APPLICATION NOTE

ANTEK *byPAC*

MultiTek® Nitrogen in Polymer by Chemiluminescence

- **Rapid and Accurate Nitrogen Determination for Solids**
- **Programmable Inlet furnace for Combustible and Non-combustible Sample Matrices**
- **Excellent Sensitivity and Stability**
- **Large Sample Volume for Improving Repeatability of Results**

Keywords:
MultiTek®, Chemiluminescence, Nitrogen, Polymer, Inlet Furnace, Solid Samples

INTRODUCTION

The analysis of total nitrogen can be utilized in the plastic and polymer manufacturing industry. Manufacturers of these products add a slip material in the form of an amide to lower the coefficient of friction. By determining the amount of nitrogen in the form of the amide, manufacturers can monitor production quality and extrusion efficiency.

The principle of operation for nitrogen analysis begins with the complete, high temperature oxidation of the entire sample matrix. The sample is combusted with oxygen at a temperature of 1050°C.

The combustion gases are routed through a membrane drying system to remove all water and then to the nitrogen detector module for quantization.

$$R\text{-}N + O_2 \xrightarrow{1050^{\circ}C} CO_2 + H_2O + NO + MO_x$$

Nitric oxide is reacted with O_3 (ozone), produced by an onboard ozone generator, to form NO_2^* (metastable nitrogen dioxide). As the metastable species decays, a photon of light is emitted at specific wavelengths and detected by a photomultiplier tube (PMT).

$$NO + O_3 \longrightarrow NO_2^* + O_2 \longrightarrow NO_2 + h\nu + O_2$$

EXPERIMENTAL CONDITIONS

- **Instrumentation**

Antek MultiTek® Horizontal Nitrogen, Antek Model 737-M programmable inlet furnace.

- **Instrument Parameters**

Sample Mass (mg)	~100
GFC 1- Ar/He (ml/min)	130
GFC 2- Pyro O_2 (ml/min)	450
GFC 3- Ozone O_2 (ml/min)	35
GFC 4- Carrier O_2 (ml/min)	25
GFC 6- Auxiliary Flow (ml/min)	200
Furnace (°C)	1050
Nitrogen PMT Voltage	400

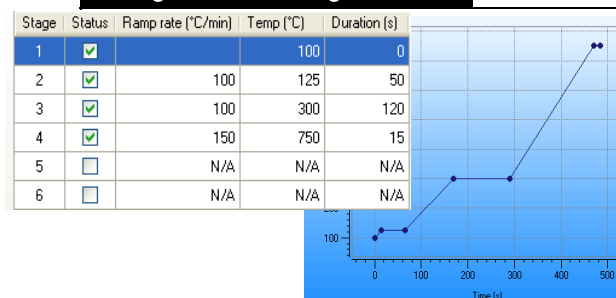| Stage	Status	Ramp rate (°C/min)	Temp (°C)	Duration (s)
1	☑		100	0
2	☑	100	125	50
3	☑	100	300	120
4	☑	150	750	15
5	☐	N/A	N/A	N/A
6	☐	N/A	N/A	N/A

Figure 1. Inlet Furnace Profile

The inlet furnace profile allows the user to control the temperature at different stages to optimize the combustion of the sample matrix and the conversion of chemically bound nitrogen to nitric oxide. Figure 1 shows the four different temperature stages.

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The MultiTek® software Timed Events Table, figure 2, allows the user to have different flow rate ratios to optimize the sample combustion and avoid coking.

Figure 2. Timed Events Table

• Calibration Curve

Calibration was performed by analyzing Nitrogen in polymer standards 500-6000 ppm as shown in figure 3.
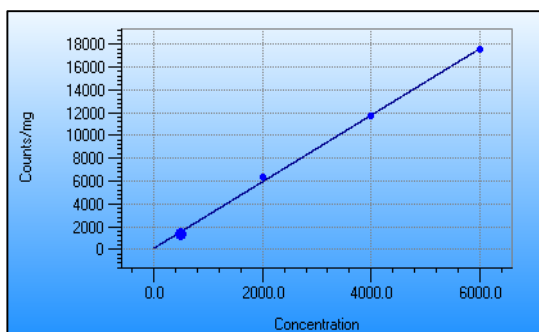

Figure 3. Calibration Results for Nitrogen
Correlation = 0.999

• Sample 1

Figure 4. Replicate 2057 ppm Overlay

• Sample Results

Polyethylene pellets sample results are shown. These samples are typical for the polymer industry.

Name	Concentration	Counts	Counts/Weight	Weight	%RSD
Sample 1	**2057**	**658650**	**6107**		**2.49**
	2117	668511	6283	106.4	
	2026	648524	6016	107.8	
	2028	658916	6023	109.4	
Sample 2	**3884**	**1247954**	**11428**		**1.17**
	3853	1235842	11338	109.0	
	3937	1272861	11582	109.9	
	3862	1235158	11363	108.7	

Table 2. Samples Run in Triplicate
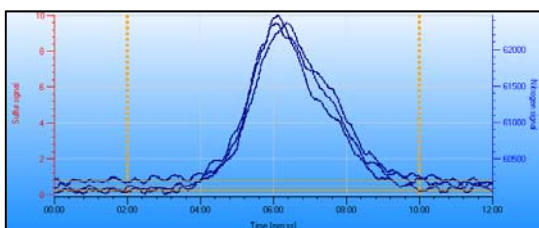
CONCLUSION

The MultiTek® Analyzer demonstrates the ability to accurately determine the total nitrogen content in polymer samples. The analysis allows the user to monitor the production process of polymers and plastic.

The Antek'MultiTek® is the only instrument on the market that combines sulfur, nitrogen, and halides analysis all in one. Compact, powerful, automated, and able to analyze gas, liquid, or solid samples, it's the perfect solution to today's increasing demand worldwide for fast, accurate detection and analysis of contaminants, and corrosive elements. Because MultiTek® delivers precise results with high sensitivity and unmatched versatility, it's a valuable process optimization tool that will deliver faster ROI and a better bottom line.

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