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Menthol Content in U.S. Marketed Cigarettes

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Abstract

Introduction—In 2011 menthol cigarettes accounted for 32 percent of the market in the United States, but there are few literature reports that provide measured menthol data for commercial cigarettes. To assess current menthol application levels in the U.S. cigarette market, menthol levels in cigarettes labeled or not labeled to contain menthol was determined for a variety of contemporary domestic cigarette products.

Method—We measured the menthol content of 45whole cigarettes using a validated gas chromatography/mass spectrometry method (GC/MS).

Results—In 23 cigarette brands labeled as menthol products, the menthol levels of the whole cigarette ranged from 2.9 to 19.6 mg/cigarette, with three products having higher levels of menthol relative to the other menthol products. The menthol levels for 22 cigarette products not labeled to contain menthol ranged from 0.002 to 0.07 mg/cigarette. The type of packaging (soft vs. hard pack) for a given cigarette product does not appear to affect menthol levels based on the current limited data.

Conclusion—Menthol levels in cigarette products labeled as containing menthol are approximately 50 to 5,000-fold higher than those in cigarette products not labeled as containing menthol. In general, menthol content appears to occur within discrete ranges for both mentholated and non-mentholated cigarettes.

INTRODUCTION

Menthol is a permitted characterizing flavor in cigarettes by the Federal Food, Drug, and Cosmetic Act. Recent reports indicate that, after a slight decline in 2009 and 2010, menthol cigarette market share in the United States rose to 32 percent in 2011.[1] In 2010–2011

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approximately 30% of current smokers in the United States reported their usual cigarette type as menthol[2]

Menthol has a distinctive flavor and cooling properties that readily transfer to the mainstream smoke during combustion; these properties have been suggested to facilitate initiation of cigarette use or discourage quitting by facilitating ease of use and depth of smoke inhalation.[3] Menthol properties may also mask symptoms of respiratory disease, potentially leading to delays in medical treatment and thereby suppressing motivation for cessation.[4]

Menthol has been added in various amounts to cigarettes since the 1920s.[5–8] To achieve a slight menthol effect (the perception of menthol flavor and cool sensation) in cigarette smoke, the amount of menthol added to tobacco filler is reported to be 0.1to 0.2% (1 to 2 mg/g of tobacco filler) for a weak effect.[9] Higher menthol levels provide more pronounced flavoring effects, with levels as high as 2% reported.[9,10] For regular cigarettes not labeled as containing menthol (nonmenthol cigarettes), added menthol is reported to be in the range of 0.003% (0.03 mg/g of tobacco filler).[10]

Despite the prevalence of sales and use of menthol cigarettes in the United States, only a few literature reports have measured menthol levels in commercial cigarettes. Celebucki et al. analyzed the menthol content for 48 mentholated cigarette products on the U.S. market before 2005.[11] The study measured menthol in the whole cigarette, which ranged from 2.35 to 7.15 mg/g of tobacco filler (1.61 to 4.38 mg/cigarette). Kreslake et al. measured menthol levels of eight U.S. menthol cigarette products.[12] The menthol content for these eight products ranged from 3.2 to 6.3 mg/g of tobacco filler. Altria Client Services measured menthol content in 68 menthol cigarette products on the U.S. market from 2008–2009.[13] The menthol levels were 2.2 – 9.8 mg/cigarette.

Menthol is an ingredient in most cigarettes and is added to cigarettes not labeled to contain menthol.[14] In these cigarettes, menthol amounts are reported to be 100- to 1000-fold lower than the amounts contained in menthol flavored cigarettes. [15] Menthol may also naturally occur in tobacco[16], or possibly be a contaminant of manufacturing processes. Therefore, menthol can be present in cigarettes even if it is not intentionally added by manufacturers. We located only one publication with data on the menthol levels measured in cigarettes not labeled to contain menthol[10]. In this study, the investigators measured flavor additives in cigarette tobacco filler of 32 cigarette products on the Swiss market in 2005, along with several homemade and reference cigarettes.[16] The menthol levels ranged from 0.019 to 13.3 μg/g, with a mean value of 0.97 μg/g.

