

# An Improved Method for the Determination of Selected Humectants in Tobacco

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## INTRODUCTION

Humectants are an important class of compounds that can be applied to tobacco for the improvement of hygroscopic properties and to act as a carrier for flavor components. Originally, Arista Laboratories validated a method for the determination of humectants based on two established methods: Health Canada method T-304 and CORESTA Recommended Method CRM 60. The objectives for a revalidation were to streamline the extraction process and expand the scope to include a variety of products (e.g. moist snuff and Kreteks).

During redevelopment it was observed that a moist snuff product appeared to be completely extracted after 30 minutes; the dry products, however, were not fully extracted as demonstrated by increasing concentration over time. The moisture content of the tobacco product affected the extraction efficiency of the humectants. Incorporating water into the extraction process resulted in elevated levels of humectants in a shorter time frame compared to a pure methanol extraction.

## METHODOLOGY

Three methods were evaluated for extraction efficiency:

1. Arista, AM-090
2. CORESTA, CRM 60
3. Health Canada, T-304

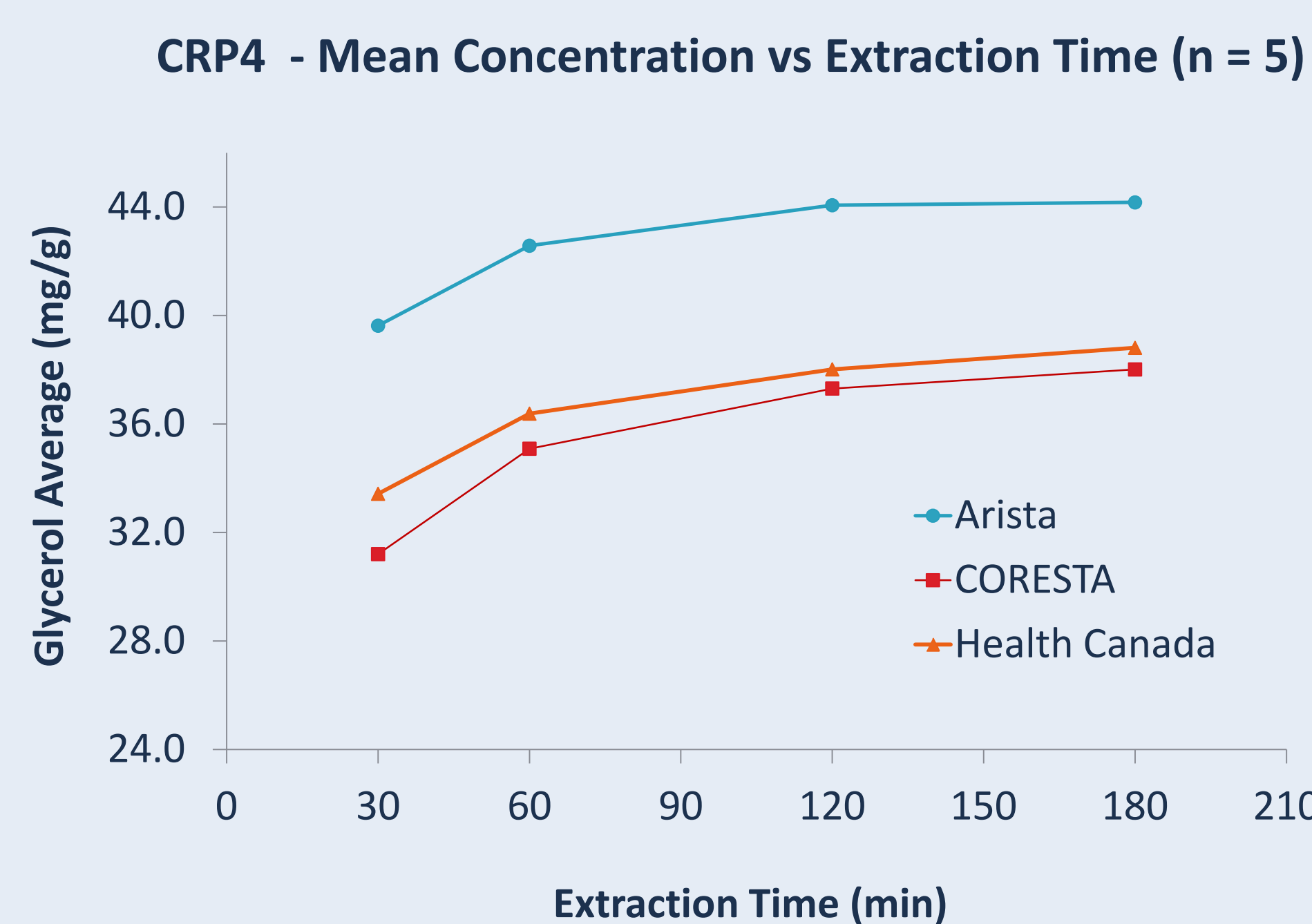
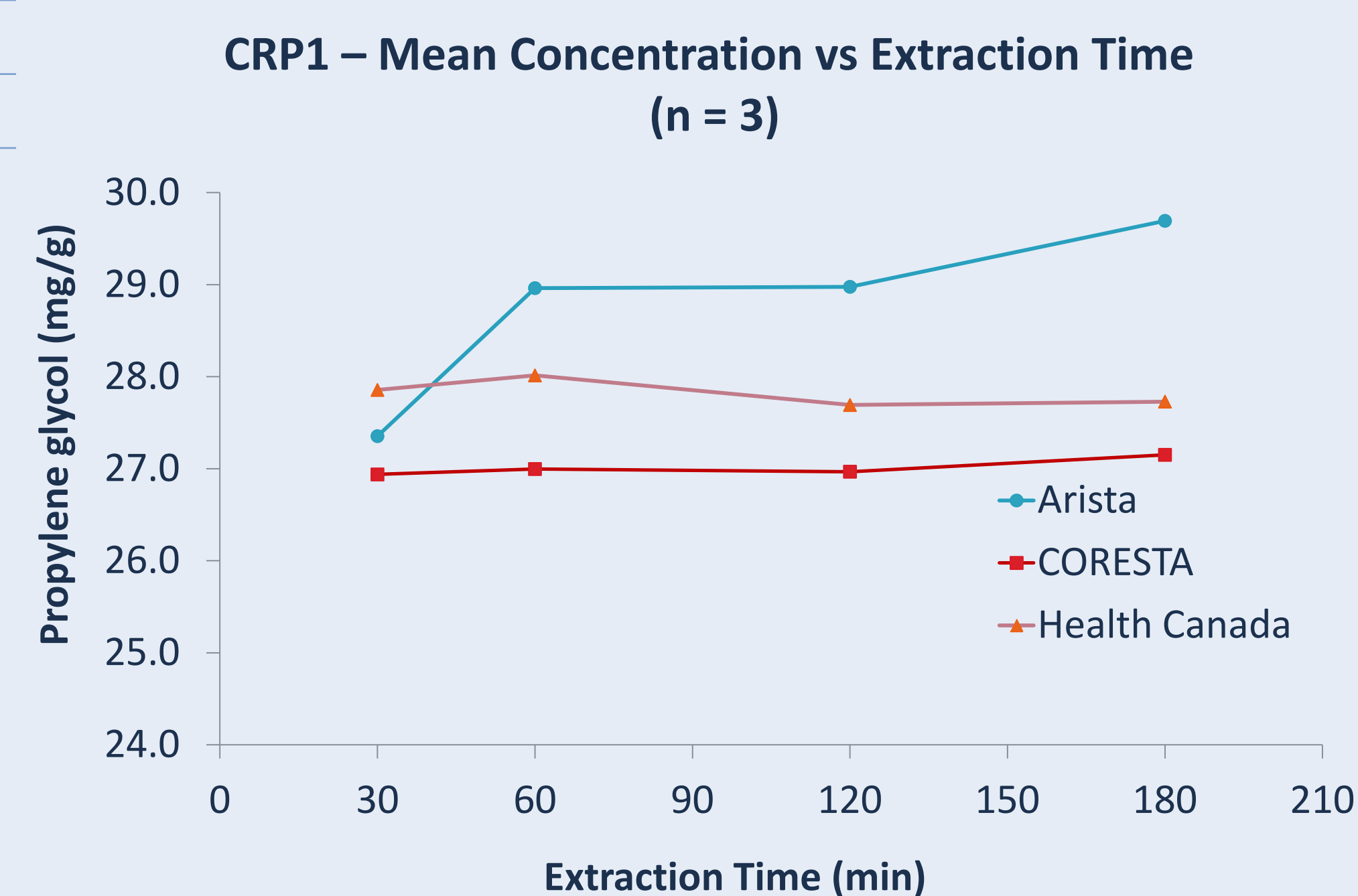
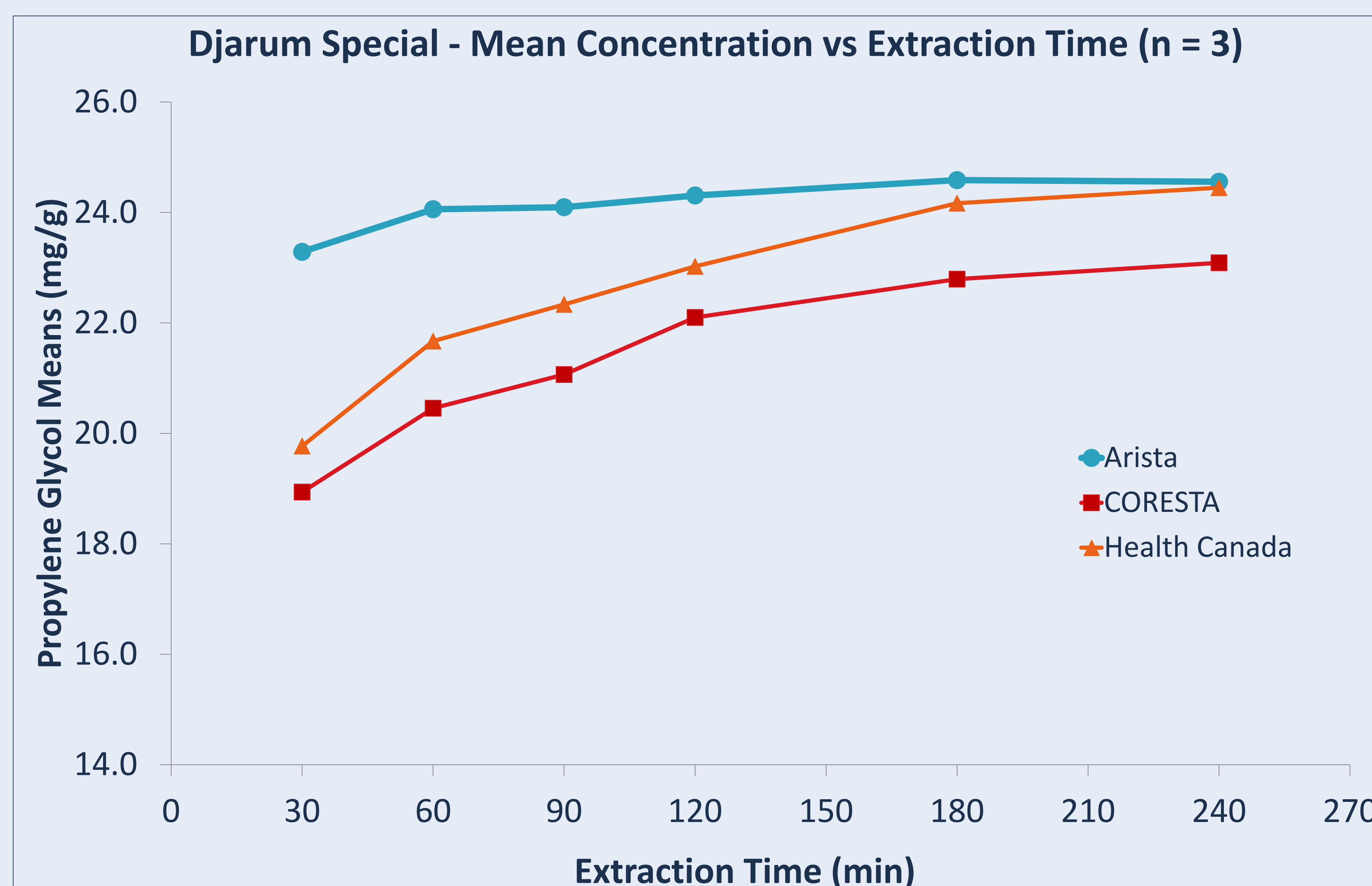
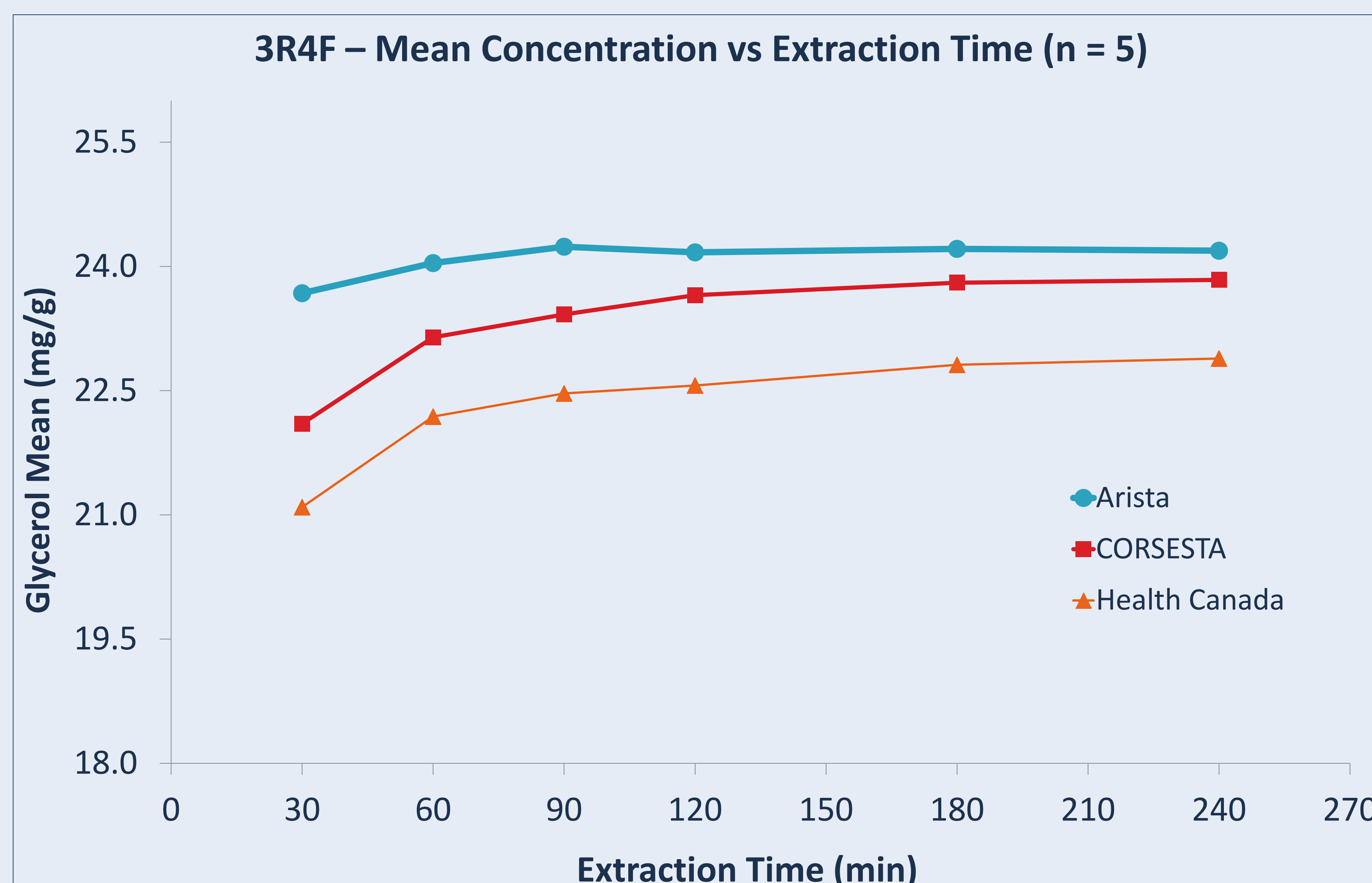
Four products were extracted in accordance with each of the methods, with the exception of extraction time. Samples were shaken for up to four hours monitoring levels at 30 minute intervals. All replicates were analyzed by AM-090. Two of the four products are considered “dry” (3R4F and Djarum Special) and the other two are considered “moist” products (CRP1 and CRP4). One product of each type contains propylene glycol (CRP1 and Djarum Special) and the others contain glycerol (CRP4 and 3R4F).

