A.I. Scientific

Application Note

Total Kjeldahl Nitrogen and Total Phosphorus in Water and Water Water

Issue: 9

Introduction

This method is adapted for use on AI Scientific's AIM600 Block Digestion System following US EPA Office of Ground Water and Drinking Water methodology.

Scope & Application

This is an acid digestion procedure to prepare ground waters, surface waters, drinking waters and industrial or domestic wastewaters for the subsequent determination of TKN (Total Kjeldahl Nitrogen) and TP (Total Phosphorus) by colorimetric methods. The range for the method is 0.1-20 mg/l as N and P respectively. Samples can be diluted to increase the range.

Note: the digestion procedure may not convert some nitrogen containing compounds sometimes found in industrial waste such amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

Apparatus

DG-U-A013* AIM500 50 place Block Digestion System (includes 50 x 100ml straight glass digestion tubes)

Alternative Digestion Tubes:
*4003006 100ml Digestion Tubes, glass, volumetric (box of 50)

Additional laboratory ware: 25ml measuring cylinder, calibrated pipettes, volumetric flasks, vortex mixer

Reagents/Chemistry required

8032173 PTFE boiling chips, 450g
Kjeltabs MQ (1.5g K_2SO_4 + 0.075g HgO), pack of 1,000
or
Kjeltabs CQ (1.5g K_2SO_4 + 0.15g $CuSO_4.{}_5H_20$), pack of 1,000 (alternative catalyst for mercury free digestion solution)
6N Sulphuric Acid - Dilute 167ml of concentrated sulphuric acid to 1 litre with reagent grade water
Reagent Grade Water

Control Program

STEP to 160°C
HOLD for 60 minutes
RAMP to 380°C at 5°C/min
HOLD for 30 minutes
End

Procedure

1. Shake sample and transfer 25ml of well mixed sample into a clean dry digestion tube.
2. For mercury catalyst: Carefully add 6ml of 6N sulphuric acid and 1 x Kjeltab MQ and mix thoroughly. or For copper catalyst: Carefully add 8ml of 6N sulphuric acid and 1 x Kjeltab CQ and mix thoroughly.
3. Add 4 - 8 PTFE boiling chips to each tube.
4. RUN the control program above on the AIM600 Control Module.
5. At the completion of the digestion, raise the digestion tubes in the tube rack to the elevated position on the cooling stand to facilitate rapid cooling.
6. When cool, dilute to 25ml with reagent grade water.

The sample is now ready for analysis for TKN by semi-automated colorimetric (1) or potentiometric ISE analysis (2) and for TP by semi-automated colorimetric analysis (3).

Safety Notes:

- The AIM600 Digestion Block should be located inside a ventilated acid-resistant fume hood. It is recommended that the AIM600 Program Controller be located outside the fume hood.
- Great care should be taken when handling acids. Always add acid to water unless otherwise directed. Protective clothing should be worn including gloves and face mask. If acids are spilled on the skin, immediately wash with copious amounts of water.
- Neutralize any acid spills with sodium carbonate (Na_2CO_3) or sodium bicarbonate ($NaHCO_3$).
- Digests must be cool before dilution water is added to avoid a violent reaction during which the acid can shoot out of the flask.

References

(1) US EPA Method 351.2, "Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry", EPA/600/R-93/100, Methods for the Determination of Inorganic Substances in Environmental Substances, Revision 2, August 1993
(2) US EPA Method 351.4, Potentiometric Ion Selective Electrode Method for determining Total Kjeldahl Nitrogen, EPA/600/4-79/020, Methods for Chemical Analysis of Water and Wastes, March 1983.
(3) US EPA Method 365.4, Colorimetric, Automated, Block Digestor method for determining Total Phosphorus, EPA/600/4-79/020, Methods for Chemical Analysis of Water and Wastes, March 1983.

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