

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Volatile Base in Tobacco by Online Distillation and Segmented Flow Analysis (SFA) Cartridge Part Number 331653CT

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 (NH_3). 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 per hour
Precision	≤5.5 % RSD at 0.04% volatile base ≤3 % RSD at 0.2% volatile base
Method Detection Limit (MDL)	0.003%

Summary of 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 NH_3 . The amount of NH_3 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.

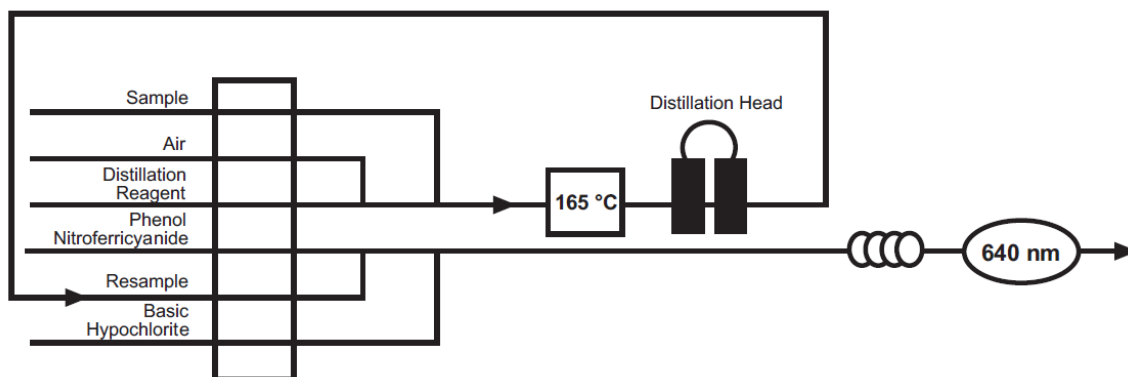


Figure 1.

Reagents and Calibrants

Chemical Name	CAS#	Chemical Formula	Part Number	Used in Prep Guide
Ammonium sulfate	7783-20-2	$(\text{NH}_4)_2\text{SO}_4$		Appendix A
Brij®-35, (21% Solution)	9002-92-0		326126	Appendix A
Deionized Water (ASTM Type I or II)				Appendix A
Ethylenediaminetetraacetic acid, disodium salt dihydrate (EDTA)	6381-92-6	$\text{C}_{10}\text{H}_{16}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$		Appendix A
Hydrochloric acid, concentrated	7647-01-0	HCl		
Phenol, solid or liquid, 88%	108-95-2	$\text{C}_6\text{H}_5\text{OH}$		Appendix A
Sodium hydroxide	1310-73-2	NaOH		Appendix A
Sodium hypochlorite, 5.25% available chlorine (household bleach)	7681-52-9	NaOCl		Appendix A
Sodium nitroferricyanide dihydrate	13755-38-9	$\text{Na}_2\text{Fe}(\text{CN})_5\text{NO} \cdot 2\text{H}_2\text{O}$		Appendix A
Sodium tetraborate decahydrate	1303-96-4	$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$		Appendix A