Menthol is a volatile chemical which readily evaporates during manufacturing and storage.[9] As a result, the quantitative comparison of the data by Celebucki et al., Altria Client Service and Kreslake et al. may not be possible since the cigarettes were manufactured in different years and the time elapsed between the manufacturing and menthol analysis is not known. Altria Client Services reported on measured menthol content of cigarettes marketed in 2008–2009. Celebucki et al. measured menthol content in cigarettes marketed before 2005, and Merckel et al. examined cigarettes marketed before 2006. Both Altria Client Services

and Celebucki et al. measured the menthol content of whole cigarettes, whereas Merckel et al. only measured menthol in the tobacco filler and the paper. As a result, there appears to be a data gap on the measured menthol content in whole cigarettes as well as in tobacco and nontobacco components of contemporary cigarettes, especially for those products marketed as nonmentholated cigarettes. In particular, more data are needed to compare the levels of menthol in cigarettes labeled as containing menthol with those not labeled as containing menthol. Existing data are not consistent in reporting menthol quantities, making it difficult to compare datasets.

In cigarettes, menthol may be applied directly to the tobacco filler, the filter, or the packaging material separately. During storage, it migrates to the other parts of the cigarette irrespective of the original application.[13] Several factors, such as storage temperature, plasticizer in filter and humectants in the filler influence the rate of migration but menthol concentration is not the influencing factor.[17] Storage temperature affects the vapor pressure of menthol, which directly influences the migration rate. Triacetin, a common plasticizer used in cigarette filters, and propylene glycol, a humectant in tobacco filler, increase the affinity of menthol for these respective components and affect menthol migration and retention. Menthol migration reaches equilibrium after prolonged storage (greater than 4 months[13] or 9–11 months[17]), with general retention in the filler and the filter of the cigarette. Because the cigarettes purchased for this study have a storage time and menthol migration has already taken place, measurements of the menthol levels in whole cigarette is necessary.

To assess recent menthol application in contemporary U.S. marketed cigarettes, the menthol levels of whole cigarettes were measured using the method previously developed by the Centers for Disease Control and Prevention (CDC).[18] Menthol levels for 45 cigarette products (23 mentholated products and 22 are not labeled to contain menthol) and two reference cigarettes (University of Kentucky 1R5F and 3R4F) were determined. The results provide insight into how menthol levels may differ within and across products and if menthol levels may be affected by packaging design.

METHOD

The analytical procedure is adopted from a previous study[18] with slight modifications.

Cigarettes

Domestic cigarette products were purchased in July 2013 and June 2014 from retail sources in metropolitan Atlanta or from wholesale locations through the Lab Depot (Dawsonville, Georgia, USA). Research cigarettes 3R4F and 1R5F were purchased from the University of Kentucky (Lexington, Kentucky, USA). Upon receipt, samples were logged into a custom database, assigned barcodes with unique identification, and stored in their original sealed packaging at room temperature until analyzed. All products were analyzed within thirty days of receipt. Table 1 provides product information with respect to brand, cigarette size, packaging, and mentholation status based on label information.

Reagents and Materials

Menthol and 3′,4′-(methylenedioxy)-acetophenone (MDA) were purchased from Sigma-Aldrich (St. Louis, Missouri, USA). MDA was used as an internal standard for quantitation. All other chemicals were of analytical grade and were purchased through Fisher Scientific (Pittsburgh, Pennsylvania, USA) unless otherwise indicated.

Sample Preparation and Analysis Procedure

New unopened packs of cigarette product were used for each analysis run to minimize menthol loss due to volatilization. Once the cigarette pack was opened, the whole cigarette was immediately sliced longitudinally through both the filter and rod to expose the inside of the cigarette. One entire cigarette (paper, filler, and filter) was then placed in a 15 mL sample vial that had been previously spiked with 50 μL MDA (17.03 mg/mL in methanol) internal standard, and the rest of the procedure is the same as previously reported.[18] Samples were analyzed in septuplicate (n=7).

Individual cigarette masses were obtained on a Cerulean (Milton Keynes, United Kingdom) C2 Range and measured in septuplicate (n=7). The average mass of each cigarette was used to assess the concentration of menthol on a mg/g of whole cigarette basis.

