



October 31, 2022

Division of Dockets Management
Food and Drug Administration
5630 Fishers Lane, Room 1061
Rockville, MD 20852

Re: Valisure Citizen Petition on Benzene in Dry Shampoo Products

Dear Sir or Madam:

The undersigned, on behalf of Valisure LLC (“Valisure” or “Petitioner”), submits this Citizen Petition (“Petition”) pursuant to Sections 301(21 U.S.C. § 331), 601 (21 U.S.C. § 361), 602 (21 U.S.C. § 362), 702 (21 U.S.C. § 372), 704 (21 U.S.C. § 374), and 705 (21 U.S.C. § 375) of the Federal Food, Drug and Cosmetic Act (the “FDCA”), in accordance with 21 C.F.R. 10.20 and 10.30, to request the Commissioner of Food and Drugs (“Commissioner”) to issue a regulation, request recalls, revise industry guidance, and take such other actions set forth below.

A. Action Requested

Dry shampoo products are considered cosmetics that are regulated by the U.S. Food and Drug Administration (“FDA”). Valisure has tested and detected high levels of benzene in specific batches of certain dry shampoo products. The Centers for Disease Control and Prevention (“CDC”) states that the Department of Health and Human Services has determined that benzene causes cancer in humans.¹ The World Health Organization (“WHO”) and the International Agency for Research on Cancer (“IARC”) have classified benzene as a Group 1 compound thereby defining it as “carcinogenic to humans.”² FDA currently recognizes the high danger of this compound and lists it as a “Class 1 solvent” that “should not be employed in the manufacture of drug substances, excipients, and drug products because of their unacceptable toxicity ... However, if their use is unavoidable in order to produce a drug product with a significant therapeutic advance, then their levels should be restricted” and benzene is restricted under such guidance to 2 parts per million (“ppm”).³ The National Institute for Occupational Safety and Health (“NIOSH”) recommends protective equipment be worn by workers expecting to be exposed to benzene at concentrations of 0.1 ppm and defines “inhalation, skin absorption,

¹ Centers for Disease Control and Prevention, *Facts About Benzene* (2018) (<https://emergency.cdc.gov/agent/benzene/basics/facts.asp>)

² International Agency for Research on Cancer and World Health Organization, *IARC Monographs on the Identification of Carcinogenic Hazards to Humans* (<https://monographs.iarc.who.int/list-of-classifications>)

³ Food and Drug Administration, *Q3C – Tables and List Guidance for Industry* (2018) (<https://www.fda.gov/media/133650/download>)

ingestion, skin and/or eye contact” as exposure routes.^{4, 5} The Environmental Protection Agency (“EPA”) has estimated that lifetime exposure to benzene inhalation at 0.4 parts per billion (“ppb”), or 0.0004 ppm, will increase the risk of developing cancer in humans at the same 1 in 100,000 exposed persons rate as FDA uses to set regulatory limits on other trace impurities like N-nitrosamines.^{6, 7}

Valisure found multiple dry shampoo products that contain levels of benzene that significantly surpass the 2 ppm conditional FDA restriction for drugs. However, the dry shampoos tested are not drugs and contain no active pharmaceutical ingredient for therapeutic purpose; therefore, any significant detection of benzene could be deemed unacceptable. Furthermore, Valisure shows data from the analysis of benzene by directly sampling contaminated air after spraying dry shampoo products, which suggests potential for short- and long-term inhalation exposure to high levels of benzene. The presence of this known human carcinogen in dry shampoo products that are regularly used indoors and in large volumes makes this finding especially troubling.

This Petition requests that the Commissioner take the following actions:

- 1) request a recall of identified batches of dry shampoo cosmetic products on the basis that, due to contamination with a known human carcinogen, these products are adulterated under Section 601 of the FDCA (21 U.S.C. § 361) and misbranded under Section 602 (21 U.S.C. § 362);
- 2) review and update regulations and published guidance for cosmetic products to include limitations on various impurities that pose known risks to human health and include benzene in such updates, and potentially include clarification that there is no acceptable level of benzene in cosmetic products and establish a reasonable detection limit;
- 3) provide information to the public regarding these products under Section 705(b) of the FDCA (21 U.S.C. § 375(b));
- 4) develop guidance documents for the analysis of benzene in dry shampoo products;
- 5) develop guidance documents defining the mass of a standard daily total application of dry shampoo, which may include multiple discrete applications, so that a daily exposure of benzene can be calculated for dry shampoo products;

⁴ Centers for Disease Control and Prevention. *The National Institute for Occupational Safety and Health (NIOSH), Benzene* (October 30, 2019) (<https://www.cdc.gov/niosh/npg/npgd0049.html>)

⁵ Centers for Disease Control and Prevention. *The National Institute for Occupational Safety and Health, BENZENE: Systemic Agent* (2011) (https://www.cdc.gov/niosh/ershdb/emergencypresponsecard_29750032.html)

⁶ Environmental Protection Agency. *Benzene; CASRN 71-43-2.* (https://iris.epa.gov/static/pdfs/0276_summary.pdf)

⁷ Food and Drug Administration (February 2021). *Control of Nitrosamine Impurities in Human Drugs.* (<https://www.fda.gov/media/141720/download>)

- 6) consider working with the United States Environmental Protection Agency on a joint initiative to address benzene contamination and potentially enter into a formal agreement committing to increase collaboration and coordination in areas of mutual interest relating to benzene contamination;
- 7) support the increasing number of independent quality testing programs in the United States by convening workshops, stakeholder meetings and providing other resources at FDA's disposal to further encourage and connect such programs; and
- 8) promulgate rules or administrative orders requiring robust independent chemical batch-level testing and verification of the chemical content of batches of regulated consumer products and, while these are pending, issue guidance requesting such testing and verification.

Background on Petitioner

Valisure operates an analytical laboratory that is accredited to International Organization for Standardization ("ISO/IEC") 17025:2017 standards for chemical testing (PJLA Accreditation Number 94238). Valisure is registered with the Drug Enforcement Administration (License # RV0484814). Valisure's mission is to help ensure the safety, quality and consistency of medications and supplements in the market. In response to rising concerns about counterfeit medications, generics, and overseas manufacturing, Valisure developed proprietary analytical technologies that it uses in addition to FDA standard methods to test medications and consumer products distributed in the United States.

In an August 7, 2018, inspection of Valisure's facilities by FDA, it was determined that since Valisure's unique testing facility is not a part of the pharmaceutical manufacturing system and does not perform release testing, stability testing or any related services for pharmaceutical manufacturers, Valisure did not require FDA registration. Valisure also received guidance that since it operates outside of the manufacturing industry using the appropriate ISO guidelines as opposed to GMPs, any product failures or concerns that Valisure identifies should be reported back to the industry. Valisure has complied with this guidance and routinely provides reports to applicable parties in the industry.

Given the potential risk to public safety, Valisure seeks to utilize this Citizen Petition to bring these concerns directly to the attention of the Commissioner and FDA, and to request that they take prompt action.

B. Statement of Grounds

In addition to the information described above, which is incorporated by reference, Valisure provides the following as its statement of grounds. FDA currently recognizes the danger of benzene and, as a result, in drug products FDA has claimed benzene should not be used in the

manufacture of any component of a drug product, and only if its use is “unavoidable” should a strict concentration limit of 2 ppm apply,³ and in cosmetic products FDA has announced recalls of various products contaminated with benzene including body sprays⁸ and certain dry shampoos^{9, 10} which are not listed in this Petition. This Petition does not list any dry shampoo products where Valisure is aware of active manufacturer efforts to address benzene contamination in the affected products.

There is a recent history of broad drug and consumer product recalls due to contamination with probable human carcinogens. Specifically, there have been a multitude of manufacturer recalls of medications, such as valsartan, irbesartan, losartan,¹¹ ranitidine, nizatidine,¹² and metformin,¹³ due to the detection of the Group 2, “probable human carcinogen” N-Nitrosodimethylamine (“NDMA”) in excess of FDA limits. FDA limits for NDMA are defined in both parts per million (“ppm”) and permissible daily intake, which is held constant at a specified nanogram level (“ng”) per day for all drug products.¹⁴

Having a constant permissible daily intake or exposure is critical when there is variability in product size and exposures per day (potentially through multiple exposure routes); a situation particularly relevant to an individual’s application of dry shampoo products. Petitioner is not aware of any FDA guidance on a permissible daily exposure for benzene in any cosmetic product, including dry shampoos, or any other FDA-regulated product categories including drugs, and requests action on behalf of FDA to issue guidance to fill this gap. Valisure’s March

⁸ Food and Drug Administration. *P&G Issues Voluntary Recall of Specific Old Spice and Secret Aerosol Spray Antiperspirants and Old Spice Below Deck Aerosol Spray Products Due to Detection of Benzene* (November 23, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/pg-issues-voluntary-recall-specific-old-spice-and-secret-aerosol-spray-antiperspirants-and-old-spice>)

⁹ Food and Drug Administration. *P&G Issues Voluntary Recall of Aerosol Dry Conditioner Spray Products and Aerosol Dry Shampoo Spray Products* (December 17, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/pg-issues-voluntary-recall-aerosol-dry-conditioner-spray-products-and-aerosol-dry-shampoo-spray>)

¹⁰ Food and Drug Administration. *Unilever Issues Voluntary U.S. Recall of Select Dry Shampoos Due to Potential Presence of Benzene* (October 21, 2022) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/unilever-issues-voluntary-us-recall-select-dry-shampoos-due-potential-presence-benzene>)

¹¹ Food and Drug Administration. *Search List of Recalled Angiotensin II Receptor Blockers (ARBs) Including Valsartan, Losartan and Irbesartan* (September 23, 2019) (<https://www.fda.gov/drugs/drug-safety-and-availability/search-list-recalled-angiotensin-ii-receptor-blockers-arbs-including-valsartan-losartan-and>).

