functions as a base and thus increases the pH in the thin electrolyte layer in front of the glass pH electrode. Since there is only a very small volume of liquid between the hydrophobic PTFE membrane and the glass electrode, the electrode reacts very sensitively to the smallest amount of ammonia.

A strongly simplified derivation leads to the following relationship between the sensor signal and the concentration of ammonia in the substance being measured: The reaction (Equation 1) is described by the law of mass action:

$$K = [\text{NH}_4^+] [\text{OH}^-] / [\text{NH}_3] \quad (2)$$

K = equilibrium constant

$[x]$ = concentration component x

The electrolyte has a relatively high concentration of ammonium chloride, so the concentration of ammonium ions in the thin electrolyte layer may be regarded as constant if a small amount of NH_3 permeates the membrane and NH_4^+ is formed in accordance with the equation given above. As a result, the concentration of ammonium ions can be included in the equilibrium constant K' .

$$K' = [\text{OH}^-] / [\text{NH}_3] \quad \text{or} \quad [\text{OH}^-] \sim [\text{NH}_3] \quad (3)$$

Applying the Nernst equation thus results in the following dependency:

$$E = E_0 - S \log[\text{NH}_3] \quad (4)$$

E = electrode potential

E_0 = standard electrode potential

S = slope, $f(T)$

The glass membrane (Figure 2, Page 8, (5)) has a flat or slightly convex shape, so that the electrolyte layer that is formed between the membrane glass and the hydrophobic membrane is as thin as possible, to ensure a fast response time and high sensitivity. The local change in pH in the thin film of electrolyte as a result in the ammonia penetration is detected by the pH electrode.

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4 pH dependence

In accordance with the above-mentioned reaction equation (Equation 1), ammonia (NH_3) in an aqueous solution is in a pH-dependent equilibrium with ammonium ions (NH_4^+). This dependence is shown in Figure 3, Page 10.