Instrumentation and Apparatus

Instrumentation and apparatus are same as previously reported with the exception of using a GC split ratio of 22:1 for this study instead of 40:1.[18]

Calibration Procedure—A standard stock solution was prepared by weighing menthol and diluting it with acetonitrile to a volume of 50 mL. A standard curve was generated by spiking approximately 400 mg of the 3R4F research cigarette filler with 200 μL of each calibration standard and 50 μL of the MDA internal standard (17.03 mg/mL in methanol). Two additional calibration standards at low menthol concentrations were added to extend the calibration range of the previously reported method,[18] to enable menthol quantification at less than microgram per gram of cigarette with a calibration range of 0.505 to 1.01×10^4 μg/g. An initial LOD for menthol was estimated as $3s_0$ where s_0 is the estimate of the standard deviation at zero analyte concentration. The value of s_0 was taken as the y-intercept of a linear regression of standard deviation versus concentration.[19] The LOD of the modified method was 0.788 μg/g.

Method Validation

Precision and accuracy were determined at five concentration levels to validate the method. Precision/accuracy data was obtained by adding menthol standards to a blank 3R4F matrix at varying concentrations (1, 5, 252, 757 and 5045 μg/g). A synthetic standard was used to assess precision and accuracy because mentholated tobacco standards were not available. A blank control was prepared by assessing five 3R4F reference cigarette filler samples with only the MDA internal standard. Table 2 lists the data from validation. The recovery range spanned 99% to 113% for all five addition levels and precision was excellent (Table 2). Samples were prepared as described above and analyzed at 30 minutes, 1 hour and 2 hours. The extraction time of 1 hour was found to be optimal because after 1 hour, extraction was

found to be complete. In general, interferences from the tobacco matrix were minor but to confirm the presence of menthol, the confirmation ion ratio was calculated and used to confirm menthol's presence rather than matrix interferences. If observed confirmation ion ratios were >10% different than found in the standard, the concentration of that sample was not reported. Relative retention time (analyte vs. MDA internal standard) was also used to confirm analyte presence.

RESULTS

The menthol levels were quantitatively determined for all commercial and reference cigarettes (Table 1). Plotting these values (Figure 1) shows that, with the exception of three products, the menthol content is within distinct ranges for both menthol and nonmenthol cigarette brands. For mentholated cigarettes, the menthol content ranged from 2.9 to 19.6 mg/cigarette. Three cigarette products, Natural American Spirit Light Green, Camel Crush King Menthol and Camel Crush King Menthol Regular Fresh, had the greatest amounts of menthol at 19.6, 14.1 and 10.8 mg/cigarette, respectively. The two Camel Crush products have menthol in capsules inside the cigarette filters. This type of cigarette is different from typical menthol cigarettes because the menthol is contained in a bead, rather than added to the cigarette during manufacturing. Menthol in the two Camel Crush products does not equilibrate through these products prior to analysis as with typical menthol cigarettes. As a result, loss from vaporization during equilibration through the product is essentially avoided in these atypical cigarettes. Similarly, Natural American Spirit Green has atypical high amount of menthol from the mentholated cigarettes. As a result, these are three niche products that have significant differences from the other menthol cigarettes in this study. Excluding these three products, the overall menthol content range was narrower (2.9 to 7.2 mg/cigarette), with an overall average menthol content of 4.75 mg menthol /cigarette. For cigarette products not labeled as mentholated, the measured menthol content ranged from 0.002 to 0.07 mg/cig with an average menthol amount of 0.0183 mg/cigarette.

This study measured menthol content for 22 nonmenthol cigarette products. In several cigarette brands, products labeled as menthol cigarette and nonmenthol cigarette with same brand name were tested for menthol content. Using the ratio of menthol amounts between menthol and nonmenthol products is a straightforward approach for studying menthol application in cigarettes. The ratio of the amount of menthol in mentholated to non-mentholated cigarettes within a brand family was also considered, and ranged from 50 (Pyramid) to ca. 5,000 (Natural American Spirit) (see last column of Table 1). The non-mentholated Pyramid product had an approximately 6-fold higher level of apparently added menthol than measured in other non-mentholated cigarette brands. Although the levels of menthol in mentholated varieties of the Pyramid brand were comparable to other mentholated cigarettes, its non-mentholated product contained more menthol relative to other non-mentholated products. As a result, the ratio between mentholated and non-mentholated cigarette varieties tested for the Pyramid brand family was relatively low (~50). One mentholated product, Natural American Spirit Light-Green, had the highest measured menthol content (19.6 mg/cigarette). The "mellow" flavored Natural American Spirit Yellow, had a measured menthol level of 0.004 mg/cigarette. As a result, the menthol to non-menthol cigarette measured menthol ratio was the highest (~ 5,000). Overall, the average

ratio of the menthol content between mentholated and non-mentholated varieties of cigarettes for the same brand family was ~1000.

