FINAL REPORT: TASK 3

COMPARISON OF CHEMICAL ANALYSES BETWEEN CROPS IRRIGATED WITH PRODUCED WATER AND CROPS IRRIGATED WITH TRADITIONAL WATER SOURCES

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The California Central Valley Region Regional Water Quality Control Board

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The front-end material in this document has been re-used and/or adapted from work conducted by Dr. William Stringfellow at Lawrence Berkeley National Laboratory. This includes the Introduction and Methods section of the 2017 Citrus Report[1]. Other work reported here is the product of work conducted by scientists at GSI Environmental.

[1] https://www.waterboards.ca.gov/centralvalley/water_issues/oil_fields/food_safety/data/crop_reports/fsp_report_citrus_083118.pdf

TABLE OF CONTENTS

AUTHORS ii
REVIEWERS iii
TABLE OF CONTENTS iv
LIST OF FIGURES v
LIST OF TABLES v
LIST OF APPENDICES vi
LIST OF ABBREVIATIONS vii
EXECUTIVE SUMMARY 1
1.0 INTRODUCTION 2
2.0 METHODS 5
2.1 Sample Collection 5
2.2 Sample Preparation and Analysis 6
2.3 Data Processing 7
2.4 Exploratory Data Analysis Methods 8
2.5 Statistical Analysis Methods – Two-Sample Hypothesis Tests 10
2.6 Statistical Analysis Methods – Multivariate Methods 11
2.6.1 Bar Charts and Stiff Plots 11
2.6.2 Correlation Matrix Plots 12
2.6.3 Hierarchical Cluster Analysis 12
2.6.4 Rank Order Statistics 13
3.0 RESULTS 13
3.1 Chemical Specific Results 14
3.1.1 Metals 14
3.1.2 Organics 17
3.2 Crop Specific Results 21
3.2.1 Almonds 21
3.2.2 Apples 22
3.2.3 Carrots 24
3.2.4 Cherries 24
3.2.5 Garlic 25
3.2.6 Grapes 26
3.2.7 Lemons 27
3.2.8 Mandarin Oranges 27
3.2.9 Navel Oranges 28
3.2.10 Valencia Oranges 29
3.2.11 Pistachios 29
3.2.12 Potatoes 30

3.2.13 Tomatoes 31
3.2.14 Classification of Chemical Profiles and Crop Groupings 32
4.0 TREATED PRODUCED WATER AND BLENDED PRODUCED WATER SAMPLING 34
4.1 Summary of Water Quality Data 34
5.0 DISCUSSION 35
5.1 Uncertainties and Data Gaps 38
6.0 CONCLUSIONS 39
7.0 REFERENCES 41

LIST OF FIGURES

Figure 1. Process for selecting datasets for exploratory data analysis and statistical analysis. CT = central tendency (mean or median); FOD = frequency of detection. 46
Figure 2. Decision process for selecting a two-sample hypothesis test. DL = method detection limit; ND = non-detect; FOD = frequency of detection; WMW = Wilcoxon-Mann-Whitney 47
Figure 3. Target analytes detected in treatment crops 48

LIST OF TABLES

Table 1: Sampling and analysis dates 49
Table 2: Full list of organic compounds measured in crops and methods of analysis, as reported in certified laboratory reports 50
Table 3: Full list of metals measured in crops and methods of analysis, as reported in certified laboratory reports 54
Table 4: 89 Crop/Chemical datasets with at least one detect in a treatment group 55
Table 5a: Summary statistics for almonds 56
Table 5b: Exploratory data analysis for almonds 57
Table 5c: Statistical analysis for almonds 58
Table 6: Summary statistics for apples 59
Table 7: Summary statistics for carrots 60
Table 8: Summary statistics for cherries 61
Table 9a: Summary statistics for garlic 62
Table 9b: Exploratory data analysis for garlic 63
Table 9c: Statistical analysis for garlic 64
Table 10a: Summary statistics for grapes 65
Table 10b: Exploratory data analysis for grapes 66
Table 10c: Statistical analysis for grapes 67
Table 11a: Summary statistics for lemons 68
Table 11b: Exploratory data analysis for lemons 69

Table 11c: Statistical analysis for lemons..... 70
Table 12a: Summary statistic for mandarins 71
Table 12b: Exploratory analysis for mandarins 72
Table 12c: Statistical analysis for mandarins 73
Table 13a: Summary statistics for navel oranges..... 74
Table 13b: Exploratory data for navels oranges..... 75
Table 13c: Statistical analysis for navel oranges..... 76
Table 14: Summary statistics for Valencia oranges..... 77
Table 15a: Summary statistics for pistachios 78
Table 15b: Exploratory data analysis for pistachios 79
Table 15c: Statistical analysis for pistachios 80
Table 16: Summary statistics for potatoes 81
Table 17a: Summary statistics for tomatoes 82
Table 17b: Exploratory data analysis for tomatoes 83
Table 17c: Statistical analysis for tomatoes 84
Table 18: Summary of crops with similar chemical profiles for metals and organics 85
Table 19: Chemicals detected in crops irrigated with blended produced water with respective MCLs for Drinking Water..... 86
Table 20: Summary of analytic results for treated produced water samples (effluent water) 87
Table 21: Summary of analytic results for blended produced water samples..... 88

LIST OF APPENDICES[1, 2]

Appendix A: Data Summaries of Exploratory Data Analysis: Summary Statistics, Box-and-whisker Plots, and Rank-order Plots of Combined Control/Treatment
Appendix B: Bar Charts
Appendix C: Stiff Charts
Appendix D: Pairwise Correlation Matrix Plots
Appendix E: Dendrograms
Appendix F: Lab Reports

[1] Each appendix contains a series of figures that are referenced in the text and captioned with the corresponding appendix letter and a number, e.g. A-5 in Appendix A, B-4 in Appendix B, C-2 in Appendix C, etc. Reference to the figures in the text may not follow serial order because appendices can contain a comprehensive reporting of each chemical result that did not need to be discussed in the text, but the results are nonetheless reported in the appendices.

[2] Appendices could not be included in this PDF document due to State requirements for ADA compliance. Please contact the Central Valley Regional Water Quality Control Board to obtain electronic copies, contact information below:

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https://www.waterboards.ca.gov/centralvalley/water_issues/oil_fields/food_safety/

LIST OF ABBREVIATIONS

2-CEVE - 2-chloroethyl vinyl ether

APPL - Agriculture & Priority Pollutants Laboratories

ATSDR - Agency for Toxic Substance and Disease Registry

CV - Coefficient of Variation

d - day

DWEL - Drinking Water Equivalent Levels

EDA - Exploratory Data Analysis

FDA - Federal Drug Administration

FDR - False Detection Rate

FOD - Frequency of Detection

FWER - Family Wise Error Rate

GOF - Goodness-of-Fit

GSI - GSI Environmental Inc.

IQR - Interquartile Range

kg - kilogram

MCL - Maximum Contaminant Level

MDL - Minimum Detection Level

mg - milligram

NA - Not Applicable

ND - Non-detect

NHANES - National Health and Nutrition Examination Survey

OEHHA - Office of Environmental Health Hazards Assessment

PTDI - Provisional Tolerable Daily Intake

PTTIL - Provisional Total Tolerable Intake Level

QA/QC - Quality Assurance and Quality Control

RfD - Reference Dose

SAP - Sampling and Analysis Plan

SE - Standard Error

TVD - Toxicity Value Dose

VOC - Volatile Organic Compound

WD - Water District

WHO - World Health Organization

WWEIA - What We Eat In America

US EPA - U.S. Environmental Protection Agency

EXECUTIVE SUMMARY

This data report summarizes an evaluation of chemical concentrations in crops irrigated with treated produced water blended with other suitable irrigation water (treatment) and conventional irrigation water sources (control). The primary purpose of the data evaluation is to determine if there is a difference in chemical profiles in crops irrigated with these two water sources. A secondary goal is to evaluate the potential human health risks from exposure to specific chemicals that may have higher concentrations in crops irrigated with produced water. Samples from thirteen crops were collected from various fields throughout the San Joaquin Valley during growing seasons in 2017, 2018, and 2019. Crops were selected to evaluate a range of agricultural practices and plant uptake potential. Crop groups include, root and tuber vegetables, bulb vegetables, fruiting vegetables, citrus, pome and stone fruit, berry and small fruit, and tree nuts. A total of 113 constituents—18 metals and 95 organic compounds—were selected for analysis based on their potential association with produced water.

Data were grouped by crop and type of irrigation water (treatm`ent or control) to examine patterns in chemical residues between groups. Results were paired to common sample identification codes, which have been deidentified to maintain confidentiality agreements with participating growers. Exploratory data analysis and statistical analysis methods were applied using techniques consistent with current US EPA guidance and standard practice.

The analysis supports a conclusion that the overall chemical concentration profiles were similar between crops irrigated with produced water compared with other water sources. The majority of the 113 target analytes were not detected in most crops. A total of 24 chemicals were detected in one or more samples collected from the 13 crops irrigated with produced water. Of these, 10 chemicals were detected in just one crop, 9 chemicals were detected in 2 to 6 crops, and 5 chemicals were detected in 7 to 13 crops. The total number of chemicals detected in one or more samples grouped by crop ranged from 4 (in apples) to 11 (in both almonds and carrots). Both the average detected concentrations and proportion of non-detects of inorganic and organic chemicals provided a chemical profile for each crop. The profiles were relatively consistent across certain crop groups and may be useful for defining baseline concentrations in crops grown under similar irrigation and crop management practices. In addition, a review of the detected organic chemicals identified sources unrelated to produced water that may have contributed to the observed concentrations in food crops.

The sampling design yielded a possible 1,469 crop/chemical datasets (i.e., 13 crops x 113 chemicals) to assess the study objectives. Of these, a total of 89 crop/chemical datasets could be compared based on detections in one or more samples collected from fields irrigated with produced water. In 84 of 89 cases, samples from fields irrigated with produced water had the same or lower mean and median concentrations compared with samples collected from the same crop irrigated with other water sources. The mean or median concentration in 5 of 89 sample comparisons were statistically higher for crops

irrigated with produced water ($p < 0.05$), but these differences are not sufficiently large enough to suggest that crops irrigated with produced water are different than those commonly found in the U.S. marketplace. These differences were observed for barium and zinc in almonds, and for strontium in garlic, grapes, and lemons.

Data were evaluated using standard statistical methods and exploratory data analysis techniques. A variety of graphical analyses and multivariate methods were applied to supplement the findings of the statistical analysis.

Too few samples were available to support statistical analyses of differences between test results of treated vs control samples of apples, carrots, cherries, potatoes and Valencia oranges; other types of evaluations were applied to the test results from these crops as a means of evaluating any differences in chemical concentration in treated vs control crops. Depending on the availability of data, the test results from these crops were evaluated using a variety of methods including, graphical evaluations; comparisons with reported concentrations in marketplace foods; qualitative comparisons, including comparisons of these crops to the other crops sampled in this project. For apples and potatoes, which did not have directly comparable control samples, we evaluated the toxicological significance of the levels of chemicals detected in these crops. In all of these evaluations, we did not find any evidence that the crops irrigated with some treated produced water were significantly different than crops wholly irrigated with conventional irrigation water, or that typical consumption of these crops posed a health hazard.

This data report also summarizes information on the within-field variability of concentrations based on a subset of samples collected from the same fields (i.e., field duplicates) as well as potential concentration outliers for each crop. Statistical analyses were rerun with potential outliers removed from control and/or treatment datasets to confirm that conclusions were not affected by outliers.

1.0 INTRODUCTION

This report describes work completed under Task 3 of the "Memorandum of Understanding Between the Central Valley Regional Water Quality Control Board and the Permit Holders Governing the Solicitation, Management and Review of Academic, Technical and/or Scientific Studies Related to the Irrigation of Crops with Oil Field Produced Water." Task 3 is the third of a three-task project to research and evaluate the safety of using treated, produced water for the irrigation of food crops. The primary objective of Task 1 was to conduct a hazard assessment of chemicals that may be present in the water that comes out of an oil well, along with oil, when crude oil is produced (i.e., produced water). A second objective was to develop a prioritized list of these chemicals for further study in the context of the beneficial use of produced water for the irrigation of food crops. Task 2 entailed a literature search for information on the properties and occurrence of the chemicals identified in Task 1 that supported the further evaluation and understanding of the safety aspects of using produced water for irrigation. Task 3 entailed testing crops to determine if there were chemical differences in crops irrigated with

produced water, when compared against those that were irrigated with conventional sources of irrigation water and, if so, to determine if differences were attributable to the use of produced water as an irrigation water source. The enumeration of Tasks 1, 2, and 3 does not reflect the temporal order in which each task was started. Work activities under Task 3 were some of the first to take place in this project. They supplement other crop sampling that was overseen by the Central Valley Regional Water Quality Control Board (Central Valley Water Board) staff in 2017. Results from this earlier sampling are already reported on the Central Valley Water Board website. The substantive work from Task 1 was completed before starting work on Task 2, which required the identification of the Chemicals of Interest. Some early results from this task (Task 3) were used in Task 1 to identify the Chemicals of Interest. In addition, some results from Task 1 are used to inform the evaluation performed under Task 3.

California's San Joaquin Valley is a major oil producing area. In 2013, approximately 150 million barrels of oil (42 gallons/barrel) were produced along with nearly 2 billion barrels of produced water (about 250,000 acre-feet). Produced water is treated to remove or reduce the concentrations of petroleum hydrocarbons, and the oil is sent off for refining. Much of the produced water is disposed of by such methods as reinjection or evaporation/percolation and is not suitable for beneficial use because of high salinity. Produced water from the areas east and north of Bakersfield, however, tends to have low salinity and has been reused for irrigation for at least the last 30 years. The produced water supplements other traditional sources of irrigation water in the area and generally meets agricultural objective for salinity and boron.

The Central Valley Water Board regulates the discharge and reuse of produced water for irrigation under waste discharge requirements (WDRs) that require the analyses of produced water for a variety of chemicals, including chemicals that are associated with additives used during petroleum exploration, production, or treatment. The analyses required under the WDRs are completed by certified third-party laboratories and results are submitted to the Central Valley Water Board for review. Cawelo Water District (WD), North Kern Water Storage District, Jasmin Ranchos Mutual Water Company, and Kern-Tulare WD (collectively referred to as Districts) are the four Districts that currently receive produced water from a total of four oil companies. Cawelo WD and parts of Kern-Tulare WD, including operation of the Jasmin Ranchos Mutual Water Company reservoir, have the longest history reusing produced water for irrigation. This report is part of an ongoing evaluation by the Central Valley Water Board concerning the beneficial reuse of produced water.

