Volatile Base in Tobacco by Online Distillation and Segmented Flow Analysis (SFA)

SEGMENTED FLOW ANALYSIS (SFA) SERIES

Cartridge Part Number: 331653CT
Channel Part Number: 331652

Scope and Application

This method is used for the determination of volatile base in tobacco leaf samples. The Method Detection Limit (MDL) is 0.003% volatile base as ammonia (NH3). The applicable range is 0.02 – 0.40% volatile base as NH_3. The range extends to analyze higher concentrations using sample dilution.

Method Performance

Range	0.02 – 0.40%
Rate	24 samples/hour
Precision	≤5.5 % RSD at 0.04% volatile base ≤3 % RSD at 0.2% volatile base
Method Detection Limit (MDL)	0.003 %

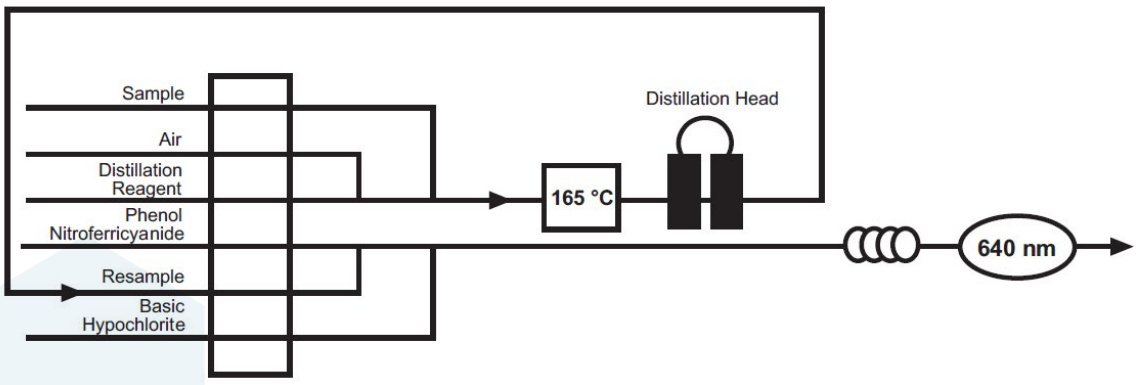

Figure 1.

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Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
Ammonium molybdate tetrahydrate	12054-85-2	$(NH_4)_6Mo_7O_{24} \bullet 4H_2O$	
Potassium antimonyl tartrate trihydrate	28300-74-5	$K(SbO)C_4H_4O_6 \bullet ½H_2O$	
Ascorbic acid	50-81-7	$C_6H_8O_6$	
DOWFAX® 2A1	12626-49-2		328852
Hydrochloric acid, concentrated	7647-01-0	HCl	
Phenylphosphate disodium salt dihydrate	66788-08-3	$C_6H_5OP(O)(ONa)_2 \bullet 2H_2O$	
Potassium Persulfate	7727-21-1	$K_2S_2O_8$	
Potassium phosphate monobasic	7778-77-0	KH_2PO_4	
Sodium hydroxide	1310-73-2	NaOH	
Sodium pyrophosphate decahydrate	13472-36-1	$Na_4O_7P_2 \bullet 10H_2O$	
Sodium tripolyphosphate	7758-29-4	$Na_5O_{10}P_3$	
Sulfuric acid, concentrated	7664-93-9	H_2SO_4	
Trimethylphosphate	512-56-1	$(CH_3O)_3P(O)$	
Water, deionized, ASTM Type I or II		H_2O	

Summary of Method

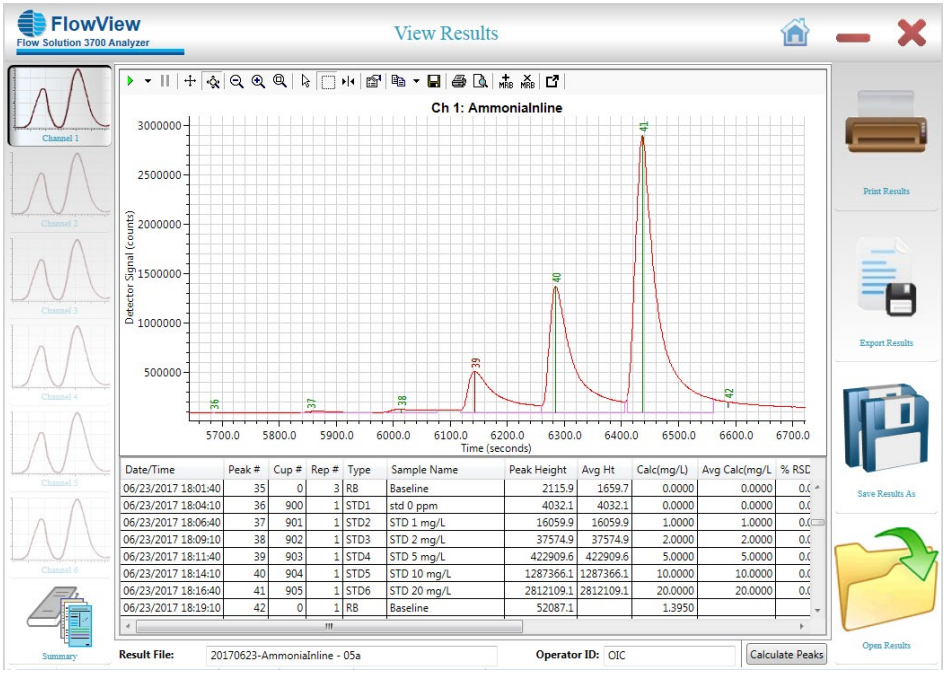
Method

- Treat tobacco leaf samples with 0.12 M hydrochloric acid to extract ammonia compounds. Distill at 165 °C and a buffered pH of 9.5. At pH 9.5 all ammonium ions quantitatively convert to NH3. The amount of NH3 obtained through distillation represents the volatile base.
- Ammonia reacts with alkaline phenol and hypochlorite to form indophenol blue in an amount that is proportional to the NH_3 concentration. Sodium nitroferricyanide intensifies the blue color. Measure the absorbance at 640 nm. [1, 2]
- Assure the analysis quality through reproducible calibration and testing of the segmented flow analysis (SFA) system.
- A general flow diagram of the SFA system is shown in Figure 1.