¹² Food and Drug Administration. *FDA Updates and Press Announcements on NDMA in Zantac (ranitidine)* (April 16, 2020) (<https://www.fda.gov/drugs/drug-safety-and-availability/fda-updates-and-press-announcements-ndma-zantac-ranitidine>)

¹³ Food and Drug Administration. *FDA Updates and Press Announcements on NDMA in Metformin* (October 5, 2020) (<https://www.fda.gov/drugs/drug-safety-and-availability/fda-updates-and-press-announcements-ndma-metformin>)

¹⁴ Food and Drug Administration. *FDA updates table of interim limits for nitrosamine impurities in ARBs* (February 28, 2019) (<https://www.fda.gov/drugs/drug-safety-and-availability/fda-updates-and-press-announcements-angiotensin-ii-receptor-blocker-arb-recalls-valsartan-losartan>)

24, 2021 Citizen Petition on benzene contamination in hand sanitizer,¹⁵ Valisure's May 24, 2021 Citizen Petition on benzene contamination in sun care products,¹⁶ Valisure's November 3, 2021 Citizen Petition on benzene contamination in body spray products,¹⁷ and the recent multiple recalls of certain hand sanitizers,^{18, 19} sunscreens^{20, 21} anti-fungal sprays^{22, 23} and antiperspirants^{24, 25} due to the presence of benzene further underscores the necessity to better regulate benzene and its apparent broad prevalence in product supply chains.

Although the dangers and carcinogenic potential of nitrosamines, like the aforementioned compound NDMA, have been well documented since the 1960s, a direct link to cancer in humans has not yet been established. In contrast to nitrosamines, benzene has long been directly

¹⁵ Valisure's Citizen Petition on Hand Sanitizer Products Containing Benzene Contamination (filed March 24, 2021) (<https://www.regulations.gov/document/FDA-2021-P-0338-0001>).

¹⁶ Valisure's Citizen Petition on Benzene in Sunscreen and After-sun Care Products (filed May 24, 2021) (<https://www.regulations.gov/document/FDA-2021-P-0497-0001>).

¹⁷ Valisure's Citizen Petition on Benzene in Body Spray Products (filed November 3, 2021) (<https://www.regulations.gov/document/FDA-2021-P-1193-0001>).

¹⁸ Food and Drug Administration. Scentsational Soaps & Candles, Inc. Voluntarily Expands Nationwide Recall of Scented Hand Sanitizers Due to the Presence of Methanol (Wood Alcohol), Benzene and Acetaldehyde (May 13, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/scentsational-soaps-candles-inc-voluntarily-expands-nationwide-recall-scented-hand-sanitizers-due#recall-announcement>).

¹⁹ Food and Drug Administration. *Best Brand Consumers Products, Inc. Issues Voluntary Recall of Mickey Mouse Hand Sanitizer Ethyl Alcohol 68% Due to Presence of Methanol and the Mandalorian Hand Sanitizer Ethyl Alcohol 68% Due to the Presence of Benzene* (April 1, 2022) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/best-brand-consumers-products-inc-issues-voluntary-recall-mickey-mouse-hand-sanitizer-ethyl-alcohol>).

²⁰ Food and Drug Administration. *Johnson & Johnson Consumer Inc. Issues Voluntary Recall of Specific NEUTROGENA® and AVEENO® Aerosol Sunscreen Products Due to the Presence of Benzene* (July 14, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/johnson-johnson-consumer-inc-issues-voluntary-recall-specific-neutrogena-and-aveeno-aerosol>).

²¹ Food and Drug Administration. *Coppertone® Issues Voluntary Nationwide Recall of Specific Lots of Pure & Simple SPF 50 Spray (2021 Launch), Sport Mineral SPF 50 Spray (2021 Launch), and Travel-Size Coppertone® Sport Spray SPF 50 (1.6OZ) Aerosols Sunscreen Sprays Due to the Presence of Benzene*. (September 30, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/coppertone-issues-voluntary-nationwide-recall-specific-lots-pure-simple-spf-50-spray-2021-launch>).

²² Food and Drug Administration. *Odor-Eaters® Issues Voluntary Nationwide Recall of Specific Lots of Odor-Eaters® Spray Powder and Odor-Eaters® Stink Stoppers® Spray Due to Benzene Contamination* (November 17, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/odor-eaters-issues-voluntary-nationwide-recall-specific-lots-odor-eaters-spray-powder-and-odor>).

²³ Food and Drug Administration. *Bayer Issues Voluntary Recall of Specific Lotrimin® and Tinactin® Spray Products Due to the Presence of Benzene* (October 1, 2021) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/bayer-issues-voluntary-recall-specific-lotrimin-and-tinactin-spray-products-due-presence-benzene>).

²⁴ Food and Drug Administration. *TCP HOT Acquisition LLC dba HRB Brands Issues Voluntary Nationwide Recall of Sure and Brut Aerosol Sprays Due to the Presence of Benzene* (February 16, 2022) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/tcp-hot-acquisition-llc-dba-hrb-brands-issues-voluntary-nationwide-recall-sure-and-brut-aerosol>).

²⁵ Food and Drug Administration. *Unilever Issues Voluntary Nationwide Recall of Suave 24-Hour Protection Aerosol Antiperspirant Powder and Suave 24-Hour Protection Aerosol Antiperspirant Fresh Due to Presence of Slightly Elevated Levels of Benzene* (March 30, 2022) (<https://www.fda.gov/safety/recalls-market-withdrawals-safety-alerts/unilever-issues-voluntary-nationwide-recall-suave-24-hour-protection-aerosol-antiperspirant-powder>).

associated with cancer in humans by epidemiological studies with persistent exposure as low as 0.8 ppm.²⁶ The hematotoxicity of benzene²⁷ has been described as early as 1897. A study from 1939 on benzene stated that “exposure over a long period of time to any concentration of benzene greater than zero is not safe,”²⁸ which is a comment reiterated in a 2010 review of benzene research specifically stating, “There is probably no safe level of exposure to benzene, and all exposures constitute some risk in a linear, if not supralinear, and additive fashion.”²⁹ In an October 15, 2021 recall of sunscreen products due to the presence of benzene, Canadian health regulator Health Canada stated “there is no safe level of benzene.”³⁰ According to the American Cancer Society:³¹

IARC classifies benzene as “carcinogenic to humans,” based on sufficient evidence that benzene causes acute myeloid leukemia (AML). IARC also notes that benzene exposure has been linked with acute lymphocytic leukemia (ALL), chronic lymphocytic leukemia (CLL), multiple myeloma, and non-Hodgkin lymphoma.

After Valisure’s previous detection of benzene in various consumer products, reports from FDA³² and industry³³ have confirmed this issue, though methodology for the analysis of contaminated aerosol sprays has not been published, to Petitioner’s knowledge, and may pose unique challenges. For the majority of measurements in this Petition, Valisure elected to utilize industry standard gas chromatography and detection by mass spectrometry (“GC-MS”) instrumentation that allows mass spectral separation and utilizing selected ion chromatograms, along with other orthogonal approaches for confirmation of a few select products including high performance liquid chromatography (“HPLC”) with UV detection. Gas chromatography conditions followed USP <467> with modifications to reduce run time that closely mirror those

²⁶ Glass, Deborah et. al. (2003). Leukemia Risk Associated With Low-Level Benzene Exposure. *Epidemiology* (Cambridge, Mass.). 14. 569-77. 10.1097/01.ede.0000082001.05563.e0. (https://journals.lww.com/epidem/Fulltext/2003/09000/Leukemia_Risk_Associated_With_Low_Level_Benzene.11.aspx)

²⁷ Santesson GG. 1897. Uber chronische Vergiftungen mit steinkohlen Benzin. Vier todes falle. *Arch. Hyg.* 31: 336–76

²⁸ Hunter, F.T. (1939). Chronic Exposure to Benzene (Benzol). II. The Clinical Effects. *Journal of Industrial Hygiene and Toxicology*. 1939 Vol.21 pp.331-54 (<https://www.cabdirect.org/cabdirect/abstract/19402700388>)

²⁹ Smith, Martyn T. (2010). Advances in Understanding Benzene Health Effects and Susceptibility. *Annual Review of Public Health*. 2010 Vol. 31:133-148 (<https://www.annualreviews.org/doi/full/10.1146/annurev.publhealth.012809.103646>)

³⁰ Health Canada (October 15, 2021). *Ombrelle Garnier Complete Dry Mist Spray sunscreen recalled due to elevated benzene levels* (<https://healthycanadians.gc.ca/recall-alert-rappel-avis/hc-sc/2021/76663a-eng.php>)

³¹ American Cancer Society. *Benzene and Cancer Risk* (January 5, 2016) (<https://www.cancer.org/cancer/cancer-causes/benzene.html>)

³² Food and Drug Administration (October 4, 2021). *FDA updates on hand sanitizers consumers should not use*. (<https://www.fda.gov/drugs/drug-safety-and-availability/fda-updates-hand-sanitizers-consumers-should-not-use>)