The sources of water distributed by Cawelo WD include the Kern River, State Water Project, pumped groundwater, and produced water generated from oil production operations (Robles, 2016). Cawelo WD receives approximately 32,000 acre-feet of produced water a year from regional oil producers under Waste Discharge Requirements Order Nos. R5-2012-0058 and R5-2012-0059 adopted by the Central Valley Water Board. The produced water is received into Cawelo WD's water distribution facilities and blended

with other suitable irrigation water sources (surface and/or groundwater) prior to being delivered to agricultural fields for irrigation (Enviro-Tox Services, 2017); throughout this report, we refer to this water as blended produced water. The blended produced water distributed by Cawelo WD is used to irrigate food crops that include citrus, nuts, grapes, apples, and row crops (e.g., garlic and carrots). During periods of low demand for irrigation water, blended produced water may be discharged to the Famoso Basins (recharge basins regulated under the WDRs) in addition to reducing the volume of surface water and groundwater that is blended with produced water (Wood, 2019).

The Kern-Tulare WD relies upon surface water, groundwater, and produced water generated from oil production operations. The Jasmin Ranchos Mutual Water Company operates independently from the Kern-Tulare WD, but is located within the service territory of Kern-Tulare WD and receives blended produced water from Kern-Tulare WD. In accordance with Waste Discharge Requirements Order No. R5-2019-0043 (formerly regulated under Waste Discharge Requirements Order No. 98-205), Kern-Tulare WD and the Jasmin Ranchos Mutual Water Company are partnered with Hathaway LLC to receive a maximum of 2,640 acre-feet per year of produced water from the Jasmin Oil Field (US Bureau of Reclamation, 2017). The maximum volume may increase to 3,320 acre-feet upon the Discharger submitting a technical report to the Central Valley Water Board, for review and approval, that demonstrates the facility is adequately designed to accommodate the additional flow. Produced water used by Kern-Tulare WD and Jasmin Ranchos Mutual Water Company is blended with other water suitable for irrigation and used to irrigate citrus.

The North Kern Water Storage District historically relied upon the Kern River and groundwater for irrigation supplies; excess surface water and produced water is used in spreading basins for groundwater recharge. North Kern Water Storage District receives approximately 9,600 acre-feet of produced water (according to 2019 monitoring reports submitted to the Central Valley Water Board). Per Waste Discharge Requirements Order R5-2015-0127 adopted in 2015, the North Kern Water Storage District also has authorization to use treated produced water, blended prior to use, from the Kern Front Oil Field for irrigation and groundwater recharge. The Order allows produced water to be used on 55,000 acres of irrigated farmland and 608 acres of spreading basins. Most of the irrigated acreage within the water district is planted with nuts, grapes, and fruit.

Recycling of water is encouraged by State policy as a means to supplement California's limited water supply, provided the water is suitable for the intended use. The Central Valley Water Board permits the recycling of produced water for irrigation, where suitable for reuse. Since food safety is outside the expertise of the Central Valley Water Board, their staff has taken a proactive approach by convening a Food Safety Panel of experts in human health, agriculture, risk assessment, and environmental toxicology to ascertain the possible food safety risks associated with using produced water on crops for human consumption. The Central Valley Water Board has also contracted with a science advisor from the Lawrence Berkeley National Laboratory to develop and coordinate and/or

implement food safety related work plans, and further advise the Central Valley Water Board staff on food safety related issues. The Central Valley Water Board staff has also engaged GSI Environmental as a neutral third-party source of technical support for the project (Central Valley Regional Water Quality Control Board, 2017).

In 2017, 2018, and 2019, food crops samples were collected from fields that had received produced water (treated sites) and fields that did not receive produced water (control sites) as part of their irrigation supply. There were 13 different crops sampled during this time: cherries, carrots, garlic, Valencia oranges, navel oranges, lemons, mandarins, almonds, apples, grapes, pistachios, potatoes, and tomatoes. Food crop samples were analyzed for a variety of organic and inorganic constituents that were selected based on their potential association with produced waters. The purpose of this sampling was to determine concentrations of inorganic and organic constituents in food crops from treated and control areas with the objective of comparing fruit from treated and control areas in the context of food safety. The purpose of this report is to document the results of the sampling of this crop in 2018-19, in combination with previously published crop sampling results from 2017, and to present a preliminary scientific interpretation of the results of this sampling. As is discussed in greater detail below, with the exception of a few specific crop/chemical combinations, the overall chemical profiles for crops irrigated with produced water as part of the irrigation water were the same as chemical profiles for crops irrigated strictly with other local sources of irrigation water.

2.0 METHODS

A summary of the methods used for sample collection, sample preparation/chemical analysis, and data analysis are described below. The overarching principle in selecting sample collection and preparation methods was to collect and prepare samples in a manner that would represent fruit, vegetable, and nut crops at the time of harvest, to minimize contamination by ambient sources of chemicals, and to use standard, agency-approved analytical methods. Methods were also designed to be consistent with those used in previous sampling of crops irrigated with produced water in the study area to facilitate a comparison of results from this study to results from the previous testing. More details on the sample collection are provided in the Sampling and Analysis Plan (SAP). More details regarding sample preparation and analysis are provided in laboratory reports generated over the course of this study.

2.1 Sample Collection

Samples were collected in accordance with the Citrus Fruit SAP prepared by Enviro-Tox Services, Inc. that was initially reviewed and commented on by Central Valley Water Board staff (Enviro-Tox Services, 2017). As the Food Safety Project went forward, Central Valley Water Board staff distributed the Citrus Fruit SAP to the Panel, Science Advisor, and GSI for review and consideration. Table 1 reports sampling and analysis periods for each crop. Following the SAP, samples were collected in the interior portions of the fields (at least 100 feet from the edge) to minimize potential effects of contamination from road

dust, diesel fumes, or any other identified sources of hydrocarbons. Individual samples were whole, edible crop samples, which were cut or picked from the branch and were not pulled because pulling might damage the sample. Likewise, root, tuber, and bulb vegetables (i.e., carrots, potatoes, and garlic) were carefully removed from the ground, so as not to damage the sample.

Collected samples were placed inside sample containers specified in the SAP. Multiple sample containers were used at some sampling locations to accommodate larger crop samples required for sample preparation and analysis. When multiple containers were used, the contents of the sample containers were composited at the analytical laboratory before extraction and analyzed as described below. Sample containers were closed and sealed with tape. Samples were labelled and placed on ice in a cooler immediately after sample collection. Samples in coolers were shipped by an overnight shipping service to Weck Laboratories, Inc., (Weck Laboratories) in City of Industry, CA for analysis for all samples except for carrots sampled in 2019, which were delivered to Agriculture & Priority Pollutants Laboratories, Inc (APPL Inc.) in Clovis, CA by Central Valley Water Board staff. Additionally, for the analysis of volatile organic compounds in cherries, Weck Laboratories sent prepared and extracted samples to APPL Inc. for analysis. This was due to malfunctioning equipment at Weck Laboratories that could not be repaired within the time required to ensure that holding times for extracted samples were not exceeded. Extracted cherry samples were kept on ice and shipped to APPL Inc., within eight hours of extraction. Maximum holding times, however, have not been established for intact food samples analyzed by the analytic methods employed in this study.

Samples were maintained at 4ºC +/- 2ºC until received by the laboratory. Chain of custody was maintained throughout the sampling and shipping process. Central Valley Water Board staff observed the crop sampling events and retained possession of the samples, with the exception of the control group potato sample that was purchased at a local grocery store in Bakersfield by Cawelo WD staff (see Section 3.2.12). Field duplicate samples were collected for quality control. Field duplicates were collected from the same location as the primary sample and were collected at both treated and control fields.

2.2 Sample Preparation and Analysis

Food crop samples were cleaned in the field using lint-free tissue and processed in the laboratory so that the edible portion of the food crop was used for analysis. The edible portion from each sample from each site were composited into one sample for extraction and analysis using U.S. Environmental Protection Agency (US EPA) approved methods. When the food crop had an edible skin that may in some circumstances be peeled (e.g., apples, carrots, potatoes), care was maintained to ensure the surface was as clean as possible and the whole edible portion was processed, including the skin. For crops like garlic and citrus, the paper skin and peel were removed prior to processing.

Field duplicate samples were collected at the same time and sample location. They were analyzed independently, as per accepted practice for certified analytical laboratories.

Samples were labelled so the lab could not identify control samples from treated or duplicates. Field duplicates were collected to assess the degree of homogeneity of the concentrations within crops grown in the same field. As such, field duplicates provide a measure of within-field variability, rather than a measure of precision of analytic methods.

Food crop samples were analyzed by Weck Laboratories and APPL Inc. Both labs participate in California's State Water Resources Control Board Environmental Lab Accreditation Program to provide water quality analysis services. There is no state laboratory certification, however, for the analysis of chemicals in food. Samples were analyzed for major classes of hydrocarbons and heavy metals associated with produced water. In total, concentrations for 113 analytes were quantified, which included 18 metals and 95 organic compounds. Table 2 and Table 3 report the lists of organics and metals and their associated analytical methods, respectively.

2.3 Data Processing

Figure 1 and Figure 2 illustrate the data processing steps used to develop the datasets used in the exploratory data analysis (EDA) and statistical analysis. In Step 1, the data were filtered to exclude the following:

- Laboratory quality control samples (e.g., spikes)
- Duplicates (i.e., only parent samples were retained for statistical analysis); parent/duplicate pairs were evaluated to assess within-field variability.

Results for values qualified as non-detects (NDs) were set equal to the sample method detection limit (MDL) unless otherwise noted. In the case of NDs of 1,4 dioxane for which the MDL was not reported, results were set equal to the method reporting limit (MRL). Results qualified as estimated (e.g., J flag) were grouped with detects rather than NDs and qualified accordingly[1]. Further processing of NDs for parameter estimation, graphical analysis, and statistical analysis was performed using methods consistent with US EPA guidance. Unit conversions were applied to present all results in units of mg/kg.

In Step 2, the frequency of detection (FOD) was assessed for both the control and treatment groups. Chemicals that were not detected in a treatment group of a specific crop were excluded from further analysis for that crop. Chemicals that were detected in 1, 2, or 3 samples in a treatment group were retained for qualitative evaluation. Table 4 and Figure 3 summarize the **89 crop/chemical** groups for which a chemical was detected in at least one sample from the treatment group. Table 5 through 17 provide summary statistics (e.g., sample size, frequency of detection [FOD], range, arithmetic mean and standard deviation [SD], and selected percentiles) for the control and treatment groups by crop. For censored datasets (i.e., FOD<100%), the arithmetic mean and SD were

[1] J-Flag results were only reported in samples of carrots where arsenic, cadmium, chromium, cobalt, copper, lead, molybdenum, nickel, selenium, vanadium, and zinc were detected in treated samples. All of these metals were detected in both treated and control samples, with the exception of cadmium (detected in 2 of 7 of treated). These data were screened during Step 2 of the analysis framework due to the low number of detections (n ≤ 2).

estimated with Kaplan-Meier statistics, consistent with US EPA guidance for analysis of censored data (US EPA, 2015).

US EPA guidance regarding minimal sample sizes required for inferential statistics range from four observations per group for hypothesis testing (US EPA, 2009) to 8 to 10 observations for parameter estimation (US EPA, 2015). Accordingly, crop/chemical data groups consisting of at least four detects in both the control and treatment groups were retained for further exploratory data analysis (Step 3), which is described in greater detail below. The following five of thirteen crops had fewer than four observations in either the control or treatment groups: cherries (2 control and 2 treatment [i.e., 2/2]); Valencia (3/3), potato (3/3), apple (0/4), and carrots (3/6). A total of **36 crop/chemical** datasets (consisting of both control and treatment sample results) were retained. This dataset consists of the following 11 chemicals:

Type of Chemical	Chemical Name
Metals:	• barium • copper • strontium • zinc
Volatile organic compounds (VOCs):	• acetone • acrolein • 2-butanone • ethyl acetate • methyl tert-butyl ether (MTBE) • p-isopropyltoluene
Alcohol:	• methanol

The following eight crops were evaluated in Step 3: almonds, garlic, grapes, lemons, mandarins, navels, pistachios, and tomatoes. For these crops, the tabular summaries are divided into a set of three consecutive tables. For example, almonds are summarized in Table 5a, 5b, and 5c; garlic is summarized in Table 9a, 9b, and 9c, and so on. Apples are not explored further because samples were not collected from a control group. Cherries, carrots, and potatoes were not explored further because no chemicals were detected in at least four samples of control and treatment groups for these three crops.

2.4 Exploratory Data Analysis Methods

The concentration data collected from the chemical analyses of crop samples collected in 2017, 2018, and 2019 were tabulated, plotted, explored graphically, and analyzed using descriptive and inferential statistics. The goal of the data analysis is to determine if there is a difference in the chemical profiles of crops irrigated with produced water blended with other irrigation water (i.e., treatment group) and crops irrigated with water containing no produced water (i.e., control group).

The following exploratory data analysis (EDA) steps were conducted on each of the 36 crop/chemical datasets (both control treatment samples):

- Calculation of FOD (%)
- Outlier screening using both a quantitative metric based on the interquartile range (IQR) (i.e., 75th percentile + 3 x IQR), as well as a graphical analysis using normal quantile-quantile (Q-Q) plots
- Calculation of the ratio of the arithmetic means and ratio of medians
- Evaluation of goodness-of-fit (GOF) for normal distributions using both a graphical analysis (Q-Q plot) and statistical analysis (Shapiro-Wilks test)
- Box-and-whisker plot showing control and treatment groups side-by-side for each crop/chemical dataset
- Rank-order plots of combined control and treatment datasets

The “b” series of the table for the eight crops provides a tabular summary of the first four approaches listed above. That is, results are summarized in the following tables: Table 5b (almonds), Table 9b (garlic), Table 10b (grapes), Table 11b (lemons), Table 12b (mandarins), Table 13b (navels), Table 15b (pistachio), and Table 17b (tomato). These summary statistics plus the box-and-whisker plots and rank-order plots of the combined control and treatment datasets are provided as a set of one-page summaries in Appendix A.

One objective of the EDA is to inform the selection of appropriate hypothesis tests for evaluating the differences between the distributions of concentrations in samples from the two groups (control and treatment) for each crop/chemical dataset. Hypothesis tests are discussed in greater detail below. Both the GOF evaluation of normal distributions and the ratio of the central tendency (i.e., mean or median) sample statistics are key elements of Step 3 of the EDA.

If both the control and treatment datasets are approximately normally distributed with FOD=100%, then the ratio of the means is evaluated in Step 4. For ratios > 1 (i.e., the sample mean for the treatment is greater than the sample mean for control), a hypothesis test is used to determine if the difference is statistically significant. Similarly, for datasets that are either FOD<100% or non-normal, a non-parametric hypothesis test is used to evaluate differences in the medians, but only for ratio>1. Intuitively, as noted in Step 4 of Figure 1, a formal statistical test is unnecessary if the ratio of the relevant central tendency summary statistics is less than one.