Very low menthol amounts were detected in several non-mentholated cigarettes and the 3R4F reference cigarette. The menthol content for four tested products (Fortuna Red: 0.0018 mg/cigarette; Natural American Spirit Yellow: 0.0040 mg/cig; and Rave Red: 0.0018 mg/cigarette, USA Gold: 0.0045 mg/cigarette) were close to the detection limit of the analytical method (0.0008 mg/cigarette). The relative standard deviations from seven replicates of those four products were relatively high (24%, 25%, 31% and 15%). The menthol content is 0.0058 mg/cigarette in 3R4F reference cigarettes while the menthol content of the 1R5F reference cigarette was below the detection limit.

The effect of product packaging on menthol content was examined. Menthol content for three mentholated products (Marlboro Menthol King, Newport Gold King and Newport Green 100′s) was available for both flip-top box and soft pack varieties. The menthol content in Marlboro Menthol King was 4.41 mg/cigarette in the box case and 4.29 mg/cigarette in the soft pack. For Newport Gold King, menthol contents were 4.21 mg/cigarette in the box case and 4.85 mg/cigarette in the soft pack. For Newport Green 100′s, menthol contents were 5.30 mg/cigarette in the box case and 5.40 mg/cigarette in the soft pack. While comparative data were only available for three products, there was not a consistent pattern between packaging and menthol content. A possibility is that intra-brand flip top box and soft pack varieties may be from similar manufacturing batches. Alternatively, some manufacturers adjust the amount of menthol added to the product based on the physical properties of the packaging, Menthol retention was not examined for products whose packages were opened for an extended period of time.

DISCUSSION

The menthol amounts for a variety of U.S. marketed cigarettes have been determined. Because menthol migrates from the application point to other components of cigarettes after manufacturing, the menthol content in whole cigarettes has been measured. Among 23 mentholated products, menthol in whole cigarettes has a range of 2.9–19.6 mg/cigarette. Two Camel Crush products and the Natural American Spirit Light Green have extraordinarily high levels of menthol relative to the other mentholated products tested. If these three products are excluded, the menthol range is 2.9 – 7.2 mg/cigarette, which is higher, but comparable to the data provided by Celebucki[11] and Altria Client Service.[13] For 22 nonmenthol products, the measured menthol range is 1.8 – 73.5 μg/cigarette.

The menthol content in so-flavored cigarettes measured in this study is much higher than the amount needed to have a slight menthol sensory effect (1 – 2 mg/g[9,10] or approximately 0.6 – 1.5 mg/cigarette, based on a tobacco filler weight of 0.6 – 0.75 g/cigarette). Nineteen of the 21 menthol cigarettes (excluding two Camel Crush products) are tested to have menthol levels higher than that needed for a strong menthol effect (2.5 – 4.5 mg/g[9,10] or approximately 1.75 – 3.4 mg/cigarette). Tobacco manufacturers add more menthol to cigarettes which have ventilation or use high permeability paper.[13] Cigarette smoke is diluted by air when the ventilation is not blocked that results in a smoking machine low tar

yield. Similarly, menthol transfer yield to the mainstream smoke is also decreased by ventilation. Therefore tobacco manufacturers add more menthol to the cigarette to compensate the decreased smoke yield of menthol.[13] One example in this study is that Natural American Spirit Dark Green has a menthol level of 7.2 mg/cigarette Natural American Spirit Light Green, has a menthol level of 19.6 mg/cigarette.

The menthol range 1.8 to 73.5 μg/cigarette that was obtained for the twenty-two "nonmenthol labeled" cigarette products in this study are greater than the results obtained by Merkel et al. [16] cigarette. In the study by Merckel et. al.[16] the menthol range in tobacco filler of 32 Swiss nonmenthol cigarettes was 0.019 to 13.3 μg/cigarette. Menthol is typically added to non-menthol cigarettes at approximately 0.03 mg/g of tobacco filler[10] or approximately 20 μg/cigarette. This value is consistent with the mean (18 μg/cigarette) of the measured menthol contents of the 22 nonmenthol cigarettes in this study.