³³ Consumer Reports. *CDER Health Hazard Evaluation*. (July 7, 2021) (https://article.images.consumerreports.org/prod/content/dam/CRO-Images-2021/Health/12Dec/FDA_Benzene_in_Sunscreen_Assessment)

recommended by FDA in its August 24, 2020 guidance for impurities detection in hand sanitizer, which includes benzene analysis.³⁴

As Valisure has noted in previous FDA Citizen Petitions, some GC-MS methodologies can lead ingredients to break down into a suspected analyte due to elevated GC oven temperatures. Valisure identified such a situation in its September 13, 2019 FDA Citizen Petition regarding the drug ranitidine, and Valisure therefore developed modifications to the existing methodologies to lower temperature and prevent degradation.³⁵ The GC-MS methodologies described in this petition utilized body temperature (37°C) for oven incubation. 40°C has been previously used for benzene analysis from liquid pharmaceuticals and beverages, and reduced false positive results compared with higher-temperature incubation.^{36, 37}

Valisure acquired dry shampoo product samples from many retailers and in many different formulations. Although Valisure has made good faith efforts to obtain a broad selection of samples from the general supply, many brands and formulations are not included in Valisure's analysis presented in this Petition. Even in this limited survey of certain available dry shampoo products within the United States, multiple samples contained significantly detectable benzene and some samples contained up to about 170 times the conditionally restricted limit for drugs. Moreover, the dry shampoos tested are cosmetics, not drugs, and contain no active pharmaceutical ingredient for therapeutic purpose; therefore, any significant detection of benzene could be deemed unacceptable.

There was significant variability from batch to batch, even within a single brand, underscoring the importance of batch-level chemical analysis and the necessity of overall increased quality surveillance of these consumer products. Unlike other aerosol products that Valisure previously tested such as sunscreens and body sprays, some dry shampoo products also displayed significant variability between subsequent sprays from a single bottle, suggesting inconsistent product composition and/or aerosolization in some products. It was also visually apparent for some products that the initial sprays from the bottle were primarily gaseous and dispersed into the air while final sprays before a bottle emptied contained higher volume of particulate material, despite shaking the bottle before each spray. Therefore, the values for the first spray and any subsequent sprays are shown individually in Tables 2-5 instead of being averaged together. Also

³⁴ Food and Drug Administration. FDA Guidance Document (August 24, 2020) *Direct Injection Gas Chromatography Mass Spectrometry (GC-MS) Method for the Detection of Listed Impurities in Hand Sanitizers* (<https://www.fda.gov/media/141501/download>)

³⁵ Valisure FDA Citizen Petition Requesting to Recall Ranitidine (filed September 13, 2019) (<https://www.regulations.gov/docket?D=FDA-2019-P-4281>)

³⁶ Kyoung, H. et al. (2008). Evaluation of headspace-gas chromatography/mass spectrometry for the analysis of benzene in vitamin C drinks; pitfalls of headspace in benzene detection. *Biomedical Chromatography*, Vol. 22, p. 900-905 (<https://analyticalscience.wiley.com/do/10.1002/sepspec.19271ezine/full/>)

³⁷ Liu, H. et al. (2011) A general static-headspace gas chromatographic method for determination of residual benzene in oral liquid pharmaceutical products. *J Pharm Biomed Anal.* Vol. 54(2), p. 417-21. doi: 10.1016/j.jpba.2010.09.006. (<https://www.sciencedirect.com/science/article/abs/pii/S0731708510005182?via%3Dihub>)

of note, roughly 4% of dry shampoo samples analyzed by Valisure contained potentially confounding peaks at benzene's target retention time and m/z. This observation appeared to be brand/formulation specific, and these lots and brands are not included in this Petition as they are still being investigated by Valisure and will likely benefit from higher mass accuracy instrumentation to better determine the potential presence of benzene, if any.

Beyond the significant concern for public health, there is also evidence that benzene poses a serious risk to the environment, marine ecosystems, and United States waterways. Scientific papers published by the National Oceanic and Atmospheric Administration ("NOAA") have shown that benzene can be rapidly absorbed by fish³⁸ and short-term exposure (48 hr) to concentrations of benzene at parts per billion levels can significantly reduce survival of certain fish eggs.³⁹ The depth of experience with benzene regulation at EPA and the concern over environmental impact of benzene contamination offers a rational basis for collaboration between FDA and EPA to expeditiously address the current lack of much needed benzene regulation. Such collaboration could efficiently result in regulations applicable for all FDA regulated products. Precedence for FDA formally working with EPA through the execution of an agreement committing to increase collaboration and coordination in areas of mutual interest is found in the October 18, 2019 announcement of a Memorandum of Understanding between FDA, EPA and the United States Department of Agriculture ("USDA") regarding food waste.⁴⁰

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Strict EPA regulations on benzene are detailed in a report authored by the Agency for Toxic Substances and Disease Registry ("ATSDR"),⁴² which stated:

EPA has set 5 ppb [equivalent of 0.005 ppm] as the maximum permissible level of benzene in drinking water. EPA has set a goal of 0 ppb for benzene in drinking water and in water such as rivers and lakes because benzene can cause leukemia.

...

EPA recommends 200 ppb [equivalent of 0.2 ppm] as the maximum permissible level of benzene in water for short-term exposures (10 days) for children.

³⁸ S Korn, N Hirsch, J W Struthsaker (1976). UPTAKE, DISTRIBUTION, AND DEPURATION OF 14C-BENZENE IN NORTHERN ANCHOVY, ENGRAULIS MORDAX, AND STRIPED BASS, MORONE SAXATILIS. *Fishery Bulletin*. 1976 March Vol 74, No. 3: 545-51 (<https://spo.nmfs.noaa.gov/sites/default/files/pdf-content/1976/743/korn.pdf>)

³⁹ J W Struthsaker (1977). EFFECTS OF BENZENE (A TOXIC COMPONENT OF PETROLEUM) ON SPAWNING PACIFIC HERRING, CLUPEA HARENGUS PALLASI. *Fisher Bulletin*. 1977 Vol 75, No. 1: 43-49 (<https://spo.nmfs.noaa.gov/sites/default/files/pdf-content/1977/751/struthsaker.pdf>)

⁴⁰ Food and Drug Administration (October 30, 2019). *MOU 225-19-033*. (<https://www.fda.gov/about-fda/domestic-mous/mou-225-19-033>)

⁴¹ Environmental Protection Agency (May 27, 2020). *Winning on Reducing Food Waste Federal Interagency Strategy* (<https://www.epa.gov/sustainable-management-food/winning-reducing-food-waste-federal-interagency-strategy>)

⁴² Agency for Toxic Substances and Disease Registry (August 2007). *Toxicological Profile for Benzene*. (<https://www.atsdr.cdc.gov/toxprofiles/tp3.pdf>)

Furthermore, the long-established epidemiological data in humans is utilized by EPA to determine that a lifetime exposure of 0.4 ppb, or 0.0004 ppm, of benzene in air will likely lead to one additional cancer case in 100,000 exposed persons. Although this concentration in air does not directly apply to the level of contamination inside an aerosol product, which is not likely inhaled in its entirety, it does provide rational guidance for investigation of potential inhalation exposure by analyzing air contaminated by the spray of dry shampoo products containing benzene.

To investigate benzene levels directly from air contaminated by spray from dry shampoo products containing benzene, this Petition details additional testing performed on certain lots of two dry shampoo brands by Syft Technologies utilizing their Selected Ion Flow Tube – Mass Spectrometry (“SIFT-MS”) analysis system that directly analyzes volatile organic compounds and inorganic gasses in the air, including benzene. Although GC-MS is an industry standard approach and was utilized by Valisure in this Petition, the sample preparation required in GC-MS analysis may allow some benzene to escape detection and, therefore, potentially underestimate the amount of contamination. By contrast, SIFT-MS does not require sample preparation, thereby enabling real-time quantitative analysis of dry shampoo spray directly, allowing the investigation of real-world conditions and the potential risks consumers are exposed to with these kinds of contaminated products.

Personnel at Syft placed a SIFT-MS instrument in a closed mobile laboratory with an air volume of approximately 550 cubic feet (15.6 cubic meters), which is similar to a large bathroom's air volume. An operator inside the closed vehicle then sprayed a dry shampoo product approximately one foot away from the SIFT-MS inlet for ten seconds before exiting and reclosing the vehicle. This created a measurable spike of benzene as the contaminated “cloud” of spray hit the machine’s detector and established how much benzene fills the closed space and lingers after the initial spray.

The data from the SIFT-MS testing suggests short-term exposure of approximately 1,600 ppb of benzene from the “cloud” of sprayed dry shampoo and potential long-term exposure up to 36 ppb of benzene by raising the benzene level for all the air in a 550 cubic foot space. 36 ppb is 90 times the previously mentioned EPA calculated threshold for increased cancer risk by long-term inhalation exposure to benzene. Furthermore, calculating the total benzene present in the air from the stabilized, increased benzene level in the 550 cubic foot volume of air, up to 1.8 mg of benzene was present in a single spray of approximately 10 seconds. Through the measurement of the total spray volume in a bottle, up to 340 ppm of benzene was present in the first spray from a contaminated bottle. Such high detections of benzene from a direct detection methodology suggests that Valisure’s GC-MS analyses in this Petition, and perhaps previous Petitions investigating aerosol products, significantly underestimate the concentrations of benzene in such products due to the need to prepare samples from aerosolized product. Therefore, the widespread benzene contamination observed in consumer products may be of even greater risk to consumers than previously presented to FDA by Valisure’s petitions involving aerosol products.