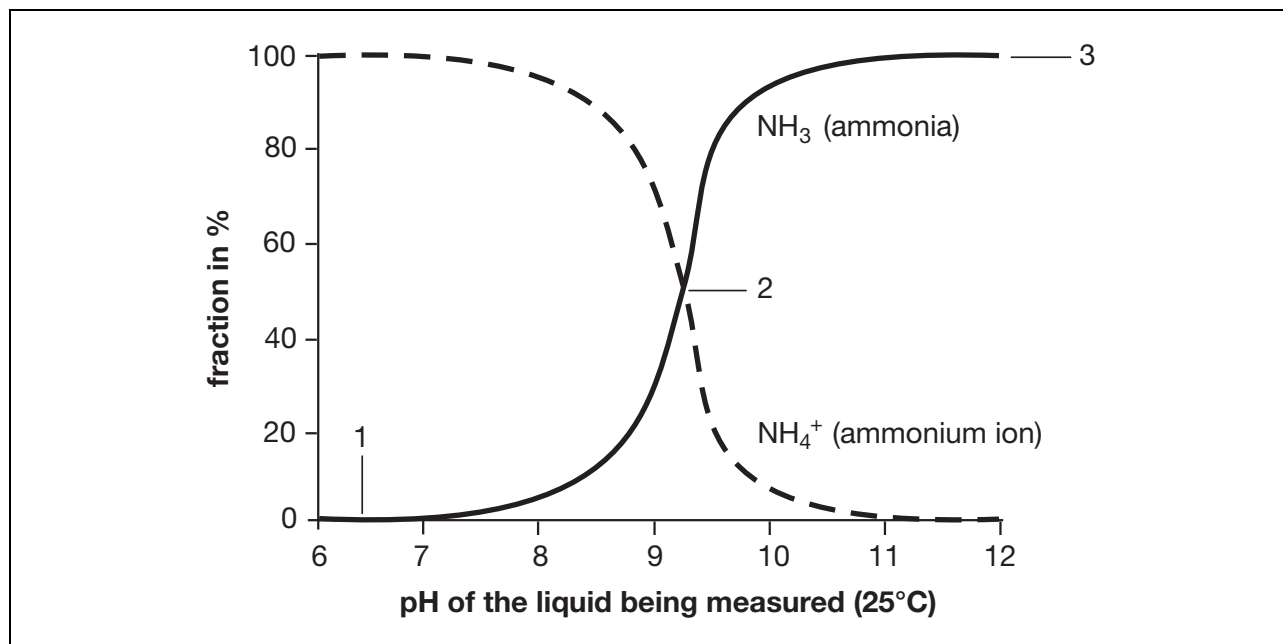


Fig. 3: pH dependence of the equilibrium between ammonia and ammonium ions

- | | | | |
|---|--|---|--|
| 1 | Only NH_4^+ ions (ammonium) are present | 2 | The ratio of NH_4^+ ions (ammonium) to NH_3 (ammonia) is 1:1 |
| 3 | Only NH_3 (ammonia) is present | | |

If ammonium ions are predominant in the solution, as is the case in the acidic range, then they must be converted into ammonia (NH_3) by the addition of a strong alkali (e. g. NaOH, adding 10 N NaOH solution to the liquid being measured for a $\text{pH} \geq 11$), since the membrane is only permeable for ammonia (water in a liquid aggregate or ionic constituents such as ammonium ions cannot pass through the membrane).

5 New JUMO ammonia sensor

The advantage of the new JUMO sensor for ammonia is that prefabricated membrane caps are available, thus avoiding the manual fitting under tension of the sensitive membrane. The membrane cap for the JUMO sensor is replaced as a complete unit (see Figure 1, Page 7). For the systems usually available today, the membrane has to be manipulated using gloves and tweezers. This method of manual membrane replacement must be applied with great care, to avoid mechanical damage or contamination of the membrane (by touching it with bare hands, for instance). During manual fitting, care must also be taken that the membrane does not develop folds or become overstretched. This time-consuming (and often faulty) test of patience is not needed for JUMO ammonia sensors, since they only require a simple replacement of the membrane cap as a unit.

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6 Concentration range

The potentiometric measurement of ammonia shows a linear characteristic over a wide range of concentration on a semi-logarithmic plot, as is indicated in the following conversion table for various units of concentration.

| mol/l | ppm as N | ppm as NH ₃ |
|--------------------|----------------------|------------------------|
| 5×10^{-7} | 7×10^{-3} | 8.5×10^{-3} |
| 10^{-6} | 1.4×10^{-2} | 1.7×10^{-2} |
| ... | ... | ... |
| 10^{-1} | 1400 | 1700 |
| 1 | 14000 | 17000 |

Table 1: Conversion table for various units of concentration

At especially low levels of concentration, the response time can be considerably longer, as shown in the table above. It may be possible to improve the response time by diluting the electrolyte by a factor of 10, using distilled water.

And, particularly at high concentration, there is a risk that readings are too low because of ammonia losses. Samples should therefore be measured as soon as possible. Loss of ammonia can be reduced by a cover or by using the apparatus in an enclosed vessel. If alkali is added, then this should be done immediately before measurement.

7 General advice on performing ammonia measurements

Possible calibration procedures for the potentiometric measurement of ammonia are: direct calibration with a series of calibration solutions, or the addition method. For the latter method, a known quantity of a standard solution is added to a sample of the liquid with the unknown concentration, and from the change in signal one can work back to calculate the initial concentration.

During calibration and measurement, care must be taken that the sample and the calibration solution have the same temperature, otherwise the transport of water vapor through the membrane can cause drifting of the electrode. Furthermore, the temperature also affects the slope of the sensor characteristic (which is temperature-dependent in accordance with the Nernst equation). Basically, the ammonia sensor can be used over the temperature range 0 to 50°C. Ammonium chloride is recommended for making up calibration solutions. A strong alkali is used to adjust the pH of the calibration solutions to $\text{pH} \geq 11$. The sample solutions are prepared in the same way.

