# The THERMALOX™ ${ }^{\text {T4 }}$ Total-N 

An Elemental Analyser
Manufactured by Analytical Sciences Limited of Overbury, England


Nitrogen in water has become of increasing concern not only to environmentalists but also to manufacturers, particularly in the pharmaceutical and food industries. Excess nitrates and nitrites in water are toxic as well as promoting rapid microbial growth. Mounting pollution from nitrogen based fertilisers has greatly increased the need to monitor wastewater as its effect on our rivers, estuaries and seawater becomes more pronounced,

- Catalytic thermal oxidation. The only way to measure 'real 'samples containing particulate or difficult to oxidize materials
- Lower Detection Limit of 50 ppb
- Upper Detection Limit of 200 ppm
- Selectively measures only Nitrogen
- Analysis time less than two minutes
- Complete recovery of Nitrogen, including suspended solids
- Handles seawater and particulate easily
- Holds up to 88 samples
- Complete washing between samples ensures no carry over
- Totally software driven from a Windows ${ }^{\text {TM }}$ based platform
- Automatic preparation of calibration standards
- The perfect instrument for wastewater, surface, seawater and groundwater applications


## Sample Digestion <br> Kjeldahl decomposition, UV or Thermal Oxidation?

The conventional method for determining organic nitrogen is based on a neutralization titration - the Kjeldahl method. In this method, the sample is decomposed in hot, concentrated sulphuric acid to convert the bound nitrogen to ammonium ions. The resulting solution is then cooled, diluted, and made basic. The liberated ammonia is distilled, collected in an acidic solution, and determined by a neutralization titration. The critical step in any elemental analysis is the decomposition. In the Kjeldahl method this is with sulphuric acid, which oxidizes the carbon and hydrogen in the sample to carbon dioxide and water. What happens to the nitrogen, however, depends on its state of combination in the original sample. For example, amine and amide nitrogens are quantitatively converted to ammonium ion. However, nitro, azo, and azoxy groups may well yield elemental nitrogen or various oxides of nitrogen, all of which are lost from the hot acidic medium. This loss may be avoided by pre-treating the sample with a reducing agent to form products that
behave as amide or amine nitrogen. The decomposition step is frequently the most timeconsuming aspect of a Kjeldahl determination. Some samples can take heating periods in excess of an hour. It should be noted that Total Kjeldahl Nitrogen (TKN) measurements actually include ammonia as well as the purely organic content of a sample.

In some elemental determination methods it is necessary to destroy the organic materials completely. Examples are heavy metal determinations in wastewaters or liquids such as wine and beer. Oxidative decomposition with the addition of hydrogen peroxide followed by UV radiation can be used in these cases. Compared with the Kjeldahl method described alongside, UV decomposition requires only small amounts of acid in addition to hydrogen peroxide without a significant increase in temperature. This keeps the contamination resulting from heavy metal levels in the reagents at a low level.

Highly reactive chemical radicals are produced by UV radiation, and ozone is generated. In the secondary reactions, organic substances that bind to heavy metals are decomposed.

However, UV irradiation does not offer the certainty of complete oxidation, indeed most suspended solids are untouched. The European standard BS EN 1484 of 1997 specifically rules out the use of UV techniques for measurements where difficult to oxidize materials, such as humic compounds or suspended solids, may be present. It should be remembered that most measuring techniques rely on measuring oxides, so if there is any doubt that complete oxidation has taken place the
accuracy of results cannot be regarded with confidence. With thermal oxidation the technique for measuring TN relies on introducing the sample into a high temperature furnace (reactor) in the presence of a catalyst and oxygen. The nitrogen in the sample is converted to nitric oxide, NO.

Thermal oxidation digestion techniques are becoming the standard worldwide for TOC measurements and are now becoming recognized as the way forward for nitrogen measurements as well, as this method ensures complete oxidation at speed and is proving to be the only reliable way to provide economic measurement of large numbers of samples.

## The Measurement

The T4 uses a sensitive chemiluminescent analyzer to detect the NO. Nitric oxide combines with ozone $\left(\mathrm{O}_{3}\right)$ to yield an excited state of NO, which decays and gives off a photon which is detected by the analyzer. The reaction is highly specific and sensitive to NO. During this phase the NO concentration, plotted against time, follows a

## Features

The Thermalox ${ }^{\text {TM }}$ is equipped, as standard, with the following:

- One high temperature reactor for total nitrogen oxidation;
- Peltier Cooler and acids filter
- Precision Electronics Mass Flow Control of Carrier Gas
- Fast response, highly sensitive chemiluminescent
typical curve called a 'peak'. The concentration rises, quickly reaches a maximum level, and then slowly drops back to the initial (base) value. The more NO present in the sample the larger the peak. The peak shape can be observed as it rises on the system's monitor. The 'Trace' version of the T4 uses a hisensitivity vacuum type chemiluminescence detector.
- Option of adding total carbon measurement as well
- Stand-by Mode to limit carrier gas consumption
- RS232C serial interface to enable networking capability
- Nitrogen-free injection method
- TN with options for TOC and TP

The Thermalox ${ }^{\text {TM }}$ Total-N Specification

| Analysis | TN, with options to add TC, TOC and TP |
| :---: | :---: |
| Method | Total substance: Thermal catalytic oxidation at $740^{\circ} \mathrm{C}$. Chemiluminescence Detector |
| Ranges and analyzer | Nitrogen: $50 \mathrm{ppb}-200 \mathrm{mg} / \mathrm{N}$. Auto dilution facility provided for ranges to $20,000 \mathrm{mg} / \mathrm{l}$ |
| Cycle time | Typically 2 minutes per replicate |
| Precision | Standard deviation: $\leq 10 \%$ of full scale for ranges from 100 ppb to 5 ppm ; and $\leq 2 \%$ of full scale for ranges to 200ppm |
| Accuracy | Better than $\pm 10 \%$ below $1 \mathrm{ppm} ; \pm 5 \%$ between 1 and $10 \mathrm{ppm} ; \pm 3 \%$ above 10 ppm |
| Sample matrix | Aqueous samples, including those containing suspended solids and seawater. Consult your dealer for applications with non-aqueous matrices such as acids, caustic solutions or flammable materials. Not all are recommended or suitable. LODs and accuracy will vary dependent on the application matrix. |
| Injection volume | 1 to $500 \mathrm{\mu l}$ |
| Sample injection | Automatically from 88 position vial tray, or manually |
| Carrier gas | Oxygen (grade 5.5 or better) or other special gases depending on sample; flow rate is $\leq 10 \mathrm{lt} / \mathrm{hr}$ |
| Power supply | $230 \mathrm{VAC} \pm 10 \%, 50 \mathrm{~Hz} \pm 1 \%$; or 115 V AC $\pm 10 \%, 60 \mathrm{~Hz} \pm 1 \%$ |
| Ambient temperature | $5-35^{\circ} \mathrm{C}$ |
| Dimension and weight | $525{ }^{\mathrm{W}} \times 410^{\mathrm{D}} \times 420{ }^{\text {H }} \mathrm{mm}$ footprint; 45kg; (all figures exclude autosampler and PC) |
| Complies with | BS EN 1484 of 1997; EMI class Euro 50081/50082; EU requirements for TN measurements |

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