

## **NEW Lachat Dual Digestion methods for Total Nitrogen and Total Phosphorus**

In addition to single parameter digest methods, Lachat Instruments introduces new Dual Digestion methods for the determination of Total Nitrogen and Total Phosphorus in a single Manual Persulfate digest.

These methods utilize an off-line digestion to convert all forms of nitrogen into nitrate and all forms of phosphorus into orthophosphate using an alkaline and acidic persulfate digestion. BOTH TP and TN are measured from the same solution following the digestion.

These methods have several advantages:

- Allows Total Nitrogen and Total Phosphorus to be analyzed from a single digest.
- Samples containing particulates can be digested with these methods.
- A total of 46 samples and standards can be digested simultaneously when using Lachat's BD-46 Block Digester.
- Methods can also be used with an autoclave.

	<b>Total Nitrogen</b>	<b>Total Phosphorus</b>
<b>Lachat Method #</b>	10-107-04-4-B	10-115-01-4-B
<b>Method Range:</b>		
<b>Low Range</b>	0.02 - 5.00 mg N/L	0.005 to 1.0 mg P/L
<b>High Range</b>	1.00 - 40.0 mg N/L	0.25 to 10 mg P/L
<b>Method Detection Limit:</b>		
<b>Low Range</b>	0.006 mg N/L	0.60 µg P/L

To order these methods, or for more information, please contact Lachat Instruments:

[sales@lachatinstruments.com](mailto:sales@lachatinstruments.com)

US: 800-247-7613

International: 970-669-3050

### **Digestion details:**

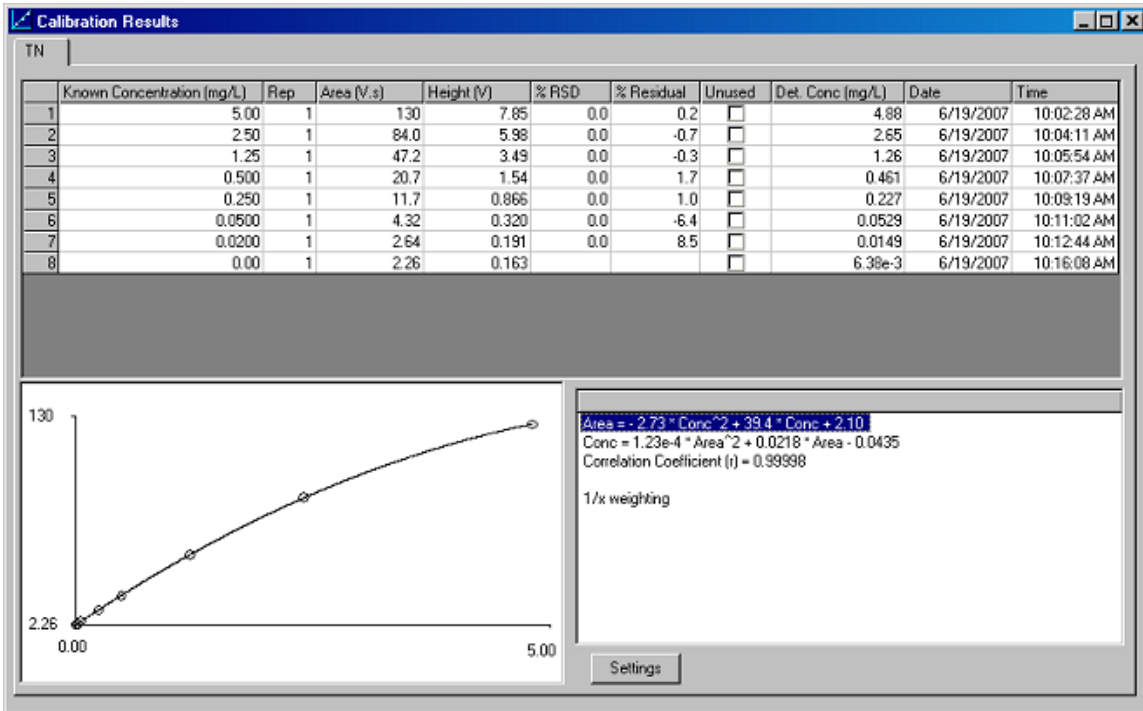
- The samples must be taken through the entire digestion process, even if one of the analytes will not be measured. This is to correctly match the sample matrix to the carrier.
- Procedure:
  - 20 mL of sample are placed into a 25x100 mm screw cap culture tube. 5 mL of an alkaline persulfate reagent are added to this. The tube is sealed and placed into the Lachat BD-46 at 150°C for 15 minutes. The tubes are removed from the block and cooled slightly. Next, 0.5 mL of 11N H<sub>2</sub>SO<sub>4</sub>

is added. The tube is resealed and placed on the block for an additional 30 minutes. The contents of the tube are then analyzed for TN and TP.