Although FDA has recently released further guidance to industry and the public regarding benzene contamination in drug products,^{43, 44} there is comparably a lack of similar regulation or guidance for cosmetic products, such as dry shampoos. As FDA has acknowledged, the FDCA prohibits the marketing of adulterated or misbranded cosmetics in interstate commerce and specifies a product as adulterated if “it bears or contains any poisonous or deleterious substance which may render it injurious to users under the conditions of use prescribed in the labeling thereof, or under conditions of use as are customary and usual.”⁴⁵ However, specifically defined limits of “poisonous or deleterious” substances, such as benzene, are not defined and should be reviewed and addressed by FDA.

Petitioner urges the Commissioner and FDA to expeditiously request recalls on the affected batches of products and to take other such actions outlined in this Petition as deemed appropriate.

Analytical Methods: GC-MS

The method USP <467> Residual Solvents Procedure A was modified from flame ionization detection (FID) to mass spectrometry (MS) detection for benzene in dry shampoo products. The sample preparation and headspace (HS) gas chromatography (GC) methods were also modified to fit dry shampoo product matrices and to allow shorter run time. Identification of benzene is based on the retention time matching to certified reference standards and mass spectral matching to benzene. Quantification of benzene is performed by comparing peak area of benzene in a sample to a validated 11-point calibration curve. Results in parts per million are determined by dividing the micrograms of benzene detected per sample by the grams of material used for each sample.

Materials and Methods

Agilent 7890B GC equipped with 7697A headspace autosampler coupled with 5977B MS was utilized for sample analysis, and a DB-Select 624 UI, 60m × 0.32mm × 1.8µm GC column (Agilent Technology, Santa Clara, CA) was used to separate benzene from other compounds. Dimethyl sulfoxide (DMSO, GC Grade) was used for sample preparation (Thermo Fisher Scientific, Waltham, MA). Standard of benzene (99.8 % purity) and isotopic labeled benzene standard (d₃-, 99.8% purity) was used for retention time verification (Sigma-Aldrich, St. Louis, MO). USP Class 1 residual solvents mixture was used for calibration confirmation (USP, Rockville, MD). All volumetric glassware used are Class A certified.

⁴³ Food and Drug Administration (June 9, 2022). *FDA alerts drug manufacturers to the risk of benzene contamination in certain drugs.* (<https://www.fda.gov/drugs/pharmaceutical-quality-resources/fda-alerts-drug-manufacturers-risk-benzene-contamination-certain-drugs>)

⁴⁴ Food and Drug Administration (June 9, 2022). *Frequently Asked Questions on Benzene Contamination in Drugs.* (<https://www.fda.gov/drugs/drug-safety-and-availability/frequently-asked-questions-benzene-contamination-drugs>)

⁴⁵ Food and Drug Administration (March 8, 2021). *FDA Authority Over Cosmetics: How Cosmetics Are Not FDA-Approved, but Are FDA-Regulated* (<https://www.fda.gov/cosmetics/cosmetics-laws-regulations/fda-authority-over-cosmetics-how-cosmetics-are-not-fda-approved-are-fda-regulated>)

Standard and Sample Preparation

Benzene standard was diluted in DMSO. Calibration standards were prepared in 20-mL GC headspace vials to a total of 5 mL volume. Dry shampoo products were manually shaken and samples were dispensed into the GC headspace vials at approximately 500 mg and weighed, followed by adding 4.5 mL of DMSO to make up the final volume to approximately 5 mL and gently vortexing to mix. Five (5) mL of DMSO was used as blank samples.

Instrumental Analysis

Table 1 summarizes the major instrumental parameters used for analysis of benzene in the dry shampoo samples.

Table 1. Instrumental parameters optimized for benzene detection in dry shampoo samples.

| HS Autosampler | | GC | | MS | |
|-------------------------|------------------|--------------------|--------------------------------------|------------------|--------|
| Oven temperature (Temp) | 37 °C | Carrier gas | Helium | Source Temp | 230 °C |
| Loop Temp | 55 °C | Inlet Temp | 220 °C | Quad Temp | 150 °C |
| Transfer line Temp | 175 °C | Column flow | 2 mL/min | Acquisition type | SIM |
| Vial equilibration | 20 minutes (min) | Split ratio | 5:1 | Gain factor | 1 |
| Injection time | 1 min | Oven Temp Gradient | 60 °C (12 min) > 240 °C at 40 °C/min | Solvent delay | 3 min |
| Vial shaking | 71 shakes/min | GC run time | 18.5 min | | |
| Fill pressure | 15 psi | | | | |

Quality Assurance and Quality Control

Linear non-forcing through zero calibration curve was generated from the peak areas of the 11-point calibration standards. Calibration curve was accepted when the coefficient of determination R^2 was equal or greater than 0.995. Lower limit of detection (LLOD) and lower limit of quantification (LLOQ) were determined from linear regression and reference standard control samples prepared at the concentration of the lowest calibration standard that were ran with each experiment. LLOD was 0.026 μg (equivalent to 0.05 ppm in dry shampoo products) and LLOQ was 0.088 μg (equivalent to 0.18 ppm in dry shampoo products). The measurement uncertainty is concentration dependent and determined to be 32% near the LLOQ, 16% near 2 ppm, and 14% near 20 ppm. USP Class 1 residual solvent mixture was analyzed against the calibration and result of benzene agreed with certified concentration. Values are given for quantification over LLOQ or 0.18 ppm. For the data presented in this petition, Valisure is using the nomenclature that any benzene detection of 0.18 ppm or above is “significantly detected,” and any detection below this value or lack of benzene detection is described as “< LLOQ.”

Some dry shampoo samples with benzene concentration above the quantification threshold of 0.18 ppm were re-analyzed in triplicates and reported herein. In many cases variability was high and might be due to inconsistent dry shampoo product compositions and high sample volatility from inactive ingredients like butane and propane.

Analytical Methods: SIFT-MS

All operation of SIFT-MS technology was conducted by personnel at Syft Technologies and the below description of Materials and Methods and Instrumental Analysis was provided by Syft Technologies and not independently verified by Petitioner. Raw data from SIFT-MS analysis of dry shampoos was collected and interpreted by Syft Technologies and shared with Valisure to help generate this Petition and data reports contained within it.

Materials and Methods

A Syft Technologies Voice200*ultra* mass spectrometer equipped with an auto-validation inlet (AVI) was utilized for measurement of benzene in real time during and after an approximate 10 second spray of dry shampoo. This instrument was located inside the Syft Technologies mobile laboratory vehicle in the San Francisco Bay Area, CA. Two air conditioning units were running with recirculating air during the testing to maintain the temperature of the mobile laboratory. Individual dry shampoo products were sprayed at the wall, at a distance of approximately one foot from the inlet of the SIFT-MS.

Instrumental Analysis

The two product ions listed in Table 2 were used to measure the concentration of benzene before, during and after the spray process for each dry shampoo sample.

Table 2. SIFT-MS parameters for the measurement of benzene in real-time during and after an approximately 10 second spray event.

| Reagent Ion | Product Ion Formula | Product Ion Mass |
|-------------------------------|---|------------------|
| H ₃ O ⁺ | C ₆ H ₆ ⁺ H ⁺ | 79 |
| O ₂ ⁺ | C ₆ H ₆ ⁺ | 78 |

Benzene concentrations (ppb) were plotted in real time over a 15-minute run for each of the dry shampoo samples using LabSyft analysis software, and each measurement cycle was four seconds.

Analytical Findings: GC-MS

Using the GC-MS method described above for the determination of benzene, Valisure analyzed 148 unique batches from 34 brands of dry shampoo products with the results detailed below. In summary, 3 lots of dry shampoo products from 1 brand contained spray with over 100 ppm of benzene; 11 lots from 3 brands contained spray with quantifiable benzene over 20 ppm; 18 lots from 10 brands contained spray with quantifiable benzene between 2 - 20 ppm; 71 lots from 20 brands contained spray with quantifiable benzene between 0.18 - 2.0 ppm and benzene in 45 lots from 23 brands was either not detected or below the LLOQ through initial analysis of at least one sample. Due to the aforementioned inconsistencies observed in some dry shampoo product mixtures and detection of benzene in subsequent sprays from some bottles, each analysis for each individual spray from a dry shampoo product is detailed in Tables 3 – 6 with no averages or statistical calculations performed. Although inconsistencies exist in the general product category of dry shampoo, summary results presented in Table 6 include instances of two different bottles

analyzed from the same lot, and in these four cases the detected benzene levels for the first spray are relatively consistent, which suggests consistency in measurement and within the same lot of a given dry shampoo product.