The crop/chemical data groups for which hypothesis testing is warranted are summarized in the “c” series tables. These tables include the **18 crop/chemical data groups** that are evaluated in Step 5 (statistical analysis), as discussed below. This is exactly half of the 36 crop/chemical groups that were carried forward from Step 3 to Step 4. These 18 groups include the same **eight crops** evaluated in Steps 3 and 4, but with fewer chemical

combinations. **Eight chemicals** are retained in these groups: three metals (barium, copper, strontium), four organics (2-butanone, acetone, MTBE, and p-isopropyltoluene), and one alcohol (methanol). Therefore, 3 of 11 chemicals (i.e., zinc, acrolein, and ethyl acetate) evaluated in Step 4 were not carried forward to Step 5.

2.5 Statistical Analysis Methods – Two-Sample Hypothesis Tests

Two sample hypothesis tests are commonly used to evaluate differences in one variable (e.g., concentration of one chemical) between two or more groups (e.g., treatment and control).

Figure 2Figure 2 summarizes the decision process used in this analysis to select a hypothesis test based on the statistical properties of the 18 crop/chemical data groups. As noted in US EPA guidance (US EPA 2002; 2007; 2009; 2015), the choice of hypothesis tests largely depends on the FOD and goodness-of-fit to normal distributions. If both control and treatment samples are FOD=100% and normally distributed, a Student's *t*-test is used to evaluate the differences in the sample arithmetic means, assuming the assumption of equal variance is reasonable. (If the ratio of sample SDs is greater than 3.0, then the Welch-Satterthwaite Test is used in lieu of the Student's *t*-test). For datasets with FOD between 50% and 100%, US EPA (2015) recommends applying a non-parametric hypothesis test to evaluate differences in the sample medians. The Wilcoxon-Mann-Whitney (WMW) test is used for cases when the MDL is consistent for all NDs (inclusive of control and treatment groups), and the Gehan test is used for cases when multiple MDL values are present. Since a test of the medians may be impacted by the MDLs for datasets with FOD<50%, the Fisher's Exact test is used to evaluate differences in the FOD for the more highly censored crop/chemical data groups. For this analysis, US EPA's software called ProUCL v5.1 was used to implement the hypothesis tests.

For both parametric and non-parametric hypothesis tests, the conclusions can vary depending on how the null hypothesis is defined. US EPA (2002, 2015) discusses the use of both "Test Form 1" and "Test Form 2" for one-sided, two-sample hypothesis tests. The two approaches can yield different conclusions when the difference between the means (or medians) is relatively small. Expressions for the null hypothesis are given below:

- Test Form 1: H_0: $\mu_{treatment} \leq \mu_{control}$
- Test Form 2: H_0: $\mu_{treatment} > \mu_{control} + S$

(Equivalent expressions for the population medians are used for the non-parametric tests). There are two key differences between Form 1 and Form 2. First, with Form 1, we assume the mean for the treatment group is less than or equal to the mean of the control group. This places the burden of proof on the data to reject the null hypothesis, so if the power of the test is not sufficient (given the sample size and pooled SD), we may not achieve the desired error rates (i.e., we may fail to conclude there is a difference with a greater probability than expected). For this analysis we used the standard error rates for Type 1 (α=0.05) and Type 2 (β=0.20). With Form 2, we assumed the mean for the

treatment group is greater than or equal to the mean of the control group by some amount "*S*", which is referred to as a statistically significant difference. This difference may be specified *a priori*, as part of the sampling and analysis plan, before collecting the samples. Alternatively, as was done for this analysis, a post-hoc power analysis can be conducted using a range of values for *S* (US EPA, 2015). In this analysis, the first analysis for *S* was based on the observed difference in sample statistics. This was accompanied by a power test using the pooled SD and equations given in the statistics literature as summarized in US EPA guidance (US EPA, 2015). If the power test indicated the sample size was too small to achieve the desired error rates, then the value of *S* that can be assessed was calculated based on the sample sizes and pooled SD. Results for evaluations using Form 1 and Form 2 null hypotheses are also included in the "c" series tables.

2.6 Statistical Analysis Methods – Multivariate Methods

In addition to evaluating treatment and control groups one chemical at a time, several multivariate graphical and statistical analysis methods were applied. These methods are listed below along with the investigation questions that each method addresses.

Multivariate Method	Investigation Question(s)
Bar charts and Stiff Plots	• Are the chemical profiles similar between treatment and control groups for a given crop? • Are the chemical profiles similar between two or more crops?
Correlation Matrix Plot	• Which chemicals exhibit moderate to strong correlations across samples? • Are similar patterns in correlations observed across two or more crops?
Hierarchical Cluster Analysis	• When all of the chemistry data for the treatment and control group samples for a crop are combined, to what extent do individual field samples from each group cluster together?
Rank Order Statistics	• Are there specific fields in either control or treatment groups for which multiple chemicals are consistently detected at higher concentrations?

2.6.1 Bar Charts and Stiff Plots

Two graphical methods were used to evaluate patterns in relative magnitudes of concentrations of multiple chemicals detected in the same crop – bar charts and stiff plots.

Side-by-side bar charts are a simple but effective technique to compare the relative magnitudes of multiple chemicals measured in two groups. Twelve figures (one for each crop, excluding apples) in Appendix B show bars illustrating the arithmetic mean concentrations plus two times the standard error (SE) for 14 chemicals (7 metals and 7

organics) that are more frequently detected in multiple crops. This includes the eight chemicals evaluated using two sample hypothesis testing. For censored datasets (i.e., observations include one or more NDs), the mean and SE were estimated using Kaplan-Meier methods. Each plot illustrates the relative magnitude of concentrations detected in samples for one crop. Two ordinate (y-axis) scales were used – the primary axes (on the left) is for the metals and the secondary axis (on the right) is for the organics. The ranges used for each scale were modified, as needed, in order to most easily interpret the profile for an individual crop. Observations are summarized in Table 18.

Stiff plots are commonly used to illustrate relative magnitudes of geochemical constituents in groundwater. Three figures in Appendix C show side-by-side plots for control and treatment groups for 11 crops (excluding apples, which do not have control group samples, and cherries for which the samples sizes are relatively small). Each plot illustrates the relative magnitude of concentrations of multiple constituents when crops are plotted on the same scale. Each crop is depicted by a set of four polygons to show control and treatment groups side-by-side, each represented by two chemical classes. The top polygon represents four metals (barium, copper, strontium, zinc) and the bottom polygon represents four organics (acetone, acrolein, ethyl acetate, and p-isopropyltoluene). Non-detects are included at the maximum MDL of the samples qualified as NDs within a crop/chemical group.

2.6.2 Correlation Matrix Plots

Pair-wise correlations are informative when concentrations are measured in the same set of samples. Correlation matrix plots provide a one-page summary of pairwise correlations for a suite of chemicals that are all measured in the same samples for a given crop. Pairwise correlation matrix plots, in Appendix D, report correlations for eight crops with at least four samples in both control and treatment groups: almonds, garlic, grapes, lemon, Mandarin oranges, Navel oranges, pistachios, and tomato. The plots show x-y scatterplots of individual observations (after combining control and treatment groups together), histograms for each chemical (along the diagonal), and Spearman rank correlation coefficients, which are mathematically equivalent to Pearson correlations calculated with the ranks rather than numeric values of each observations. Spearman rank correlation is less sensitive to substituted values for NDs (i.e., the MDLs) as well as potential high-end outliers.

2.6.3 Hierarchical Cluster Analysis

Agglomerative hierarchical clustering is a type of multivariate data analysis in which a relationship between sampling units (e.g., crop samples) can be established by reducing a large number of variables (e.g., chemicals) to a coefficient that represents the degree of dissimilarity between sampling units. Although there are many different methods for calculating coefficients and establishing clusters, each method is generally applied in the following sequence:

1. Calculate a matrix of dissimilarities between all pairs of sampling units.
2. Form the first cluster between two sampling units with the smallest dissimilarity.
3. Calculate dissimilarities between the first cluster and the remaining sampling units.
4. Form the second cluster between the first cluster and the sampling unit with the smallest dissimilarity to the first cluster.
5. Continue until all sampling units are linked in clusters.

For this analysis, we calculated dissimilarity coefficients using a Euclidian method and applied a “complete linkage” procedure, which is a furthest neighbor technique in which the dissimilarity between two clusters is measured by the maximum dissimilarity between all combinations of samples.

The output from the cluster analysis is called a dendrogram, which is a tree-like diagram that shows the dissimilarity coefficients for each sample identification code. In this analysis, samples that are linked in the same cluster exhibit the greatest similarities in chemical concentrations. Clusters that have the shortest links (or distances) are most similar. The samples from the control and treatment groups were color coded differently and prefixes were added to the sample identification codes to facilitate comparisons.

Separate dendrograms are included in Appendix E for the following eight crops: almonds, garlic, grapes, lemon, mandarin, navel, pistachios, and tomato. The main utility of the dendrograms is to determine if the control and treatment samples cluster together or separately. A mix of control and treatment samples at the same branches of the tree diagram is an additional line of evidence that the overall chemical profiles are similar for crops irrigated with produced water and other water sources. Conversely, if the control and treatment samples cluster separately, this is an indication of differences in the chemical profiles between the two groups.

2.6.4 Rank Order Statistics

For each crop/chemical/irrigation water source (control or treatment) group for the eight crops carried forward to Step 5, the chemical concentrations were rank ordered in descending order so that the highest ranked value is “1”, the second highest is “2”, and so forth. Non-detects were excluded from the ranking. The ranks were then summed across chemicals for each sample identification code. Two key summary statistics were calculated: 1) average rank score (noting that crops with higher FODs will have higher average rank scores); and 2) sum of ranks. Sample identification codes with the lowest sum of ranks correspond with samples that more consistently have higher chemical concentrations. This analysis provides an indication of field-to-field variability within crops.

3.0 RESULTS

The sampling design yielded a possible 1,469 crop/chemical datasets (i.e., 13 crops x 113 chemicals) to assess the study objectives. A total of 89 crop/chemical datasets could

be compared based on detections in one or more samples collected from fields irrigated with produced water. Table 4 and Figure 3 show the distribution of the 89 crop/chemical datasets grouped into three chemical classes: metals, organics (VOCs), and alcohols. Of the 113 chemicals that were measured, 24 chemicals were detected among the crops that were irrigated with produced water. Chemicals detected in at least seven crops included three metals (strontium, copper, and barium) and two organics (acetone and acrolein).

The sample sizes for the treatment groups varied from 2 to 21 across the 13 crops. In general, crops with a greater number of samples also tended to have a greater number of detected chemicals. For example, as summarized in Table 4, almonds and pistachios had among the highest number of samples (i.e., 20 and 21, respectively) and also the highest number of chemicals detected in at least one sample (i.e., 11 and 9, respectively). Similarly, crops with fewer than five samples from fields irrigated with produced water, including cherries (n=2), potatoes (n=3), Valencia (n=3), and apples (n=4), had among the fewest chemicals detected in at least one sample (i.e., 4 to 6 chemicals). Notable exceptions to this generalization included carrots (11 chemicals detected in 6 samples) and grapes (7 chemicals detected in 21 samples).

The variability in sample sizes across crops is an important consideration when evaluating findings from EDA and assessing the statistical power of tests used to evaluate differences in mean/median concentrations between control and treatment groups. Potential implications for findings from this study are discussed below in the subsection on crop-specific results.

3.1 Chemical Specific Results

Chemical-specific results are described below. The majority of the 24 chemicals detected were metals and organic compounds commonly found in crops. Limonene, a citrus terpene, was identified in the 2017 Citrus Sampling Report as a result of quality control activities related to incorrect identification of 1,2,4-trimethylbenzene in both treated and control samples. This terpene was not a target analyte, but its detected concentrations have been presented in this report. Limonene and related terpenes are naturally occurring compounds in citrus fruit and are a component of citrus flavor and aroma (Perez-Cacho and Rouseff, 2008; Favela-Hernandez et al., 2016).

3.1.1 Metals

As shown in Figure 3, of the 11 metals detected in one or more samples from a treatment group, the most frequently detected across the 13 crops in this study are strontium, copper, and barium. Strontium and copper are detected in all crops, often at 100% detection in both control and treatment groups. Although copper is reported as ND in each of the three treatment group samples of Valencia (and, therefore, excluded from Table 4 and Figure 3), copper is detected in one field duplicate sample.

For strontium, the arithmetic mean or median concentrations were higher in treatment groups of garlic, grapes, lemon, mandarin, and pistachios. The differences were statistically significant for garlic (Table 9c), grapes (Table 10c), and lemon (Table 11c). The sample sizes provided sufficient power to evaluate the observed differences for these datasets.

For copper, the arithmetic mean or median concentrations were higher in treatment groups of almonds, garlic, and mandarin, but none of these differences were statistically significant. For almonds, the difference in means was so small (i.e., 8.01 and 8.02 mg/kg in control and treatment groups, respectively) that a sample size of at least 300,000 would be needed to evaluate the statistical significance (Table 5c). As shown in the side-by-side box plots and rank ordered values of the combined datasets (see Figure A-4), there was substantial overlap in the two distributions. Similarly, for garlic, at least 182 observations in both control and treatment samples would be needed to evaluate the statistical significance of the observed difference in arithmetic means (2.32 and 2.38 mg/kg in control and treatment groups, respectively). The current sample sizes (5 and 6 observations) and the pooled SD were sufficient to conclude the means are not different by more than 0.32 mg/kg (Table 9c). As shown in the side-by-side box plots and rank ordered values of the combined datasets (see Figure A-31), there was substantial overlap in the two distributions. Furthermore, for mandarins, at least 20,000 observations in both control and treatment samples would be needed to evaluate the statistical significance of the observed difference in medians (0.55 and 0.56 mg/kg in control and treatment groups, respectively). The current sample sizes (10 observations in each group) and the pooled SD were sufficient to conclude that the medians were not different by more than 0.47 mg/kg (Table 12c). As shown in the side-by-side box plots and rank ordered values of the combined datasets (see Figure A-50), there is substantial overlap in the two distributions and indications of one extreme outlier (2.4 mg/kg) in the control group. Although the medians are robust to the presence of a single outlier, this value does contribute to the relatively high ratio of SDs (5.0), which can affect the reliability of the two-sample hypothesis test (Table 12c). The concentration of copper observed in these crops was within the range expected in nut, fruit, and vegetable crops (ATSDR, 2004b).

Barium was detected in almonds, carrots, garlic, mandarin, navel, pistachio, and Valencia. The median concentration of barium was higher in the treatment group of almonds and the difference was statistically significant. Given that FOD<50% for barium in control and treatment groups of pistachios, the detection frequency was evaluated instead of the difference in means or medians. The difference in FOD (48% for treatment, 35% for control) was not statistically significant (see Table 15c) and the ranges of detects substantially overlapped (see Figure A-62).

