

Ethanolamines/Glycol Ethers

Tier 3 Procedure

Section Number: 612
Issue Number: 1.5
Original Date: 12-17-91 **Author:** P.L. Bourgeois
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Title: Percent Water Determination Of Glycol Ethers via Karl Fisher Method
References: Glossary Of Equipment Identification (Text # 114)

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612. Percent Water Determination Of Glycol Ethers via Karl Fisher Method

1.0 Objective:

1.1 To provide instructions to all operators for consistency and accuracy in determining percent water.

2.0 Special Notations:

2.1 Karl Fisher reagent is a mixture of iodine, sulphur dioxide and methanol. Upon addition of this reagent to water, iodine is reduced by sulphur dioxide. The brown color of the reagent changes to yellow as long as water is available to permit the reaction to proceed. Handle karl fisher with care: it has a noxious odor and is corrosive.

3.0 Procedure:

3.1 Before each sample round, empty the titration vessel of the existing solution.

3.2 Flush the vessel with methanol. Pump methanol from the pipette provided.

3.3 Close the stopcock and add 45 ml of methanol (one pump on the methanol pipette) and 0.5 ml of monoethanolamine acetic acid mixture (one pump on the small pipette).

3.4 Before titrating, be sure to zero the digital counter and be sure the buret is full.

3.5 Press start to titrate karl fisher reagent with the methanol and mea/acetic acid mixture. Solution is dry when the red light comes on.

3.6 Before adding sample, check to be sure the red light is on and the meter is reading 50% or above. If below 50%, press the start button to ensure a dry solution.

3.7 Inject 5cc of sample into the titration vessel with a plastic syringe. Be careful not to touch the solution with the syringe or to splash any sample on the vessel walls.

3.8 Zero the counter then press start to begin titration. Titration is complete when the solution is once again dry. This is indicated by the red light.

3.9 Calculate the percent water as follows:

3.9.1 Amount karl fisher x factor = % water

3.10 The factor is posted on the relief board at operators desk.

4.0 Revisions:

Revision No. 1	02-19-93	Changed 612.3.10 (location)
Revision No. 2	12-21-94	Yearly review
Revision No. 3	02-24-95	January/Yearly review
Revision No. 4	12-29-95	Yearly Review
Revision No. 5	02-12-96	Changed Title