In the study of Merckel et al.[16] six cigarette products without any additives (no menthol added), have a range of measured menthol from 0.033 to 0.18 μg/g of cigarette filler with a mean value at 0.08 μg/g. Based on their results, the authors assumed that menthol was intentionally added to cigarettes when its concentration exceeded 0.23 μg/g (0.08 μg/g plus three standard deviations). [16] In the present study, the menthol levels measured in Fortuna Red, Natural American Spirit Yellow, USA Gold, and Rave Red (1.8 to 4.5 μg/cigarette), were lower than that measured menthol in reference cigarette 3R4F (5.8 μg/cigarette). All these values are much higher than 0.23 μg/g set by Merckel et al.[16], which may correspond to menthol that may be either naturally occurring or contamination at the manufacturing facility. According to the University of Kentucky reference cigarette program, no flavors, including menthol, are added to the reference cigarette 3R4F. [21] Absent additional information, the measured menthol in these reference cigarettes may either be due to naturally occurring menthol in the tobacco or from contamination of residual menthol during manufacturing from other cigarette products. As indicated by Altria Client Services, menthol and nonmenthol cigarettes can be manufactured in the same facility, which can cause trace amounts of menthol to be present in the nonmentholated cigarettes.[13] The menthol content in those four non-menthol cigarettes with menthol levels lower than that of reference cigarette 3R4F may also represent naturally occurring menthol in the tobacco or the carryover of residue menthol in the manufacturing facilities.

Menthol measured in nonmenthol cigarettes that is significantly higher than that of the 3R4F reference cigarette are likely to be intentionally added menthol in the manufacturing process. Among the 22 nonmenthol cigarette products measured in this study, 18 of them (excluding those four with menthol levels lower than that of 3R4F) have a measured menthol range of 9 – 73 μg/cigarette with a mean value of 22 μg/cigarette. The findings suggest that menthol may be added to cigarettes that are not labeled as menthol flavored, albeit at much lower concentrations than in products labeled as containing menthol.

The ratios of menthol content between the mentholated and the non-mentholated cigarettes within brands ranges from 50 for Pyramid, to nearly 5,000 for Natural American Spirit. For those cigarette brands with the ratio greater than 1,000-fold, the non-mentholated products may contain naturally occurring menthol in tobacco or residual menthol contamination at the

manufacturing facilities without any added menthol. In general, the ratios of menthol content between the mentholated and the non-mentholated cigarettes are approximately 100-fold for most cigarette brands included in this study.

In this study, three cigarette brands sold as hard pack and soft pack varieties showed minimal difference in measured menthol levels. Assuming comparable product age, this may indicate that this packaging difference does not affect menthol loss from cigarettes in an unopened product. More detailed studies, to include a larger sample base, the manufacturing date and shelf-storage duration, and conditions are needed to conclusively determine if packaging affects post manufacture menthol retention.

Perhaps most important, this study demonstrates with current commercial US cigarette products that menthol cigarettes and cigarettes not labeled as containing menthol all contain some menthol, but within fairly discrete ranges. In cigarettes not labeled to contain menthol as a characterizing flavor, the measured amounts were within a range which appears to be less than the previously reported threshold limit of 1 mg/g of cigarette filler (approximately 0.6 mg/cig) [9] to impart sensory effects. In contrast, the menthol flavored cigarettes in this study contain menthol in amounts that exceed sensory threshold amounts by 5- to 10-fold. Continued investigation may be useful to enhance understanding the implications of low levels of menthol in cigarettes.

