

Waterpipe Tobaccos, Part I. Composition of Forty Brand-Styles of Contemporary Waterpipe Tobacco Available on the US Market in 2020–2022 *

by

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SUMMARY

The compositions of waterpipe tobaccos available for retail sale on the US market are relatively unknown compared with more popular products such as cigarettes. Indeed, the phrase “waterpipe tobaccos” is used only in some governmental regulations and in journal articles dealing with regulatory aspects of those products. Commercially, the terms “shisha”, “hookah tobaccos”, and “flavored tobaccos” are used. In addition to the differences in commercial terminology, there is also confusion about the composition of such tobaccos, with the term “waterpipe tobacco” also being applied to products on the market that do not contain enough glycerol to prevent combustion during use. Therefore, during the past several years, 40 samples of shisha products on the US market were sent to an ISO 17025 accredited laboratory for the determination of glycerol, propylene glycol, fructose, glucose, and sucrose, in addition to the usual tobacco analytes and water using the Karl Fischer method. Moreover, two surrogate samples of shisha tobacco, one based on flue-cured tobacco and the other based on dark air-cured tobacco, were analyzed by the same laboratory along with samples of the starting tobaccos. The main finding from these analyses was that there were two very different types of shisha tobaccos on the market. One type was based on dark air-cured tobacco and the other type was based on flue-cured tobacco. Among the brand-styles based on flue-cured tobacco, some had higher levels of glycerol and lower levels of added

sugars than others that had higher added sugars and lower levels of glycerol. Another important point of differentiation was that the products based on dark air-cured tobacco had much smaller tobacco particle sizes than did those based on flue-cured tobacco. The results of this research as well as other research that will be presented in two subsequent reports showed that waterpipe tobaccos cannot be considered as a single product category. This is particularly true for the determination of emissions using the instrumentation specified in ISO 22486:2019 (Water pipe tobacco smoking machine — Definitions and standard conditions). [Contrib. Tob. Nicotine Res. 33 (2024) 148–156]

KEYWORDS

Waterpipe tobacco; shisha; hookah tobacco; flavored tobacco, glycerol; propylene glycol; fructose; glucose; sucrose; water; flue-cured tobacco; dark air-cured tobacco

ZUSAMMENFASSUNG

Die Zusammensetzung von Wasserpfeifentabaken, der auf dem US-amerikanischen Markt für den Einzelverkauf angeboten werden, ist im Vergleich zu populäreren Produkten wie Zigaretten relativ unbekannt. Tatsächlich wird der Begriff “Wasserpfeifentabak” nur in einigen staatlichen Verordnungen und in Zeitschriftenartikeln verwendet, die

*Received: 19th October 2023 – accepted: 15th February 2024

sich mit regulatorischen Aspekten dieser Produkte befassen. Im Handel werden die Begriffe “Shisha”, “Hookah-Tabak” und “aromatisierte Tabake” verwendet. Zusätzlich zu den Unterschieden in der kommerziellen Terminologie gibt es auch Unklarheiten bezüglich der Zusammensetzung solcher Tabake, wobei der Begriff “Wasserpfeifentabak” auch für Produkte auf dem Markt verwendet wird, die nicht genügend Glycerin enthalten, um die Verbrennung während des Gebrauchs zu verhindern. Daher wurden in den vergangenen Jahren 40 Proben von Shisha-Produkten auf dem US Markt an ein nach ISO 17025 akkreditiertes Labor geschickt, um neben den üblichen Tabakanalysen und Wasser auch Glycerin, Propylenglykol, Fruktose, Glukose und Saccharose nach der Karl-Fischer-Methode bestimmen zu lassen. Darüber hinaus wurden zwei Surrogatproben von Shisha-Tabak, eine auf der Basis von flue-cured Tabak und die andere auf der Basis von dunklem luftgetrocknetem Tabak, vom selben Labor zusammen mit Proben der Ausgangstabake analysiert. Die wichtigste Erkenntnis aus diesen Analysen war, dass es zwei sehr unterschiedliche Arten von Shisha-Tabak auf dem Markt gibt. Der eine Typ basiert auf dunklem, luftgetrocknetem Tabak, der andere auf flue-cured-Tabak. Bei den Markenprodukten auf der Basis von flue-cured-Tabak wiesen einige einen höheren Glycerin- und einen niedrigeren Gehalt an zugesetztem Zucker auf als andere, die einen höheren Gehalt an zugesetztem Zucker und einen niedrigeren Glycerin-Gehalt hatten. Ein weiteres wichtiges Unterscheidungsmerkmal war, dass die Produkte auf der Basis von dunklem, luftgetrocknetem Tabak viel kleinere Tabakpartikelgrößen aufwiesen als die Produkte auf der Basis von flue-cured-Tabak. Die Ergebnisse dieser und anderer Untersuchungen, die in zwei weiteren Berichten vorgestellt werden, zeigen, dass Wasserpfeifentabake nicht als eine einzige Produktkategorie betrachtet werden können. Dies gilt insbesondere für die Bestimmung der Emissionen mit den in ISO 22486:2019 spezifizierten Instrumenten. [Contrib. Tob. Nicotine Res. 33 (2024) 148–156]

