

# Appendix F

## Thiolamine Analytical

**QUANTITATIVE ANALYSIS OF 2-DIISOPROPYLAMINOETHANETHIOL**  
**USING GAS CHROMATOGRAPHY FLAME IONIZATION DETECTION**  
**REGIONAL ANALYTICAL SERVICES**  
**JACKSON LABORATORY, RM 2202**

**DESCRIPTION**

The following method is used for the quantitative analysis 2-diisopropylaminoethanethiol of received, post-oxidation, and precipitated NCH samples.

**CALIBRATION STANDARD PREPARATION**

A 1000-ppm stock solution is prepared by accurately weighing 25 mg of 98% pure 2-diisopropylamino ethanethiol hydrochloride salt standard into a 50-mL glass vial. The amount of 2-diisopropylamino ethanethiol is calculated by the following:

$$\text{ethanethiol (mg)} = \text{weight (mg)} \times 98\% (\text{purity}) \times 81.56\% (\text{ethanethiol in ethanethiol HCl})$$

Accurately pipet the same number of mL of pH-adjusted water (pH ~6.5) as mg of ethanethiol as calculated above into the vial. A series of calibration standards are prepared according to the following:

vial #	vol. of stock soln. (μL)	vol. of pH-adj. water (μL)	conc. ethanethiol (ppm)
1	0	1000	0
2	5	995	5
3	25	975	25
4	100	900	100
5	500	500	500
6	1000	0	1000

A calibration curve is to be run before analysis of samples.

**SAMPLE PREPARATION**

16% NCH As Received

Pipet 5 μL of 16% NCH into a GC vial and dilute to 1 mL with pH-adjusted water.

16% NCH After Oxidation and After Precipitation

The oxidized and precipitated samples are analyzed neat.

All samples are analyzed in duplicate.

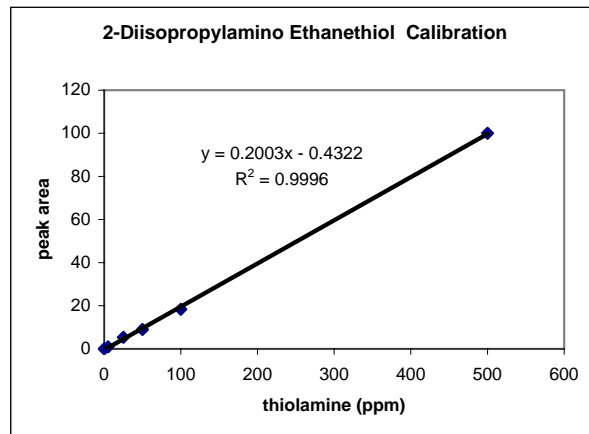
## INSTRUMENT PARAMETERS

The samples are quantified using a GC-FID instrument with the following operating conditions:

Detector	Flame ionization Hydrogen: 40.0 mL/min Air: 450.0 mL/min
Split Ratio	50:1
Injection Temperature	150°C
Detector Temperature	325°C
Initial Column Temperature	40°C
Pre-Program Hold	0 min
Temperature Rise	5°C/min to 190°C
Post-Program Hold	0 min
Working Column	30.0m x 0.25mm I.D. fused silica coated with 1.00µm film thickness, 5% phenyl methyl siloxane

## RESULTS

The calibration standards yielded the following curve:



Using the above calibration curve, concentrations of the following samples were determined:

As Received	42A	3.09%
As Received	42B	7.24%
Oxidized	58	<5 ppm
Precipitated	58P	<5 ppm
As Received	42C	3.43%
Oxidized	60	<5 ppm
Precipitated	60P	<5 ppm
As Received	42D	3.26%
As Received	42E	2.97%
Oxidized	59	<5 ppm
Precipitated	59P	<5 ppm