While there was some evidence of differences in strontium and barium levels in treatment groups for some crops, these levels were within the concentration ranges expected in fruit and vegetable crops (ATSDR, 2004a; ATSDR, 2007a). Chemical profiles including these metals for each crop are discussed further in Section 3.2 (Crop Specific Results).

Antimony, molybdenum, nickel, and zinc were detected in samples from the same three crops – almonds, pistachios, and garlic. Antimony and nickel were detected in one control sample of pistachios and were not detected in any treatment group samples, so were not included in the counts in Table 4 and Figure 3. Nickel and molybdenum are each detected in 1 of 20 treatment group samples of almonds, and the detection was in the same sample (Alm09-20170808-1115). Of these metals, zinc had the highest within-crop FOD, with detections in all samples of both control and treatment groups for almonds, garlic, and pistachios. The arithmetic mean concentration of zinc was higher in almonds (but not garlic or pistachios) collected from fields irrigated with produced water and the difference was statistically significant. However, the concentration was within the range expected in fruit and vegetable crops (ATSDR, 2005a).

Antimony was detected in 2 of 20 (10%) samples from the control group and 7 of 20 (35%) samples from the treatment group of almonds. These levels were higher than has been reported from national surveys of fruits and vegetables (ATSDR, 2017a). The range for the control group (i.e., 0.64 to 0.73 mg/kg) was comparable to that of the treatment group (0.52 to 0.95 mg/kg) when the maximum of the treatment group (1.8 mg/kg) is excluded (see Figure A-2). The sample for which the maximum was measured happens to be part of a parent/duplicate pair, and the result for the field duplicate sample was 0.77 mg/kg. Therefore, accounting for the within-field variability, there was no difference in the distribution of antimony between control and treatment crops of almonds. Antimony was also detected in 2 of 6 (33%) samples from the control group and 1 of 5 (20%) samples from the treatment group of garlic. The maximum concentrations (0.61 mg/kg) were the same.

Molybdenum was detected in 1 of 20 (5%) samples from both control and treatment groups of almonds, as well as 1 of 5 (20%) samples from the treatment group of garlic. The concentrations were all below the range of molybdenum levels typically reported for crops in the U.S. of 1 to 2 mg/kg (ATSDR, 2017b).

Nickel was detected in 5 of 20 (25%) samples from the control group and 1 of 20 (5%) samples from the treatment group of almonds. The concentrations in the control group were all higher than that of the treatment group (see Figure A-6). Nickel was also detected in 1 of 20 (5%) and 1 of 21 (5%) samples of pistachios from the control and treatment groups, respectively. The results were 1.6 mg/kg for the treatment group and 1.0 mg/kg for the control group. Nickel concentrations detected in tree nuts were within the range expected in food crops (ATSDR, 2005b).

Arsenic, cadmium, chromium, and lead were each detected in the same sample (A-C1-20190725-0820) collected from carrots irrigated with produced water. The detected concentrations were less than the MRL but greater than the MDL, and the sample was one half of a sample pair containing a duplicate. The results for field duplicates were all within 50% of the parent sample and likewise reported as estimated detects. Similarly,

arsenic, chromium, and lead were each measured in a single field duplicate sample (B-C2-20190725-0935) from the control group for carrots and qualified as estimated.

3.1.2 Organics

The most frequently detected organics among the 13 crops in this study were acetone, acrolein, ethyl acetate, p-isopropyltoluene, and methanol (Table 4 and Figure 3). These organic compounds are common and often naturally occur in food crops (ATSDR, 1994; ATSDR, 2007b; US EPA, 2013; US EPA 2011; OEHHA, 2012).

Acetone was the only one of the 13 organics for which there was some evidence from statistical tests that levels detected in the treatment group may be elevated compared with the control group for tomatoes. Acetone was detected in all five control samples and all six treatment samples (see Table 17a to 17c as well as Figure A-74). Both the side-by-side box plots and the rank ordered combined dataset indicated that concentrations in the treatment group were more variable with a slightly higher arithmetic mean. The difference in means was small (i.e., 0.46 mg/kg and 0.63 mg/kg in control and treatment groups, respectively) and a sample size of at least 22 in each group (rather than the 5 and 6 observations per group) would be needed to evaluate the statistical significance (Table 17c). The conclusions from two-sample hypothesis tests depend on how the null hypothesis is defined. Using Form 1 of the null hypothesis, the difference in means was *not* statistically significant (p=0.12), however, the test lacks the power to detect the observed difference given the small sample sizes. Using Form 2, a difference in means of at least 0.34 mg/kg (the minimum difference that the test can evaluate with α=0.05) cannot be ruled out (p=0.11) given the pooled SD. However, this result must also be qualified as uncertain given that the ratio of SDs is 4.7, which indicates that the assumption of homogeneity of variance required for the Student's *t*-test is violated. Acetone is discussed in further detail in crop-specific summaries (see Section 3.2).

Acrolein and ethyl acetate were detected in the same set of seven crops: carrots, potatoes, garlic, tomatoes, apples, grapes, and almonds. Ethyl acetate was detected in all three samples for the control group for garlic but not in any samples from the treatment group, so garlic is not indicated on Table 4 and Figure 3. Mean concentrations, in descending order (highest to lowest) for treatment groups are as follows (underlined crops indicate that the mean concentration is higher in the treatment group than the control group):

- Acrolein: garlic > <u>carrots</u> > <u>almonds</u> > <u>potatoes</u> > tomatoes > apples > grapes
- E. acetate: potatoes > apples > almonds > <u>carrots</u> > grapes > tomatoes

The concentrations of the two chemicals were moderately correlated in tomatoes and almonds with Spearman rank correlation coefficients of 0.5 and 0.4, respectively (see Figures D-1 and D-7). For most of the crop/chemical pairs for these two chemicals, no statistical analysis was needed because the mean or median for the control group was

clearly greater than that of the treatment group. This was true for acrolein in garlic, tomatoes, and grapes, and for ethyl acetate in potatoes, almonds, grapes, and tomatoes. Apples was not further evaluated because the dataset lacks control group samples.

For acrolein in carrots, almonds, and potatoes, there were fewer than 4 detects in control and treatment groups, which precluded formal statistical tests. Acrolein was detected in 2 of 3 (67%) and 5 of 6 (83%) samples of control and treatment groups of carrots, respectively. The concentrations overlapped with the exception of the maximum concentration in the treatment group (1.3 mg/kg), which was approximately three times that of the control group (0.45 mg/kg) and slightly less than the outlier screening threshold (1.4 mg/kg) (Figure A-25). Acrolein was detected in 2 of 20 (10%) and 6 of 20 (30%) samples of control and treatment groups of almonds, respectively (Table 5a). These concentration ranges overlapped and were lower than most of the MDLs of the NDs due to differences in dilution factors (Figure A-10). Acrolein was detected in 2 of 3 (67%) and 3 of 3 (100%) samples of control and treatment groups of potatoes, respectively (Table 16). The median concentrations were comparable (0.12 mg/kg and 0.17 mg/kg in control and treatment groups), though slightly higher in the treatment group.

Ethyl acetate was detected in all samples of carrots (2 for control group and 6 for treatment group). The median concentrations were comparable (0.47 mg/kg and 0.56 mg/kg for control and treatment groups, respectively) and the maximum concentration for the treatment group (1.10 mg/kg) was approximately two times that of the control group (0.54 mg/kg) (Table 7). The sample sizes are too small to quantitatively compare the distributions for the treatment and control groups. Based on visual inspection of the box-and-whisker plots (Figure A-26), the range for the control group is contained within the range of the treatment group, which would be expected if the distributions were the same, given differences in sample sizes.

There are many potential sources of organics that may be unrelated to concentrations present in water used to irrigate the crops in this study. The following are considerations when evaluating seven of the organic chemicals detected in treatment samples, presented here in the same sequence as the complete list of detected chemicals in Table 4. Chemical names are highlighted in bold to facilitate review.

Acrolein is a product of the ripening process and is commonly found in fruits and vegetables (WHO, 2002). In addition, acrolein is also present in a variety of cooked and uncooked foods (WHO, 2002; ATSDR, 2007b). Because of uncertainties associated with measuring the level of acrolein in foods, and in reconstructing typical diets, it is difficult to estimate total daily exposure to acrolein using dietary information. One research group measured urinary metabolites to estimate daily consumption of acrolein and reported daily exposure to be “roughly a few” µg/kg/day. The same researchers also observed that such levels seemed unlikely to pose a health risk (Abraham et al., 2011). If we interpret “roughly a few” µg/day to mean 3 µg/kg/day, a 70 kg person would have a dietary intake of about 210 µg of acrolein. Below, in Section 3.2.2, we present a screening level

exposure assessment of the likely intake of acrolein detected in apples irrigated with blended produced water. This screening level exposure assessment, while uncertain, suggests the level of acrolein detected in apples is within the range of levels expected as part of the normal diet.

p-Isopropyltoluene is a naturally occurring aromatic terpene present in carrots and citrus products such as orange juice and lemon oil (Stofberg and Grundschober, 1987). It is also used as a flavoring agent and is approved for direct addition to food for human consumption (US EPA, 2011).

p-Isopropyltoluene was detected in all of the samples of carrots (Table 7) and between 27% and 70% of samples collected from the four citrus crops (Table 11 through Table 14). The small sample sizes and/or low FOD preclude the use of a two-sample hypothesis test for all but navel oranges. This organic chemical is detected in 4 of 13 (31%) samples from the control group and 4 of 15 (27%) samples from the treatment group of navels. The Fisher's Exact test confirms that this small difference in detection frequency is not statistically significant (Table 13c). The side-by-side box-and-whisker plots and rank ordered values indicate that the distributions for both groups substantially overlap for navel (Figure A-61), as well as lemon (Figure A-47) and mandarin (Figure A-53). For Valencia, p-isopropyltoluene is detected in 1 of 3 (33%) samples in both control and treatment groups (Table 14). The concentration in the control group sample is approximately two times higher. For carrots, the median concentrations are comparable (0.22 mg/kg and 0.26 mg/kg in control and treatment groups, respectively), however, the maximum value for the treatment group (1.5 mg/kg) is approximately three times greater than that of the control group (0.58 mg/kg), and it marginally exceeds the outlier screening threshold of 1.1 mg/kg (Table 7 and Figure A-28). Overall, the available data support a conclusion that the distributions of p-isopropyltoluene are similar in control and treatment groups in the 5 crops where it is detected.

Two alkanones were detected, one in grapes (**2-hexanone**) and one in pistachios (**2-butanone**). Both of these organic chemicals have been reported as naturally present in a wide range of nuts, fruits, and vegetables including roasted filberts (nuts), intact tree-ripened nectarines, dried beans, split peas, lentils, and soybeans (US EPA, 2003a; ATSDR, 2018). Exploratory data analysis indicated that these compounds were detected with approximately the same frequency and concentration ranges in both control group and treatment group samples. 2-Hexanone was detected in 2 of 19 (11%) samples from the control group and 4 of 21 (19%) samples from the treatment group of grapes (Table 10a). The side-by-side box-and-whisker plots and rank ordered values indicated that the distributions for both groups substantially overlap (Figure A-40). 2-Butanone was detected in 10 of 21 (48%) samples from both groups, and the arithmetic mean was slightly greater in the control group of pistachios (Table 15a). The side-by-side box-and-whisker plots and rank ordered values indicated that the distributions for both groups substantially overlap (Figure A-68).

2-Chloroethyl vinyl ether is sometimes used as a ripening control agent, which could have been a potential source from this agricultural practice.

2-Chloroethyl vinyl ether was detected in 1 of 15 (7%) samples from the treatment group for navel oranges at a concentration of 1.1 mg/kg. It was not detected in any of the 13 control samples. The detected concentration (1.1 mg/kg) was equal to the MRL, which was elevated due to a 100x dilution factor. Given the low concentration and detection in just one sample, it is likely this result was an artifact of the laboratory analysis. For this and other samples included in batch 9B15061, the analytical laboratory report from Weck Laboratories states, "*2-CEVE produced low recoveries for lab control spiked QC samples at the mid-level and a high bias at the low-level lab control spiked sample. Any 2-CEVE detections above the MRL would be suspect…*". This suggests that the reported result may be invalid. Considering these lines of evidence collectively, it is unlikely that 2-chloroethyl vinyl ether detected in one sample of navels was related to crop irrigation practices.

Methyl tert-butyl ether (MTBE) is not known to have natural sources and is commonly considered a soil and groundwater contaminant associated with historical uses as a gasoline additive. This use of MTBE was banned in California in 2004. MTBE is also used in some medical applications and as a laboratory solvent (NJDEP, 2014; CDC, 2020).

MTBE was detected in 10 of 21 (48%) samples from both control and treatment groups of pistachios (Table 15a). The side-by-side box-and-whisker plots and rank ordered values indicate that the distributions for both groups substantially overlap and that the concentrations detected in the treatment group may be higher by approximately 10% (Figure A-70). A complete review of all laboratory results, including quality control sample and calibration curves, confirmed that the results were valid as reported. Both control and treatment groups have field duplicates that correspond with a parent sample in which MTBE was detected. The relative percent difference (i.e., difference divided by the mean of the parent/duplicate pair) was low, ranging from 10% to 13%. Given the FOD<50%, a two-sample hypothesis test was precluded, and since there was no difference in the detection frequency, a Fisher's Exact test naturally supports a conclusion that there was no difference in the distributions.

Methanol may accumulate in fruits and vegetables as a natural breakdown product of pectin by pectinesterase (Koch et al., 1999). OEHHA recognizes this potential source when interpreting hazards and risks of methanol content in consumed fruits and vegetables (OEHHA, 2012). Once a sample is processed, methanol concentrations in the sample can increase over a period of hours. In one study, after three hours in storage at 4º C, the concentration of methanol in tomato juice processed from fresh tomatoes rose from 110 mg/L at the start to 202 mg/L after three hours (Hou et al., 2008), which equates to an equal mass-per-mass [mg/kg] concentration assuming tomato juice has a density approximately the same as water. In the same study, lemon and Valencia orange juices had concentrations of methanol as high as 58 mg/L and 89 mg/L, respectively, in samples

refrigerated at 4º C. In samples kept at 30º C for three hours, concentrations of methanol in tomatoes, lemons, and Valencia oranges were observed at 241 mg/L, 56 mg/L, and 145 mg/L, respectively (Hou et al., 2008). Unlike samples reported in Hou et al. (2008), which sampled freshly squeezed juice, samples tested in Task 3 were whole-fruit samples. All of the Task 3 samples are expected to have additional pectin associated with fruit solids; this has been specifically characterized in the pith of citrus fruit (Ojewumi et al., 2018). Additional pectin in whole fruit is therefore likely to contribute to more methanol in samples if there is sufficient pectinesterase. This process may be sufficient to account for the observed concentrations in tomato (360 [treatment group] to 430 [control group] mg/kg) (Table 17a and Figure A-71), citrus (excluding lemons) (<20 to 380 mg/kg) (Table 12a, Table 13a, Table 14 and Figures A-48, A-55), and almonds (detected in 1 of 40 samples in the treatment group at a detectable concentration of 170 mg/kg) (Table 5a and Figure A-1). Based on an evaluation of the frequency of detection, concentration ranges, and rank ordered values, the distributions of methanol detected in these crops was similar for the control and treatment groups.