RESUME

Les compositions des tabacs pour pipes à eau disponibles pour la vente au détail sur le marché américain sont relativement peu connues par rapport à des produits plus populaires tels que les cigarettes. En effet, l'expression “tabacs pour pipe à eau” n'est utilisée que dans certaines réglementations gouvernementales et dans des articles de journaux traitant des aspects réglementaires de ces produits. Dans le commerce, les termes shisha, tabacs de narguilé et tabacs aromatisés sont utilisés. Outre les différences de terminologie commerciale, il existe également une confusion quant à la composition de ces tabacs, le terme “tabac pour pipe à eau” étant également appliqué aux produits commercialisés qui ne contiennent pas suffisamment de glycérol pour empêcher la combustion pendant l'utilisation. C'est pourquoi, au cours des dernières années, 40 échantillons de produits de chicha sur le marché américain ont été envoyés à un laboratoire accrédité ISO 17025 pour la détermination du glycérol, du propylène glycol, du fructose, du glucose et du saccharose, en plus des analytes habituels du tabac et de l'eau, à l'aide de la méthode Karl

Fischer. En outre, deux échantillons de substitution de tabac à chicha, l'un basé sur du tabac flue-cured et l'autre sur du tabac dark air-cured, ont été analysés par le même laboratoire en même temps que des échantillons du tabac original. La principale conclusion de ces analyses est qu'il existe deux types de tabacs à chicha très différents sur le marché. Un type était basé sur du tabac brun séché à l'air et l'autre sur des tabacs séchés à la flamme. Parmi les styles de marque basés sur le tabac flue-cured, certains présentaient des teneurs en glycérol plus élevées et des teneurs en sucres ajoutés plus faibles que d'autres qui présentaient des teneurs en sucres ajoutés plus élevées et des teneurs en glycérol plus faibles. Un autre point de différenciation important est que les produits à base de tabacs bruns séchés à l'air ont des particules de tabac beaucoup plus petites que les produits à base de tabacs séchés à la flamme. Les résultats de cette recherche ainsi que d'autres recherches qui seront présentées dans deux rapports ultérieurs ont montré que les tabacs de pipe à eau ne peuvent pas être considérés comme une catégorie de produits unique. Cela est particulièrement vrai pour la détermination des émissions à l'aide de l'instrumentation spécifiée dans la norme ISO 22486:2019. [Contrib. Tob. Nicotine Res. 33 (2024) 148–156]

INTRODUCTION

Based on references given in SHIHADDEH *et al.* (1) and MAZIAK *et al.* (2), the predecessors of the shisha products currently on the US market were developed during the 1990's, with the key difference between the new products and the prior products being sufficient glycerol in the composition to prevent combustion of the tobacco and provide the source of the aerosol that was produced when the shisha was heated (3, 4). There were several articles on toxicants in shisha smoke in the following years until the publication by SCHUBERT on relationship between glycerol (VG) and propylene glycol (PG) levels in waterpipe tobaccos and carbonyl levels in smoke when the products were heated (5). However, only two commercial samples from the same manufacturer (Nakhla) were included in the sample set. VG, PG, and nicotine levels in another series of Nakhla products were reported by WAGENER *et al.* (6) in 2019. According to Supplemental Table 1 in WAGENER *et al.* (6), nicotine, propylene glycol, and glycerol by flavor and condition, products listed as having humectants, had VG contents ranging from 15% to 37%, while those products listed as not having humectants, had VG contents ranging from 1.6% to 24%. Data on VG and PG levels in a set of commercial products were reported by JACCARD and coworkers in 2020 (7). However, that report did not identify the brand-styles associated with the analytical data, although it is likely that all brand-styles listed in JACCARD's Table 1 except for T7, T9, and T10 contained sufficient VG and PG to perform in a similar manner to the leading brands of contemporary waterpipe tobaccos available for sale in the US. By contemporary brands, we mean waterpipe tobaccos that do not combust when used as intended in a waterpipe. It is important to note that in US retail markets (local smoke shops as well as internet-based merchants), the terms “waterpipe”, and “waterpipe tobacco”, are not used. A waterpipe is known as a “hookah” or “hookah pipe”. Water-