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}

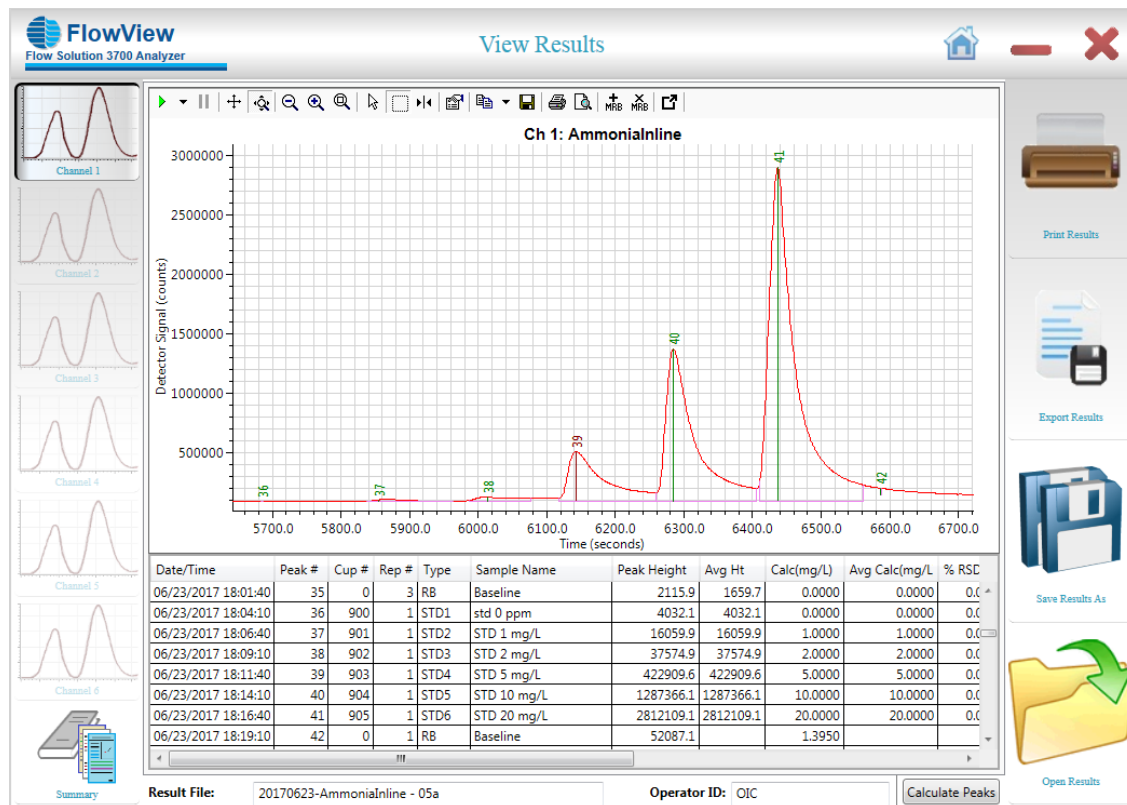


Figure 5. Volatile Base in Tobacco by Online Distillation and SFA Calibration Series

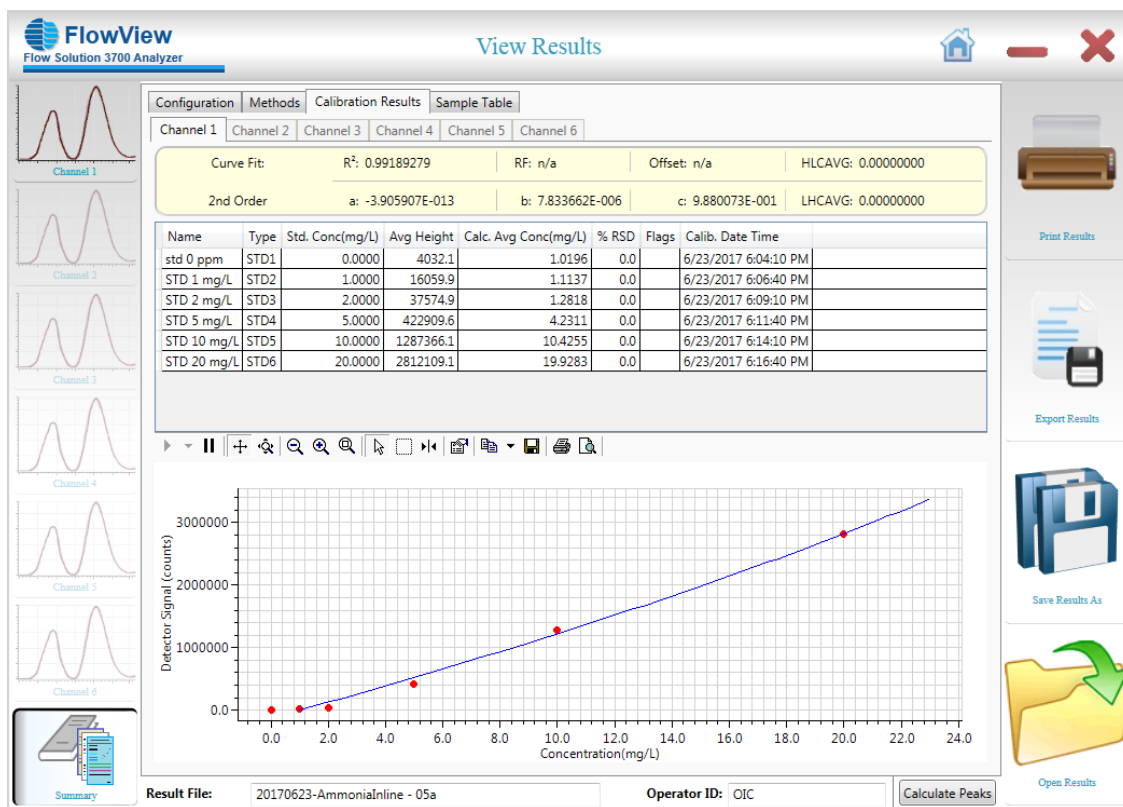


Figure 6. Volatile Base in Tobacco by Online Distillation and SFA Calibration Curve and Statistics

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