8 Special aspects

Coloration or opacity of the liquid being measured does not affect the measurement. Most ionic substances in the sample will also have no effect, since they cannot pass through the hydrophobic membrane. But volatile amines, for instance, may affect measurement. These include various substances, e. g. methylamine, ethylamine und hydrazine. Such substances also exhibit an alkaline reaction in the sensor electrolyte. The presence of such substances in addition to ammonia leads to an erroneously high concentration reading.

If the liquid being measured contains solvents or tensides from cleaning agents or detergents that can affect the hydrophobic properties of the membrane, then measurements should be made in a

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gas space above the liquid. This “head space” method requires that the ammonia sensor is built into a completely gas-tight fitting. The gas-tight fitting can, for instance, be an Erlenmeyer flask with a bored-out stopper. The ammonia sensor is pushed into the borehole, so that it is above the surface of the liquid in the Erlenmeyer flask. A head space measurement is also advisable if oil or grease is present, since these would also block the membrane and thus adversely affect the response time of the electrode.

The sensor membrane is very delicate and must not be touched in any circumstances. Cleaning by mechanical means is not possible. Measurements cannot be performed under pressure, because the compression of the inevitable air bubbles in the sensor electrolyte can cause distortion and damage to the inelastic membrane. If the liquid being measured is under pressure, then JUMO recommends using a quick-change fitting (see Figure 6, Page 14). This reduces the pressure of the liquid to atmospheric pressure.

9 Purpose of ammonia measurement

Ammonia measurement can have various aims:

- Measurement of the relative concentration of ammonia, with reduced accuracy requirements, e. g. for coolant monitoring. To detect the absence/presence of ammonia or an increase/decrease in the ammonia concentration.
- Measurement of the absolute concentration of ammonia, with high accuracy requirements. For laboratory applications, with the aim of determining the ammonia concentration as accurately as possible.

10 Application: coolant monitoring

For coolant monitoring, the plant operator is more concerned with the fast detection of an ammonia leak in the primary circuit via a qualitative ammonia detection in the secondary circuit. The measurement of the exact concentration in the secondary circuit is of minor importance, since the main objective is to take fast action to prevent damage to the installation and its surroundings.

In this case, using an ammonia sensor for monitoring the secondary circuit makes sense, because the possible buffering effect of the coolant in the secondary circuit means that an ammonia sensor provides much faster response than, for instance, a pH measurement. The pH of the coolant must, of course, be higher than about 8, so that there is at least a small amount of ammonia in equilibrium with ammonium ions (Figure 3, Page 10).

In the JUMO transmitters dTRANS Rd 01, type 202535/10-888-000-23-00/000, and AQUIS 500, type 202560, the relationship between the electrode voltage and the concentration of ammonia in water is stored in the form of a typical characteristic (Figure 5, Page 13). Both types of transmitter are thus able to calculate the ammonia concentration from the electrode voltage measurement and display the result directly in the concentration unit “parts per million” (ppm) (Figure 4, Page 13).

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Fig. 4: Ammonia concentration display on the JUMO AQUIS 500

The characteristic curve that is stored in the transmitter is, of course, valid for certain specified conditions, such as the composition of the liquid and the pH value, and individual sensor characteristics also play a part. If conditions differ from those specified, or different electrodes are used, then this will affect the displayed value for the concentration. However, this is unimportant for coolant monitoring, since the objective is merely to detect a relative increase in the ammonia concentration. The individual characteristics of the particular sensor are allowed for in the zero point calibration. The functionality of the software is especially adapted to coolant monitoring. First of all, the sensor voltage is measured in the presence of ammonia, and the curve shown in Figure 5, Page 13 is shifted accordingly. The slope is calculated according to the Nernst equation. This requires either an automatic temperature measurement with a Pt 100 or Pt 1000 sensor, or a manual temperature entry.