- **Note:** Refractory nitrogen compounds, such as nicotinic acid, may require a 30-minute digestion under alkaline conditions.

**Total Nitrogen method details:**

For the TN measurement, Nitrate in the digestate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite thus produced is then determined by diazotizing with sulfanilamide followed by coupling with N- (1-naphthyl) ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color, which is read at 520 nm.



**Figure 1. Example of calibration for low-range Total Nitrogen method**

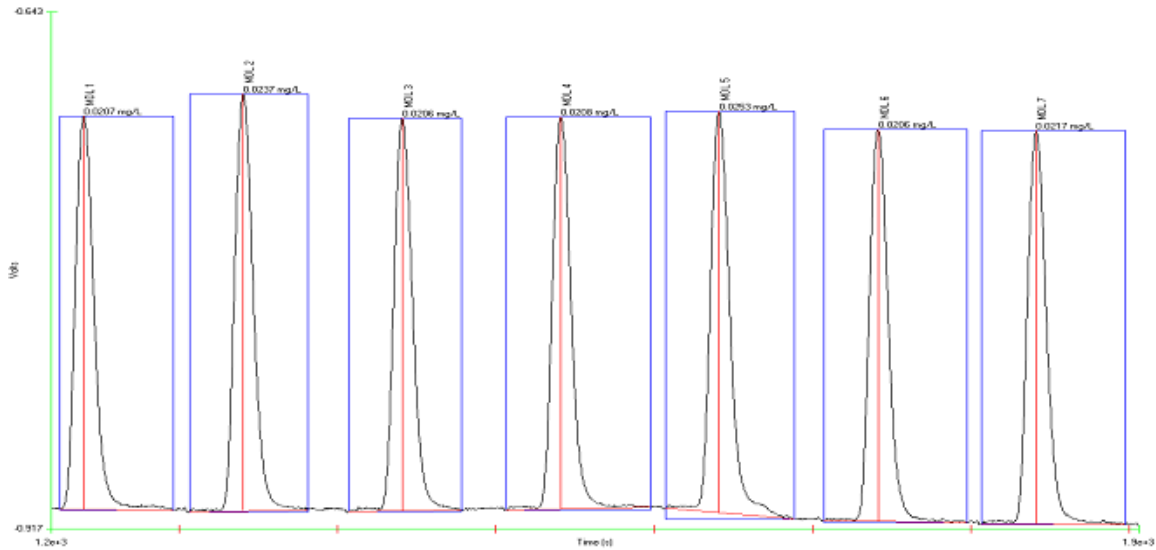


Figure 2. Method Detection Limit for low range Total Nitrogen using 0.02 mg N/L standard

**Total Phosphorus method details:**

For the TP measurement, the orthophosphate ion ( $PO_4^{3-}$ ) produced in the digestion reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex. This complex is reduced with ascorbic acid to form a blue complex, which absorbs light at 880 nm. The absorbance is proportional to the concentration of orthophosphate in the sample.