The remaining 4 organics (**bis(2-ethylhexyl)phthalate**, **bromomethane**, **chloromethane**, **sec-butylbenzene**) were detected in just 1 or 2 samples from treatment groups and 0 to 2 samples from control groups of the crops listed in Table 4. The count of detects per crop/chemical dataset were not sufficient to assess potential differences in chemical profiles for control and treatment groups.

3.2 Crop Specific Results

The evaluation of results by crop focuses on the overall chemical profiles using a variety of multivariate graphical and statistical analysis methods. The methods are designed to facilitate side-by-side comparisons of control and treatment group results, and to identify any specific combinations of chemicals or samples that may explain differences in the observed patterns. As noted in Section 2.3, fewer than four samples were collected in control and/or treatment groups for the following five of thirteen crops: cherries (2 control and 2 treatment [i.e., 2/2]); Valencia (3/3), potatoes (3/3), apples (0/4), and carrots (3/6). While small sample sizes for these crops precluded a more rigorous statistical analysis, observations based on visual inspection of control and treatment group results are included in the summaries of crop-specific results below.

3.2.1 Almonds

The chemical profile for almonds was similar to pistachios and garlic and characterized by a relatively high number of chemicals detected in at least one sample (11 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for four metals and one organic chemical. The side-by-side bar chart (Figure B-1) showed that the overall chemical profile of metals and organics is very similar for both control and treatment groups. For metals, the average profile was comprised of a peak for zinc at a mean concentration of approximately 25 mg/kg, followed by approximately 3-to 4-fold lower mean concentrations of copper and strontium, and relatively low (≤ 1 mg/kg) levels of

antimony, barium, molybdenum, and nickel. The error bars (given by two times the standard error for the sample mean) were relatively narrow and the same for both control and treatment groups, indicative of high sample sizes (n=20) and low variability (coefficient of variation [CV] < 1) (Table 5a). For organics, the profile was comprised of three chemicals: ethyl acetate, followed by 2-fold lower levels of acetone, and 3-fold lower levels of acrolein. The error bars for ethyl acetate were relatively wide for both control and treatment groups, which reflected both the smaller sample sizes (n=10) and greater variability (CV>1) compared with acetone and acrolein (both n=20 and CV<1) (Table 5a).

The treatment group means appeared slightly higher than the control group for some chemicals. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in means (or medians), while marginal, were statistically significant for barium and zinc.

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For almonds (Figure E-1), the majority of the samples were well mixed, indicating no differences in overall chemical profiles. Two groupings were noted in Figure E-1. The treatment group exhibited a cluster for four samples, which were indicative of combinations of low and mid-ranked concentrations for selected metals and organics. The control group also exhibited a cluster of four samples, indicative of a combination of low and high ranked concentrations for some of the same suite of chemicals, notably barium.

3.2.2 Apples

Four compounds were detected in at least one sample for apples (Table 6). No control samples were collected as no apple orchards appropriate for a control group were identified during the study period. The chemical profile consisted of copper, strontium, acrolein, and ethyl acetate. Compared to other crops, the ranges of concentrations detected in samples of treatment group apples were relatively low for both metals and organics. Nevertheless, the potential human health risks associated with consumption of apples with this chemical profile was evaluated.

The health risk was assessed by calculating the ingestion rate that would result in an average daily dose that matches the risk-based toxicity reference values. In other words, given the concentrations measured, we determined the mass of apples a person would need to consume on a daily basis to ingest a dose equivalent to the toxicity screening level adopted for each chemical in the Task 1 report. Assuming each of the four detected chemicals is present at the maximum concentration reported, the amount of apple a 70 kg adult would need to consume each day to ingest the screening level dose is 0.84 kg for copper, 67 kg for strontium, 0.23 kg for acrolein, and 15 kg for ethyl acetate. The 90th percentile consumption rate of apples in the general US population (0.5 kg/d) is lower than the consumption rates required to reach the toxicity reference level for copper, strontium, and ethyl acetate. This estimate of the 90th percentile consumption includes

the consumption of apples in the form of fruit (0.091 kg/day), apple sauce (0.053 kg/day), and apple juice (0.36 kg/day) (CDC, 2003; CDC, 2015).

The calculated ingestion rate for acrolein (0.23 kg/d) is approximately one-half of the 90th percentile ingestion rate for apples and, therefore, warrants closer examination. If we use the Tolerable Daily Intake (TDI) of 0.0075 mg/kg/day developed by the WHO for ingested acrolein (instead of the more conservative screening level developed for acrolein in Task 1), a person could consume approximately 3.5 kg/day of apples without exceeding the TDI. The screening level of 0.0005 mg/kg/day selected in Task 1 is the US EPA Reference Dose (RfD) for ingested acrolein. While WHO and US EPA considered the same toxicity study data (WHO, 2002; US EPA, 2003b), the two organizations selected different studies to use as the basis of their recommended exposure limit. In their final evaluation, the WHO used a 13-week gavage study in Fischer rats and mice (NTP, 1998) to calculate a TDI, while US EPA used a 2-year rat study (Parent et al., 1992) to calculate a RfD. The WHO did not use the Parent et al. (1992) study to derive their TDI because the study was unable to report a cause for the increase in mortality, and no other adverse effects were observed. They also point to patterns in the observed results that are inconsistent with dose dependent mortality (WHO, 2002).

Several points provide perspective on the assessment of health risks from acrolein detected in apples. First, acrolein was also detected across six other crops (Table 4 and Figure 3), and in no cases did the levels in the treatment group appear to be elevated relative to the control group, following statistical analysis (see Section 3.1.2). Second, the range of concentrations detected in apples was consistent with levels found in other food. Such dietary exposure levels are not expected to present an unacceptable health risk to the general population. Acrolein accumulates in fruit as part of the natural ripening process, and the concentrations measured in four samples collected as part of this study (i.e., <0.035 to 0.15 mg/kg) (see Table 6) are consistent with levels reported in the literature (WHO, 2002). As the samples analyzed for this study were collected from the packing house, the amount of time between picking and analysis was extended, which also extends the ripening time. If the irrigation water was an additional source, we would expect to measure higher concentrations.

Finally, even though acrolein was identified as an oil field additive used in the San Joaquin Valley (see Appendix B of the Task 1 report), the physical properties of acrolein (e.g., high volatility and reactivity, readily biodegradable) suggest that it is unlikely that acrolein used in oil production would persist long enough to be present in the blended produced water. Considering these factors, WHO stated that acrolein, “reacts with sulfides in oil/water mixtures to form a non-hazardous, water-soluble product…” (WHO, 2002). Empirical evidence from the current study supports this statement. Acrolein was not detected in the treated produced water or blended produced water used on the crops sampled as part of this study. Thus, it is not likely that produced water was the source of the acrolein detected in the apple samples collected as part of this study.

3.2.3 Carrots

The chemical profile for carrots was similar to citrus characterized by a relatively high number of chemicals detected in at least one sample (11 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for three metals and four organic chemicals (Table 7). The side-by-side bar chart (Figure B-2) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups. For metals, the average profile was comprised of a peak for strontium at a mean concentration of approximately 4.5 mg/kg, followed by approximately 2-fold lower mean concentration for barium, and 4-fold lower concentration for copper. The error bars (given by two times the standard error for the sample mean) were relatively narrow and the same for both control and treatment groups, indicative of the low variability (CV ≤ 0.5) (Table 7). For organics, the profile was comprised of four chemicals of approximately equal proportions: acetone, acrolein, ethyl acetate, and p-isopropyltoluene. The control and treatment group profiles were similar except for acetone, which appeared elevated in the control group and had a much wider error bar. As shown in Figure A-24, this was a result of a small sample size (n=3) and a maximum detected concentration that is nearly 10 times greater than that of the treatment group. The treatment group means appeared slightly higher than the control group means for the other detected chemicals. The small samples sizes (n=3 and 6) precluded formal two-sample statistical tests of means, medians, and detection frequencies.

3.2.4 Cherries

Five compounds were detected in at least one treatment group sample for cherries (Table 8). Copper and strontium were detected in all samples (Figure B-3). Also, cadmium, bromomethane, and chloromethane were detected in one of two treatment group samples. Due to the small number of cherry samples and in accordance with guidance from the statistical analysis plan, these findings were excluded from the bar charts presented in Appendix B. Compared to other crops, the ranges of concentrations of copper and strontium were similar to samples of grapes, potatoes, and tomatoes. Detected levels of copper and strontium in cherries are also within the range of concentrations expected in food crops (ATSDR, 2004a; ATSDR, 2004b). The small samples sizes (n=2 per group) precluded further exploratory data analysis and formal two-sample statistical tests of means, medians, and detection frequencies. See Section 3.1.1 for additional summaries of copper, strontium, and other metals across the crops evaluated. Section 5 also discusses levels of strontium measured in this study in the context of national occurrence data for strontium in groundwater and food crops.

The reporting of a detection of cadmium in a single treated sample of cherries—but not in any of the control samples—was a result of small sample size and an analysis plan that reserved duplicate samples for an independent analysis of within-field variability. A review of the two reserved duplicate samples found that cadmium was detected in the control sample, but not in the treated sample. When the duplicate samples are combined with the set of samples used in the main analysis, cadmium was detected in 1 of 3 treated

and in 1 of 3 control samples at concentrations of 0.26 mg/kg and 0.28 mg/kg, respectively. Given the similar concentrations and detection frequencies in the two groups, there does not appear to be evidence that cadmium accumulated in cherries irrigated with blended produced water at a higher rate than cherries irrigated with conventional water. Also, cadmium was only detected in 54 blended produced water samples at a concentration of 0.004 mg/L, which is below the drinking water standard concentration level of 0.005 mg/L (See Section 4).

Chloromethane and bromomethane in cherries are likely unrelated to the use of produced water for irrigation. Chloromethane was not detected in any of the blended produced water samples and bromomethane was only detected in 1 of 69 blended produced water quality samples at a concentration of 0.0014 mg/L. Additionally, the fate and transport characteristics of chloromethane and bromomethane will reduce their concentrations in water. Chloromethane is “readily biodegradable” in water, based on the OECD 301D biodegradation test, and therefore unlikely to be consistently present in blended produced water at high levels (ECHA, Chloromethane). Bromomethane undergoes photo-hydrolysis with a half-life of 6.6 hours in outdoor surface waters (Wegman et al., 1981). The degradation products of bromomethane are bromide ions and methanol: methanol is readily biodegradable (ECHA, Methanol). Thus, both chloromethane and bromomethane are unlikely to be present in blended produced water when it is applied to food crops.

3.2.5 Garlic

The chemical profile for garlic was very similar to that of almonds and pistachios, although the magnitude of the concentrations was generally lower. The garlic chemical profile was characterized by a moderate number of chemicals detected in at least one sample (8 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for three metals and two organic chemicals. The side-by-side bar chart (Figure B-4) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups. For metals, the average profile was dominated by a single peak for zinc at a mean concentration of approximately 12 mg/kg, followed by approximately 5-fold lower mean concentrations of copper and strontium, and relatively low (≤ 1 mg/kg) levels of antimony, barium, molybdenum, and nickel. The error bars were relatively narrow and the same for both control and treatment groups, indicative of low variability (CV ≤ 0.5) (Table 9a). For organics, the profile was comprised of two chemicals: acrolein, followed by 3-fold lower levels of acetone. Ethyl acetate was detected in the control group (not shown in the profile in Figure B-4 or Table 9a), but not in the treatment group. Acrolein was elevated in the control group and had a much wider error bar. As shown in Figure A-36, this was a result of a small sample size (n=6) and two detected concentrations greater than 40 mg/kg.

The treatment group means appeared slightly higher than the control group for some chemicals. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in means was statistically significant for strontium.

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For garlic (Figure E-2), multiple separate groupings were noted at the first level of the dendrogram indicating that there were likely combinations of chemicals that exhibited some differences between control and treatment group samples. The treatment group exhibited two separate clusters, one that associated three samples based on relatively higher concentrations of strontium and acetone, and a second that associated two samples based on relatively high concentrations of barium, molybdenum, and strontium. None of these compounds exhibited particularly high correlations across samples based on Spearman rank correlations (Figure D-2). The control group also exhibited two separate clusters, one that associated three samples based on relatively higher concentrations of barium, nickel, and methanol, and a second that associated two samples based on relatively higher concentrations of nearly all of the detected metals and organics, including acrolein which was elevated as discussed above.

3.2.6 Grapes

The chemical profile for grapes was very similar to that of potatoes and tomatoes. The grapes chemical profile was characterized by a moderate number of chemicals detected in at least one sample (7 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for two metals and one organic chemical. The side-by-side bar chart (Figure B-5) shows that the overall chemical profile of metals and organics was very similar for both control and treatment groups, with slightly higher mean concentrations in the control group for most chemicals. For metals, the average profile was comprised of a peak for copper at concentrations ranging from approximately 1.25 to 2.25 mg/kg, followed by approximately 2-fold lower mean concentration strontium. The error bars were relatively wide for copper and narrow for strontium, reflecting differences in the variability (Table 10a). For organics, the profile was comprised of three chemicals: ethyl acetate, followed by 1.5-fold lower levels of acetone, and 3-fold lower levels of acrolein. The error bars were consistently wider for organics detected in the control group, which reflected greater variability (Table 10a).

The treatment group mean appeared slightly higher than the control group for strontium. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in medians (rather than means due to FOD<100%) was statistically significant. This finding was consistent with the side-by-side box-and-whisker plots (Figure A-38), which showed a group of 3 of 21 observations in the range of 1 to 2 mg/kg, whereas the detects for the control group were clustered between 0.5 and 1 mg/kg.

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For grapes (Figure E-3), the majority of the samples were well mixed, indicating no differences in overall chemical profiles. Two groupings were noted in Figure E-3. The treatment group exhibited a cluster of five samples, which were indicative of combinations of relatively higher concentrations

of organics. The control group exhibited a cluster of three samples, indicative of a combination of higher concentrations of copper, acetone, and ethyl acetate.