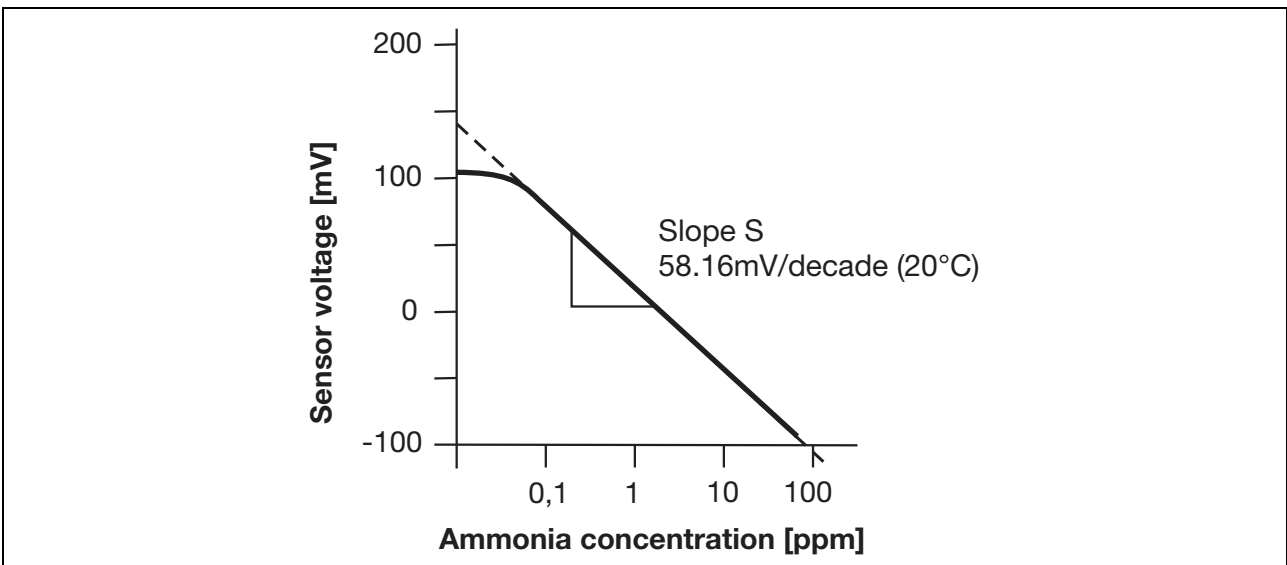


Fig. 5: JUMO dTRANS Rd 01 and JUMO AQUIS 500
Relationship between the electrode voltage and the ammonia concentration

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11 Background information on ammonia leakage monitoring in cooling circuits

The JUMO ammonia sensor can be used for the monitoring of ammonia leakage in cooling circuits. In large cooling plant (or also for ice-skating rinks), ammonia is frequently used as the coolant in the primary cooling circuit. In the secondary circuit, on the other hand, substances are used that are easier to handle and less dangerous, such as brine or various coolants based on a water-glycol mixture, or aqueous solutions of organic salts (some typical trade names are: Glykosol[®], Pekasol[®], Tyfoxit[®], ...)¹.

