

Project No. 8627

Principle

ALANAP, also commonly known as the sodium salt of naptalam (2-[(1-naphthylamino) carbonyl] benzoic acid, sodium salt), CAS No. 132-66-1, is hydrolyzed in a boiling sodium hydroxide solution and 1-naphthylamine (1-NA) is recovered by steam distillation. The distillate is injected into the HPLC and analyzed for 1-NA using an electrochemical detector fitted with a glassy carbon electrode.

Method

1. Reagents

Water: Distilled and HPLC-grade

NaOH: 50% solution

Titanous trichloride: 20% solution, technical

Mossy zinc: Technical

Silicone Surfactant: Antifoam B[®], J.T. Baker Chemical Co.
(or equivalent)

Phosphoric Acid: 85% solution, reagent-grade

1-naphthylamine, 99.4%, AC-917-58

ALANAP, 89.4%, AC-921-54

2. Apparatus

One-liter, flat-bottomed boiling flasks

Distillation heads with water condensers suitable for distillation of water under an atmospheric pressure and receivers of 250 mL capacity, such as graduated Erlenmeyer flasks.

Magnetic stirrer, hot-plates

Magnetic stir bars

High pressure liquid chromatograph with an electrochemical detector

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Method (Continued)

3. HPLC Operating Conditions

Pump: Model M-6000 Solvent Delivery System, Waters Associates
(or equivalent)

Injector: Model 7120, Rheodyne, 100 μ L loop (or equivalent)

Recorder: Strip chart type, Linear Instruments
Chart speed 20 cm/hour

Detector: Electrochemical detector model EC/230, IBM (or
equivalent)

Electrode: Glassy carbon working electrode set at +850 mV vs.
Ag/Ag Cl reference electrode and 5 to 10 μ A (depending
on the sensitivity required).

Column: PRP-1, GCS, Semi-preparative, 10 μ m, 305 x 7 mm,
Hamilton, protected by a 0.45 μ m inlet filter.

Mobile Phase: 30/70% (v/v) acetonitrile/0.15 M phosphoric acid
filtered through a 0.45 μ m membrane filter and
degassed.

Injection Volume: 100 μ L

Flow Rate: 1.5 mL/minute

1-NA Retention Time: 10 minutes

4. Sample Preparation

Soil samples are removed from the freezer and allowed to thaw for
one to two hours. The whole sample is placed into an aluminum foil pan
and allowed to dry at ambient temperature overnight. To insure
uniformity, the whole dried sample is ground to a fine powder and
thoroughly mixed.

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Method (Continued)

5. Hydrolysis and Distillation

A sample of the dry homogenized soil, approximately 50 g, is accurately weighed on a calibrated balance into a 1-L, flat-bottomed flask containing a magnetic stir bar. Distilled water, 100 mL, is added and the flask is placed on the magnetic stirrer, hot-plate. While the mixture is stirring, the following are added:

Massy zinc:	about 1 g
Antifcam B®:	0.5 to 1.0 mL
50% NaOH Solution:	250 mL
20% Titancus Trichloride:	5 mL

The boiling flask is fitted with the distillation apparatus and the distillate is collected in a 250 mL Erlenmeyer flask until 200 mL is distilled. It takes about two hours to complete the distillation.

6. Determination

The analyte, 100 µL, is injected into the HPLC. The 1-NA peak height is measured and the concentration, µg/mL 1-NA, is obtained from a standard calibration plot, or preferably, using a linear regression analysis.

Calculation:

$$\text{ppm ALANAP in the sample} = \frac{k \times C}{W \times R}$$

where,

$$k = 437.6 = \frac{\text{analyte volume, mL}}{\text{mol. wt., 1-NA}} \times \text{mol. wt., ALANAP}$$

$$C = \text{1-NA, } \mu\text{g/mL}$$

$$W = \text{Sample weight, g}$$

$$R = \text{1-NA recovery} = \frac{\text{parts recovered}}{\text{parts added}}$$

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Method (Continued)

7. Standard Solution for a Calibration Plot

A stock solution is prepared by accurately weighing on a calibrated analytical balance about 50 to 60 mg 1-NA standard into a 100 mL volumetric flask. The flask is filled with acetonitrile to the mark and sonicated briefly. A series of dilutions with IC-water are made to obtain standard solutions of approximately 0.15, 0.10, 0.05 and 0.025 $\mu\text{g/mL}$ concentration. One hundred μL of each solution is injected into the HPLC to obtain a calibration plot. 1-NA standard solutions are stable for at least a week when kept in the refrigerator.

8. Standard Solution for Recovery Determinations

A stock solution is prepared by accurately weighing on a calibrated analytical balance approximately 800 mg of ALANAP standard into a 100 mL volumetric flask. The flask is filled with water to the mark and sonicated briefly. The stock solution is serially diluted with IC-water 1/10 twice to give a solution of about 8 $\mu\text{g/mL}$ ALANAP.

9. Linearity of Response and Limit of Detection

A linear relationship between the peak height and 1-NA concentration was observed over the range from about 0.15 to 0.025 $\mu\text{g/mL}$, which is the expected range of concentrations in the analyte from the soil analysis. The limit of detection (signal to background ratio of 3) was found to be 0.1 ppm ALANAP in a sample.

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