Table 3a. Product description and results (first observation and follow-up triplicates) of benzene analysis on various batches of dry shampoo products in which benzene was detected at 2.00 ppm or higher.

| Brand | UPC | Lot | Description | Benzene Concentration (ppm) | | | |
|-----------------------------|---------------|---------------|--|-----------------------------|--------------|-------------|--------------|
| | | | | First Spray | Second Spray | Third Spray | Fourth Spray |
| Not Your Mother's | 688047130500 | 21090 | Beach Babe Texturizing Dry Shampoo - Toasted Coconut - 7 oz | 158 | 122 | 45.0 | 20.5 |
| Not Your Mother's | 688047130098 | 21145 | Clean Freak Refreshing Dry Shampoo - Original - 7 oz | 143 | 88.8 | 107 | 92.8 |
| Not Your Mother's | 688047130296 | 21070 | Clean Freak Refreshing Dry Shampoo - Unscented - 7 oz | 69.6 | 34.1 | 11.5 | 19.9 |
| Paul Mitchell Invisiblewear | 009531127866 | 20274 | Brunette Dry Shampoo - 4.7 oz | 35.2 | 27.9 | 16.6 | 21.5 |
| Sun Bum | 871760001824 | 21056 | Beach Formula Dry Shampoo - 4.2 oz | 30.4 | 26.6 | 28.3 | 10.4 |
| Not Your Mother's | 688047130975 | 20356 | Triple Threat Brunette Dry Shampoo - Hint of Brunette Tinted Powder - 7 oz | 27.6 | 5.04 | 0.57 | 0.11 |
| Batiste | 5010724529836 | RF1125 | Dry Shampoo Bare - 4.23 oz | 14.9 | 1.44 | 0.95 | 0.91 |
| Not Your Mother's | 688047130296 | 21188 | Clean Freak Refreshing Dry Shampoo - Unscented - 7 oz | 9.41 | 18.4 | 26.7 | 15.2 |
| Sebastian | 070018053978 | 4307144504 | Dry Clean Only Dry Shampoo - 1.7 oz | 7.76 | 4.96 | 4.44 | 3.65 |
| Redken | 884486431233 | WWU80WL | Deep Clean Dry Shampoo - 5 fl oz | 7.55 | 2.46 | 3.73 | 2.45 |
| dp Hue | 810001821012 | 2101501:32:29 | Apple Cider Vinegar Dry Shampoo - 5 oz | 7.52 | 4.56 | 3.61 | 5.91 |
| OGX | 022796640819 | 1461D07 | Extra Strength Dry Shampoo Coconut Miracle Oil - 5 oz | 3.98 | 3.85 | 3.43 | 3.80 |
| Sol de Janeiro | 810912031623 | 21216A | Brazilian Joia Dry Shampoo - 4 oz | 3.15 | 2.07 | 1.55 | 1.70 |
| Klorane | 3282770208702 | FR709 | Dry Shampoo with Nettle Oil Control - Oily Hair - 3.2 oz | 3.07 | 1.95 | 1.12 | 1.64 |
| Batiste | 5010724529836 | RF1155 | Dry Shampoo Bare - 4.23 oz | 2.61 | 1.16 | 1.94 | 0.99 |
| OGX | 022796640819 | 1181D06 | Extra Strength Dry Shampoo Coconut Miracle Oil - 5 oz | 2.56 | 1.07 | 1.15 | 1.63 |
| IGK | 854253008616 | 19196 E | Direct Flight Multi-Tasking Matcha Dry Shampoo - 2 oz | 2.49 | 1.65 | 1.17 | 1.20 |

Table 3b. Product description and single-observation results of benzene analysis on various batches of dry shampoo products in which benzene was detected at 2.00 ppm or higher.

| Brand | UPC | Lot | Description | Benzene Concentration (ppm) |
|-----------------------------|---------------|---------------|---|-----------------------------|
| Not Your Mother's | 688047130517 | 21141 | Plump for Joy Body Building Dry Shampoo - Orange Mango - 7 oz | 132 |
| Not Your Mother's | 688047130500 | 21124 | Beach Babe Texturizing Dry Shampoo - Toasted Coconut - 7 oz | 85.4 |
| Not Your Mother's | 688047130500 | 21179 | Beach Babe Texturizing Dry Shampoo - Toasted Coconut - 7 oz | 73.1 |
| Not Your Mother's | 688047130517 | 21181 | Plump for Joy Body Building Dry Shampoo - Orange Mango - 7 oz | 55.3 |
| Not Your Mother's | 688047130098 | 20280 | Clean Freak Refreshing Dry Shampoo - Original - 7 oz | 51.2 |
| Not Your Mother's | 688047130500 | 21063 | Beach Babe Texturizing Dry Shampoo - Toasted Coconut - 7 oz | 40.1 |
| Not Your Mother's | 688047130296 | 21009 | Clean Freak Refreshing Dry Shampoo - Unscented - 7 oz | 39.5 |
| Not Your Mother's | 688047130951 | 20353 | Blonde Moment Dry Shampoo - Hint of Blonde Tinted Powder - 7 oz | 22.9 |
| dp Hue | 810001821036 | 2101417 | Apple Cider Vinegar Dry Shampoo - 1 oz | 7.11 |
| dp Hue | 810001821012 | 2101501:32:32 | Apple Cider Vinegar Dry Shampoo - 5 oz | 6.20 |
| Klorane | 3282770200874 | FR4002 | Dry Shampoo with Oat Milk Ultra Gentle - All Hair Types - 1 oz | 5.72 |
| Not Your Mother's | 688047132290 | 21194 | Clean Freak Refreshing Dry Shampoo - Original - 1.6 oz | 4.46 |
| Batiste | 5010724529836 | RF0113 | Dry Shampoo Clean & Light Bare - 6.73 fl oz | 3.30 |
| Paul Mitchell Invisiblewear | 009531127866 | 19309 | Brunette Dry Shampoo - 4.7 oz | 2.88 |
| Paul Mitchell Invisiblewear | 009531127866 | 19007 | Brunette Dry Shampoo - 4.7 oz | 2.15 |

Table 4a. Product description and results (first observation and follow-up triplicates) of benzene analysis on various batches of dry shampoo products in which benzene was detected at 0.18 ppm to 2.00 ppm.

| Brand | UPC | Lot | Description | Benzene Concentration (ppm) | | | |
|-----------|---------------|------------|--|-----------------------------|--------------|-------------|---------------------|
| | | | | First Spray | Second Spray | Third Spray | Fourth Spray |
| Sebastian | 070018053893 | 9199144541 | Dry Clean Only Dry Shampoo - 4.9 oz | 1.77 | 0.92 | 0.78 | 0.82 |
| Batiste | 5010724527467 | RF1054 | Dry Shampoo Plus Brilliant Blonde - 6.73 fl oz | 1.70 | 0.70 | 0.43 | 0.78 |
| Batiste | 5010724527399 | RF9077 | Dry Shampoo Floral & Flirty Blush - 1.6 fl oz | 1.47 | 1.45 | 0.81 | Insufficient sample |

| | | | | | | | |
|-------------------|---------------|--------------|--|------|--------|--------|--------|
| Not Your Mother's | 688047130296 | 21195 | Clean Freak Refreshing Dry Shampoo - Unscented - 7 oz | 0.90 | 1.17 | 1.08 | 0.89 |
| CHI | 633911826959 | 21C08206 | VIBES Wake + Fake Soothing Dry Shampoo - 5.3 oz | 0.86 | 0.61 | 0.51 | 0.52 |
| OGX | 022796640918 | 1331D06 | Extra Strength Dry Shampoo Argan Oil of Morocco - 5 oz | 0.80 | 0.77 | 0.79 | 0.47 |
| Garnier Fructis | 603084580088 | HSU97WW | Texturizing Invisible Dry Shampoo Beach Tonic - 3.67 oz | 0.80 | 0.55 | 0.45 | 0.31 |
| Batiste | 5010724529836 | RF1181 | Dry Shampoo Bare - 4.23 oz | 0.73 | 0.71 | 0.58 | 0.32 |
| Not Your Mother's | 688047130098 | 21235 | Clean Freak Refreshing Dry Shampoo - Original - 7 oz | 0.67 | 0.31 | 0.20 | 0.30 |
| Pureology | 884486437600 | HCT80W 20227 | Style + Protect Refresh & Go Dry Shampoo - 5.3 oz | 0.66 | 0.30 | 0.31 | 0.24 |
| Kerastase | 884486442550 | HCU30W | Fresh Affair Refreshing Dry Shampoo Vitamin E + Rice Starch - 1.2 oz | 0.51 | 0.19 | 0.18 | < 0.18 |
| Amika: | 840117804272 | 21232 21209 | Perk Up Dry Shampoo - 5.3 oz | 0.47 | 0.25 | < 0.18 | < 0.18 |
| Hask | 071164373286 | 1917915181 | Dry Shampoo Monoi Coconut - 6.5 oz | 0.41 | 0.29 | 0.23 | 0.19 |
| Drybar | 819204019158 | 07169/21111 | Detox Clear Invisible Dry Shampoo Blanc Scent - 3.5 oz | 0.40 | 0.23 | < 0.18 | 0.23 |
| Biolage | 884486462251 | WWU40WM | All-In-One Intense Dry Shampoo - 5 fl oz | 0.39 | < 0.18 | < 0.18 | 0.252 |
| Drybar | 819204016119 | 10006/21298 | Detox Dry Shampoo Original Scent - 3.5 oz | 0.37 | 0.43 | 0.48 | 0.37 |
| Eva NYC | 840117802063 | 21257 21435 | Freshen Up Invisible Dry Shampoo - 5.3 oz | 0.25 | 0.21 | < 0.18 | < 0.18 |
| Kristin Ess | 840797129702 | 21139 AA01 | Brunette Dry Shampoo - 4 oz | 0.22 | 0.23 | < 0.18 | < 0.18 |
| Bumble and Bumble | 685428027152 | A51 21131 | Pret-A-Powder Tres Invisible Dry Shampoo - 1.3 oz | 0.20 | < 0.18 | < 0.18 | < 0.18 |
| Klorane | 3282770200898 | FR4015 | Dry Shampoo with Oat Milk Ultra Gentle - All Hair Types - 3.2 oz | 0.20 | ND | ND | ND |
| Cake | 899363003526 | 19 101 | The 'Do Gooder Volumizing Dry Shampoo - 4.2 oz | 0.19 | < 0.18 | < 0.18 | 0.25 |

