

DETERMINATION OF WATER AND NICOTINE IN TOBACCO SMOKE BY GAS CHROMATOGRAPHY USING CAPILLARY COLUMNS INSTEAD OF PACKED COLUMNS

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Abstract:

The standard procedure to determine Water and Nicotine in tobacco smoke is gas chromatography with two packed columns for the separation. The analysis time is between 4 and 6 minutes. The quality of packed columns is variable from one production to another (hand-made preparation by supplier) and ghost peaks are even present on some occasions. To eliminate this problem, capillary columns were evaluated to perform routine water and nicotine determinations in tobacco smoke. Capillary columns are produced by machine and are less variable to production. The goals of this method improvement were to use the same standard concentrations, internal standards and only one injection for both columns. The procedure was optimized to reduce the analysis time. Finally, same or better method characteristics such as Limits of Quantitation (LOQ) and separation system robustness.

Results are not significantly different with packed or capillary columns under ISO and Canadian Intense smoking regimes. The chromatogram is very simple and is acquired with the TCD for water for the first minute and then changed for the FID for the last portion of it. This approach has the advantage to reduce the disk space for data storage and to generate only one chromatogram which minimize the time for the data processing. The analysis time was reduced to less than 2 minutes. The LOQ are 0.2 and 0.3 mg/cig for water and 0.05 and 0.03 mg/cig for nicotine respectively with packed and capillary columns.

Gas chromatograph:

Packed column injection port
Capillary column injection port



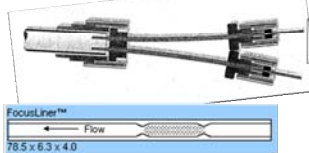
Packed columns:

Water: Haysep Q 80/100 5'X1/8"
Nicotine: 16% Apiezon+2% KOH+2% Carb-20M 5'X1/8"



Capillary columns:

Water: CP-Poraplot Q 12.5 m X 0.53 mm X 20 µm
Nicotine: CP-Wax 52 CB 25 m X 0.53 mm X 2 µm



GRAPHPACK® 2M dual column inlet adapter for GERSTEL®
For Agilent Technologies Split/splitless injector
Part No. 007533-007-00

Split/Splitless FocusLiner for HP 6.3 mm OD p/w Quartz Wool
Part No. 2879801-U
Applications: Dirty samples, Improved reproducibility

Experimental conditions:

- Samples were generated under ISO and Canadian Intense smoking regimes
- Samples were analyzed on 2 GC's with packed columns (P_A & P_B) and on 1 GC with capillary columns in a random fashion as described in the test schedule

Test Schedule:

Day	Tray	Tray placed on		
		GC # P _A	GC # P _B	GC # Cap
		Packed columns	Packed columns	Capillary columns
Monday	A	A	-----	-----
Tuesday	B	-----	A	B
Wednesday	C	B	C	A
Thursday	-----	-----	B	C
Friday	-----	C	-----	-----

First week: ISO
Second week: Intense*

- Two repeats of a minimum of 240 samples were analyzed on the 3 GC's
- Results were compared for both regimes independently
- Results were compared for differences between GC P_A, GC P_B and GC Cap

Results (ISO):

Concentration range (mg/cig)	Water				Nicotine			
	P _A vs P _B	Capil. vs P _A	Capil. vs P _B	Concentration range (mg/cig)	P _A vs P _B	Capil. vs P _A	Capil. vs P _B	
< 1	0.22	0.23	0.13	< 0.5	0.03	0.04	0.02	
1-2	0.35	0.31	0.17	0.5-1.0	0.05	0.19	0.07	
2-3	0.37	0.42	0.21	1.0-1.5	0.09	0.16	0.12	
3-4	0.47	0.52	0.18	1.5-2.0	0.05	0.02	0.12	
4-7	0.46	0.46	0.22					

Introduction:

The standard procedure to determine water and nicotine in tobacco smoke is gas chromatography with two packed columns for the separation. Capillary columns were evaluated to perform this routine analyses. A splitter at the injection port was used to inject on both columns simultaneously. The same standard concentrations and Internal standards were used. An isothermal mode and injection port program were developed to reduce the analysis time. The new procedure was evaluated with two smoking regimes (ISO and Canadian Intense*).