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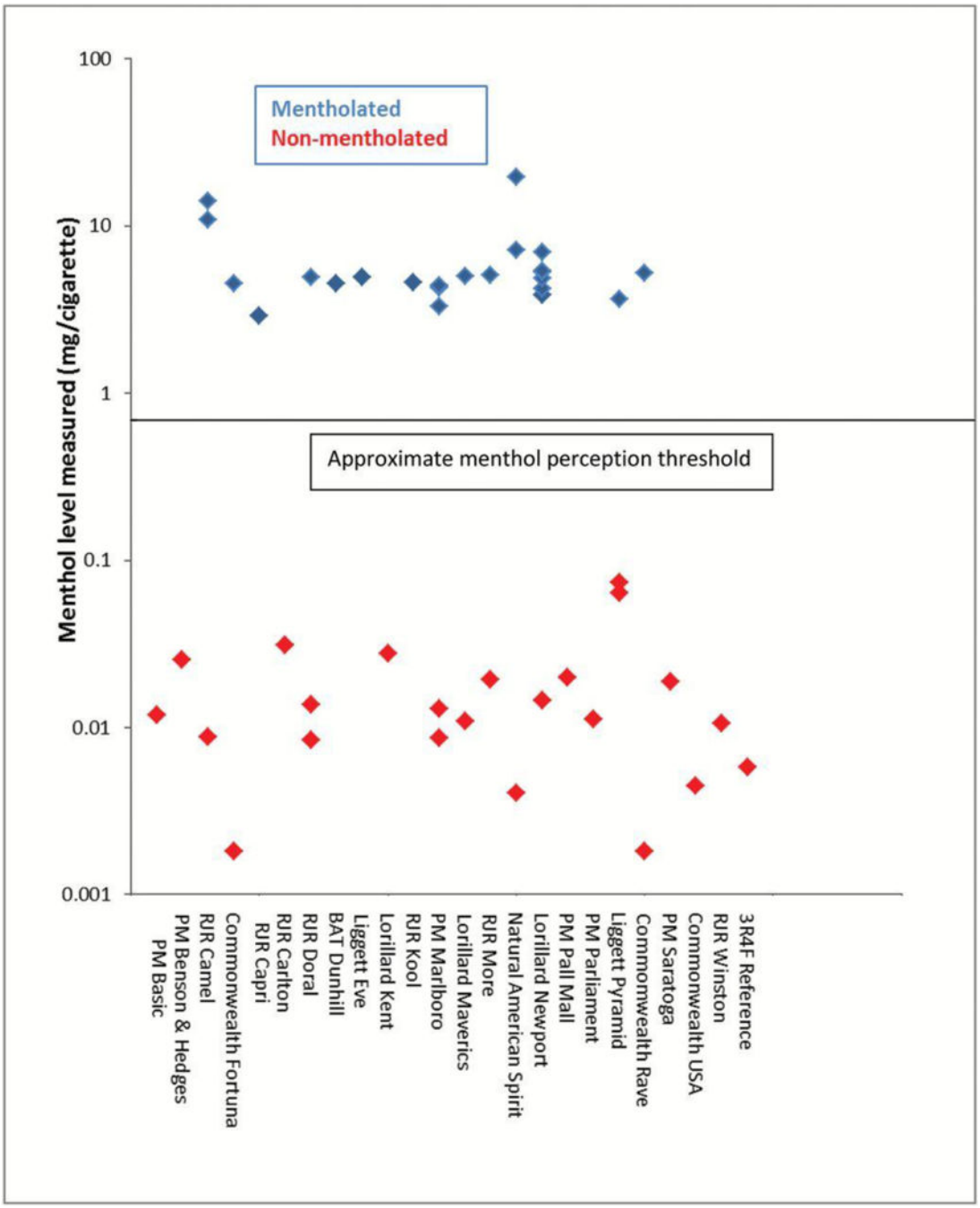

Figure 1.

Menthol content plot of 45 cigarette products and reference cigarette 3R4F in units of mg/cigarette. Blue dots are cigarettes labeled as mentholated. Red dots are cigarette products without menthol labeling. The vertical axis is in logarithm scale.

