Hach 9586 sc Hydrazine Analyzer Calibration

Introduction

The Hach 9586 sc Hydrazine Process Analyzer should be calibrated when used in matrices other than high purity water. A routine calibration with an appropriate laboratory test will ensure that the analyzer is producing accurate results, even in the presence of common boiler treatment chemicals. Hach Hydrazine Laboratory Method 8141 measures hydrazine from 4-600 µg/L, and can be used to calibrate the 9586 sc analyzer. This application note describes how to perform this routine calibration.

Background

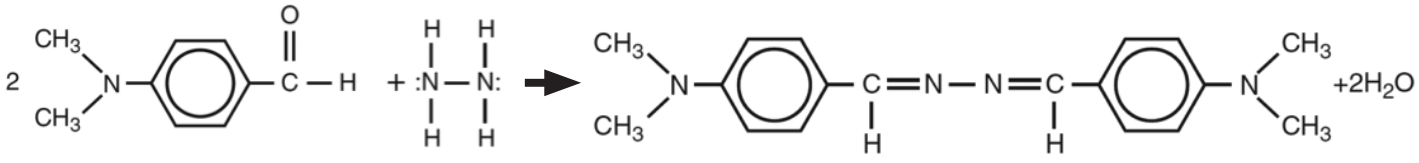
The Hach 9586 sc Hydrazine Process Analyzer uses 3-electrode amperometry to measure hydrazine and other oxygen scavengers in high purity steam-cycle water. Hydrazine is oxidized at the platinum working electrode surface through application of a constant polarization voltage. This oxidation results in the generation of a current which is proportional to the concentration of hydrazine in the sample. The chemical reaction is shown in Figure 1. As the oxidation is enhanced in an alkaline environment, alkaline amine vapors are introduced to the measuring cell through a venturi.

Figure 1 – Oxidation of Hydrazine

$$N_2H_4 + 4OH^- \rightarrow N_2 + 4H_2O + 4e^-$$

Hach Hydrazine Laboratory Method 8141 uses the p-dimethylaminobenzaldehyde method for colorimetric determination of hydrazine concentration, Figure 2. Under acidic conditions, hydrazine combines with p-dimethylaminobenzaldehyde to form a yellow complex. This intensity of the color of this complex is proportional to the hydrazine concentration. Color is measured with a DR-series spectrophotometer (DR1900, DR3900, DR6000), and the concentration is calculated with the built-in calibration curve.

Figure 2 – p-Dimethylaminobenzaldehyde Reaction



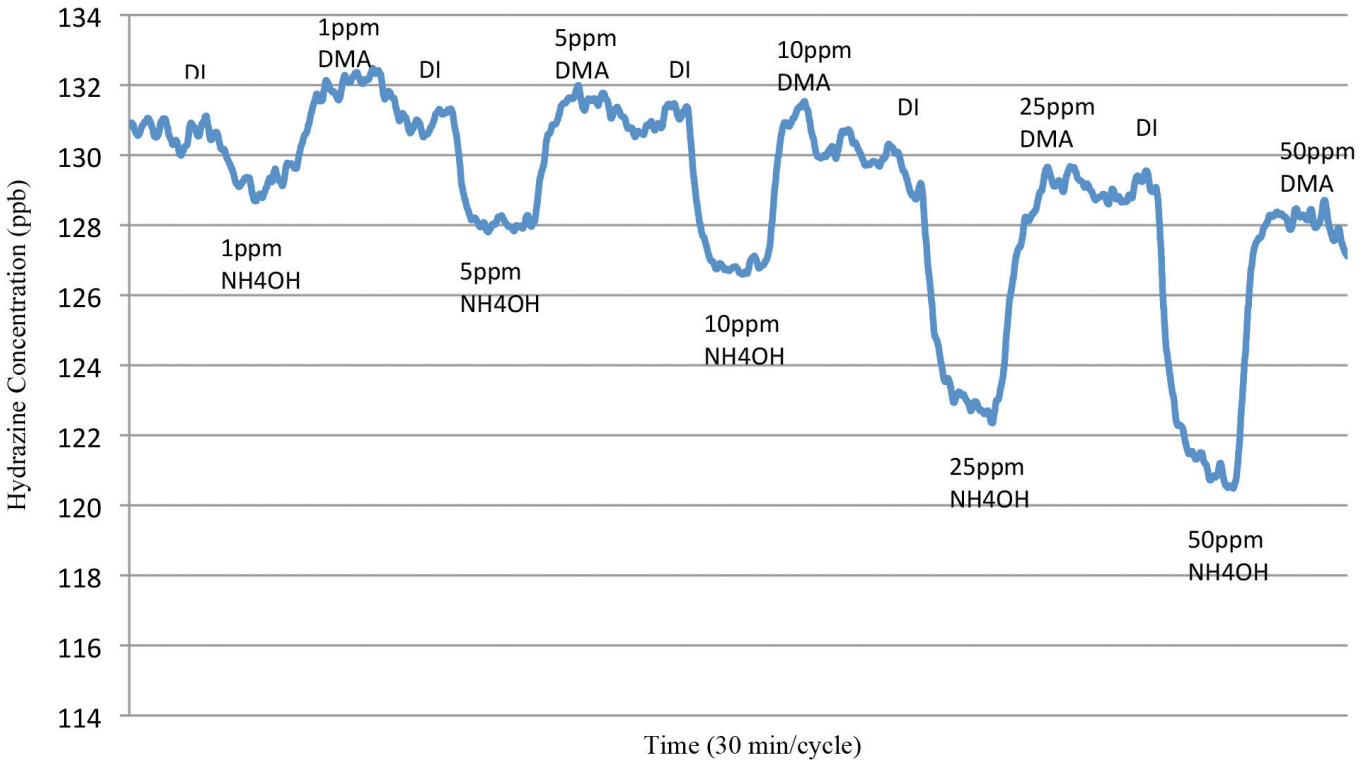
p-Dimethylaminobenzaldehyde *Hydrazine* *Yellow Azine Complex*

HYDRAZINE CALIBRATION IN POWER APPLICATIONS

Steam-cycle pH is frequently controlled via dosing of ammonia and volatile amines. The high pH created by these amines provides protection against corrosion. Treatment programs generally maintain a concentration of amines in the low µg/L range. At these concentrations, the matrix can effect the 9586 sc hydrazine measurement. This effect is illustrated in the chart in Figure 3.