3.2.7 Lemons

The chemical profile for lemons was very similar to that of other citrus samples, with a minor difference that barium was only detected in samples from the control group, and so was excluded from Figure B-6. The lemons' chemical profile was characterized by a relatively small number of chemicals detected in at least one sample (5 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% only for strontium. The side-by-side bar chart (Figure B-6) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups, with slightly higher mean concentrations in the treatment group for most chemicals. For metals, the average profile was comprised of a peak for strontium at concentrations ranging from approximately 1.7 to 2.3 mg/kg, followed by approximately 5-fold lower mean copper concentration. The error bars were relatively narrow, reflecting low variability (CV≤1) and moderate sample sizes (n=9 and 10) (Table 11a). For organics, the profile was comprised of three chemicals: a peak for p-isopropyltoluene, followed by trace levels (<0.5 mg/kg) of acetone and limonene. The error bars were relatively wide for p-isopropyltoluene for both groups, which reflected high variability (CV>1.5) (Table 11a).

The mean of the treatment group appeared slightly higher than the control group for each chemical. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in means was statistically significant for strontium. This finding was consistent with the side-by-side box-and-whisker plots (Figure A-45), which showed concentrations appear approximately 50% higher on average in the treatment group.

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For lemons (Figure E-4), the majority of the samples were well mixed, indicating no differences in overall chemical profiles. Two groupings were noted in Figure E-4. The treatment group exhibited a cluster of four samples, which were indicative of combinations of relatively higher concentrations of copper and strontium. The control group exhibited a cluster of four samples, indicative of a combination of higher concentrations of methanol and moderate levels of strontium.

3.2.8 Mandarin Oranges

The chemical profile for mandarin was very similar to that of other citrus. The mandarin chemical profile was characterized by a moderate number of chemicals detected in at least one treatment group sample (7 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for two metals and one organic. The side-by-side bar chart (Figure B-7) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups, with slightly higher mean concentrations in the treatment group for most chemicals. For metals, the average profile was comprised of a peak for strontium at approximately 2 mg/kg, followed by

approximately 4-fold lower mean concentration copper, and 8-fold lower mean concentration of barium. The error bars were relatively narrow, reflecting low variability (CV≤1) and moderate sample sizes (n=10) (Table 12a). For organics, the profile was comprised of two chemicals: p-isopropyltoluene, followed by trace levels (<0.5 mg/kg) of acetone. The error bars were relatively wide for p-isopropyltoluene for both groups, which reflected high variability (CV>1) (Table 12a). Methanol was detected in 4 of 10 samples in both the control and treatment groups, with treated samples having slightly higher estimated mean concentrations (Table 12a). While the frequency of detection was too low to perform statistical tests, we present additional material supporting the conclusion that the methanol we observe in these crop samples is due to the breakdown of pectin by pectinesterase (below, see Section 5).

The control and treatment group means were very similar, and the error bars overlapped in each case. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in means and medians were not statistically significant.

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For mandarin (Figure E-5), the samples were well mixed with no evidence of separate clusters, indicating no differences in overall chemical profiles.

3.2.9 Navel Oranges

The chemical profile for navel oranges was very similar to that of other citrus samples and was characterized by a moderate number of chemicals detected in at least one treatment group sample (8 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% only for strontium. The side-by-side bar chart (Figure B-8) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups, with slightly higher mean concentrations in the control group for most chemicals. For metals, the average profile was comprised of a peak for strontium at approximately 2 to 2.5 mg/kg, followed by approximately 5-fold lower mean concentration barium, and 6-fold lower mean concentration of copper. The error bars were relatively narrow, reflecting low variability (CV≤1) and moderate sample sizes (n=13 and 15) (Table 13a). For organics, the profile was comprised of three chemicals: a peak for p-isopropyltoluene, followed by trace levels (<0.5 mg/kg) of acetone and limonene. The error bars were relatively wide for p-isopropyltoluene for both groups, which reflected high variability (CV>1.5) (Table 13a).

The control and treatment group means were very similar, although the error bars did not quite overlap for barium and copper, both of which were elevated in the control group. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in means and medians were not statistically significant.

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For navels, the majority of the

samples were well mixed, indicating no differences in overall chemical profiles. Three groupings were noted in Figure E-6. The treatment group exhibited two clusters of samples. The first group (A) associated five samples based on relatively higher concentrations of barium, copper, strontium, methanol and a single detect of 2-chloroethyl vinyl ether. The second group (B) associated three samples based on relatively higher concentrations of methanol and moderate concentrations of barium and strontium. The control group exhibited one cluster of three samples that were associated based on relatively higher concentrations of methanol and moderate concentrations of barium and strontium.

3.2.10 Valencia Oranges

The chemical profile for Valencia oranges was very similar to that of other citrus samples. The Valencia oranges' chemical profile is characterized by a relatively low number of chemicals detected in at least one treatment group sample (6 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for barium and strontium. The side-by-side bar chart (Figure B-9) showed that the overall chemical profile of metals and organics have some similarities between control and treatment groups. For metals, the average profile was comprised of strontium at approximately 3 to 4 mg/kg, followed by approximately 4-fold lower mean concentration of barium and copper (which was detected in the control group only). The error bars were relatively narrow, reflecting low variability (CV≤1) (Table 14a). For organics, the profile was comprised of three chemicals: a peak for p-isopropyltoluene, followed by trace levels (≤0.5 mg/kg) of acetone and limonene. No error bars were shown because each chemical was detected in just 1 of 3 samples, which was insufficient to estimate the SD and SE (Table 14a). Methanol was detected in 1 of 3 samples in both the control and treatment groups, at approximately the same concentration.

The small number of detects (n=3 per group) precluded formal statistical tests or hierarchical cluster analysis of concentrations in Valencia oranges. The control and treatment group ranges and means were very similar. Control and treatment group maximum results were less than a factor of 1.5 higher in treatment groups for metals (barium and strontium) and less than a factor of 2 higher in control groups for organics (limonene, acetone, and p-isopropyltoluene).

3.2.11 Pistachios

The chemical profile for pistachios was similar to almonds and garlic and characterized by a relatively high number of chemicals detected in at least one sample (9 of the 24 chemicals detected in treatment groups across all crops) and FOD≥70% for three metals but lower FODs for organics. The side-by-side bar chart (Figure B-10) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups. For metals, the average profile was dominated by a single peak for zinc at a mean concentration of approximately 12 mg/kg, followed by approximately 2-fold lower mean concentrations of copper, 6-fold lower concentration in strontium, and

relatively low (≤ 1 mg/kg) levels of barium and nickel. The error bars were narrow and the same for both control and treatment groups, indicative of high sample sizes (n=20 and 21) and low variability (CV< 1) (Table 15a). For organics, the profile was comprised of three chemicals: a peak for 2-butanone, followed by 2-fold lower levels of acetone, and trace levels (≤0.5 mg/kg) of MTBE. The error bars for 2-butanone were relatively wide for both control and treatment groups, which reflected greater variability (CV>1) compared with acetone and MTBE (both n=20 and CV<1) (Table 15a).

The treatment group means appeared slightly higher than the control group for some chemicals. The results of two-group hypothesis testing discussed in Section 2 indicated that the differences in means (or medians) were not statistically significant (Table 15c).

A hierarchical cluster analysis provided an indication of the sample-by-sample similarities when both control and treatment groups were combined. For pistachios, the majority of the samples were well mixed, indicating no differences in overall chemical profiles. Three groupings were noted in Figure E-7. The first group (A) associated five treated samples based on relatively higher concentration of barium, strontium, zinc, acetone, and MTBE. The second group (B) associated six treated samples based on low levels of copper, strontium, and zinc. The last group (C) associated four samples (two treated and two control) based on relatively higher concentrations of zinc, moderate levels of copper, and lower levels of strontium.

3.2.12 Potatoes

Purple potatoes were collected by Cawelo WD within the North Kern Water Storage District. Three samples irrigated with blended produced water were collected from a cold storage facility subsequent to harvest, as notification of the harvest came late. There were no available purple potato samples collected from farms that had been irrigated with conventional water sources. Instead, three samples of purple potatoes from a local grocery store were purchased and used as controls. The product labels indicated these purple potatoes were grown and harvested in Montana.

The chemical profile for potatoes was very similar to that of grapes and tomatoes, but with greater variability between control and treatment groups, which was likely attributable to the smaller sample sizes. The potatoes chemical profile was characterized by a low number of chemicals detected in at least one sample (5 of the 24 chemicals detected in treatment groups across all crops), but FOD≥70% for each chemical. The side-by-side bar chart (Figure B-11) shows that, when detected, the overall chemical profile of metals and organics was similar for both control and treatment groups. For metals, the average profile was comprised of copper and strontium at concentrations ranging from approximately 0.7 to 1 mg/kg. Strontium was detected in all three samples from the treatment group, and none from the control group. No field duplicate samples were available for potatoes. The error bars were relatively narrow, reflecting very low within-group variability (CV< 0.1) (Table 16). For organics, the profile was comprised of three chemicals: a peak for ethyl acetate, followed by 8-fold lower levels of acetone, and 15-

fold lower levels of acrolein. Some error bars were wider for organics than for metals, which reflected greater variability (CV ≥ 1) (Table 16).

The small number of detects (n=3 per group) precluded formal statistical tests or hierarchical cluster analysis of concentrations in potatoes. The treatment group mean for copper appeared approximately 1.2 times higher than the control group. The treatment group maximum for copper was 1.1 mg/kg, compared with the control group maximum of 0.90 mg/kg. Strontium was not detected (i.e., <0.25 mg/kg) in the three control groups samples and was detected in the three treatment group samples at concentrations ranging from 0.65 to 0.75 mg/kg. Strontium detected in Task 3 samples of potatoes are approximately 3-fold lower than the average concentration reported by ATSDR (2004a) for potatoes of 2.6 mg/kg (ATSDR, 2004a). Maximum concentrations of organics were higher in control groups for acetone and ethyl acetate, and higher in the treatment group for acrolein (Table 16).

In potatoes, the maximum concentration of acrolein in the treatment group (0.2 mg/kg) is only slightly higher than the highest concentration observed in apple samples (0.15 mg/kg). Based on the screening analysis presented in Section 3.2.2 for acrolein in apples, we can also conclude that acrolein levels in potatoes are of limited concern. The screening analysis showed that 3.5 kg of apples per day would have to be consumed to reach a dose equivalent to the TDI. Similarly, one would have to consume 2.6 kg of potatoes to reach a dose equivalent to the TDI. Chronic consumption rates of this magnitude are unlikely for both of these crops.

See Section 3.1.1 for additional summaries of copper and strontium across the crops evaluated. Section 5 also discusses levels of strontium measured in this study in the context of national occurrence data for strontium in groundwater and food crops

3.2.13 Tomatoes

The overall chemical profile for tomatoes was very similar to that of grapes and potatoes. The tomatoes chemical profile was characterized by a low number of chemicals detected in at least one sample (6 of the 24 chemicals detected in treatment groups across all crops), and FOD=100% for each chemical except strontium. The side-by-side bar chart (Figure B-12) showed that the overall chemical profile of metals and organics was very similar for both control and treatment groups. For metals, the average profile was comprised of copper and strontium at concentrations ranging from approximately 0.3 to 0.7 mg/kg. The error bars were relatively narrow, reflecting very low within-group variability (CV< 0.5) (Table 17a). For organics, the profile was comprised of three chemicals: two peaks for acetone and ethyl acetate, followed by 2-fold lower levels of acrolein. The error bars were also relatively narrow for organics, reflecting very low within-group variability (CV< 0.5) (Table 17a).

The control group means appeared slightly higher than the treatment group for chemicals except acetone. The results of two-group hypothesis testing for acetone, discussed in

Section 2, indicated that the differences in means of 0.17 mg/kg was not statistically significant using Form 1 of the null hypothesis. However, the *t*-test lacked sufficient power to detect a difference with the small sample sizes (n=5 and 6). At least n=22 in both control and treatment groups would be needed to evaluate the observed difference in means given the pooled SD of 0.22 mg/kg and specified error rates. With Form 2, the sample sizes were sufficient to evaluate a difference in means of 0.34 mg/kg, and to conclude that the treatment mean may be elevated (p=0.11). Notably, the sample variance of the treatment group was nearly 5-fold greater than the control group, which introduced uncertainty in findings from hypothesis testing. Both the side-by-side box plots and the rank ordered combined dataset indicated that concentrations in the treatment group were more variable with a slightly higher arithmetic mean (Figure A-74). The range of concentrations for the control and treatment groups overlapped, but the treatment group range was wider (0.2 to 1.1 mg/kg).

A hierarchical cluster analysis provides an indication of the sample-by-sample similarities when both control and treatment groups are combined. For tomatoes (Figure E-8), the majority of the samples were well mixed, indicating no differences in overall chemical profiles. One grouping was noted for two samples from the treatment group. The association for these samples was attributable to relatively higher concentrations of copper and strontium.

3.2.14 Classification of Chemical Profiles and Crop Groupings

If the environmental conditions were similar and the primary sources of the target analytes in this study were associated with irrigation water, then crops that exhibit similar uptake mechanisms may also exhibit similar chemical profiles. A key assumption in this study was that the variability in environmental conditions, crop management practices, and crop uptake mechanisms were similar across control and treatment fields, so that the main variable that explained the chemical profiles could be attributed to differences in the chemical composition of irrigation water along with differing chemical uptake efficiencies across crops.

Table 18 synthesizes the information from the crops-specific analysis presented above. This table summarizes key observations regarding the relative proportions of the concentrations of chemicals detected within a crop as well as between crops. Crops with few samples (e.g., cherries) were excluded because the small sample sizes introduce uncertainty in the associations across crops. To facilitate an analysis of between-crop profiles, a series of stiff plot diagrams was used. As described in the Methods (Section 2.6.1), each plot illustrates the relative magnitude of concentrations of multiple constituents when crops are plotted on the same scale. Each crop was depicted by a set of four polygons to show control and treatment groups side-by-side, each represented by two chemical classes. The top polygon represented four frequently detected metals (barium, copper, strontium, zinc) and the bottom polygon represented four frequently detected organics (acetone, acrolein, ethyl acetate, and p-isopropyltoluene). Non-detects were included at the maximum MDL of the samples qualified as NDs within a

crop/chemical group. Figure C-1 shows arithmetic mean stiff plots for almonds, carrots, garlic, and grapes. Figure C-2 combines the citrus crops. Figure C-3 shows pistachios, potatoes, and tomatoes.

Based on the relative bar chart profiles, chemistry profiles could be classified into three categories for metals (labeled A, B, and C on Table 18) and two categories for organics (labeled D and E). Four crops (carrots, garlic, cherries, and pistachios) showed unique organic chemical profiles and were excluded from the classification. Crops were visually grouped based on relative peak height patterns (i.e., without regard to differences in the magnitude or mean concentration of the analyte).

Group A for metals consisted of tree nut crops (almonds and pistachios) and garlic. This grouping was characterized by high levels of zinc and copper with lesser concentrations of strontium, barium, antimony, and molybdenum. Relative antimony and barium concentrations were low for this crop grouping and absent altogether in pistachios. Also, the absolute concentration of copper for almonds was twice that of other crops in this group. Garlic and pistachios had similar profiles for metals except the copper concentration, which was approximately two times greater in pistachios.

