

DETERMINATION OF EQUIVALENT  
WEIGHT OF AMINES

Walter S. Selig

January 8, 1987

Lawrence  
Livermore  
National  
Laboratory

This is an informal report intended primarily for internal or limited external distribution. The opinions and conclusions stated are those of the author and may or may not be those of the Laboratory.

Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

---

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

#### **DISCLAIMER**

**This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial products, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.**

Printed in the United States of America

Available from

National Technical Information Service

U.S. Department of Commerce

5285 Port Royal Road

Springfield, VA 22161

<b><u>Price Code</u></b>	<b><u>Page Range</u></b>
A01	Microfiche
<b><u>Papercopy Prices</u></b>	
A02	001 - 050
A03	051 - 100
A04	101 - 200
A05	201 - 300
A06	301 - 400
A07	401 - 500
A08	501 - 600
A09	601

UCID--20962

DE87 005203

DETERMINATION OF EQUIVALENT WEIGHT OF AMINES

Walter S. Selig  
Condensed Matter and Analytical Sciences Division  
Chemistry and Materials Science Department

January 8, 1987

1598L

MASTER

*EB*  
DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

## 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  $\text{HClO}_4$  in glacial acetic acid. Mix 4.25 mL of 72%  $\text{HClO}_4$  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  $\text{HClO}_4$  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  $\text{HClO}_4$  titrant normality according to

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

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.

4. Titrate potentiometrically against 0.05 N  $\text{HClO}_4$  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.