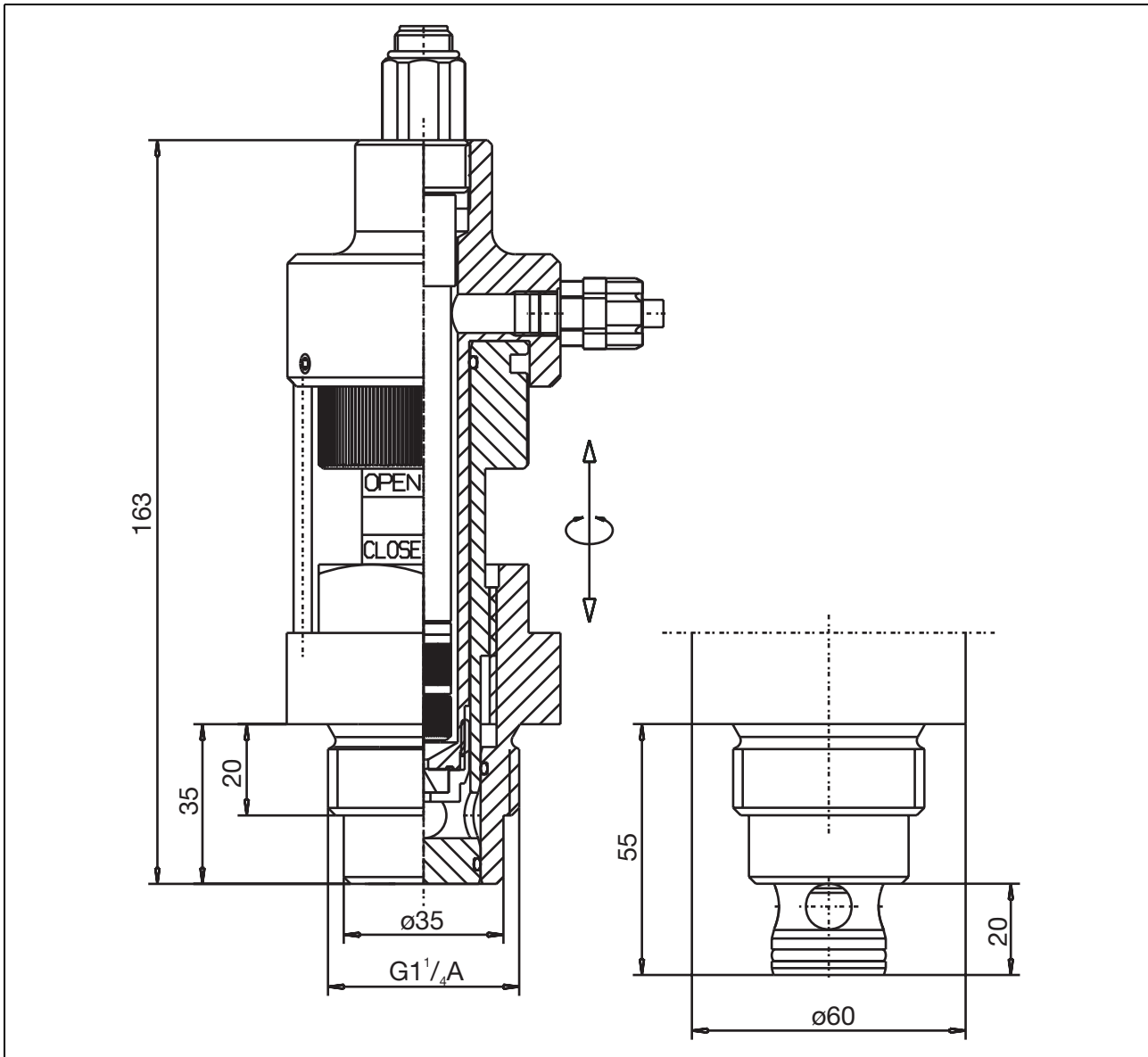


Fig. 6: Quick-change fitting for use in cooling circuits

¹ Trademarks mentioned in the text:
Glykosol[®], Pekasol[®]: proKÜHLSOLE GmbH
Tyfoxit[®]: TYFOROP Chemie GmbH

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In cooling systems it is vitally important that any ammonia leak is detected as fast as possible, in order to prevent corrosion of copper or non-ferrous piping in the secondary circuit. Although it would be conceivable to use pH measurement for this purpose, it must be excluded, because the minimum detection threshold is considerably increased because of the buffering effect of the liquid being measured. Even very low concentrations of ammonia can cause corrosion that perforates the piping system as a result of the formation of the stable copper-tetramine complex $[\text{Cu}(\text{NH}_3)_4]^{2+}$.