pipe tobacco is known as “shisha”, “hookah tobacco”, or “flavored tobacco”. There is no indication that unflavored shisha is available commercially. However, it can be produced in the laboratory (see “MATERIALS AND METHODS”). The aerosol (smoke) from unflavored shisha is without taste.

Several years ago, we were approached by a representative of several small manufacturers and importers of shisha, and we were asked how they should get information on the chemical composition of their products. We suggested use of an ISO 17025 accredited laboratory that could do the traditional tobacco analytes as well as VG, PG, fructose, glucose, sucrose, and water by the Karl Fischer method. We also suggested that samples of relevant competitive products be sent to the same laboratory too. Furthermore, we obtained samples of the products and the tobaccos used to make them for our own analytical work. In addition, we prepared a flue-cured (FC) surrogate shisha and a dark air-cured (DAC) surrogate shisha. They were sent to the same laboratory for analysis along with samples of the starting tobaccos. The results of the laboratory analyses are shown in Table 1 (see page 153).

The following codes are used in Table 1.

BQL Results below the limit of quantitation for that analyte
 NM Results of calculation are not meaningful when one or more of the needed values were not reported or below the limit of quantitation for an analyte
 NR Information not reported by the laboratory or analyses were not run.

The data in Table 1 show that there were major differences among the waterpipe tobaccos analyzed. These differences will be explained the “RESULTS AND DISCUSSION” section of this report.

MATERIALS AND METHODS

As noted earlier, the samples analyzed by the laboratory came from several manufacturers and importers of waterpipe tobaccos. These were contemporary waterpipe tobaccos in that they were all flavored and contained sufficient glycerol or a mixture of glycerol and propylene glycol such that they would be heated, but not combusted during normal operation of a waterpipe. In addition, we asked those organizations to provide samples of their starting tobaccos. We received several samples of flue-cured tobacco and one sample of dark air-cured tobacco. The analytical data we received with the samples are shown below in Table 2.

Table 2. Analytical data for flue-cured (FC) and dark air-cured (DAC) tobaccos.

Sample	Moisture (%)	Alkaloids (% DWB)	Reducing sugars (% DWB)	Total sugars (% DWB)
FC 1	13.6	0.88	22.1	NR
FC 2	NR	0.82	19.2	29.2
FC 3	12.5	0.97	NR	32
DAC 1	NR	0.80	NR	NR

NR means the laboratory did not report a result for that analyte.

A typical formula for shisha is 60% glycerol, 20% tobacco, 10% invert sugar syrup, and 10% propylene glycol (PG). This PG simulates the PG and flavor concentrates that are used when making a shisha that has flavors with low transfer rates such as *para*-tolualdehyde, *cis*-3-hexen-1-ol, benzaldehyde, *para*-anisaldehyde, and menthol (8). While glycerol levels in commercial products range from 30 to 70% of product weight, we made our recipe with 60% glycerol (VG).

The experimental procedure for the preparation of the high-VG FC surrogate shisha is as follows:

1. Glycerol (600 g) and invert sugar syrup (100 g) were mixed in 5-qt Pyrex bowl of KitchenAid KSM155GBCA Mixer and Flat Beater at slowest speed on the mixer.
2. FC tobacco (200 g) was added to the mixture of glycerol and invert sugar syrup in 50-g portions with the mixture being stirred as the tobacco was being added.
3. Propylene glycol (100 g) was then added with stirring.
4. After adding the propylene glycol, the mixture was still too thick for good mixing and water (100 g) was added.
5. The beater was removed from the mixing bowl and the bowl was covered with aluminum foil.
6. After 24 h, the mixture was stirred for a short time and the shisha packed off into mason jars containing 500 g of product (2 jars) with the remainder placed in a third jar for in-house analytical work.

A similar surrogate product was made with DAC tobacco.