Group B for metals consisted of a combination of fruit and vegetable crops: cherries, grapes, potatoes, and tomatoes. This grouping was characterized by relatively high copper and strontium concentrations for which copper equaled or exceeded strontium. Absolute mean concentrations for potato and tomato were approximately 80% and 50% that of cherries, respectively. Mean concentrations for grapes were effectively the same as cherries except the copper treatment concentration, which was twice that of the cherries.

Group C for metals consists of citrus and carrots. This grouping was characterized by relatively higher strontium than copper concentrations and variable lower concentrations of barium. The mean concentrations of strontium and copper in carrots and Valencia oranges were twice that of lemons, mandarin, and navel oranges.

Group D for organics closely resembled Group B for metals except almonds replaced cherries: almonds, grapes, potatoes, tomatoes. This grouping was characterized by a peak concentration of ethyl acetate, followed by lower concentrations acetone, and even lower levels of acrolein. Mean concentrations of organics were approximately the same for almonds, grapes, and tomatoes. The profile for potatoes had a two-folder higher peak for ethyl acetate and acetone, and roughly the same level of acrolein.

Group E for organics consisted of citrus (similar to Group C for metals). This grouping was characterized by a peak concentration of p-isopropyltoluene, followed by lower concentrations of acetone. The mean concentrations were roughly equivalent for lemon, navels, and Valencias. Concentrations for mandarins were approximately 2-fold lower.

The similarities in chemical profiles across multiple crops, coupled with the observations that the profiles were very similar between control and treatment groups in nearly every dataset, provided a strong line of evidence that there was no systematic difference in chemical profiles based on sources of irrigation water. Small differences in means and medians, some of which were statistically significant, were present in the crop/chemical datasets, however, such differences would be expected to be more systematically observed across multiple crops if the key factor was a difference between chemicals in irrigation water. The chemical profiles of the irrigation water are reviewed in the next section.

4.0 TREATED PRODUCED WATER AND BLENDED PRODUCED WATER SAMPLING

GSI reviewed water quality data collected by the Central Valley Water Board collected in accordance with WDRs mandated for the beneficial reuse of produced water for agricultural irrigation. Under the WDRs, data were collected for both treated produced water and blended produced water before it is used for irrigation. The collection and review of this data by the Central Valley Water Board is to ensure that the Discharger is in compliance with the WDRs. Part of these requirements are meeting daily and/or annual water quality limits specified in each of the WDRs (unique to each of the WDRs).

The remainder of this section reports comparisons of chemical concentrations observed in treated produced water and blended produced water to drinking water standards. In the absence of agricultural water quality standards designed to protect human health, we have used maximum contaminant level (MCL) drinking water standards as a reference level to identify concentrations of chemicals in water that may be elevated. Table 19 provides a summary of the MCLs available for the 24 chemicals detected in treated crops. Drinking water standards, however, are not available for many of the detected chemicals.

4.1 Summary of Water Quality Data

Water quality data were compiled for the period ranging 1967 through September 2019; most of the available data are from samples collected from 2014 onwards. Table 20 and Table 21 provide summary statistics of the water quality data for treated produced water and blended produced water, respectively. In Tables 20 and 21, we report summary statistics that include sample size, number of non-detects, FOD, range of detected values, arithmetic mean, and the count and percentage of detections that exceed WQOs outlined in Table 19.

In both treated produced water and blended produced water samples, seven of 24 chemicals were reported as non-detects. For treated produced water samples, these seven chemicals were 2-chloroethyl vinyl ether, acrolein, cadmium, chloromethane, ethyl acetate, methanol, and MTBE. For blended produced water samples, these seven chemicals were 2-butanone, 2-chloroethyl vinyl ether, 2-hexanone, acrolein, chloromethane, ethyl acetate, and MTBE.

Where drinking water standards were available, chemical concentrations in treated produced water and blended produced water did not exceed MCLs for most of the chemicals detected in food crop samples. However, arsenic exceeded the MCL in 75% of the treated produced water samples. In blended produced water, the MCL for arsenic was exceeded 53% of the time; and there was one sample in which antimony exceeded the MCL of 0.006 mg/L, with a reported concentration of 0.011 mg/L.

Arsenic was the only chemical detected in irrigation water that frequently exceeded the drinking water quality standard. Nevertheless, for the crops sampled in this study, arsenic was detected just once, in 1 of 6 samples of carrots in the treatment group with an estimated concentration of 0.081 mg/kg (MDL = 0.07 mg/kg and MRL = 0.5 mg/kg) (see Figure A-12). Levels detected in this blended produced water were marginally higher than the relevant drinking water standards. The current federal and state drinking water MCL for arsenic is 0.01mg/L and the maximum concentration reported in blended produced water was 0.065 mg/L. The current MCL was lowered from 0.05 mg/L to 0.01 mg/L by US EPA in January 2006 and by California in November 2008.

As with many watersheds in the U.S., arsenic is a common naturally occurring metal found in the San Joaquin Valley ground water sources. The levels of arsenic measured in effluent and blended irrigation were similar to levels reported from water samples collected from shallow aquifers within the Tulare Basin of the San Joaquin Valley (Fuji and Swain, 1995). Concentrations vary by subzones within the study area, with medians ranging 1 to 20.5 µg/L and maximums ranging 12 to 2,600 µg/L (Fuji and Swain, 1995). Given that arsenic was only detected at trace levels in a few carrot samples, it does not appear that arsenic in blended produced water was affecting levels of arsenic in irrigated food crops.

While MTBE was not detected in any water samples. Nevertheless, it was detected in pistachio samples collected in 2019, which are approximately half of those collected in both treated and control samples (Table 15a). Given that MTBE is not detected in irrigation water, and that MTBE was only detected in one crop, the source of MTBE is unclear.

While the majority of the 24 chemicals detected in the 13 crops were also present in treated produced water and blended produced water, there was no apparent correlation with observed concentrations, detection frequency, or rates of exceedance of MCLs of these chemicals in treated produced irrigation water, prior to and after blending.

5.0 DISCUSSION

In support of the purpose of the project, which was to address the Central Valley Water Board's goal "to investigate and develop additional knowledge to address public concerns regarding the safety of irrigating food crops with treated produced water.", this report presents the results of chemical analyses comparing food crops irrigated with blended produced water (treated) and crops irrigated with conventional irrigation water sources

(controls). Samples of 13 crops were collected from a variety of fields in 2017 through 2019 and tested for 113 target analytes associated with oil and gas production. Of these 113 target analytes, 24 were detected in one or more samples from the treatment groups, resulting in a total of 89 crop/chemical datasets that were evaluated following a systematic process involving multiple data analysis and statistical analysis methods. A total of 36 crop/chemical datasets had a sufficient number of detects in both control and treatment groups to evaluate quantitatively. This dataset consists of the following 11 chemicals:

Type of Chemical	Chemical Name
Metals:	• barium • copper • strontium • zinc
Volatile organic compounds (VOCs):	• acetone • acrolein • 2-butanone • ethyl acetate • methyl tert-butyl ether (MTBE) • p-isopropyltoluene
Alcohol:	• methanol

We analyzed the data using a variety of approaches using methods consistent with US EPA guidance for statistical analysis of environmental data. These approaches included outlier screening, parameter estimation of censored data, correlation analysis, and hypothesis testing. Multivariate analyses included hierarchical cluster analysis and graphical analysis with bar charts and dendrograms.

The majority of chemicals detected are naturally occurring in fruits, vegetables, and nuts, and the concentration ranges were consistent with ranges observed in food crops found in the marketplace. Furthermore, results of the exploratory data analysis and statistical analysis indicated that for most of the 36 crop/chemical datasets, the distribution of concentrations was the same between control and treatment groups. In the following six cases, there was some evidence that the distributions are not the same and the difference in means/medians was statistically significant:

- strontium in garlic, grapes, lemons
- barium and zinc in almonds
- acetone in tomatoes

In the case of methanol in citrus fruit, we observed that the estimated mean concentrations in treated samples of Mandarin, Navel, and Valencia oranges were higher than control samples, but there was no evidence that these differences were statistically different. Below, we further discuss the presence of methanol in citrus samples.

For the remaining chemicals listed above, the weight of evidence, including statistical analysis and graphical analysis, support the conclusion that differences between treatment and control groups were not statistically significant. Small samples sizes (i.e., n<4) precluded the use of statistical analysis to compare distributions for cherries, Valencia oranges, potatoes, carrots, and apples. Maximum concentrations were higher in treatment groups for some of the same chemicals: barium in carrots and Valencia oranges; strontium in cherries, Valencia oranges, and potatoes; acetone in Valencia oranges (but lower in potatoes); and copper in cherries and potatoes.

Strontium was detected in all 13 crops and, with the exception of garlic, grapes, and lemons, the distributions for control and treatment groups appeared to be the same. Blended produced water may have contributed an additional source of strontium to some crops irrigated in the San Joaquin Valley, albeit a small source. Based on a review of the water quality data, strontium was detected in 87% of blended produced water samples with detected values ranging from 0.018 – 0.46 mg/L. In comparison, the National Contaminant Occurrence Database reports that in 4,353 of 4,383 groundwater samples in the United States the average concentration was 1.6 mg/L (range, 0.0009–200 mg/L); in lakes and reservoirs this database reports that the average concentration was 1.09 mg/L (range, 0.002–170 mg/L) (US EPA, 2002b). Among samples collected by the United States Geological Survey, as published on the California Water Boards' Groundwater Ambient Monitoring and Assessment Program (GAMA) website, strontium levels in municipal water source wells in Kern County have mean concentrations of 0.73 mg/L (range, 0.008 – 2.8 mg/L) (CWB, 2015). These data all suggest that strontium levels in produced water were low compared with other potential sources of irrigation water.

Uptake of strontium by plants is typically a passive process (Isermann, 1981). Consequently, higher concentrations of strontium in irrigation water will typically lead to higher concentrations in the plants. Strontium, however, is also a naturally occurring element in soil and the observed concentrations of strontium in these food crop samples were consistent with what has been observed in food crops nationally (ATSDR, 2004a).

While the statistical results suggested that barium and zinc in almonds were higher in the treated samples, evidence suggested that these levels were within those expected for almonds. Observed mean concentration of barium in treated almonds was 1.45 mg/kg with an observed maximum concentration of 2.4 mg/kg. Rodushkin et al. (2011) reported that median concentration of barium in almonds is 2 mg/kg. For additional context, concentrations of barium expected in fruit and vegetables range from 0.047 to 3.75 mg/kg (ATSDR, 2007a). The observed mean concentration of zinc in treated almonds was 26.5 mg/kg with an observed maximum concentration of 39.0. Rodushkin et al. (2011) reported

that the median concentration of zinc in almonds is 33 mg/kg with a standard deviation of 3.3. Assuming a near normal distribution, the standard deviation of 3.3 would imply that the maximum concentration of zinc in almonds is likely to approach or exceed 39 mg/kg. Based on the above noted literature, concentrations of barium and zinc in almonds irrigated with blended produced water were similar to what might be naturally expected in this nut crop.

Acetone was unlikely to be associated with true differences in concentrations in sources of irrigation water, given that it was frequently detected across all crops except apples and cherries, and the only observed difference in distributions was for tomatoes. Acetone is also known to occur in tomato fruit, but quantified concentrations are not available (USDA, 2020).

Methanol is known to be naturally occurring in fruit and the results we observe here are congruent with findings reported elsewhere. As discussed earlier, methanol was observed in freshly squeezed tomato and citrus juice samples (Hou et al., 2008). The pattern of findings in Hou et al. (2008) further confirm the conclusion that methanol identified in crop samples irrigated with produced water is due to the breakdown of pectin by pectinesterase. In Hou et al. (2008), methanol was observed in tomatoes, Valencia oranges, and lemons, but after samples were held for three hours, methanol concentrations in tomatoes and Valencia oranges were elevated while in lemon juice they were greatly reduced over a three-hour period (Hou et al., 2008). This is similar to observations in samples irrigated with produced water, where methanol was detected in samples of tomatoes, Mandarin oranges, Navel oranges, and Valencia oranges, but not in lemons.

For apples and potatoes, which lacked standard control samples, we conducted consumption-based hazard assessments. In the case of apples, no control samples were collected; in the case of potatoes, the control samples were purchased and sourced from Montana. Results from both of these hazard assessments indicate there was minimal risk from consuming either of these crops.

Chemicals that were infrequently detected among the 13 crops and evaluated qualitatively include four metals (arsenic, cadmium, chromium, and lead) and nine organics (limonene, bis(2-ethylhexyl)phthalate, 2-butantone, 2-chloroethyl vinyl ether, 2-hexanone, bromomethane, chloromethane, MTBE, and sec-butylbenzene). Since these compounds were detected infrequently, the likelihood that they are indicators of differences between irrigation water sources was judged to be low. Some of these compounds are naturally occurring as part of the food ripening process or may be introduced in agricultural practices.

5.1 Uncertainties and Data Gaps

To the extent practicable, the fields selected to represent the control and treatment groups were matched by growing season and general geographical areas. However, there are

numerous factors that may be different across the two groups, and these differences could contribute to decision errors. While sampling included control and treatment groups, there were a few uncontrolled variables in this empirical study that might have affected the results. The following additional points are important to keep in mind when evaluating conclusions from this study.

- **Water Use Practices**. One of these variables was the amount of produced water applied to each crop, as farmers typically use other sources of irrigation water in combination with the blended produced water. Another related variable may have been the total amount of blended produced water applied to each field over time or information on specific application rates.

- **Chemical Composition of Conventional Irrigation Water**. In addition, we do not have information on the variability of the chemical composition of conventional irrigation water (surface water and groundwater) used on crops in the control groups.

- **Homogeneity of Field Soil Conditions**. Differences in soil between treated and control fields may have influenced the results, as well. Although sites were picked in an attempt to obtain the same farmer for control and treatment sample sites, there may be different agricultural practices between farmers or in treated and control fields, including application of soil amendments, tillage practices, use of pesticides, and use of fertilizers.

- **Lack of Control Groups**. As discussed, there were no control group samples for apples, and the control group for potatoes is based on samples purchased from a grocery store and sourced in Montana instead of California.

- **Small Sample Sizes**. Uncertainty in parameter estimation varies depending on sample sizes, frequency of detection, and magnitude of variance across sample results. Sample sizes were insufficient ($n<4$) to conduct hypothesis testing on results for cherries, Valencia oranges, potatoes, apples, and carrots.

For most of the crop/chemical combinations, these potential confounding factors do not appear to have contributed substantially to the variability in concentrations measured in the 13 crops.

6.0 CONCLUSIONS

Exploratory data analysis and statistical analysis applied to the chemical residue data, along with supporting information from national food surveys and chemical profiles of produced water supported the following conclusions:

- Levels of metals and organics detected in crops of fruits, vegetables, and nuts grown in the San Joaquin Valley were within ranges expected for food supplies in the U.S.

