Organic Application Note

Nitrogen in Oil/Amines

Accessories 502-186 Tin Foil Cups, 501-614 Spatula, Disposable Eyedroppers, 501-439 Paraffin Oil, 501-426 COM-AID

Sample Weight 0.1 g

Calibration Standard 502-092 EDTA, Glycine Solution (see reverse side for detailed instructions), or other suitable standard

Furnace Temperature 950°C

Flow Profile All High

Atmospheric Blank (N) 0.04 (verified by analyzing paraffin oil with COM-AID)

Crucible Changing Interval ~100 analyses using 614-961-110 Porous Crucible

Analysis Time ~170 seconds

Procedure

1. Prepare the instrument by following the procedure as outlined in the operator's instruction manual (i.e. check gas supplies, perform any required maintenance, perform leak checks, etc.).
2. Analyze blanks (gas) until a plateau is reached. Analyze three to five additional blanks and set blank using these data.
3. Analyze five EDTA standards at 0.2 g and drift correct (if using the PC option). *NOTE: Each method on PC requires prior calibration with multiple weights of EDTA (0.035 to 0.4 g). If PC is not installed, analyze five EDTA standards and calibrate using the DSP screen menu.*
4. Weigh ~0.1 g oil into a 502-186 Tin Foil Cup containing ~0.1 g 601-427 COM-AID, add 0.4 to 0.5 g additional COM-AID, seal tin foil to avoid trapping air or losing sample, and analyze.
5. Analyze a standard at end of set to verify calibration.

Typical Results

Sample	*Weight (g)*		*% Nitrogen*
Amine #1	0.0982		1.00
	0.1074		0.98
	0.1074		1.00
	Average	**=**	**0.99**
	Std. Dev.	**=**	**0.014**
Amine #2	0.0901		0.143
	0.0951		0.131
	0.0876		0.141
	Average	**=**	**0.138**
	Std. Dev.	**=**	**0.007**

Sample	*Weight (g)*		*% Nitrogen*
Amine #3	0.1072		4.80
	0.0931		4.82
	0.1030		4.84
	0.1054		4.83
	0.1079		4.79
	0.1107		4.79
	Average	**=**	**4.81**
	Std. Dev.	**=**	**0.021**
Lube Oil	0.1041		0.101
	0.1059		0.111
	0.1050		0.100
	0.1054		0.100
	Average	**=**	**0.103**
	Std. Dev.	**=**	**0.005**

FP-528

GLYCINE SOLUTION PREPARATION

1. The following formula can be used to make a specific concentration:

$$G = \frac{C}{(0.99^{\dagger} \bullet 0.18658)}$$

where: C = desired nitrogen concentration as percent
G = grams of glycine powder

Example for 1% solution:

$$G = \frac{1}{(0.99^{\dagger} \bullet 0.18658)} = 5.414$$

NOTE: A quick reference chart, shown below, shows the grams of glycine powder needed to reach given concentrations.

2. Place a flask on the balance and tare. The flask should be large enough to hold 100 ml (where 100 g = 100 ml).
3. Add the amount of glycine calculated in step 1 and record the mass.
4. Add distilled water until the total mass equals 100 g, then record the mass (W).
5. Seal the flask and mix the contents.
6. To figure the exact concentration:

$$\% \text{ Nitrogen} = \frac{G\,(18.658 \bullet 0.99^{\dagger})}{W}$$

where: G = mass in grams of glycine recorded in step 3
W = mass in grams of water and glycine powder recorded in step 4

7. If the distilled water is not pure, determining the nitrogen concentration may be necessary.
   a. Analyze five samples of distilled water.
   b. Average the nitrogen content of the five samples (A).
   c. Add this average to % nitrogen calculated for the calibration solution.

Example: To make a calibration solution of approximately 0.3% nitrogen:
where: G = 1.672 g
W = 99.824 g
A = 0.004%

$$\frac{1.672(18.654)}{(99.824)} + 0.004 = 0.316\%\ \text{N}$$

QUICK REFERENCE CONCENTRATION TABLE

Nitrogen Concentration	Grams of Glycine†
0.10%	0.541
0.30%	1.624
0.50%	2.707
0.75%	4.060
1.00%	5.414

†*Assuming 99.0% purity of glycine powder.*

LECO Corporation
3000 Lakeview Ave. • St. Joseph, MI 49085-2396
Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • *ISO-9001 No. FM 24045*