1. Glycerol (420 g) and invert sugar syrup (70 g) were mixed in 5-qt Pyrex bowl of KitchenAid KSM155GBCA Mixer and Flat Beater at slowest speed on the mixer. Then water (70 g) was added and stirred in.
2. Attached stem was removed from DAC tobacco before use. Next two 70-g portions of the destemmed leaf were added to the mixture of glycerol and invert sugar syrup with the mixture being stirred as the tobacco was being added.
3. Propylene glycol (70 g) was then added with stirring.
4. The beater was removed from the mixing bowl and the bowl was covered with aluminum foil.
5. After 24 h, the mixture was stirred for a short time and the shisha packed off into a mason jar containing 500 g of product with the remainder placed in a second jar.

Unlike the FC surrogate shisha and commercial FC shishas, the DAC surrogate was composed of small particles as is the case with commercial DAC shishas. Also, on standing, there was a noticeable separation between the liquid phase on the bottom of the container and the particles above. This is typical behavior for many commercial DAC shishas. The FC tobacco used in many shisha products is specially grown and processed to have, “high absorption capacity of molasses” (9). Molasses is a shisha industry term that refers to sugar syrups of all types added to the tobacco.

There are several factors to consider when reviewing the data in Table 1. First, the samples are “point-in-time” samples. They may or may not be representative of the products available to consumers. There are no standards for the collection and processing of samples such as there are for cigarettes as given in ISO 8243 (10). Shisha products

tend to separate into a tobacco-rich portion on top and a liquid-rich portion on the bottom once the products are packaged. As with sampling, there are no generally accepted procedures for sampling the samples of shisha once they are removed from the packaging. Moreover, there are no standard methods applicable for the sampling and analysis of shisha such as the CORESTA Recommended Methods (CRM) (11). The closest method is CRM 61 (12). The laboratory reported that it used a method similar to that and validated it for the ranges of VG, PG, fructose, glucose, and sucrose found in the shisha samples.

RESULTS AND DISCUSSION

Table 1 shows the results of the analyses performed by the ISO 17025 accredited laboratory. The laboratory selected the analytes that in its view were essential for characterizing waterpipe tobaccos. These analytes included some of the harmful and potentially harmful constituents (HPHC) typically determined on the tobacco-containing products as well glycerol, propylene glycol, fructose, glucose, and water (not oven moisture). There are other analytes in greater concentration than some of those just mentioned, but they were not on the list recommended by the laboratory. These include the higher saccharides such as found in corn syrup, flavor carriers such as ethanol and triacetin, and citrus oils that are both flavors in themselves as well as solvents for other flavors.

Unlike most other tobacco-containing products, the tobacco content of the commercial and surrogate products listed in Table 1 is less than 30% of product weight, and in some cases it is less than 20% of product weight. The purpose of the tobacco is to serve as a carrier for the glycerol that is needed to form the aerosol and to provide a source of nicotine. Glycerol is most always present at more than 30% of product weight, but many products have glycerol contents between 45 and 60%. The main purpose of the glycerol is to provide the aerosol that is the smoke produced by heating the shisha with charcoal briquettes or electric heaters. The reported transfer rate for glycerol is 17% (8) under the conditions specified in ISO 22486:2019 (20) and ISO TS 22487 (21). Thus, a glycerol inclusion rate of 30% should provide sufficient glycerol for a 10 g sample to provide aerosol for smoking under the conditions specified in the aforementioned ISO documents. One manufacturer, M2 in Table 1, added an approximately equal weight of propylene glycol to its loading of glycerol. Propylene glycol has a much lower boiling point than glycerol and a much higher transfer rate than does glycerol (8). This allows the smoker to get early puffs as the shisha is heated to the temperature required for glycerol alone. Inspection of the data in Table 1 for Manufacturers M1 through M11, show that some manufacturers use a higher proportion of sugar syrups than others and consequently have a lower proportion of glycerol when not used in conjunction with propylene glycol as in the case of M2. Added sugar syrups (i.e., high fructose corn syrup, partially inverted sucrose syrup, corn syrup) have two functions. Generally, 5–10% added sugar syrup is needed to hold the

tobacco particles together when the user transfers the product from the package to the bowl of the hookah pipe, and to minimize the loss of tobacco particles during smoking when Egyptian-style bowls are used [Egyptian-style bowls are the commercial equivalent of the waterpipe tobacco holder specified in ISO 22486:2019 (20)]. Added sugars in excess of 5–10% are used to dilute the tobacco so that nicotine concentration is about 0.1%.

