#### solidpartners provensolutions



#### Trace N Determination in Catalytic Naphtha by Standard Addition and Combustion Chemiluminescence Analysis

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Cambridge Viscosity\*

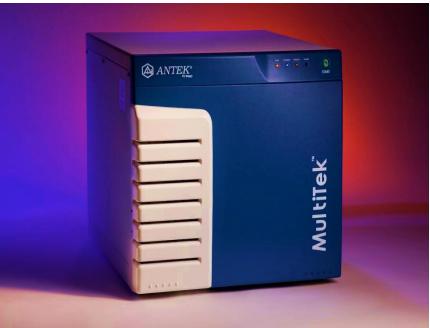


# Introduction



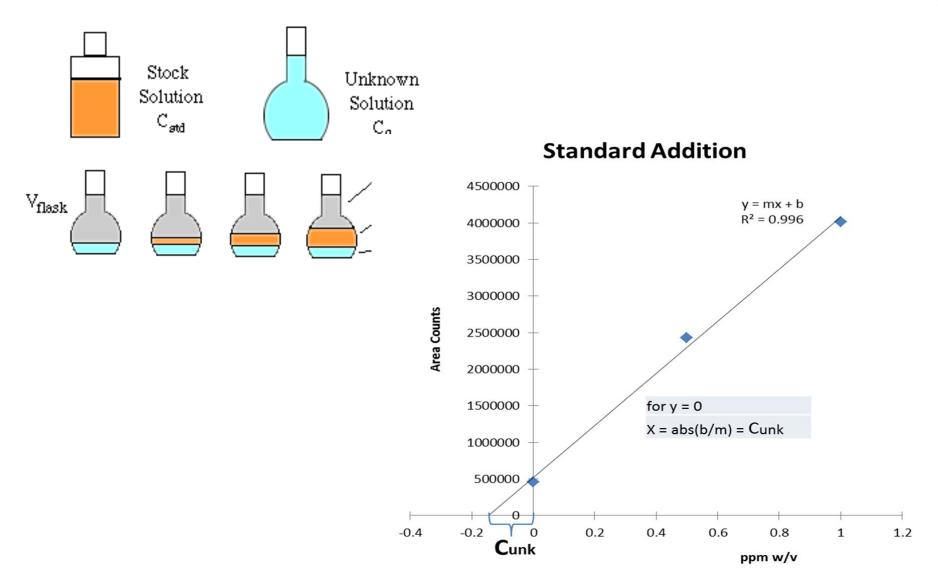
#### **STANDARD ADDITION METHOD**

- Need to eliminate matrices effects
- No need for calibration
- No need for real blanks
- Needs Linearity response of detector
- Can reach very low LOD's



# **Standard Addition Method**



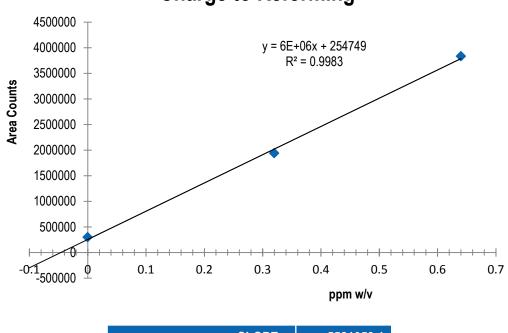


## Experimental



R-N	V + O <sub>2</sub>	$\rightarrow CO_2 + H_2O + NO + MOx  (1)$
NO	+ O <sub>3</sub>	$\rightarrow NO_2^* + O_2^* (2)$
N	O <sub>2</sub> *	$\rightarrow NO_2 + hv$ (3)
Sample Volume (µL <b>)</b>	20	
GFC 1- Ar carrier (ml/min)	130	
GFC 2- Pyro O <sub>2</sub> (ml/min)	450	
GFC 3- Ozone O <sub>2</sub> (ml/min)	35	
GFC 4- Carrier O <sub>2</sub> (ml/min)	25	
GFC 5- Auxiliary O <sub>2</sub> (ml/min)	25	Multife
Furnace (°C)	1050	2
Nitrogen PMT voltage (V)	700	

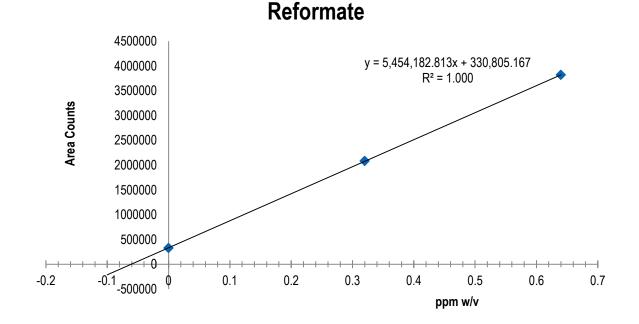




#### Charge to Reforming

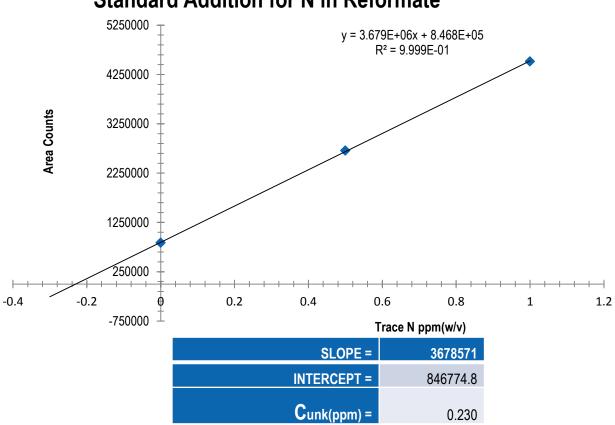
SLOPE =	5521053.1
INTERCEPT =	254748.7
Cunk(ppm) =	0.046