Gas Chromatograph conditions:

T_{Oven}: 160°C
Init. Time: 2.2 min
Back inlet: Splitless mode
Temp.: 250°C
Pressure: 24 psi
Purge Time: 0.05 min
Purge flow: 10.0 mL/min
Total flow: 43.0 mL/min
Gas Saver: Off

Front det. (FID)
Temp.: 250°C
H₂ flow: 45 mL/min
Air flow: 400 mL/min
Mkup (He): 3.0 mL/min

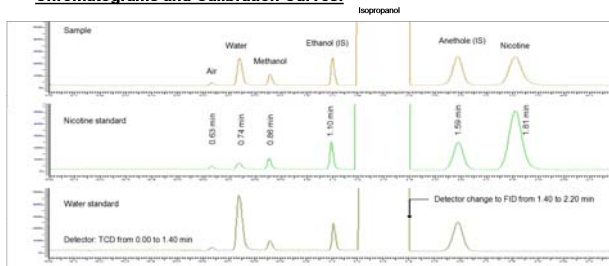
Back det. (TCD)
Temp.: 300°C
Ref. flow: 20.0 mL/min
Mkup (He): 3.0 mL/min

Run Table
Time: 0:00
Type: Sig 1 defn
Defn: Back
Sig 1 range
Setpoint: 0
Time 1:40 min
Type: Sig 1 range
Setpoint: 3
Time: 1:40 min
Type: Sign 1 defn
Defn: Front

Column 2 (He)
Flow: 46.7 mL/min
Velocity: 309 cm/sec
Mode: Ramped pres.
Init. Pres: 24.0 psi
Init. Time: 1.25 min
Rate 1: 50.0 mL/min
Final Pres 1: 35.0 psi
Final Time: 1.00 min

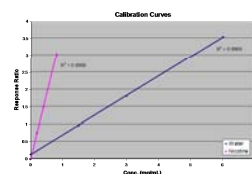
* Puff volume: 55 mL, Puff duration 2 sec, Frequency Interval 30 sec., Ventilation holes blocked (taped)

Chromatograms and Calibration Curves:



Standard concentrations:

Std #	Concentration (mg/mL)		Smoking Regime
	Water	Nicotine	
1	0.25	0.08	ISO
2	0.50	0.20	ISO
3	1.50	0.40	ISO
4	1.50	0.20	Intense
5	3.00	0.40	Intense
6	6.00	0.80	Intense



Results (Intense):

Concentration range (mg/cig)	Water				Nicotine			
	P _A vs P _B	Capil. vs P _A	Capil. vs P _B	Concentration range (mg/cig)	P _A vs P _B	Capil. vs P _A	Capil. vs P _B	
< 1.5	0.03	0.08	0.10	< 1.5	0.03	0.08	0.10	
1.5-2.2	0.18	1.18	0.63	1.5-2.2	0.17	0.30	0.17	
2.2-2.7	0.63	0.68	0.72	2.2-2.7	0.23	0.23	0.22	
2.7-3.2	0.83	0.65	0.92	2.7-3.2	0.30	0.28	0.27	
3.2-3.7	0.75	0.93	0.90	3.2-3.7	0.30	0.30	0.27	

Acceptable differences: Differences between packed and capillary columns should be of the same order of magnitude as differences between two packed columns independently of the concentrations or smoking regimes

Conclusion:

- Results are the same using capillary or packed columns and with the same quality results (LOQ, repeatability)
- Same standard concentrations and internal standards can be used
- The total analysis time is around 2 min. which is a time reduction of 50 to 66 % in comparison with packed columns
- GRAPHPACK® 2M dual column inlet adapter from GERSTEL® allows the injection of one sample vial into two capillary columns. It is robust and well suited for routine work
- The FocusLiner™ retains impurities preventing column contamination. It was still good after more than 1700 injections
- The column lifetime has still to be determined. After around 5000 injections, no signs of degradation were observed