Also, unlike most other tobacco products, waterpipe tobaccos require mixing before use, as some of the tobacco particles, even though they are coated with glycerol, sugar syrups, and flavors tend to float on that mixture when allowed to stand for a day or so. However, even if samples are finely ground by processing through a food processor such as the Robot Coupe Blixer 2, there is still separation of the phases. Another factor to consider when reviewing the data in Table 1 is that we have no information on the batch-to-batch variation when two different batches of the same flavor product are analyzed. By different batches, we mean different lots of starting tobacco, different preparation of the base blend, and different flavoring and packaging operation.

The products of the first eleven manufacturers (M1–M11) manufacture or import products based on FC tobacco. Reportedly, the FC tobaccos used in the manufacture of waterpipe tobacco are grown and cured under conditions that keep nicotine levels around 1% and reducing sugar levels in excess of 20% (9). Based on the data in Table 1 and in the data in JACCARD's Table 1 (7) (for all his samples except T7, T9, and T10), most manufacturers of FC products are targeting a nicotine level of 0.1%. Since most manufacturers offer a wide variety of flavors, and some of these flavors involve changing the type and amounts of flavors and flavor carriers, relatively consistent nicotine levels across a manufacturer's product line may be the best indication of manufacturing consistency. Products from manufacturers M1 through M7 exemplified such consistency. Products from M8 showed greater variation not only in nicotine content, but also in levels of glycerol. We do not have any information to explain the compositional variations reported for M8's products. Products from Manufacturers M9 and M10 appear similar to other FC-based products. Products from M11 are unique in that the fructose content is much higher than the glucose and sucrose contents. We have no information to explain the high fructose content found in M11's products.

Manufacturers M12 and M13 specialize in manufacture of waterpipe tobaccos based on dark air-cured tobaccos. There are no detectable sugars in DAC tobaccos. Thus, the sugars found in the one sample from M12 and the two samples from M13 come from added sugars. In the case of M12, the sugar source may have been partially inverted sucrose syrup. The sugar source for the two M13 products cannot be determined based on the available data.

The analytical data for the two surrogate samples compare favorably with the starting tobaccos and ingredients added to those tobaccos. The high tobacco for the DAC surrogate shisha relative to the commercial DAC products may be due to the fact that no citric acid was added to the formulation. Citric acid is believed to be a common additive use in the manufacture of DAC shishas.

CONCLUSIONS

The data in this report shows that shisha (waterpipe) tobaccos vary widely in their composition and physical properties. It is likely that such differences in composition will adversely affect any attempts to determine emissions using the instrumentation specified in ISO 22486:2019. Moreover, the data in this report show that the analytical ranges for ingredients added to tobacco during shisha manufacture are outside those that have been validated for existing consensus standard methods. Furthermore, new methods are needed to determine levels of other commonly used ingredients such as corn syrup, triacetin, citric acid, and the citrus oils that are used as both flavors and carriers of other flavors.

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Table 1.

