Spectrophotometric Determination of Hydroquinone and Phenidone in First Developers

ECR-440B

INTRODUCTION

Hydroquinone and Phenidone (1-phenyl-3-pyrazolidinone) are spectrophotometrically determined simultaneously. They are extracted into water-saturated ethyl acetate. Aliquots of the extract are diluted and measured on a spectrophotometer. The concentration of the two components is determined by solving two three-variable linear regression equations which were derived for fresh samples of known composition.

Prior to being water-saturated, the ethyl acetate should be checked for ultraviolet absorbance. If the absorbance of a 1-cm silica cell filled with ethyl acetate exceeds 0.150 (measured against an air blank at 295 and 315 nm), the ethyl acetate is not suitable for use. Reagent ACS, spectro grade ethyl acetate, is recommended for use in this analysis, but it must be checked and it must meet the indicated absorbance criteria. Any grade ethyl acetate that meets the indicated absorbance criteria may be used.Locally acceptable practices should be used when discarding waste ethyl acetate.

This method requires handling potentially hazardous chemicals. Consult the Material Safety Data Sheet for each chemical before use. MSDS’s are available from your chemical supplier.

RELIABILITY

Five first developer laboratory standard mixes were prepared containing the following combinations of hydroquinone and Phenidone.

<table>
<thead>
<tr>
<th>Hydroquinone, g/L</th>
<th>Phenidone, g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.00</td>
<td>0.200</td>
</tr>
<tr>
<td>3.00</td>
<td>0.200</td>
</tr>
<tr>
<td>5.00</td>
<td>0.400</td>
</tr>
<tr>
<td>7.50</td>
<td>0.200</td>
</tr>
<tr>
<td>7.50</td>
<td>0.600</td>
</tr>
</tbody>
</table>

They were analyzed by seven analysts in two laboratories for a total of 35 data points for each component. The 95 percent confidence limits for an individual determination of a fresh tank or replenisher sample are:

± 0.12 g/L Hydroquinone and ± 0.02 g/L Phenidone

SPECIAL APPARATUS

- Spectrophotometer with UV lamp
- 1-cm Silica Cell

Note: Use pipets and volumetric glassware meeting the “Class A” definition by the National Institute of Standards and Technology (NIST).

REAGENTS

Use ACS Reagent Grade reagents unless specified otherwise.
- Glacial Acetic Acid, CH₃COOH, Reagent ACS
- Ethyl Acetate, Reagent ACS, Spectro-grade, water saturated
- 1.0 N Sulfuric Acid, H₂SO₄

Note: Prior to being water-saturated, the ethyl acetate should be checked for ultraviolet absorbances. If the absorbance of a 1-cm silica cell filled with ethyl acetate exceeds 0.150 when measured against an air blank at 295 and 315 nm, it is not suitable for use.
PROCEDURE

Extraction
1. Pipet, wipe before leveling, 25.0 mL of sample into a 250-mL separatory funnel.
2. Add, from a tip-up pipet, 4 mL of glacial acetic acid to the funnel.
3. Swirl the unstoppered funnel to allow the rapidly forming gases to escape.
4. Pipet (wipe) 50.0 mL of water-saturated ethyl acetate into the separatory funnel; stopper and shake the funnel for a few seconds, then vent through the stopper, continue to shake vigorously, venting occasionally, for 30 seconds.
5. Allow the phases to separate; do not remove the stopper.

Acidic Dilution
1. Add approximately 200 mL of distilled water to a 250-mL volumetric flask and add, by tip-up pipet, 25 mL of 1.0 N sulfuric acid to the flask.
2. Add approximately 20 mL of distilled water to a 50-mL volumetric flask and add, by tip-up pipet, 5 mL of 1.0 N sulfuric acid to the flask.
3. Remove the stopper from the separatory funnel and pipet (wipe) 2.00 mL of the upper (ethyl acetate) layer into both the 250-mL and 50-mL volumetric flasks.
4. Swirl both volumetric flasks until the ethyl acetate is completely dissolved.
5. Dilute both volumetric flasks to volume with distilled water; stopper the flasks and mix the contents by inverting 6 to 10 times.

Absorbance Measurements
1. Rinse and fill a clean 1-cm silica cell with the solution in the 250-mL volumetric flask. Measure the absorbance of the solution in the cell versus air on a spectrophotometer at 288 nm; record as $A_{288}$.
2. Discard the solution in the 1-cm silica cell and rinse with at least 6 portions of the solution in the 50-mL volumetric flask, and then fill the cell. Measure the absorbance of the solution in the cell versus air on a spectrophotometer at 250 nm; record as $A_{250}$.

Calculations

\[
\text{Hydroquinone, } \text{g/L} = 12.01(A_{288}) - 0.117(A_{250}) - 0.59
\]

\[
\text{Phenidone, } \text{g/L} = 1.35(A_{250}) - 0.35(A_{288}) - 0.091
\]