For this application, the JUMO ammonia sensor can be installed in a quick-change fitting (Figure 4, Page 13) that has been optimized for cooling circuits. The pressure in the medium, which may reach up to 6bar (ideally 2 – 3bar), is reduced here to atmospheric pressure, so that the membrane-covered ammonia sensor can be used without being put under pressure. Excessive coolant that enters the quick-change fitting through a porous PTFE disk used for pressure reduction is led out through an overflow, to which a discharge hose may be attached. If, in special circumstance, the pressure relationships go beyond those outlined above, then the flow of liquid through the fitting will have to be adapted by varying the thickness or porosity of the PTFE disk. On the one hand, not too much of the liquid being measured should be lost through the fitting, on the other, a satisfactory minimum threshold must be achieved for ammonia detection.

12 Application: laboratory measurements with calibration

The sensor is, of course, also suitable for measurement in situations where the user needs to determine the ammonia concentration as accurately as possible. In this case, more complex means are required for the calibration, in order to account for the various influences on the measurement. Measurements can be made by means of a pH meter working in the voltage-display [mV] mode (e. g. a JUMO dTRANS pH or Rd or a JUMO AQUIS 500) or an “ion meter” [ppm].

There are two ways of making a quantities measurement of ammonia:

- direct measurement, with a multi-point calibration
- the standard addition method.

The first method requires the setting up of a calibration curve, using reference solutions with known ammonia concentration levels. The concentration levels of the reference solutions should cover the expected concentration range of the samples. The calibration solutions and sample solutions must be treated in the same way: in order to achieve maximum measurement accuracy, the chemical equilibrium between the ammonium ions and the ammonia must be shifted towards ammonia.

This is done by adding an alkali, which is used to achieve a pH value ≥ 11 . The adjustment of the pH is carried out not only for the sample solution (with the unknown concentration) but also for the calibration solutions. Furthermore, the measurement of the samples and reference solutions must be performed at the same temperature, because the results will otherwise be influenced by the temperature.

Calibration starts off with the reference solution that has the lowest concentration, and proceeds through increasingly higher concentrations. Before changing over to the next sample vessel, the sensor must be flushed with ammonia-free distilled water and dried off by dabbing with a soft paper tissue (without touching the membrane!).

The result of the calibration measurements is a calibration curve that can be evaluated with normal analytical methods. In some laboratory instruments (ion meters), the instrument itself performs the evaluation, so that the concentration can be displayed directly after calibration. For lengthy series of measurements, the calibration should be repeated at intervals.

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With the standard addition method, a known amount of ammonia (in the form of ammonium chloride) is added to the sample with the unknown concentration. From the increase in the signal after this augmentation, one can work back to derive the unknown initial concentration, whereby the increased volume of the total augmented solution must be taken into account in the calculation. With the standard addition method, the user can exclude matrix-specific influences that are not accounted for when setting up a calibration curve (the first method). The remarks made previously about the shift of the chemical equilibrium in the ammonia direction also apply to the standard addition method.

13 Procedure for laboratory measurements

In order to ensure that the chemical equilibrium between ammonium ions and ammonia is shifted towards ammonia as completely as possible, alkali must be added to the samples with the unknown concentration and the standard solutions just before starting measurement. One can, for instance, proceed as follows: 100ml of standard solution resp. 100ml of the sample have 1 ml 10-mol caustic soda added until the pH value has reached at least 11 (samples with a low pH may require the addition of a substantial amount of caustic soda to reach a pH of at least 11. If in doubt, the pH must be measured).

Samples and standard solution should be stirred with a magnetic mixer and mixer paddle, so that the measurement stabilizes quickly. But care must be taken that the samples and standard solutions are not warmed up by action of the magnetic mixer. If necessary, a thermally insulating plate must be placed between the vessel and the mixer plate. The ammonia sensor should be mounted on a stand in such a way that it is immersed in the liquid in the sample vessel placed on the mixer, but does not touch the magnetic mixer paddle or the bottom of the vessel. Any air bubbles on the membrane must be removed.