Interferences

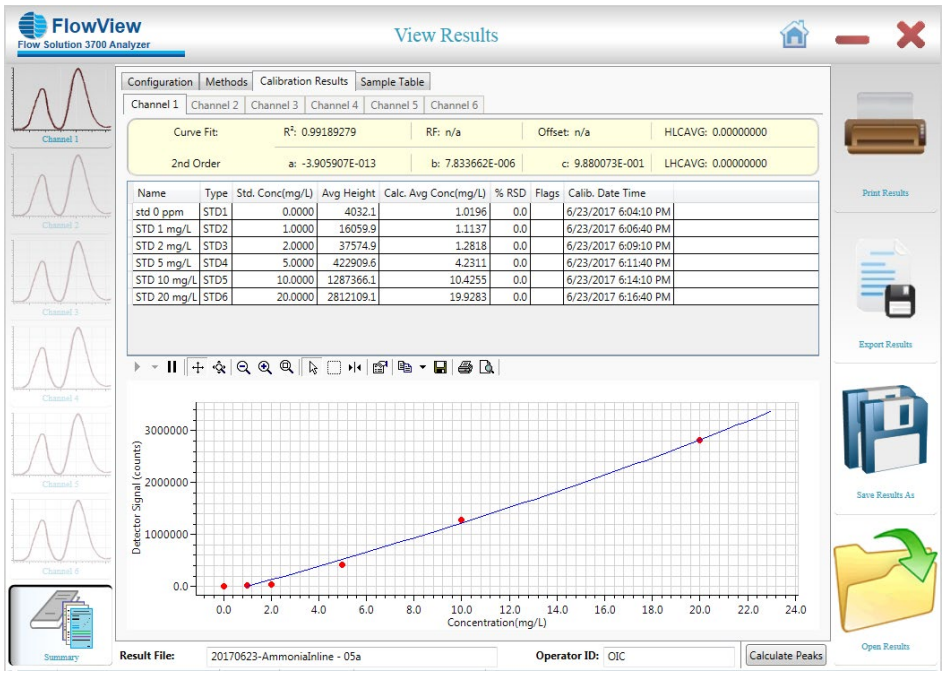
- Eliminate precipitation in the distillation tubing by adding ethylenediaminetetraacetic acid (EDTA).
- Filter turbid samples prior to analysis.
- Samples with background absorbance at the analytical wavelength may interfere. [2,3]

Figure 2. Volatile Base in Tobacco by Online Distillation and SFA Calibration Series



Date/Time	Peak #	Cup #	Rep #	Type	Sample Name	Peak Height	Avg Ht	Calc(mg/L)	Avg Calc(mg/L	% RSD
06/23/2017 18:01:40	35	0	3	RB	Baseline	2115.9	1659.7	0.0000	0.0000	0.0
06/23/2017 18:04:10	36	900	1	STD1	std 0 ppm	4032.1	4032.1	0.0000	0.0000	0.0
06/23/2017 18:06:40	37	901	1	STD2	STD 1 mg/L	16059.9	16059.9	1.0000	1.0000	0.0
06/23/2017 18:09:10	38	902	1	STD3	STD 2 mg/L	37574.9	37574.9	2.0000	2.0000	0.0
06/23/2017 18:11:40	39	903	1	STD4	STD 5 mg/L	422909.6	422909.6	5.0000	5.0000	0.0
06/23/2017 18:14:10	40	904	1	STD5	STD 10 mg/L	1287366.1	1287366.1	10.0000	10.0000	0.0
06/23/2017 18:16:40	41	905	1	STD6	STD 20 mg/L	2812109.1	2812109.1	20.0000	20.0000	0.0
06/23/2017 18:19:10	42	0	1	RB	Baseline	52087.1		1.3950		

Figure 3. Volatile Base in Tobacco by Online Distillation and SFA Calibration Curve and Statistics



Name	Type	Std. Conc(mg/L)	Avg Height	Calc. Avg Conc(mg/L)	% RSD	Flags	Calib. Date Time
std 0 ppm	STD1	0.0000	4032.1	1.0196	0.0		6/23/2017 6:04:10 PM
STD 1 mg/L	STD2	1.0000	16059.9	1.1137	0.0		6/23/2017 6:06:40 PM
STD 2 mg/L	STD3	2.0000	37574.9	1.2818	0.0		6/23/2017 6:09:10 PM
STD 5 mg/L	STD4	5.0000	422909.6	4.2311	0.0		6/23/2017 6:11:40 PM
STD 10 mg/L	STD5	10.0000	1287366.1	10.4255	0.0		6/23/2017 6:14:10 PM
STD 20 mg/L	STD6	20.0000	2812109.1	19.9283	0.0		6/23/2017 6:16:40 PM

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