Table 4b. Product description and single-observation results of benzene analysis on various batches of dry shampoo products in which benzene was detected at 0.18 ppm to 2.00 ppm.

| Brand | UPC | Lot | Description | Benzene Concentration (ppm) |
|---------|---------------|--------|---|-----------------------------|
| Batiste | 5010724527450 | RF1120 | Dry Shampoo Fresh Breezy Citrus - 4.23 oz | 1.94 |
| Klorane | 3282770200850 | FR330 | Dry Shampoo with Oat Milk Ultra Gentle - Dark Hair - 3.2 oz | 1.89 |
| Batiste | 5010724527443 | RF1131 | Dry Shampoo Plus Divine Dark - 6.73 fl oz | 1.81 |
| Batiste | 5010724527375 | RF0231 | Dry Shampoo Floral & Flirty Blush - 6.73 fl oz | 1.33 |

| | | | | |
|-------------------|---------------|-----------------|---|------|
| Batiste | 5010724527443 | RF9345 | Dry Shampoo Plus Divine Dark - 6.73 fl oz | 1.20 |
| Batiste | 5010724527443 | LR0083 | Dry Shampoo & a Hint of Colour for Dark Hair - 6.73 fl oz | 1.11 |
| Batiste | 5010724533123 | RF1167 | Dry Shampoo Dark Hair - 6.35 oz | 1.09 |
| IGK | 854569007402 | 21270A1 | Jet Lag Invisible Dry Shampoo - 6.3 oz | 0.94 |
| IGK | 854253008470 | 21021 A | Direct Flight Multi-Tasking Matcha Dry Shampoo - 6.3 oz | 0.92 |
| Batiste | 5010724527511 | RF1259 | Dry Shampoo Tropical Exotic Coconut - 4.23 oz | 0.83 |
| Not Your Mother's | 688047130487 | 21232 | Clean Freak Tapioca Dry Shampoo - Warm Sugar - 7 oz | 0.83 |
| Batiste | 5010724527481 | RF1103 | Dry Shampoo Original Classic Clean - 4.23 oz | 0.79 |
| Garnier Fructis | 603084580088 | HSU96WS | Texturizing Invisible Dry Shampoo Beach Tonic - 3.67 oz | 0.75 |
| Batiste | 5010724527481 | RF0167 | Dry Shampoo Clean & Classic Original - 6.73 fl oz | 0.70 |
| IGK | 810021400518 | 2023516721 | First Class Charcoal Detox Dry Shampoo - 2 oz | 0.70 |
| Batiste | 5010724529836 | RF0352 | Dry Shampoo Clean & Light Bar - 6.73 fl oz | 0.64 |
| Redken | 884486431264 | WWU60WV | Invisible Dry Shampoo - 5 fl oz | 0.61 |
| Pureology | 884486369703 | HCS30W 19085 | Style + Protect Refresh & Go Dry Shampoo - 1.2oz | 0.54 |
| Batiste | 5010724528150 | RF1200 | Dry Shampoo Plus Divine Dark - 1.6 fl oz | 0.54 |
| Batiste | 5010724527450 | LR9099 | Dry Shampoo Light & Breezy Fresh - 6.73 fl oz | 0.51 |
| Batiste | 5010724527399 | RF7132 | Dry Shampoo Floral & Flirty Blush - 1.6 fl oz | 0.46 |
| IGK | 810021400518 | 212542134914:12 | First Class Charcoal Detox Dry Shampoo - 2 oz | 0.45 |
| IGK | 810021400518 | 20293 | First Class Charcoal Detox Dry Shampoo - 2 oz | 0.43 |
| Batiste | 5010724527535 | RF8361 | Dry Shampoo Coconut & Exotic Tropical - 1.6 fl oz | 0.43 |
| OGX | 022796640819 | 0751D04 | Extra Strength Dry Shampoo Coconut Miracle Oil - 5 oz | 0.41 |
| Bumble and Bumble | 685428023314 | AC1 21347 | Pret-A-Powder Tres Invisible Dry Shampoo - 3.1 oz | 0.40 |
| Redken | 884486431233 | WWU50WT | Deep Clean Dry Shampoo - 5 fl oz | 0.39 |
| Amika: | 840117803299 | 21174 20726 | Perk Up Dry Shampoo - 1.8 oz | 0.38 |
| Not Your Mother's | 688047130098 | 21210 | Clean Freak Refreshing Dry Shampoo - Original - 7 oz | 0.36 |
| Not Your Mother's | 688047130098 | 21277 | Clean Freak Refreshing Dry Shampoo - Original - 7 oz | 0.36 |
| Redken | 884486431233 | WWU30WN | Deep Clean Dry Shampoo - 5 fl oz | 0.35 |
| Not Your Mother's | 688047130098 | 21211 | Clean Freak Refreshing Dry Shampoo - Original - 7 oz | 0.34 |
| Drybar | 819204016126 | 07877/21173 | Detox Dry Shampoo Original Scent - 1.4 oz | 0.33 |
| Batiste | 5010724527399 | RF8253 | Dry Shampoo Floral & Flirty Blush - 1.6 fl oz | 0.30 |
| Drybar | 819204019196 | 08750/21228 | Detox Dry Shampoo Coconut Colada - 3.5 oz | 0.30 |
| Not Your Mother's | 688047130296 | 21317 | Clean Freak Refreshing Dry Shampoo - Unscented - 7 oz | 0.28 |
| Not Your Mother's | 688047130296 | 21221 | Clean Freak Refreshing Dry Shampoo - Unscented - 7 oz | 0.27 |
| Hask | 071164373200 | 2117620991 | Volumizing Dry Shampoo Chia Seed - 9.6 fl oz | 0.27 |

| | | | | |
|-----------|---------------|--------------|--|------|
| Pureology | 884486437600 | HCU50W 21146 | Style + Protect Refresh & Go Dry Shampoo - 5.3 oz | 0.27 |
| Biolage | 884486462251 | WWU20W8 | All-In-One Intense Dry Shampoo - 5 fl oz | 0.26 |
| Redken | 884486431264 | WWT70W2 | Invisible Dry Shampoo - 5 fl oz | 0.23 |
| L'Oreal | 071249334980 | 173311545 | Extraordinary Clay Dry Shampoo - 4 oz | 0.23 |
| L'Oreal | 071249334980 | 173311546 | Extraordinary Clay Dry Shampoo - 4 oz | 0.23 |
| Drybar | 819204016126 | 06564/21105 | Detox Dry Shampoo Original Scent - 1.4 oz | 0.22 |
| Redken | 884486431257 | WWSD0W5 | Deep Clean Dry Shampoo - 1.3 oz | 0.22 |
| Drybar | 819204016126 | 07877/21174 | Detox Dry Shampoo Original Scent - 1.4 oz | 0.21 |
| Batiste | 5010724533048 | RF1204 | Dry Shampoo Original Classic Fresh - 6.35 oz | 0.20 |
| Batiste | 5010724527535 | RF1350 | Dry Shampoo Tropical Exotic Coconut - 1.06 oz | 0.20 |
| Batiste | 5010724532966 | FG1183 | Volumizing Dry Shampoo - 6.73 fl oz | 0.19 |
| Kerastase | 884486442543 | HCU40W | Fresh Affair Refreshing Dry Shampoo Vitamin E + Rice Starch - 5.3 oz | 0.18 |

Table 5. Product description and results of benzene analysis on various batches of dry shampoo products in which benzene was below the lower limit of quantitation (LLOQ).

| Brand | UPC | Lot | Description |
|-----------------|---------------|--------------|--|
| American Crew | 669316418345 | Y17X24708I | Techseries Boost Spray Dry Shampoo - 6.7 fl oz |
| Aquage | 671570000693 | 7L22 | Dry Shampoo - 8 oz |
| Aquage | 671570119517 | 9A10 | Dry Shampoo - 2 oz |
| Batiste | 5010724527504 | RF1316 | Dry Shampoo Original Classic Clean - 1.06 oz |
| Batiste | 5010724527511 | FE0164-53 | Dry Shampoo Coconut & Exotic Tropical - 6.73 fl oz |
| Batiste | 5010724533420 | FG1222 | Volumizing Dry Shampoo - 1.6 fl oz |
| Cake | 899363003526 | 19 247 | The 'Do Gooder Volumizing Dry Shampoo - 4.2 oz |
| CHI | 633911761014 | 18B04308 | Dry Shampoo - 2.6 oz |
| CHI | 633911761014 | 21006909 | Dry Shampoo - 2.6 oz |
| COLAB | 5016155119806 | 2081821 | Dry Shampoo Original Fragrance - 4.1 oz |
| COLAB | 5016155128518 | 2052220 | Dry Shampoo Paradise Fragrance - 6.7 fl oz |
| COLAB | 5016155132331 | 2102219 | Dry Shampoo Unicorn Fragrance - 6.7 fl oz |
| Drybar | 819204016126 | 03345/20191 | Detox Dry Shampoo Original Scent - 1.4 oz |
| Drybar | 819204018137 | 07272/21118 | Detox Dry Shampoo Lush Scent - 3.5 oz |
| Drybar | 819204019196 | 100097/19114 | Detox Dry Shampoo Coconut Colada - 3.5 oz |
| Eva NYC | 840117802063 | 21187 21058 | Freshen Up Invisible Dry Shampoo - 5.3 oz |
| Garnier Fructis | 3600542268264 | 14T1003 | Invisible Dry Shampoo Coco Colada - 4.4 oz |