14 Further notes on using the ammonia sensor

If the ammonia sensor has to be used in media that contain dissolved salts or solvents at high levels of concentration, such as anti-freeze, then drift effects must be expected when the standard electrolyte is used.

The drift of the sensor is caused by the diffusion of water molecules through the sensor membrane. This is an osmotic process through a semipermeable membrane, whereby water molecules diffuse from the side with the lower concentration of the species to the side with the higher concentration. Osmosis is a colligative property, that depends only on the quantity of the dissolved species (the total number of dissolved molecules and ions) and not on their nature.

When water vapor from the sensor electrolyte diffuses through the semipermeable membrane into the liquid being measured (acting to equalize the concentration of the species on both sides of the membrane), then the sensor will continue to drift as long as this process continues. It is, of course, neither feasible nor intended that the small volume of electrolyte in the ammonia sensor should equalize the concentration. Since the liquid being measured has a given mix of constituents and cannot be altered, the adjustment can normally only be made on the side with the sensor electrolyte.

For this purpose, JUMO provides a specially adapted sensor electrolyte which should be used, for instance, for coolants based on glycols or carbonic acid salts. It is, of course, not possible to match the sensor electrolyte to all possible individual mixtures that may occur. But the augmentation of the electrolyte concentration will nevertheless reduce the drift effects.

The special electrolyte can be used at temperatures down to -5°C. If necessary, the ammonia

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sensor will have to be used in combination with an impedance converter at low temperatures, since the electrical resistance of the glass membrane increases as the temperature decreases.

For laboratory measurements it is naturally possible to dilute the sample solution as required, if there are drift effects caused by high concentrations of additional constituents.

If the ammonia sensor is stored in air, the electrolyte may lose some water (as vapor). The sensor should therefore be stored in liquid electrolyte, in the storage receptacle that is provided with the sensor.

During measurement and calibration, care should be taken that no air bubbles are present on the sensor membrane since this can hinder the passage of ammonia through the membrane. For this reason, it can often be advisable to mount the sensor at an angle of about 20° from the vertical, to prevent the accumulation of air bubbles.

To avoid loss of the analyte, the surface-to-volume ratio of the sample should be low (e. g. by using a narrow-necked Erlenmeyer flask).

If samples containing ammonia are to be stored for some time before analysis, then it is recommended that they are conserved by adding acid (e. g. 1 ml 0.5-mol sulfuric acid per liter of sample). The sample can then be stored in a closed vessel. However, before making the actual measurement, the probe must be made alkaline again, as described above.

If chlorine is present at the same time as ammonia, the measurement will be affected by the formation of chloramines. The disturbing effect of chlorine in chlorinated swimming pool water or drinking water can be countered by a previous addition of sodium thiosulfite.

If any of a number of heavy metal ions are present (e. g. Cu^{2+} , Zn^{2+} , Hg^{2+}), the measurement will be affected by the formation of complex ammonia compounds. Many complex compounds, such as ethylene-diamine-tetra-acetic acid (EDTA) and its salts, are able to free the ammonia completely from the metal complexes, thus enabling measurement with the ammonia sensor. The addition of EDTA will also prevent a possible flocculation of metal hydroxides when the sample is made alkaline.

15 Testing the sensor function

To test the functioning of the sensor, prepare two test solutions that differ in their ammonium chloride concentration by a factor of 10. The recommended procedure is described below.

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16 Preparation of the required solutions

Pour 1 ml of 0.1 mol/l ammonium chloride solution into a 100 ml measuring flask, and fill it up to 100 ml with ammonia-free distilled water. For the actual ammonia measurement, transfer the solution to a glass beaker and add 1 ml of 10-mol caustic soda (= test solution 1) just before making the measurement. During the measurement at 25°C, agitate the solution with a magnetic stirrer and paddle.

Pour 10 ml of 0.1 mol/l ammonium chloride solution into a second 100 ml measuring flask, and fill it up to 100 ml with ammonia-free distilled water. After transferring this to a glass beaker and adding 1 ml of 10-mol caustic soda (= test solution 2), proceed as already described for solution 1.

