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DETERMINATION OF EQUIVALENT WEIGHT OF AMINES

Walter S. Selig

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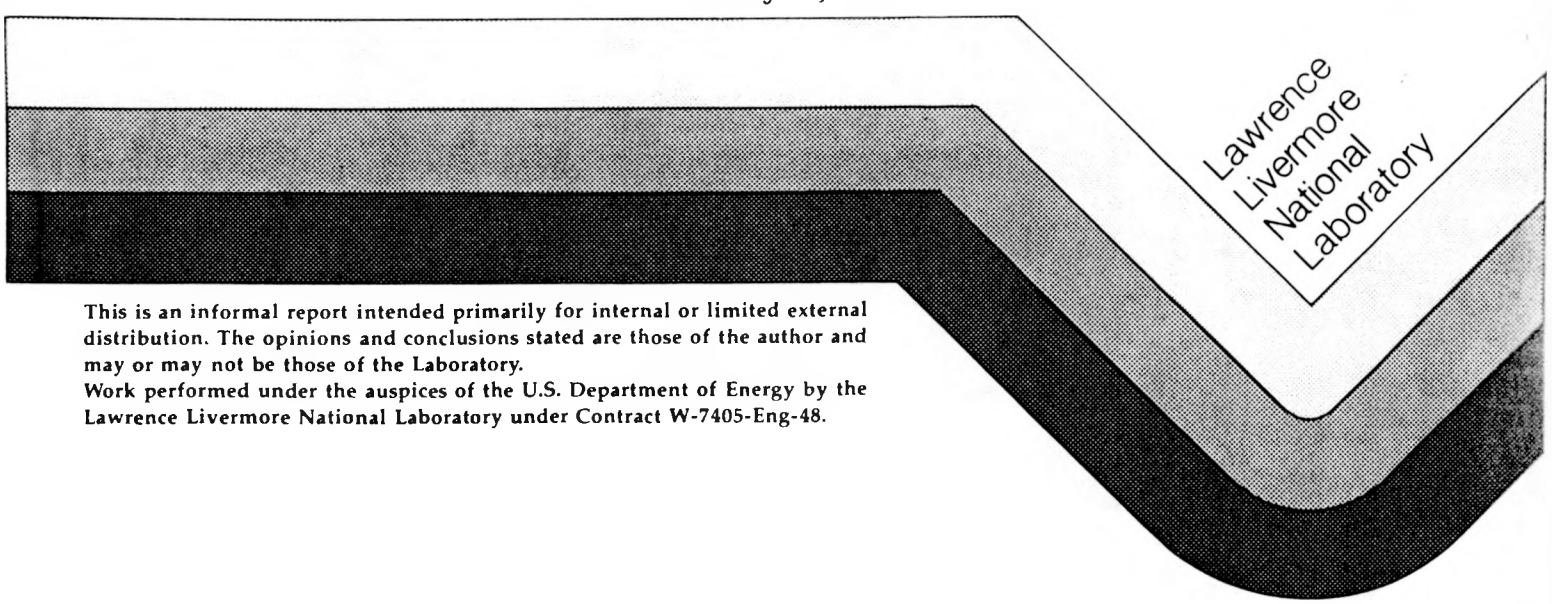

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DETERMINATION OF EQUIVALENT WEIGHT OF AMINES

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January 8, 1987

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DETERMINATION OF EQUIVALENT WEIGHT OF AMINES*

PRINCIPLE:

This procedure is based on an acid-base reaction performed in glacial acetic acid. The sum of primary, secondary, and tertiary amines are determined by titration with standard perchloric acid in glacial acetic acid.

SAFETY PRECAUTIONS:

Toxic organic solvents are used in this procedure. They should be used in a well-ventilated hood and disposed of in a special container for organic solvents. Do not discard in the sink!

REAGENTS:

Standard 0.05 N $HClO_4$ in glacial acetic acid. Mix 4.25 mL of 72% $HClO_4$ with 300 mL of glacial acetic acid and add 20 mL of acetic anhydride. Dilute to 1 liter with glacial acid and allow to stand overnight.
1,3-diphenylguanidine, reagent grade.
Glacial acetic acid.

EQUIPMENT:

Glass indicator electrode.
Ceramic fiber-junction calomel reference electrode, with a salt bridge of saturated tetramethylammonium chloride in methanol.
Automatic titrator with 10-mL buret.

STANDARDIZATION OF TITRANT:

1. Weigh approximately 250 mg of 1,3-diphenylguanidine to the nearest 0.1 mg into a 50-mL volumetric flask and dissolve in glacial acetic acid. Dilute to volume with glacial acetic acid.
2. Transfer 10.00-mL aliquots into 50-mL beakers containing stirring bars.
3. Dilute to 25 mL with acetic acid.
4. Titrate potentiometrically against 0.05 N $HClO_4$ in glacial acetic acid using the automatic titrator and the glass/modified calomel sensing couple.
5. Perform the standardization in at least triplicate and calculate the $HClO_4$ titrant normality according to

$$N = (\text{mmol of 1,3-diphenylguanidine})/(\text{mL of titrant})$$

The molecular weight of 1,3-diphenylguanidine is 211.269.

PROCEDURE:

1. If the approximate equivalent weight of the amine is known, weigh about 0.25 times its amount in milligrams to the nearest 0.1 mg into a 50-mL beaker containing a stirring bar.
2. If the equivalent weight is unknown, a preliminary "stab-in-the-dark" determination is required.
3. Dissolve the sample in 20 - 25 mL of glacial acetic acid.

*Work completed March 1986

4. Titrate potentiometrically against 0.05 N $HClO_4$ in glacial acetic acid as outlined in "Standardization of Titrant.
5. Rinse out beakers with acetone.
6. Rinse the electrodes and buret tip with glacial acetic acid.

CALCULATIONS:

Amine equivalent weight = (sample, mg)/(mmol of titration)

REFERENCES:

L. S. Siggia and J. G. Hanna, "Quantitative Organic Analysis via Functional Groups," Fourth Edition, Wiley-Interscience, New York, 1979, pp. 545.