

Total Kjeldahl Nitrogen (TKN)

FIALab standard method for the Total Kjeldahl Nitrogen (TKN) Assay using the FIALab-2500/2600/2700 system.

Assay	Typical Throughput	Concentration Range	Notes
Ammonia (Mid)	120 samples/hour	3.0 to 300 mg (N)/L H_3	10 cm flow cell

Principle:

Samples are first digested in a block digester with a sulfuric acid prior to FIA assay, converting Kjeldahl nitrogen to the ammonium cation, and measured as ammonia using the salicylate method.

The *salicylate* method is a variation of the *Berthelot-Phenate* method but does not require the use and disposal of toxic phenol. The *salicylate* method involves a three-step reaction sequence. The first reaction step involves the conversion of ammonia to monochloroamine by the addition of chlorine. The monochloroamine then reacts with salicylate to form 5-aminosalicylate. Finally, the 5-aminosalicylate is oxidized in the presence of sodium nitroferricyanide to form a blue-green colored dye that absorbs light at 650 nm.

Comments:

Water Bath Heater should be turned on to 60 C. The flow rate of the pump should be set to 45. The FIA LOV connections B should be bridged by short green tubing. Recommended wavelengths: 650 nm primary and 525 nm reference.

For most TKN assays make the sample loop from six inches of green tubing (.03" ID). **For Macro-TKNs, dilute sample 5 fold with De-ionized (DI) water.** Use all PEEK fittings in LOV manifold (normal fittings may be damaged by high NaOH content).

Low Concentration Assays: For low concentrations (below 10.0 ppm) use a 5 or 10 cm flow cell. Increase sample loop to 12" of green (.03 ID) tubing. Also, carrier matrix matching becomes critical. The carrier should be identical with the standard 0 (blank).

Interferences:

Sulfide will intensify the blue-green color and should be removed from the sample if possible. Hydrazine and glycine like sulfide can also intensify the color. High sample turbidity will give significant erroneous results and may need to be pre-filtered or run utilizing a different ammonia method. Iron will also interfere with the analysis, but can be eliminated by doping the calibrations samples with the same level of iron present in the sample. To a lesser extent the following ions may also interfere: sulfates (>300 ppm), phosphates (>100 ppm), nitrates (>100 ppm), nitrites (>12 ppm), calcium (>1000 ppm) and magnesium (>6,000 ppm).

Reagents:

Carrier: For high concentrations De-ionized (DI) water will work (use 1 cm FC). For low concentrations, the carrier matrix should be identical with the samples.

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Reagent 1: Hypochlorite Solution.

10-mL of a 6% Sodium hypochlorite solution (*common household bleach*) 50-grams NaOH (s) (**note 10 fold increase of NaOH over water Ammonia assays**)
Balance Degassed DI water (1 L).

Place a 10ml solution of bleach into a 1-liter volumetric flask and fill with 700-mL of degassed DI water. Dissolve all other chemicals and fill flask to the 1-liter mark.

Reagent 2: Salicylate/Catalyst Solution.

100-grams Sodium Salicylate [160.11 FW] 0.40-grams Sodium Nitroferricyanide (III) dihydrate [297.95 FW] *catalyst*
5-grams NaOH (s) Balance Degassed DI water(1 L)

Place the sodium salicylate into a 1-liter volumetric flask and mix with 700-mL of degassed DI water until dissolved. Add the sodium nitroferricyanide and mix until dissolved. Add NaOH to adjust the pH to the ~12.0 range. Add the Brij 35, mix and fill flask to the mark. Transfer solution into a dark, airtight glass bottle for maximum longevity. Prepare fresh weekly due to limited storage life.

Standards:

100ml ICAMU-100 (ammonia standard)
Source: 727-425-7732 – www.exaxol.com