The sensor voltage measured when using test solution 1 should be at least 50 mV higher than that obtained with test solution 2.

Preparation of the 10-mol caustic soda

40 g of NaOH are dissolved in about 80 ml of ammonia-free distilled water, in a 100 ml measuring flask. The solution will become a little warm. When it has cooled down to 20°C, top up to 100 ml with distilled water.

Preparing the 0.1 mol/l ammonium chloride solution

0.535 g of ammonium chloride is first of all dissolved in a partial quantity of ammonia-free distilled water in a 100 ml measuring beaker and then, when it has cooled down to 20°C, topped up to 100 ml.

17 Versatile measuring principle

In addition to the measurement of ammonia, gas-sensitive sensors based on the potentiometric measurement principle can also be made for other analytes. The method is suitable for all analytes in gaseous form that can pass through the membrane and alter the pH value of the electrolyte behind the membrane. These include, for instance, carbon dioxide (CO₂) and sulfur dioxide (SO₂). If you are looking for special solutions to your measurement problems, contact the JUMO analytical instruments department.

18 JUMO data sheets and operating instructions for ammonia sensors and accessories

- Data Sheet 20.1030 – JUMO ecoLine pH/Rd
pH and redox combination electrodes in versions with glass or plastic stems
- Data Sheet 20.2560 – JUMO AQUIS 500
Transmitter/controller for pH value or redox potential and temperature
- Operating instructions 20.1040 – gas-sensitive sensors for ammonia measurement

These data sheets and the operating instructions can be downloaded from www.jumo.de

19 Supplementary literature

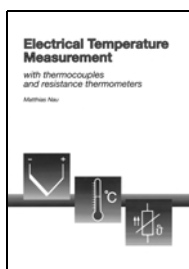
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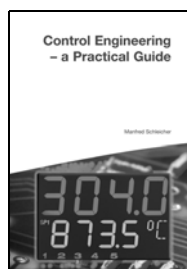
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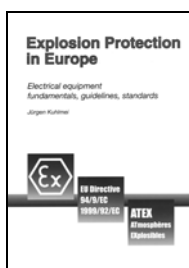
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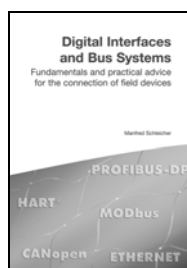
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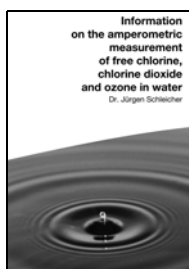
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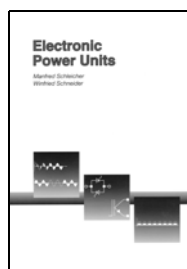
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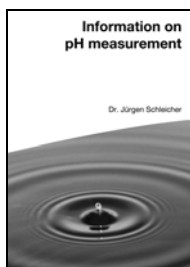
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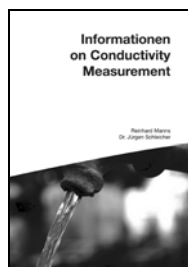
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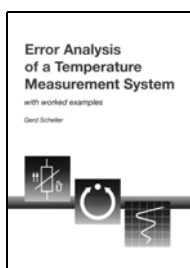
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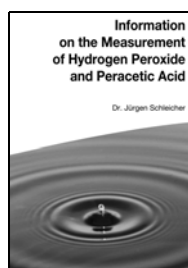
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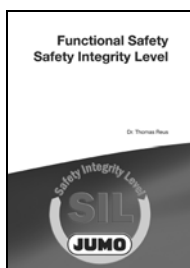
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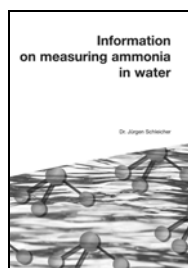
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