| | | | |
|-------------------|---------------|--------------|---|
| Garnier Fructis | 3600542268264 | 14U402F | Invisible Dry Shampoo Coco Colada - 4.4 oz |
| Garnier Fructis | 3600542268271 | 14S800G | Invisible Dry Shampoo Melon-tini - 4.4 oz. |
| Garnier Fructis | 3600542268271 | 14T200D | Invisible Dry Shampoo Melon-tini - 4.4 oz. |
| Hask | 071164373200 | 2121821257 | Volumizing Dry Shampoo Chia Seed - 9.6 fl oz |
| Hask | 071164373286 | 21291 21519 | Dry Shampoo Monoi Coconut - 9.6 fl oz |
| Klorane | 3282770200850 | FR318 | Dry Shampoo with Oat Milk Ultra Gentle - Dark Hair - 3.2 oz |
| Klorane | 3282770207514 | FR100 | Detox Dry Shampoo with Organic Aquatic Mint - Pollution Exposed Hair - 3.2 oz |
| Klorane | 3282770208702 | FR687 | Dry Shampoo with Nettle Oil Control - Oily Hair - 3.2 oz |
| L'Oreal | 3600523957002 | 14T801 | Elvive Dream Lengths Air Volume Dry Shampoo - 4.16 oz |
| L'Oreal | 3600523957002 | 14TN016 | Elvive Dream Lengths Air Volume Dry Shampoo - 4.16 oz |
| Nioxin | 070018857507 | 027205 | Instant Fullness Dry Cleanser - 4.22 oz |
| Not Your Mother's | 688047132290 | 21337 | Clean Freak Refreshing Dry Shampoo - Original - 1.6 oz |
| OGX | 022796640819 | 2171D08 | Extra Strength Dry Shampoo Coconut Miracle Oil - 5 oz |
| OGX | 022796640918 | 2781D09 | Extra Strength Dry Shampoo Argan Oil of Morocco - 5 oz |
| Awapuhi | 009531128399 | 1821312:01 | Wild Ginger Repair Dry Shampoo Foam - 5.6 oz |
| Philosophy | 3614223642540 | 125601 | Pure Grace Dry Shampoo - 4.3 oz |
| Philosophy | 3614223648306 | 8200AA | Amazing Grace Dry Shampoo - 4.3 oz |
| Philosophy | 3614225100468 | 1035AA | Amazing Grace Dry Shampoo - 1.6 oz |
| Philosophy | 3614225100543 | 125701 | Fresh Cream Dry Shampoo - 4.3 oz |
| Raw Sugar | 895697005977 | 2114139159 | Volume Dry Shampoo Watermelon + Lychee - 5 oz |
| Redken | 884486431257 | WWSD0W4 | Deep Clean Dry Shampoo - 1.3 oz |
| SGX NYC | 034044000399 | 21288 301CAC | Dry Touch Volumizing Dry Shampoo - 6.5 oz |
| SGX NYC | 034044000450 | 21287 301CAC | Happy Place Nourishing Dry Shampoo - 6.5 oz |
| Sol de Janeiro | 810912031623 | 21363A | Brazilian Joia Dry Shampoo - 4 oz |
| Virtue | 817023020232 | 20337 | Re Refresh Dry Shampoo - 1.8 oz |
| Wella | 070018078636 | 105405 | EIMI Dry Me Dry Shampoo - 4.22 oz |
| Wella | 070018078650 | 118605 | EIMI Dry Me Dry Shampoo - 1.52 oz |
| Wella | 070018078650 | 118805 | EIMI Dry Me Dry Shampoo - 1.52 oz |

Table 6. Overview of first spray analyses by brand. In some cases, two bottles from the same lot were analyzed and these duplicate lots are grouped by a black outline around the two values.

| Brand | Sample | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 23 | 24 | 25 | 26 | 27 |
|-----------------------------|---------|------|------|------|------|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|------|
| Not Your Mother's | | <LOQ | 0.3 | 0.3 | 0.3 | 0.4 | 0.4 | 0.5 | 0.7 | 0.7 | 0.8 | 0.9 | 1.1 | 4.5 | 9.4 | 23 | 28 | 40 | 40 | 51 | 55 | 70 | 73 | 85 | 132 | 143 | 158 | |
| | Sun Bum | 30 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Paul Mitchell Invisiblewear | | 2.2 | 2.9 | 35 | | | | | | | | | | | | | | | | | | | | | | | | |
| | dp Hue | 6.2 | 7.1 | 7.5 | | | | | | | | | | | | | | | | | | | | | | | | |
| Sebastian | | 1.8 | 7.8 | | | | | | | | | | | | | | | | | | | | | | | | | |
| Sol de Janeiro | | <LOQ | 3.2 | | | | | | | | | | | | | | | | | | | | | | | | | |
| Klorane | | <LOQ | <LOQ | <LOQ | 0.2 | 1.9 | 3.1 | 5.7 | | | | | | | | | | | | | | | | | | | | |
| Batiste | | <LOQ | <LOQ | <LOQ | 0.2 | 0.2 | 0.2 | 0.3 | 0.4 | 0.5 | 0.5 | 0.5 | 0.6 | 0.7 | 0.7 | 0.8 | 0.8 | 1.1 | 1.1 | 1.2 | 1.3 | 1.5 | 1.7 | 1.8 | 1.9 | 2.6 | 3.3 | 14.9 |
| Redken | | <LOQ | 0.2 | 0.2 | 0.3 | 0.4 | 0.6 | 7.6 | | | | | | | | | | | | | | | | | | | | |
| OGX | | <LOQ | <LOQ | 0.4 | 0.8 | 2.6 | 4.0 | | | | | | | | | | | | | | | | | | | | | |
| IGK | | 0.4 | 0.4 | 0.7 | 0.9 | 0.9 | 2.5 | | | | | | | | | | | | | | | | | | | | | |
| Pureology | | 0.3 | 0.5 | 0.7 | | | | | | | | | | | | | | | | | | | | | | | | |
| Amika | | 0.4 | 0.5 | | | | | | | | | | | | | | | | | | | | | | | | | |
| Kerastase | | 0.2 | 0.5 | | | | | | | | | | | | | | | | | | | | | | | | | |
| Biologie | | 0.3 | 0.4 | | | | | | | | | | | | | | | | | | | | | | | | | |
| Bumble and Bumble | | 0.2 | 0.4 | | | | | | | | | | | | | | | | | | | | | | | | | |
| CHI | | <LOQ | <LOQ | 0.9 | | | | | | | | | | | | | | | | | | | | | | | | |
| Garnier Fructis | | <LOQ | <LOQ | <LOQ | <LOQ | <LOQ | 0.7 | 0.8 | | | | | | | | | | | | | | | | | | | | |
| Drybar | | <LOQ | <LOQ | <LOQ | 0.2 | 0.2 | 0.3 | 0.3 | 0.4 | 0.4 | | | | | | | | | | | | | | | | | | |
| Kristin Ess | | 0.2 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Hask | | <LOQ | <LOQ | 0.3 | 0.4 | | | | | | | | | | | | | | | | | | | | | | | |
| Cake | | <LOQ | 0.2 | | | | | | | | | | | | | | | | | | | | | | | | | |
| Eva NYC | | <LOQ | 0.2 | | | | | | | | | | | | | | | | | | | | | | | | | |
| L'Oreal | | <LOQ | <LOQ | 0.2 | 0.2 | | | | | | | | | | | | | | | | | | | | | | | |
| Philosophy | | <LOQ | <LOQ | <LOQ | <LOQ | | | | | | | | | | | | | | | | | | | | | | | |
| SGX NYC | | <LOQ | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | |
| Awapuhi | | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| COLAB | | <LOQ | <LOQ | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | |
| Aquage | | <LOQ | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | |
| Virtue | | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| American Crew | | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Nioxin | | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Raw Sugar | | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Wella | | <LOQ | <LOQ | <LOQ | | | | | | | | | | | | | | | | | | | | | | | | |