Figure 3 was generated by feeding a constant concentration of hydrazine to the analyzer while the matrix was alternated between deionized water (DI) and increasing concentrations of ammonium hydroxide (NH_4OH) and dimethylamine (DMA). Each dosing regime is 30 minutes. Diisopropylamine (DIPA) was used to maintain the cell pH. It is evident that increasing concentrations of NH_4OH depress the hydrazine value, while DMA slightly increases the hydrazine value. The effect is more pronounced at higher concentrations.

Figure 3 –Hydrazine vs. Time

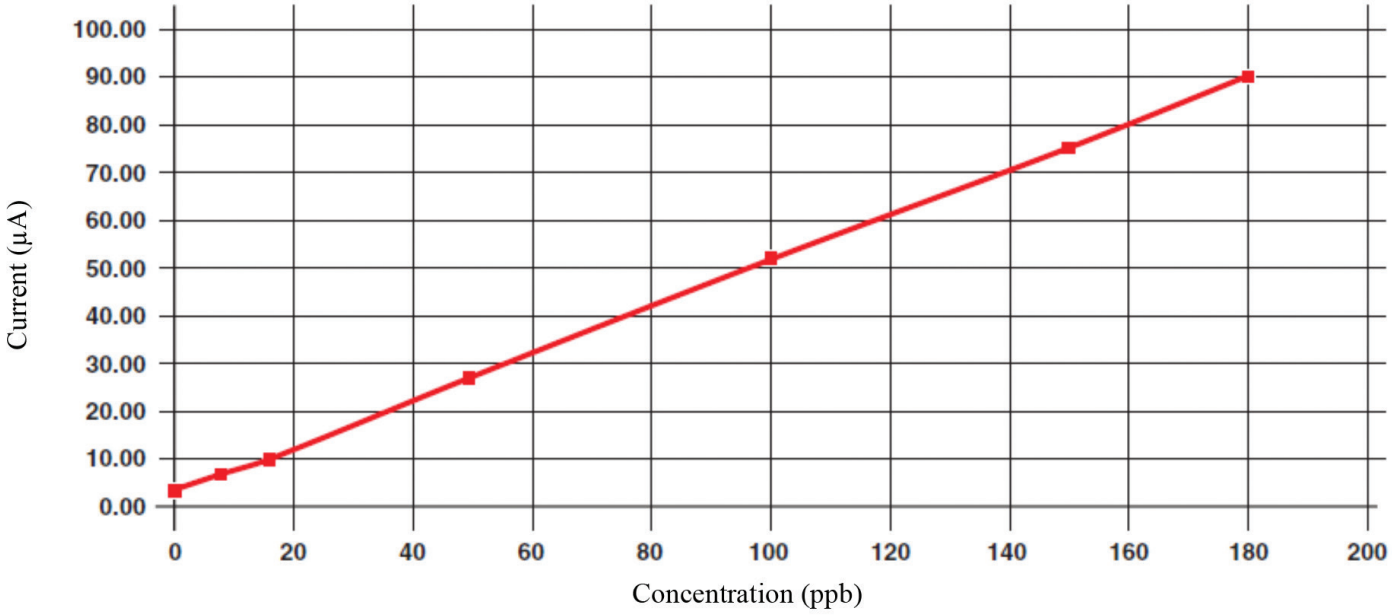


Grab Sample Calibration

In order to obtain accurate measurements, it is necessary to adjust the 9586 sc calibration curve when it is operated in these matrices. Since the 9586 sc cell current is linearly proportional to the hydrazine concentration, Figure 4, the curve can be adjusted with a single point calibration. This single point calibration changes the slope of the calibration curve so that the concentration-current correlation is accurate for the matrix.

A grab sample is taken from the process analyzer during steady-state operation. The grab sample is measured with the laboratory procedure which is not subject to the same matrix effects. The result from the laboratory procedure is compared to the steady-state analyzer result. If the values match, then the analyzer calibration is correct for the matrix. If the values don't match, then the analyzer calibration is adjusted to match the laboratory result.

Figure 4 –Hydrazine Concentration vs. Current



Calibration Procedure

1. Collect a grab sample from the analyzer inlet. The sample should be taken from the inlet as hydrazine is consumed during the amperometric measurement. Only perform the calibration during stable steady-state conditions.
2. Measure the sample hydrazine concentration with Hach Method 8141 and a DR-series spectrophotometer.
3. Enter the 9586 sc calibration menu as described in the user manual.
4. Choose **ACTIVE** output signal option.
5. During stable steady-state operation the output current should be stable. Press **ENTER**.
6. Input the measured grab sample hydrazine concentration. Press **ENTER**.
7. Return the analyzer to measurement mode.



9586 sc Hydrazine Process Analyzer and DR3900 Spectrophotometer

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