

Determination of Nitrite in Cooling Lubricants

Semiquantitative determination using a test strip impregnated with N-(1-naphthyl)-ethylenediamine

Introduction

Cooling lubricants are multi-component mixtures used during the processing of material. They are used to dissipate the heat generated when processing workpieces (cooling) and to reduce the friction that occurs (lubricating). Another aspect is the removal of filings and solids that occur during processing of the material (rinsing).¹

Since the properties of the cooling lubricants can change through contact with the workpieces and the working environment, manufacturers and regulations recommend regularly checking various properties and parameters of the cooling lubricants to ensure their functionality and to avoid a health risk for the employees.

In this context, an important parameter is the nitrite content. The nitrite content should be monitored to prevent the potential formation of carcinogenic nitrosamines. Therefore, it is important to take precautions when using cooling lubricants to prevent the formation or entrainment of nitrites.² This can occur, for example, due to microbial degradation of nitrates in the process water or through entrainment from hardening baths. A recommended limit of 20 mg/L of nitrites is applicable. If this limit is exceeded, it is recommended to consider changing or partially replacing the cooling lubricant emulsion.^{2,3}

Experimental

This application note describes the determination of nitrite in aqueous cooling-lubricant emulsions.

Method

In the presence of an acidic buffer nitrite ions react with an aromatic amine to form a diazonium salt, which in turn reacts with N-(1-naphthyl)-ethylenediamine to form a red-violet azo dye. The nitrite concentration is measured semiquantitatively by visual comparison of the reaction zone of the test strip with the fields of a color scale.

Measuring range

2 – 5 – 10 – 20 – 40 – 80 mg/L Nitrite (NO_2^-)

Applicable Sample

Aqueous cooling-lubricant emulsions

Reagents, Instruments, and Materials

Test kit

- Quant® Nitrite Test, colometric 2-80 mg/L (NO_2^-) (1.10007)

Other Reagents and Accessories

- MQuant® pH-indicator strips, pH 0-14, non-bleeding (1.09535)
- Sodium acetate anhydrous for analysis EMSURE® (1.06268)
- L(+)-Tartaric acid for analysis EMSURE® (1.00804)

Analytical Procedure

Sample Preparation

The pH must be within the range 1 – 13. If the pH is lower than 1, buffer the sample with sodium acetate. If it is greater than 13, adjust it to approx. pH 3 - 5 using tartaric acid.

Measurement

- Immerse the reaction zone of the test strip in the pretreated sample (**15 - 30 °C**) for **3 sec.**
- Shake off any excess liquid from the test strip, and after 15 sec, determine which color field on the label matches the color of the reaction zone most accurately.
- Read off the corresponding result in mg/L NO_2^- or $\text{NO}_2^- \text{N}$.

Notes on the measurement:

- The color of the reaction zone may continue to change after the specified reaction time has elapsed. This must not be considered in the measurement.
- If the color of the reaction zone matches or is darker than the darkest color on the scale, repeat the measurement using fresh, diluted samples until a value of less than 80 mg/L NO_2^- is obtained. It is important to consider the dilution when interpreting the analysis result.

Analytical Quality Assurance

To check test strips and handling: Dilute the nitrite standard solution with distilled water to 20 mg/L NO_2^- and analyze as described in "Measurement".

Conclusion

The MQuant® Nitrite Test is an **easy and fast way to analyze the nitrite concentration** in aqueous cooling-lubricant emulsions. The measurement can be performed without much experience.

References

- IFA Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung, Praxishilfen, Kühlschmierstoffe. [accessed 2022 Jun 30] <https://www.dguv.de/ifa/praxishilfen/kuehlschmierstoffe/kuehlschmierstoff/index.jsp>
- IFA Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung, Praxishilfen, Kühlschmierstoffe, Lexikon, Nitritgehalt. [accessed 2022 Jun 30] <https://www.dguv.de/ifa/praxishilfen/kuehlschmierstoffe/lexikon/nitritgehalt/index.jsp>
- BAuA – Bundesanstalt für Arbeitsschutz und Arbeitsmedizin, Technische Regel für Gefahrstoffe - TRGS 611 Verwendungsbeschränkungen für wassermischbare bzw. wassergemischte Kühlschmierstoffe, bei deren Einsatz N-Nitrosamine auftreten können. [accessed 2022 Jun 30] <https://www.baua.de/DE/Angebote/Rechtstexte-und-Technische-Regeln/Regelwerk/TRGS/TRGS-611.html>

Featured Products

Product	Cat. No.
MQuant® Tests	
Nitrite Test, colorimetric, 2-80 mg/L (NO_2^-), MQuant®	1.10007
pH-indicator strips pH 0 - 14 Universal indicator, non-bleeding, pH range 0-14, graduations and accuracy accuracy: 1 pH unit, for use with MQuant® StripScan App	1.09535
Solvents and Reagents	
Sodium acetate anhydrous for analysis EMSURE® ACS, Reag. Ph Eur	1.06268
L(+)-Tartaric acid for analysis EMSURE® ACS, ISO, Reag. Ph Eur	1.00804
Products for AQA	
Nitrite standard solution, traceable to SRM from NIST NaNO_2 in H_2O 1000 mg/L NO_2 Certipur®	1.19899

For more information on

Reflectoquant® Test strips see [SigmaAldrich.com/test-strips](https://www.sigmaaldrich.com/test-strips)

Applications see [SigmaAldrich.com/wfa-applications](https://www.sigmaaldrich.com/wfa-applications)

Order/Customer Service see [SigmaAldrich.com/order](https://www.sigmaaldrich.com/order)

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