	Report Date (mmmm-yy)	Sep-20	Sep-20	Sep-20	Aug-20	Apr-20	Apr-20	Sep-20	Aug-20	Aug-20	Aug-20	Aug-20
1	Mfg.	M1	M1	M1	M2	M2	M2	M2	M3	M3	M3	M3
2	Shisha Flavor	Lemon	Lemon/mint	Berry	NR	NR	Melon	Melon	Mint	Berry/mint	Fruit/mint	Melon/mint
3	SIZE	1 kg	1 kg	1 kg	NR	NR	NR	100 g	250 g	250 g	250 g	250 g
4	FC or DAC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC
5	VG (%)	42.8	46.3	39.0	31.9	30.7	27.2	29.3	53.8	50.8	52.2	48.5
6	PG (%)	0.987	3.05	5.77	24.8	NR	30.3	28.4	2.26	3.26	2.50	2.07
7	H2O (%)	9.50	10.2	9.30	5.86	7.85	8.08	15.0	9.55	9.75	9.68	9.32
8	VG+PG+H2O (%)	53.3	59.6	54.1	62.6	NM	65.6	72.7	65.6	63.8	64.4	52.6
9	FRU (%)	8.19	8.66	8.71	2.17	2.80	1.88	1.92	6.92	7.63	7.00	6.40
10	GLU (%)	8.22	8.50	8.87	2.29	3.04	1.57	1.91	9.05	9.42	8.8	8.95
11	FRU+GLU (%)	16.4	17.2	17.6	4.46	5.84	3.45	3.83	16.0	17.1	15.8	15.4
12	Major (%)	71.1	77.2	72.0	75.6	NM	79.2	87.1	84.0	83.3	82.6	70.5
13	SUC (%)	1.39	0.410	0.324	8.55	10.2	10.2	10.6	2.36	2.38	2.45	2.42
14	NH3 (µg/g)	11.2	BQL	BQL	57.5	65.0	72.9	BQL	32.2	31.1	30.7	35.2
15	NIC (mg/g)	1.17	1.33	1.27	1.03	0.945	0.81	0.735	0.939	1.13	1.07	0.895
16	NIC (%)	0.12	0.13	0.13	0.10	0.09	0.08	0.07	0.09	0.11	0.11	0.09
17	pH	4.60	5.25	4.83	5.58	5.40	5.42	5.39	3.98	3.96	3.98	3.97
18	NNN (µg/g)	BQL	0.03	BQL	BQL	BQL	BQL	BQL	0.015	0.013	0.014	0.017
19	NNK (µg/g)	BQL	0.050	BQL	BQL	BQL	BQL	BQL	0.013	0.014	0.015	0.02
20	As (µg/g)	0.064	0.087	BQL	0.05	BQL	BQL	BQL	0.066	0.061	0.074	0.058
21	Cd (µg/g)	0.533	0.261	0.558	0.648	NR	0.234	0.491	0.242	0.227	0.30	0.019
22	Cr (µg/g)	0.169	0.131	0.175	0.098	NR	0.141	0.102	0.064	0.055	0.051	0.056
23	Pb (µg/g)	0.085	0.072	0.106	0.071	NR	0.083	0.081	0.169	0.162	0.164	0.127

Rows are as follows:

- Code number for manufacturer
- Shisha flavor as recorded by the laboratory
- Package size for the samples received as recorded by the laboratory
- FC (flue-cured) or DAC (dark air-cured) as determined from the label or other information
- VG (%)
Percent glycerol on an as-received basis
- PG (%)
Percent propylene glycol on an as-received basis
- H2O (%)
Percent water on an as-received basis
- VG+PG+H2O (%)
This value is an indication of the amount of volatile materials in the product
- FRU (%)
Percent fructose on an as-received basis
- GLU (%)
Percent glucose on an as-received basis
- FRU+GLU (%)
This value is an indication of the amount of invert sugar or HFCS added
- Major (%)
This value is an estimate of the percentage of the product that is not tobacco
- SUC (%)
- NH3 (µg/g)
- NIC (mg/g)
- NIC (%)
- pH
- NNN (µg/g)
- NNK (µg/g)
- As (µg/g)
- Cd (µg/g)
- Cr (µg/g)
- Pb (µg/g)
- Percent sucrose on an as-received basis
- Ammonia content in micrograms per gram on an as-received basis
- Nicotine content in milligrams per gram on an as-received basis
- Percent nicotine in an as-received basis
- pH of aqueous extract of product on an as-received basis
- Nitrosomnicotine content in micrograms per gram on an as-received basis
- (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone content in micrograms per gram on an as-received basis
- Arsenic content in micrograms per gram on an as-received basis
- Cadmium content in micrograms per gram on an as-received basis
- Chromium content in micrograms per gram on an as-received basis
- Lead content in micrograms per gram on an as-received basis

