



Application Note AN-PAN-1011

Online analysis of permanganate absorption number (PAN)

Caprolactam, a vital precursor to Nylon 6, is widely used in the production of various industrial fibers, textiles, and plastics. Maintaining consistent caprolactam purity is essential for ensuring the quality of these finished products. The permanganate absorption number (PAN) analysis serves as a key indicator of caprolactam purity within the production

process, adhering to ISO 8660 standards.

This Process Application Note describes the straightforward 24/7 monitoring of the permanganate absorption number (PAN) as a measure of the caprolactam purity in the corresponding process.

INTRODUCTION

In 2023, global caprolactam production capacity was approximately 9 million tons [1], the majority of which went on to produce Nylon 6. Nylon 6 is mainly used to manufacture fibers for clothing, carpets, and industrial purposes. About 30% of produced Nylon 6 is used for resin production [1].

Caprolactam is primarily manufactured from cyclohexanone, cyclohexane, or toluene. The dominant industrial process involves converting cyclohexanone to cyclohexanone oxime, followed by Beckmann rearrangement to yield caprolactam with nearly 98% efficiency [2].

The industry standard Beckmann rearrangement process employs sulfuric acid (or oleum) as a catalyst to transform cyclohexanone oxime into caprolactam (**Reaction 1**). However, this method generates

ammonium sulfate as a byproduct. While valuable for fertilizer production, its environmental impact has led to restrictions in certain regions.



Reaction 1. Reaction mechanism from cyclohexanone to form Nylon 6.

Maintaining caprolactam quality is crucial for its downstream applications like Nylon 6 production. This can be achieved by employing a specific test – permanganate absorption number (PAN) analysis.

This standardized procedure, detailed in international references like ISO 8660, measures the presence of oxidizable impurities within the caprolactam sample. Essentially, PAN analysis measures the stability of caprolactam by evaluating its reaction with potassium permanganate. Lower PAN values indicate a purer caprolactam sample, leading to the production of better-quality Nylon 6.

Traditional laboratory analysis employing manual sampling is a viable option, but it presents certain

drawbacks. The time-consuming nature of this approach misses real-time process variations. Analyzing multiple process streams concurrently becomes labor-intensive, and the use of sulfuric acid introduces safety concerns within the laboratory environment.

In light of these limitations, online process analyzers such as the 2060 TI Process Analyzer (**Figure 2**) have emerged as the preferred solution. This process analyzer exhibits cutting-edge technology and offers continuous, high-precision analysis of caprolactam impurities, ensuring the consistent production of top-quality Nylon 6.

APPLICATION

Oxidizable impurities are monitored in caprolactam according to ISO 8660 for online purposes, with precise time- and temperature-controlled colorimetric measurements. Metrohm Process Analytics offers a multiparameter process analyzer solution for precisely measuring PAN online according to ISO 8660: the 2060 TI Process Analyzer (**Figure 2**).



Figure 2. The 2060 TI Process Analyzer is suitable for monitoring PAN during caprolactam production.

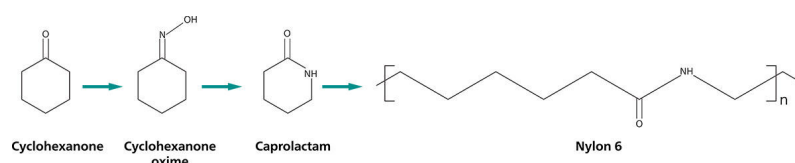
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Table 1. Typical range found for the permanganate absorption number in the caprolactam production process.

Parameter	Range
PAN	0–35

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REMARKS

Since caprolactam absorbs moisture, employing a reliable technique for water content determination is also crucial. The 2060 The NIR Analyzer from Metrohm Process Analytics stands out as an excellent solution in this regard. It delivers accurate water content measurements within seconds, eliminates the need for sample preparation, and generates no chemical

waste.

Other online applications are possible depending on the production process, including NH_4OH , NH_4NO_2 , SO_3^{2-} , alkalinity, moisture, iron, high/low acidity in cyclohexanone (Anolon), $(\text{NH}_4)_2\text{SO}_3$, H_3PO_4 , and other related components.

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CONCLUSION

Continuous online monitoring of PAN in caprolactam is essential for optimizing downstream Nylon 6 production. The Metrohm Process Analyzer 2060 TI Process Analyzer delivers precise, real-time PAN

analysis. This enhances efficiency, reduces costs, and improves product quality. Its versatility enables simultaneous monitoring of multiple process parameters for comprehensive process control.

REFERENCES

1. *Caprolactam Market Size, Share Analysis and Industry Forecast*. Prismane Consulting.
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RELATED APPLICATION NOTES

AN-PAN-1041 Inline monitoring of free isocyanate (%NCO) content in polyurethane

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AN-PAN-1041 Inline monitoring of free isocyanate (%NCO) content in polyurethane

AN-NIR-077 Moisture analysis in caprolactam

BENEFITS FOR ONLINE PROCESS ANALYSIS

- Fully automated diagnostics – automatic alarms for when samples are out of specification parameters.
- Guarantee compliance with global standards.
- Avoid unnecessary costs by measuring multiple parameters simultaneously in the process stream.
- Enhanced control over the caprolactam production process, enabling fine-tuning for optimal purity and efficiency.
- Safer working environment for employees (no handling of H_2SO_4).



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CONFIGURATION



2060 Process Analyzer

The 2060 Process Analyzer is an online wet chemistry analyzer that is suitable for countless applications. This process analyzer offers a new modularity concept consisting of a central platform, which is called a «basic cabinet».

The basic cabinet consists of two parts. The upper part contains a touch screen and an industrial PC. The lower part contains the flexible wet part where the hardware for the actual analysis is housed. If the basic wet part capacity is not sufficient enough to solve an analytical challenge, then the basic cabinet can be expanded to up to four additional wet part cabinets to ensure enough space to solve even the most challenging applications. The additional cabinets can be configured in such a way that each wet part cabinet can be combined with a reagent cabinet with integrated (non-contact) level detection to increase analyzer uptime.

The 2060 process analyzer offers different wet chem techniques: titration, Karl Fischer titration, photometry, direct measurement and standard additions methods.

To meet all project requirements (or to meet all your needs) sample preconditioning systems can be provided to guarantee a robust analytical solution. We can provide any sample preconditioning system, such as cooling or heating, pressure reduction and degassing, filtration, and many more.

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