INTRODUCTION
Ethyl acetate is used to extract the developing agent and other interfering constituents from the sample at mix pH. Carbon dioxide is evolved when acetic acid is added to lower the pH and must be completely removed before the extraction of EASTMAN Anti-fog No. 9 (AF No. 9). AF No. 9 is then extracted from the aqueous mixture with a measured amount of 4-methyl-2-pentanone. The 4-methyl-2-pentanone solution is dried over sodium sulfate. Sodium methoxide solution is pipetted into a volumetric flask, which is then filled to volume with the 4-methyl-2-pentanone solution. In the presence of sodium methoxide, AF No. 9 and 4-methyl-2-pentanone react slowly to form an unstable dye. The absorbance at 550 nm is read after the optimum reaction time of 8 minutes. A glass-stoppered silica cell is used to prevent evaporation.

Note: Water destroys the dye. The volumetric flask must be dry.

It is necessary to ensure the complete removal of carbon dioxide evolved when acetic acid is added. Any remaining carbon dioxide will be extracted by the 4-methyl-2-pentanone and will react with the sodium methoxide to give a precipitate. A small precipitate, which dissolves with mixing, will not affect the analysis, but any permanent precipitate will give excessively high answers.

The reaction is sensitive to temperature. If the laboratory temperature is below 21°C or above 27°C, it is advisable to place the volumetric flask of dye in a 27°C constant-temperature bath for most of the 8-minute reaction time.

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
Four developer mixes containing 0.10, 0.15, 0.20 and 0.25 g/L AF No. 9, respectively were analyzed by four analysts. Based on 26 data points, the 95 percent confidence limits for an individual determination are ±0.02 g/L AF No. 9. Recalibrate this method if AF No. 9 concentrations exceed 0.25 g/L.

SPECIAL APPARATUS
- Constant-temperature bath (optional)
- Spectrophotometer with a tungsten lamp
- 1-cm Silica Cell (glass-stoppered)

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

REAGENTS
Use reagents that are ACS Reagent Grade unless specified otherwise.
- 10 g/L Cetyltrimethylammonium Bromide (CTAB)
- Ethyl Acetate, water-saturated
- 2N Acetic Acid, CH₃COOH
- 4-Methyl-2-pentanone
- Sodium Sulfate, Na₂SO₄
- Sodium Methoxide in Methanol, 6 percent Solution or 59 g/L
- Methanol, Spectro-Reagent Grade

PROCEDURE
Extraction of Interferences
1. Pipet (wipe the pipet before leveling) 50.0 mL of sample into a 250-mL separatory funnel No. 1.
2. Add, from a tip-up pipet, 1 mL of 10 g/L CTAB.
3. Add, from a tip-up pipet, 50 mL of ethyl acetate, water-saturated.
4. Stopper and shake the funnel for a few seconds, then vent through the stopper. Continue to shake vigorously for 30 seconds, venting occasionally.
5. Allow the layers to separate; swirl the funnel and drain the lower (aqueous) layer into a 250-mL separatory funnel No. 2.

Note: The ethyl acetate layer remaining in separatory funnel No. 1 may now be discarded. Use locally acceptable practices for disposal of waste ethyl acetate.

7. Allow the layers to separate, swirl the funnel, and drain the lower (aqueous) layer into a clean 250-mL separatory funnel No. 3.
Extraction of AF No. 9

1. Add, from a tip-up pipet, 20 mL of 2 N acetic acid to separatory funnel No. 3.
2. Stopper the funnel and begin mixing gently; vent through the stopper, very frequently until gas evolution ceases.
3. When gas evolution stops, remove the stopper and flush the remaining carbon dioxide out of the separatory funnel with a gentle stream of air or nitrogen. (Do not bubble through the solution in the separatory funnel.)
4. Pipet (wipe) 40.0 mL of 4-methyl-2-pentanone into the separatory funnel.
5. Stopper and shake the funnel for 30 seconds.
6. Allow the layers to separate; swirl the funnel, then discard the lower (aqueous) layer.

Drying the 4-methyl-2-pentanone

1. Weigh 25 grams of anhydrous sodium sulfate to the nearest gram.
2. Drain and discard about 1 mL of the 4-methyl-2-pentanone to flush the water out of the stem of the separatory funnel No. 3.
3. Drain the 4-methyl-2-pentanone into a 150-mL beaker containing a magnetic stir bar.
4. Place the beaker on a magnetic mixer and begin stirring without splashing.
5. Add, while stirring, the 25 grams of sodium sulfate; continue stirring for 1 minute.

Dye Formation

Note: Water destroys the dye. The volumetric flask must be dry.

1. Pipet (wipe) 25.0 mL of sodium methoxide in methanol reagent into a clean dry 50-mL volumetric flask.
2. Fill the flask to volume with the 4-methyl-2-pentanone containing AF No. 9.
   Note: If necessary stir the sodium sulfate with a glass stirring rod to free more of the 4-methyl-2-pentanone.
3. Immediately start a timer for 8 minutes; stopper and invert the flask to mix.
   a. If the solution is even slightly turbid after mixing, the results will be high. See Introduction.
   b. If the laboratory temperature is below 21°C, place the volumetric flask in a 27°C constant-temperature bath for most of the 8-minute reaction time. For more information, see the INTRODUCTION.

Absorbance Measurement

1. Rinse, at least twice, a clean glass-stoppered 1-cm silica cell with methanol.
2. With the contents of the volumetric flask, rinse twice; then fill the cell; stopper the cell.
3. When the alarm sounds, measure the absorbance at 550 nm against air on a spectrophotometer (A_{550}).
   Note:
   a. During the 8-minute wait, the sample should not be warmed (or chilled) by unnecessary handling or by storing in a warm (or cold) place.
   b. The cell should not be placed in the spectrophotometer for more than a minute before reading.

Calculation:

EASTMAN Anti-Fog No. 9, g/L = 0.547(A_{550}) + 0.06