Table 1. Continued

	Report Date (mmm-yy)	Sep-20	Apr-21	Apr-21	Jun-21	Sep-20	Sep-20	Sep-20	Sep-20
1	Mfg.	M4	M4	M4	M4	M5	M6	M7	M7
2	Shisha Flavor	Vanilla	Vanilla/mint	Fruit/guava	Vanilla/mint	Vanilla	Vanilla	Vanilla	Apple
3	SIZE	250 g	250 g	250 g	250 g	250 g	250 g	250 g	250 g
4	FC or DAC	FC	FC	FC	FC	FC	FC	FC	FC
5	VG (%)	70.3	56.8	57.5	42.6	41.1	30.1	41.3	43.8
6	PG (%)	4.29	4.98	4.04	4.07	3.17	8.43	7.83	2.86
7	H2O (%)	5.21	9.64	8.96	12.5	10.0	13.9	14.2	13.5
8	VG+PG+H2O (%)	79.8	71.4	70.5	59.2	54.3	52.4	63.3	60.2
9	FRU (%)	0.676	1.75	1.61	2.37	14.4	9.83	6.99	6.79
10	GLU (%)	0.927	3.67	3.46	4.53	9.79	12.1	7.69	7.45
11	FRU+GLU (%)	1.60	5.42	5.07	6.90	24.2	21.9	14.7	14.2
12	Major (%)	86.4	79.1	77.8	70.7	79.9	75.1	81.5	82.7
13	SUC (%)	5.02	2.24	2.20	4.67	1.46	0.741	3.47	8.33
14	NH3 (µg/g)	23.1	52.7	51.4	70.0	23.7	BQL	44.5	35.6
15	NIC (mg/g)	0.659	0.924	0.997	1.03	1.11	1.10	0.691	0.792
16	NIC (%)	0.07	0.09	0.10	0.10	0.11	0.11	0.07	0.08
17	pH	5.09	4.97	4.57	5.33	5.60	4.96	5.08	4.49
18	NNN (µg/g)	BQL	0.005	BQL	BQL	0.007	0.022	0.007	0.015
19	NNK (µg/g)	BQL	BQL	BQL	BQL	0.01	0.039	0.008	0.014
20	As (µg/g)	BQL	BQL	BQL	0.066	BQL	0.053	BQL	BQL
21	Cd (µg/g)	0.429	0.489	0.499	0.492	0.393	0.443	0.207	0.149
22	Cr (µg/g)	0.098	0.258	0.245	0.461	0.063	0.055	0.164	0.131
23	Pb (µg/g)	BQL	0.094	0.094	0.114	0.133	0.072	0.055	BQL

Table 1. Continued

	Report Date (mm-yy)	Feb-21	Feb-21	Feb-21	Feb-21	Feb-21	Feb-21	Feb-21	Feb-21	Feb-22	Feb-22	Feb-22	Sep-20	Sep-20	Sep-20
1	Mfg.	M8	M8	M8	M8	M8	M8	M8	M8	M8	M8	M8	M9	M9	M9
2	Shisha Flavor	Lemon/mint	Banana	Apple	Grape	Guava	Berry	Apple	Lemon/mint	Candy	Lemon/mint	Apple	Apple	Apple	Melon
3	SIZE	250 g	50 g	50 g	50 g	250 g	250 g	250 g	250 g	250 g	100 g	100 g	100 g	100 g	100 g
4	FC or DAC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC	FC
5	VG (%)	42.2	41.3	43.2	37.1	38.3	37.6	32.8	33.1	29.9	44.3	46.1	44.5	46.1	44.5
6	PG (%)	BQL	2.01	BQL	7.55	55.8	7.31	0.521	0.289	0.504	BQL	1.38	1.37	1.38	1.37
7	H2O (%)	9.08	11.0	11.0	11.1	10.1	9.76	11.9	11.3	10.6	12.2	11.9	12.0	11.9	12.0
8	VG+PG+H2O (%)	51.3	54.3	54.2	55.8	55.8	54.7	45.2	44.7	41.0	56.5	59.4	57.9	59.4	57.9
9	FRU (%)	12.4	17.1	18.9	17.9	14.3	14.4	16.9	18.4	16.5	10.2	10.6	10.6	10.6	10.6
10	GLU (%)	10.8	15.3	15.9	15.2	11.3	11.4	13.9	15.3	15.5	10.7	11.2	11.1	11.2	11.1
11	FRU+GLU (%)	23.2	32.4	34.8	33.1	25.6	25.8	30.8	33.7	32.0	20.9	21.8	21.7	21.8	21.7
12	Major (%)	75.7	88.3	90.6	90.4	82.7	81.8	77.4	80.0	75.0	79.0	82.9	81.2	82.9	81.2
13	SUC (%)	1.26	1.55	1.63	1.55	1.3	1.29	1.40	1.62	1.99	1.55	1.70	1.61	1.70	1.61
14	NH3 (µg/g)	16.2	35.1	35.7	31.6	9.40	12.6	11.4	23.2	36.7	50.1	30.6	29.7	30.6	29.7
15	NIC (mg/g)	0.961	0.846	0.788	1.09	2.77	2.81	2.95	0.893	0.588	0.771	0.787	0.804	0.787	0.804
16	NIC (%)	0.10	0.08	0.08	0.11	0.28	0.28	0.30	0.09	0.06	0.08	0.08	0.08	0.08	0.08
17	pH	4.43	4.40	4.78	4.85	5.26	4.98	5.03	5.27	4.28	4.75	5.37	5.29	5.37	5.29
18	NNN (µg/g)	0.006	0.007	0.008	0.008	0.010	0.014	0.019	0.009	BQL	0.010	0.011	0.011	0.011	0.011
19	NNK (µg/g)	0.006	0.005	0.006	0.006	0.004	0.015	0.017	0.004	BQL	0.007	0.007	0.008	0.007	0.008
20	As (µg/g)	BQL	BQL	BQL	BQL	BQL	BQL	0.053	0.050	BQL	0.056	0.054	0.057	0.054	0.057
21	Cd (µg/g)	0.159	0.242	0.235	0.264	0.088	0.14	0.115	0.269	0.307	0.293	0.339	0.298	0.339	0.298
22	Cr (µg/g)	0.100	0.090	0.099	0.095	0.141	0.171	0.275	0.094	BQL	BQL	BQL	BQL	BQL	BQL
23	Pb (µg/g)	0.091	0.115	0.106	0.084	0.067	0.096	0.122	0.082	0.097	0.170	0.165	0.126	0.165	0.126