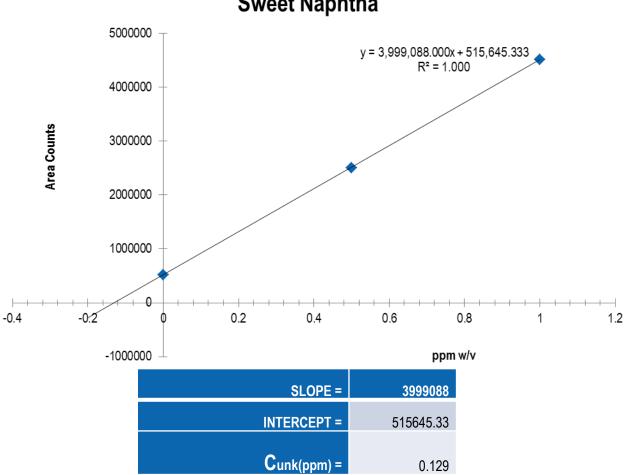
SLOPE =	5454182.8
	330805.166
INTERCEPT =	7
Cunk(ppm) =	0.061





#### **Standard Addition for N in Reformate**



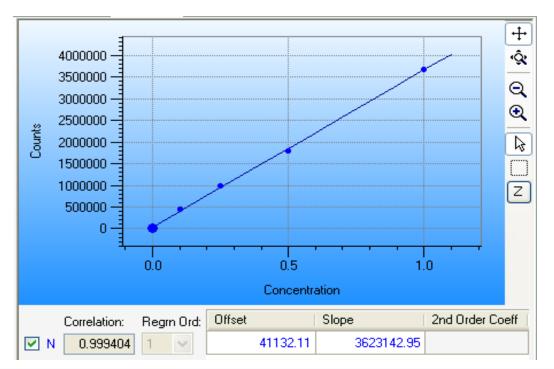


**Sweet Naphtha** 



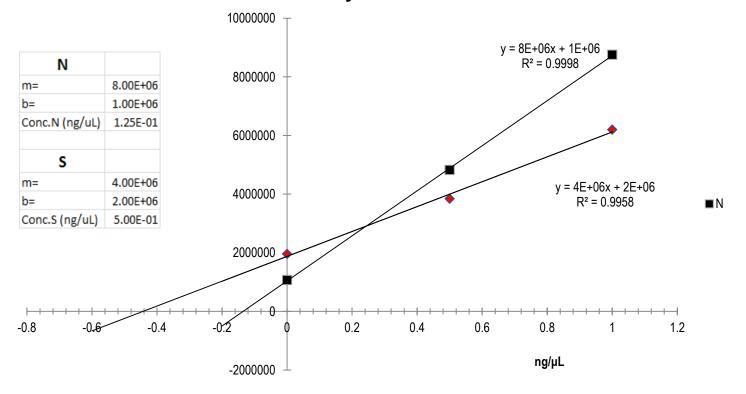
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	Sample name 1.25ppm	S-Baseline 1771089	S-Counts 14959960	S-Conc (ppm)	1.250	5-%RSD			al range	•	N	I-Basel		12881	N-Co		47385		Conc (p		1.250	N-%R	SD	_		-Cal rai	nge	
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4.4	1.25ppm	1772173	14088702		1.250		-						2	12024			47499	88		1	1.250							
	1.25ppm	1774757	13756606		1.250		-							12599			47087				1.250							
	1.25ppm	1775838	14164105		1.250		-						2	12231			47032	34		1	1.250							
4.7	1.25ppm																							_			_	
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Timestamp	Sample ID	Result ID	Method	Sequence	User	S-Col	S-Cou	S-Conc (ppm)	S-%RS	S-Cal range	N-Counts	N-Coun	N-Conc (ppm)	N-%RSD
1/4/2012 8:2	Reformate_Co	7cac2a66	N_Mod_E	N_Trace	admin	0		0.000	0.00	Unkn	840105		0.221	1.63
1/4/2012 7:5	Charge_Conoco	32c27fee	N_Mod_E	N_Trace	admin	0		0.000	0.00	Unkn	471066		0.119	16.33
1/3/2012 6:2	Std 1.00 ppm	3c6ce462	N_Mod_E	@N_Mod_E	admin	0		1.000	0.00		3680960		1.000	1.72
1/3/2012 6:1	Std 0.50 ppm	bcd804f4	N_Mod_E	@N_Mod_E	admin	0		0.500	0.00		1788676		0.500	1.57
1/3/2012 5:5	Std 0.25 ppm	e66b4cdb	N_Mod_E	@N_Mod_E	admin	0		0.250	0.00		990127		0.250	2.13
1/3/2012 5:3	0.1	8e521a00	N_Mod_E	@N_Mod_E	admin	0		0.100	0.00		448712		0.100	2.56
1/3/2012 5:0	Toluene	f08bcd74	N_Mod_E	@N_Mod_E	admin	0		0.000	0.00		0		0.000	6.80





#### Aromatic Hydrocarbon ILS

# Conclusions



- Standard addition methods have proven suitable for analyzing trace level nitrogen samples.
  - Easy to set up in a MultiTek® system
  - Provides a good alternative
- MultiTek<sup>®</sup> accurately determines very low levels of nitrogen even when not configured with a vacuum pump as demanded by ASTM standard methods D6069 and D7184.
- Results showed excellent stability, and similarities with or without the vacuum system.
- MultiTek<sup>®</sup> can optimize your refining catalytic processes by
  - Reducing operational costs savings
  - Increasing quality of final products.



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