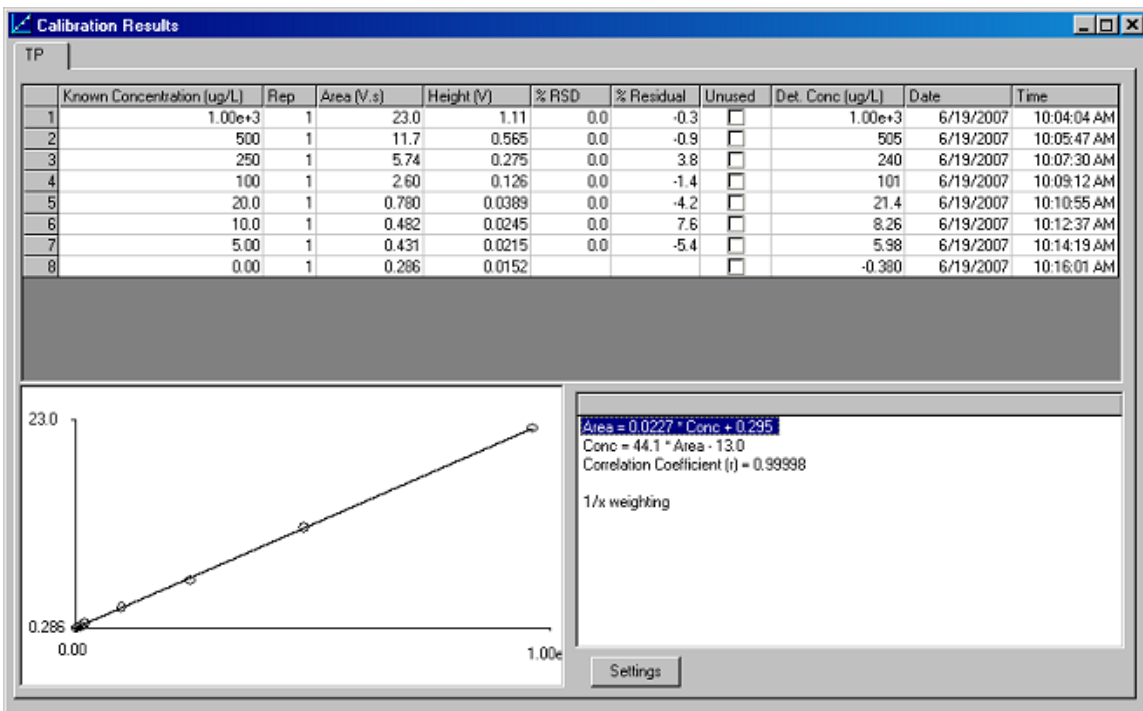


Figure 3. Example of calibration for low-range Total Phosphorus method

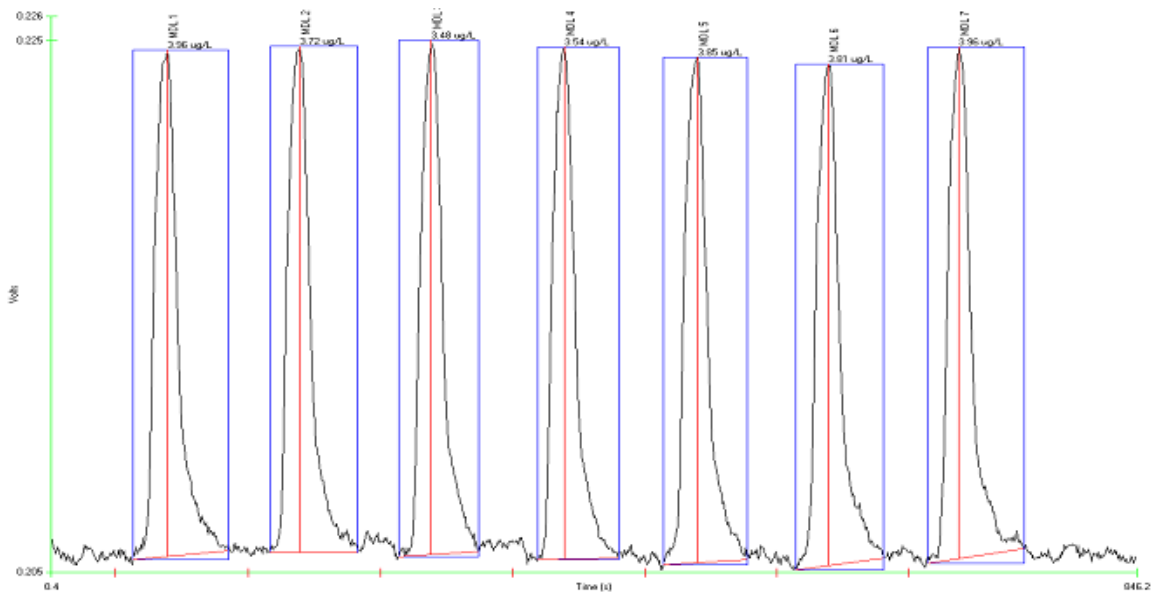


Figure 4. Method Detection Limit for low range Total Phosphorus using a 3.0 µg P/L standard

### **Differences from Standard Methods:**

These dual-digestion QuikChem methods (10-107-04-4-B and 10-115-01-4-B) are based upon Standard Methods (SM), 21<sup>st</sup> Edition 4500-P J, and Standard Methods 4500-Norg D (proposed).

Some changes were made to SM Persulfate Method 4500-Norg D:

- Acid preserved samples CAN be analyzed with these methods, as reagents have been adjusted accordingly. Standard Methods does not attempt to analyze acid preserved samples.
- Additional acid following the alkaline digestion has eliminated inconsistencies in the measurement of total phosphorus. In the Standard Method, the phosphorus results were found to be inconsistent and positively biased after the alkaline digest step, due to differences in sample make up, which in turn altered the pH.
- The buffer added after the phosphorus digest step in SM has also been eliminated. A separate line has been added to the FIA nitrate manifold board in order to neutralize the extra acid added in the phosphorus digestion step.
- When using a Lachat BD-46 Block Digester, the optimum temperature was found to be 150° C, rather than the 100 to 110° C digestion temperature in SM. This is due to the geometry of the Lachat BD-46 block digester, which accommodates 25 x 100 mm, screw capped tubes.