KEY

- > 100 ppm
- > 20 ppm
- > 2 ppm
- > 0.18 ppm
- < LLOQ

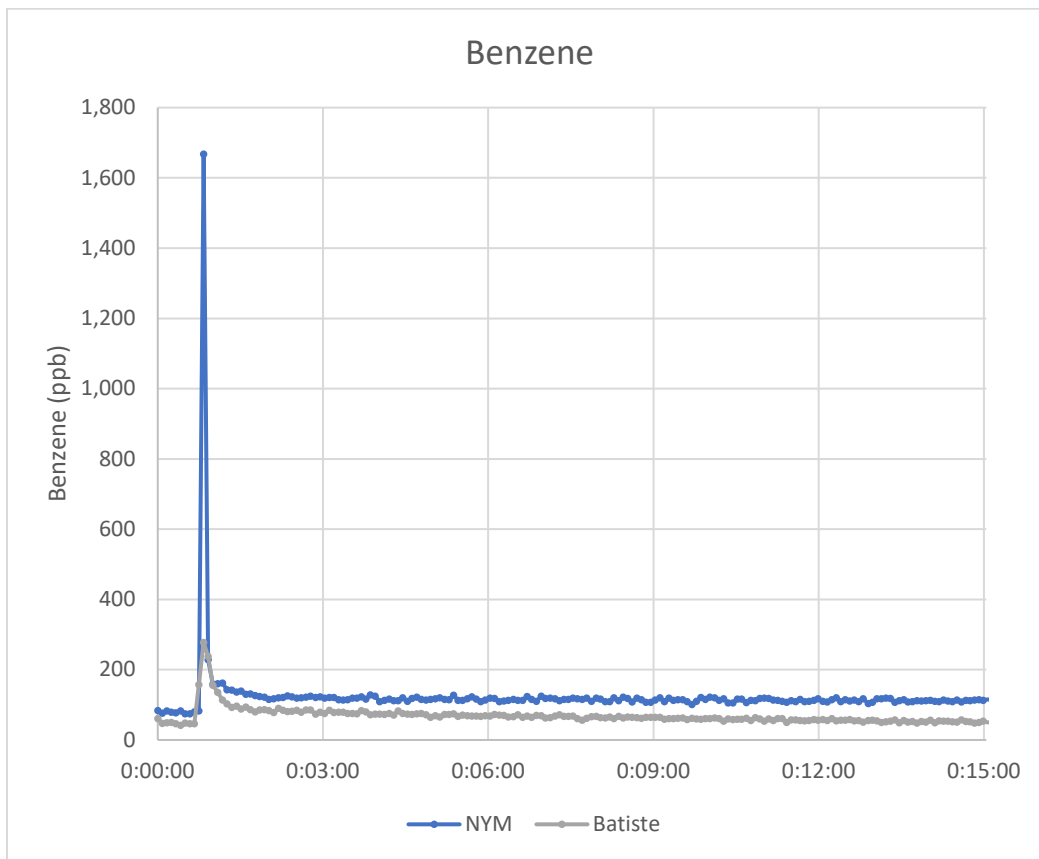
-- -- Duplicate Lots

Analytical Findings: SIFT-MS

Using the SIFT-MS method described above for the determination of benzene directly from contaminated air, presented in this Petition is Syft Technologies’ analysis of 2 unique batches from 2 brands of dry shampoo products, Batiste and Not Your Mother’s (NYM), with the results detailed below. In summary, maximum concentrations of benzene detected from the initial spray reached approximately 230 ppb and 1,600 ppb respectively, and the equilibrated level of benzene within the 550 cubic foot space rose 16 ppb and 36 ppb respectively. When Valisure measured the total spray volume of the same sized bottles and utilized the equivalence factor of 1 ppm

benzene to 3.19 mg benzene/m³ in ambient air,⁴⁶ the calculated concentration of benzene contained in the first spray using the SIFT-MS results was 67 ppm and 340 ppm respectively.

Figure 1. SIFT-MS analysis of 10 seconds of dry shampoo product spray in an approximately 550 cubic foot closed mobile laboratory vehicle for a 15 minute period, with detection every 4 seconds. One bottle from two different brands was analyzed. Shown in parts per billion (ppb).



The benzene concentration in ppm was calculated for each 10 second spray shown above by separately measuring the approximate total spray time each respective brand and bottle size contained. These calculations are detailed in Table 7 below.

⁴⁶ Environmental Protection Agency (January 2012). Benzene. (<https://www.epa.gov/sites/default/files/2016-09/documents/benzene.pdf>)

Table 7. Inputs and final output for calculation of benzene concentration in a 10 second spray of two different dry shampoo products. Total spray time was evaluated by purchasing 3 or 6 different bottles of the same size for each specific brand and spraying for 10 second increments until bottle was depleted.

| | NYM | Batiste |
|--|--------------|----------------|
| UPC | 688047130296 | 5010724529836 |
| Lot Number | 21188 | RF1155 |
| ug benzene in 10 second spray | 1797 | 827 |
| Reported Bottle Contents (g) | 198 | 120 |
| Bottle Contents (# of 10s sprays) | 37 | 10 |
| Relative Standard Deviation for Bottle Contents Measurements | 6.7% (n=6) | 6.0% (n=3) |
| g in a 10s spray | 5 | 12 |
| ug benzene/g in spray (ppm) | 340 | 67 |

A direct comparison of SIFT-MS analysis to GC-MS analysis of specific dry shampoo samples is difficult due to the aforementioned product variability; however, Valisure analyzed four subsequent sprays from the same bottles analyzed by Syft Technologies and also analyzed different batches of the same brand of product. Table 8 below details this comparison.

Table 8. Comparison of benzene concentrations detected on the first spray by SIFT-MS and four subsequent sprays by GC-MS of the same bottles of two brands described in Table 7. Summary results of first spray analyses of the same brands also provided.

| | First Spray SIFT-MS Benzene (ppm) | Subsequent Sprays GC-MS Benzene (average ppm) | GC-MS Brand Average First Spray Benzene (ppm) | Relative Standard Deviation (RSD) | GC-MS Analyzed Bottles |
|---------|--|--|--|--|-------------------------------|
| Batiste | 67 | 1.37 | 1.45 | 193% | 27 |
| NYM | 340 | 20.11 | 35.31 | 137% | 26 |

Although there are observed inconsistencies in dry shampoo products, the comparison of the SIFT-MS to GC-MS analysis of the same bottles with subsequent sprays and other bottles of the same brand, suggests that the GC-MS method described in this Petition may significantly underestimate the concentrations of benzene in such aerosol products. The SIFT-MS direct analysis system that eliminates sample prep appears to detect benzene in these aerosol products at approximately 10 – 50 times higher concentrations.

Recall Request and Other Actions

This Petition seeks to have the Commissioner and FDA request recalls for the identified batches of dry shampoo products, consistent with FDA's mandate to ensure the safety of cosmetic products in America. The 104 batches in Table 3a & b and Table 4a & b have significantly detected benzene and appropriate actions should be taken to protect American consumers.

This Petition urges FDA to request a recall of identified batches of dry shampoo products on the basis that they contain benzene, a known human carcinogen and, as such, a deleterious substance which may render the affected products injurious to users under the conditions of use in the labeling, or under customary and usual conditions. For this reason, FDA should find that these products are adulterated under Section 601 of the FDCA (21 U.S.C. § 361) and misbranded under Section 602 (21 U.S.C. § 362).

Regarding the highly variable exposure of an individual to dry shampoo products, which can relate to variations in application amount per individual, whether the application is in a small, closed area such as a bathroom, and number of applications per day, FDA should generate guidance with a daily permissible exposure limit, as is the case with nitrosamine impurities. To properly quantify daily exposure, FDA should provide further guidance on the amount of dry shampoo product and number of applications that a daily permissible exposure limit should apply to.

This Petition further requests that FDA review and update regulations and published guidance for cosmetic products and potentially include clarification that there is no acceptable level of benzene in these products, define a reasonable detection limit and include limitations on various impurities that pose known risks to human health and include benzene in such updates.

Independent, Batch-level Testing and Certification of Drug Products in the United States

Petitioner is also requesting that FDA promulgate rules or issue administrative orders requiring robust independent chemical batch-level testing and verification of dry shampoo and other products that are regulated by FDA. In the interim, while these are pending, FDA should issue formal guidance recommending such testing and verification.

Independent certification of regulated products can help prevent adulterated products from entering the market and can help ensure consumers continue to feel safe using certified brands.

In addition, Petitioner requests that FDA support the expanding number of independent quality analysis programs,⁴⁷ including that announced at The University of Kentucky,⁴⁸ through various means available to it. This may include convening new focused meetings, seminars, symposiums, and similar gatherings to connect programs and healthcare stakeholders that could benefit by learning from and augmenting such programs. It may also include adding such a topic to existing meetings, seminars, symposiums, and similar gatherings when appropriate.

As Valisure's results indicate, relying on industry to analyze and self-report is not sufficient protection from potentially dangerous contamination. A proactive drive for broad, independent testing should be combined with decisive action on the part of regulators to quickly request recalls and take other actions as appropriate.

C. Environmental Impact

Petitioner claims a categorical exclusion under 21 C.F.R. § 25.30, and believes that this Petition qualifies for a categorical exclusion from the requirement to submit an environmental assessment or environmental impact statement. To Petitioner's knowledge, no extraordinary circumstances exist.

D. Economic Impact

Pursuant to 21 C.F.R. § 10.30(b), economic impact information will be submitted by the Petitioner only upon request of the Commissioner following review of this Petition.

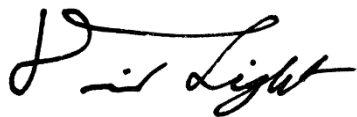
E. Certification

The undersigned certifies that, to the best knowledge and belief of the undersigned, this petition includes all the information and views on which the petition relies, and that it includes representative data and information known to the petitioner which are unfavorable to the petition.

⁴⁷ Almeter, P.J., Isaacs, J.T., Hunter, A.N. et al. FDA Approaches in Monitoring Drug Quality, Forces Impacting the Drug Quality, and Recent Alternative Strategies to Assess Quality in the US Drug Supply. *J Pharm Innov* 17, 269–282 (2022). <https://doi.org/10.1007/s12247-022-09659-5>

⁴⁸ Chapin, Elizabeth; Willett, Kristi. (October 1, 2020) UK Drug Quality Testing Leads to Petition to Recall Injectable Drug. *University of Kentucky* (<http://uknow.uky.edu/research/uk-drug-quality-testing-leads-petition-recall-injectable-drug>)

Respectfully submitted,



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