Table 1

Measured menthol content of commercial and reference cigarettes

Product	*Size*	*Pkg Type*	*Flavor*	*Mean Cigarette Mass (g)*	*Menthol Level (mg/cig)*	*RSD (%) (n=7)*[*]	*Menthol to Non-Menthol Ratio*[**]
Basic Red	King	Box	Regular	0.907	0.0119	14	N/A
Benson & Hedges Gold	100s	Box	Regular	1.09	0.0254	7.3	N/A
Camel Crush	King	Box	Menthol	0.928	14.1	2.8	1615
Camel Filters	King	Hard Pack	Regular	0.937	0.00873	4.6	
Camel Menthol	King	Box	Menthol	0.916	10.8	4.3	1237
Capri Green	N/A	Box	Menthol	0.564	2.88	5.1	N/A
Carlton	100s	Box	Regular	0.933	0.0310	11	N/A
Doral Gold	King	Box	Menthol	0.881	4.92	6.2	
	King	Box	Regular	0.878	0.00846	11	581
Doral Red	King	Box	Regular	0.900	0.0137	12	359
Dunhill Dark Green	N/A	Box	Menthol	1.02	4.51	6.7	N/A
Eve Turquoise	Slim 120s	Box	Menthol	0.925	4.91	5.2	N/A
Fortuna Green	King	Box	Menthol	0.900	4.55	8.3	2513
Fortuna Red	King	Box	Regular	0.806	0.00181	24	
Kent Gold	100s	Soft Pack	Regular	1.10	0.0278	5.8	N/A
Kool	King	Box	Menthol	0.960	4.58	4.6	N/A
Marlboro	King	Box	Menthol	0.891	4.41	6.1	507
	King	Box	Menthol	0.916	4.29	8.2	493
Marlboro Blue	King	Box	Menthol	0.884	3.29	5.6	378
Marlboro Red	King	Box	Regular	0.915	0.0129	5.0	N/A
Marlboro Silver	King	Box	Regular	0.861	0.00870	9.5	
Maverick Green	King	Box	Menthol	0.937	4.98	3.8	257
Maverick Red	King	Box	Regular	0.905	0.0109	7.4	
More	120s	Soft Pack	Menthol	0.977	5.11	5.3	263
	120s	Soft Pack	Regular	0.989	0.0194	5.0	
Natural American Spirit Dark Green	King	Box	Menthol	1.13	7.20	6.4	1791

Product	*Size*	*Pkg Type*	*Flavor*	*Mean Cigarette Mass (g)*	*Menthol Level (mg/cig)*	*RSD (%) (n=7)**	*Menthol to Non-Menthol Ratio***
Natural American Spirit Light Green	King	Box	Menthol	1.18	19.6	6.4	4875
Natural American Spirit Yellow	King	Box	Mellow	1.09	0.00402	25	
Newport Gold	King	Box	Menthol	0.907	4.21	3.1	290
	King	Soft Pack	Menthol	0.953	4.85	4.7	334
	100s	Box	Menthol	1.10	6.94	4.8	N/A
	King	Box	Regular	0.883	0.0145	14	N/A
Newport Green	King	Box	Menthol	0.932	3.84	8.0	N/A
	100s	Box	Menthol	1.11	5.30	5.5	N/A
	100s	Box	Menthol	1.09	5.40	3.6	N/A
Pall Mall Red	King	Box	Regular	0.961	0.02	9.1	N/A
Parliament Blue	King	Box	Regular	0.898	0.011	15	N/A
Pyramid Blue	100s	Box	Regular	1.01	0.073	9.9	50
Pyramid Gold	100s	Box	Menthol	1.02	3.67	6.3	
Pyramid Red	100s	Box	Regular	1.00	0.063	21	58
Rave Green	100s	Box	Menthol	0.915	5.20	9.5	2889
Rave Red	King	Box	Regular	0.773	0.0018	31	
Saratoga	120s	Box	Regular	1.13	0.019	7.5	N/A
USA Gold Red	King	Box	Regular	0.780	0.0045	15	N/A
Winston Red	King	Box	Regular	0.900	0.011	10	N/A
1R5F	N/A	Soft Pack	Regular	0.843	<LOD	N/A	N/A
3R4F	N/A	Soft Pack	Regular	1.038	0.0058	19	N/A

* n=7 means seven replicates with separate sampling from extraction to GC/MS analysis.

** For a given brand, the ratio was calculated by dividing the menthol level of the menthol product with that of non-menthol product.

Table 2

Method validation for menthol including precision and accuracy.

Spike Level	*Average (µg/g)*	*Std. Dev. (µg/g)*	*Calculated Conc. (µg/g)*	*Precision (CV)*	*Accuracy (Recovery)*
1 µg/g	1.00	0.13	1.01	13%	99.0%
5 µg/g	5.02	0.21	5.05	4.2%	99.5%
252 µg/g	277.5	5.60	252.3	2.02%	110.0%
757 µg/g	856.4	14.7	756.8	1.72%	113.2%
5045 µg/g	5479.1	128.5	5045.4	2.35%	108.6%