Extraction Parameters for Methods Evaluated

Method	Arista	CORESTA	Health Canada
Tobacco Weight	2 g	2 g	4 g
Solvent	H <sub>2</sub> O + MeOH	MeOH	MeOH
Volume	5 + 25 mL	50 mL	50 mL
Shaking Time	60 min	120 min	60 min

AM-090 Instrument Parameters

Column	Rtx-Wax (30 m x 0.32 mm; 1 μm)
Split Ratio	20:1
Injection Volume	1 μL
Inlet Temp.	250 °C
Initial Oven Temp.	100 °C
Temperature Program	20 °C/ min to 215 °C ; hold for 5 min
FID Temp.	250 °C



Comparison of Humectants at Recommended Extraction Times

	CRP4		
	Arista	CORESTA	Health Canada
Time (min)	60	120	60
Mean GLY (mg/g)	42.6	37.3	36.4
SD	0.8	1.0	1.4

	3R4F		
	Arista	CORESTA	Health Canada
Time (min)	60	120	60
Mean GLY (mg/g)	24.0	23.7	22.2
SD	0.2	0.4	0.1

	CRP1		
	Arista	CORESTA	Health Canada
Time (min)	60	120	60
Mean PG (mg/g)	29.0	27.0	28.0
SD	0.5	0.3	0.6

	Djarum Special		
	Arista	CORESTA	Health Canada
Time (min)	60	120	60
Mean PG (mg/g)	24.1	22.1	21.7
SD	0.3	0.3	0.5

## CONCLUSION

Water is necessary for complete extraction of humectants from tobacco. The CORESTA and Health Canada methods that utilize pure methanol are not as effective in determining the concentration of the propylene glycol and glycerol in tobacco.

The improved method results in a more complete extraction of humectants from tobacco in only 60 minutes. The revalidated method also extends the scope to include a wider range of tobacco products.

## REFERENCES

1. CORESTA Recommended Method, CRM 60 – Determination of 1, 2-Propylene Glycol and Glycerol in Tobacco and Tobacco Products by Gas Chromatography. [http://www.coresta.org/Recommended\\_Methods/CRM\\_60-update\(May11\).pdf](http://www.coresta.org/Recommended_Methods/CRM_60-update(May11).pdf)
2. Health Canada, T-304 – Determination of Humectants in Whole Tobacco. [http://www.hc-sc.gc.ca/hc-ps/tobac-tabac/legislation/reg/indust/method/\\_whole-entier/humectant-eng.php](http://www.hc-sc.gc.ca/hc-ps/tobac-tabac/legislation/reg/indust/method/_whole-entier/humectant-eng.php)