Table 1. Continued

	Report Date (mmm-yy)	Apr-20	Aug-20	Aug-20	Aug-20	Jan-21	Jan-21	Jun-21	Dez-20	Aug-20	Aug-20	Aug-20
1 Mfg.												
2 Shisha Flavor		M10	M11	M11	M11	M11	M11	M13	Surrogate	Surrogate	Leaf	Leaf
3 SIZE		Lemon/mint	Mint	Mint	Mint	Vanilla Mint	Apple	Berry	Unflavored	Unflavored	Tobacco	Tobacco
4 FC or DAC		250 g	250 g	250 g	250 g	250 g	250 g	250 g	NA	NA	NA	NA
5 VG (%)		FC	FC	FC	FC	FC	DAC	DAC	DAC	DAC	FC	DAC
6 PG (%)		NR	32.6	35.9	34.4	34.4	54.9	42.6	52.3	54.0	BQL	BQL
7 H2O (%)		10.1	2.19	4.10	4.10	1.50	2.20	4.97	8.66	8.17	BQL	BQL
8 VG+PG+H2O (%)		12.74	12.74	12.43	12.84	12.84	10.7	14.7	14.0	13.3	10.2	11.3
9 FRU (%)		NM	47.5	52.4	48.7	48.7	67.8	62.3	75.0	75.5	NM	NM
10 GLU (%)		9.68	28.2	29.3	28.9	28.9	2.98	0.493	2.39	1.19	BQL	BQL
11 FRU+GLU (%)		12.2	1.53	1.26	1.60	1.60	3.08	2.17	2.80	1.22	BQL	BQL
12 Major (%)		21.9	22.9	24.9	25.9	25.9	6.06	2.66	5.19	2.41	16.2	NM
13 SUC (%)		NM	71.8	78.5	75.9	75.9	81.4	67.2	85.9	81.9	NM	NM
14 NH3 (µg/g)		2.30	1.32	1.13	1.23	1.23	7.55	2.31	5.79	4.06	10.2	BQL
15 NIC (mg/g)		28.0	36.4	29.6	34.3	34.3	BQL	940	38.3	394	BQL	328
16 NIC (%)		1.09	1.37	1.33	1.03	1.03	3.07	2.19	1.27	0.953	7.09	6.04
17 pH		0.11	0.14	0.13	0.10	0.10	0.31	0.22	0.13	0.10	0.71	0.60
18 NNN (µg/g)		5.39	4.98	5.62	4.83	4.83	5.24	5.64	5.89	7.19	5.36	6.52
19 NNK (µg/g)		0.010	0.029	0.027	0.029	0.029	0.064	0.667	0.014	0.845	BQL	4.01
20 As (µg/g)		BQL	0.065	0.062	0.069	0.069	0.081	0.08	0.011	0.09	BQL	0.394
21 Cd (µg/g)		BQL	0.050	0.055	0.051	0.051	0.056	0.105	0.058	0.088	0.217	0.319
22 Cr (µg/g)		NR	0.259	0.262	0.275	0.275	0.259	0.184	0.417	0.149	2.19	0.643
23 Pb (µg/g)		NR	0.124	0.100	0.110	0.110	0.254	1.06	0.076	0.505	0.493	3.70
		NR	0.070	0.068	0.070	0.070	0.136	0.328	0.173	0.202	0.957	0.975