- In spite of statistical differences between treated and control samples that were identified for some specific crop/chemical combinations, there were no indications that these crops were different than those found in the marketplace, and the overall chemical profiles of these treated and control crops were the same.

- The chemical profiles were very similar for several groups of crops, which may help to establish baseline conditions and guide future studies with similar objectives

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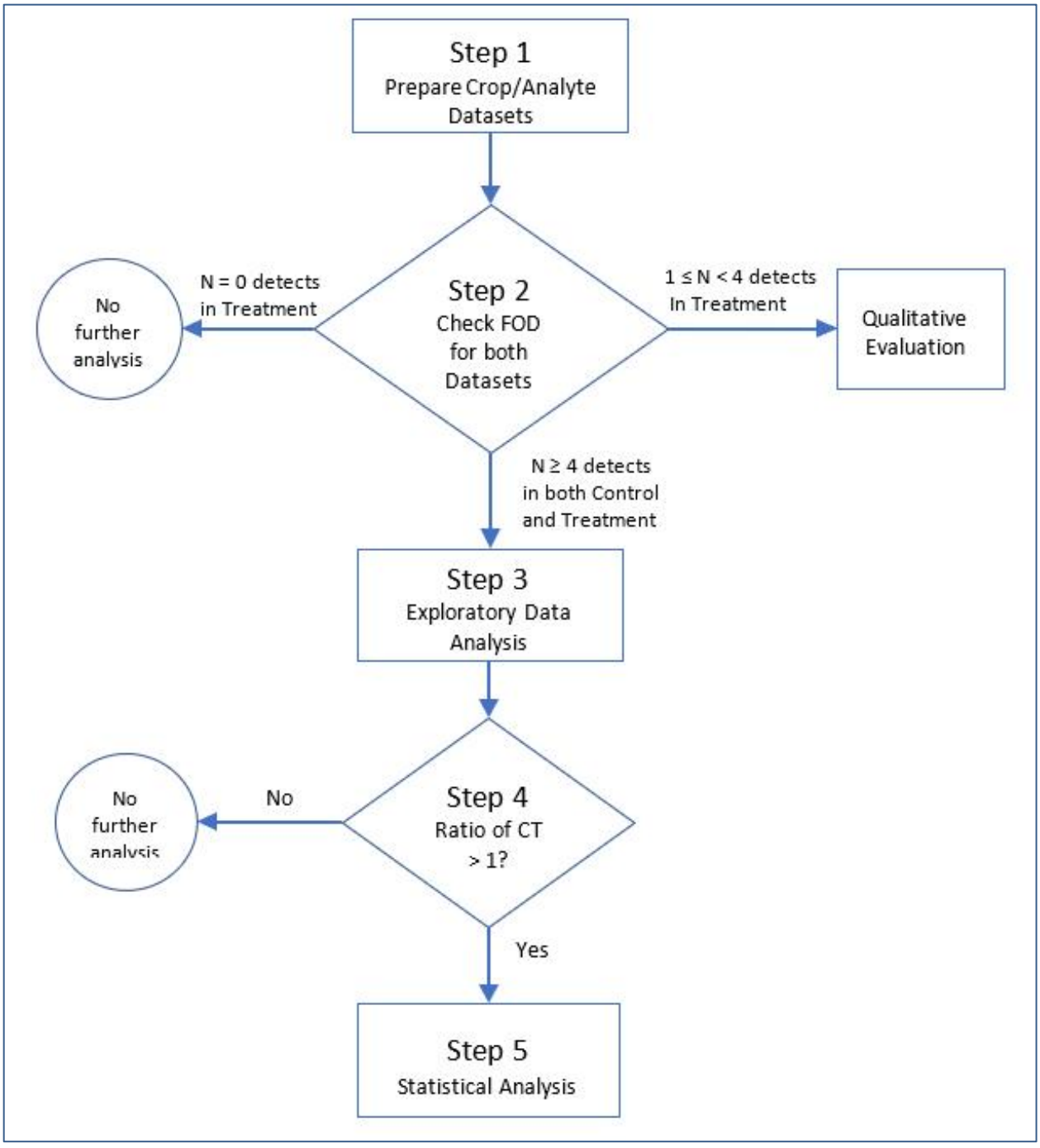

Figure 1. Process for selecting datasets for exploratory data analysis and statistical analysis. CT = central tendency (mean or median); FOD = frequency of detection.

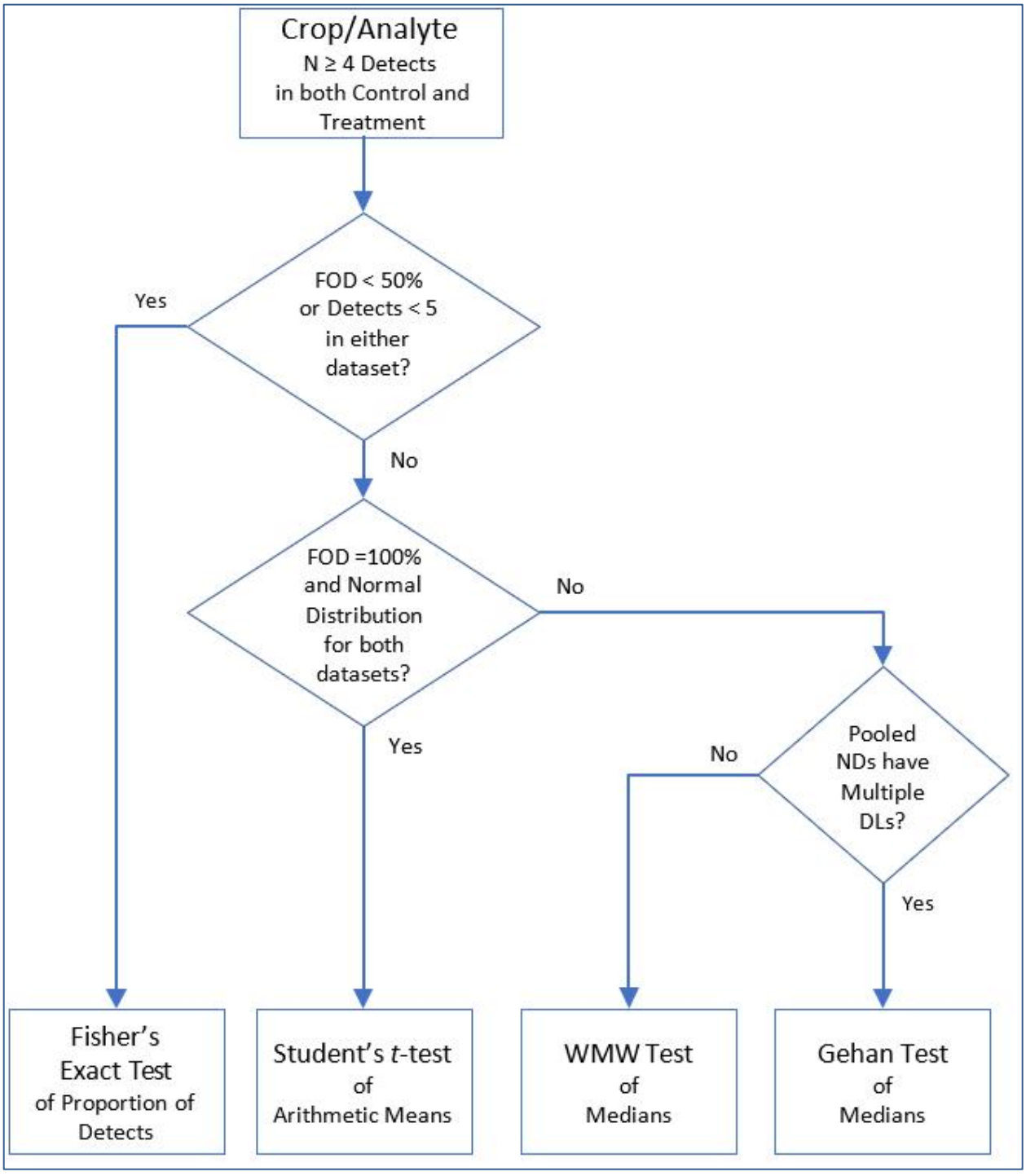

Figure 2. Decision process for selecting a two-sample hypothesis test. DL = method detection limit; ND = non-detect; FOD = frequency of detection; WMW = Wilcoxon-Mann-Whitney

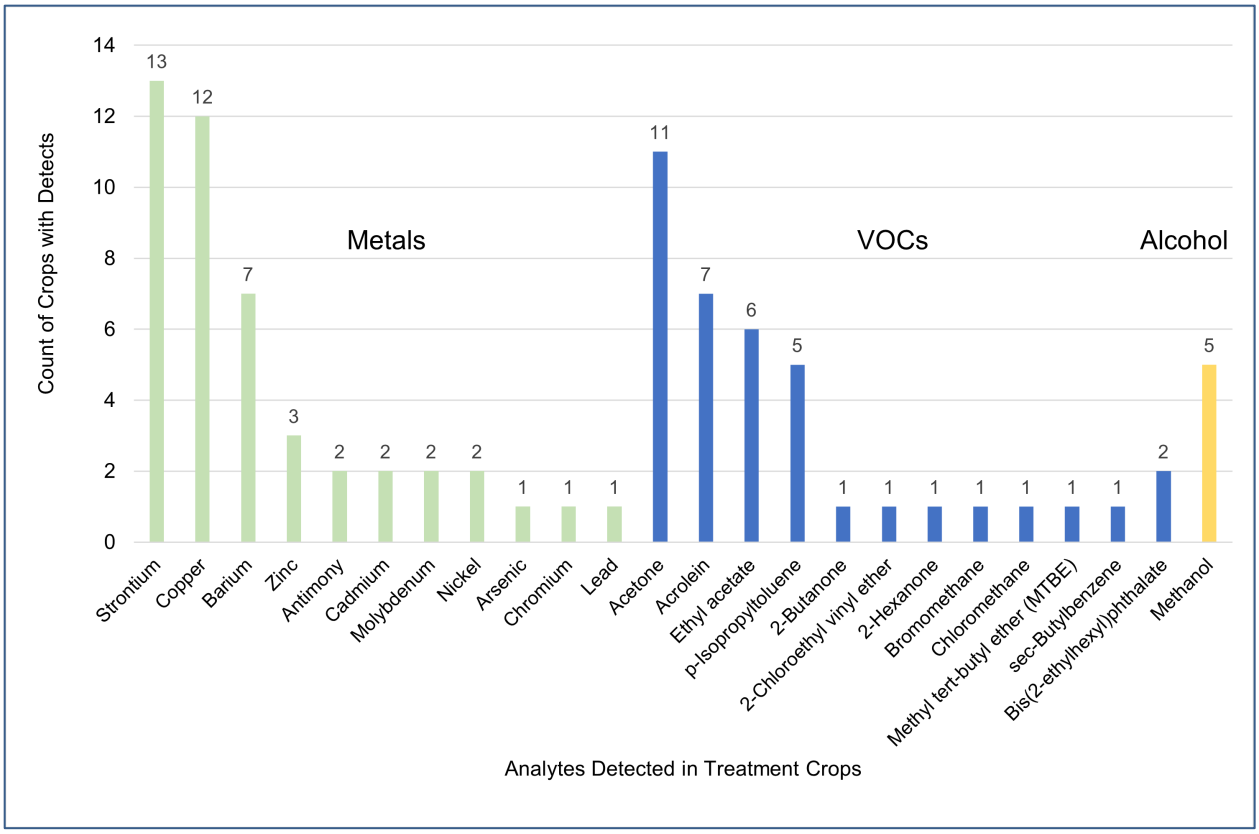

Figure 3. Target analytes detected in treatment crops

Table 1: Sampling and analysis dates

Crop Group[1]	Commodity	2017 Sampling Event	2018-2019 Sampling Event	Analysis Period for 2018-2019 Samples
Root and Tuber Vegetables	Carrot	Not applicable	11/1/18 to 12/7/19	11/1/18 to 12/7/19
Root and Tuber Vegetables	Potato	Not applicable	11/1/18 to 12/7/19	11/1/18 to 12/7/18
Bulb Vegetables	Garlic	7/18/17	7/12/18	1/3/19 to 3/10/19
Fruiting Vegetable	Tomato	9/6/17 to 9/7/17	8/9/18 to 8/13/18	10/24/18 to 12/01/18
Citrus Fruit	Lemon	3/30/2017	2/12/19 to 2/21/19	2/25/19 to 4/5/19
Citrus Fruit	Mandarin	3/30/2017	2/12/19 to 2/21/19	2/26/19 to 4/5/19
Citrus Fruit	Navel	3/30/17; 4/4/17	2/12/19 to 2/13/19	2/25/19 to 4/3/19
Citrus Fruit	Valencia	4/4/17	2/12/19	2/26/19 to 4/3/19
Pome Fruit	Apple	Not applicable	8/20/18	9/20/18 to 11/1/19
Stone Fruit	Cherry	Not applicable	5/1/19	5/13/19 to 6/1/19
Berry and Small Fruit	Grape	8/8/17 to 8/10/17	7/25/18 to 9/05/18	11/17/18 to 1/22/19
Tree Nuts	Almond	8/8/17 to 8/10/17	7/25/18 to 8/6/18	10/4/18 to 3/19/19
Tree Nuts	Pistachio	Not applicable	9/4/18 to 9/5/18	2/12/19 to 3/19/19

[1] Consistent with Code of Federal Regulations 40 CFR § 180.41 – Crop group tables. Part 180.41 provides crop tables for purposes of establishing tolerances and exemptions for pesticide chemical residues in food.

Table 2: Full list of organic compounds measured in crops and methods of analysis, as reported in certified laboratory reports

Analyte	Method
1-Methylnaphthalene	EPA 8270C SIM
1,1-Dichloroethane	EPA 8260B, EPA 8260C
1,1-Dichloroethene	EPA 8260B, EPA 8260C
1,1-Dichloropropene	EPA 8260B, EPA 8260C
1,1,1-Trichloroethane	EPA 8260B, EPA 8260C
1,1,1,2-Tetrachloroethane	EPA 8260B, EPA 8260C
1,1,2-Trichloroethane	EPA 8260B, EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 8260B, EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260B, EPA 8260C
1,2-Dibromoethane (EDB)	EPA 8260B, EPA 8260C
1,2-Dichloroethane	EPA 8260B, EPA 8260C
1,2-Dichloropropane	EPA 8260B, EPA 8260C
1,2,3-Trichlorobenzene	EPA 8260B, EPA 8260C
1,2,3-Trichloropropane	EPA 8260B, EPA 8260C
1,2,4-Trichlorobenzene	EPA 8260B, EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260B, EPA 8260C
1,3-Dichloropropane	EPA 8260B, EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260B, EPA 8260C
1,4-Dioxane	EPA 8270M, EPA 8270D
2-Butanone	EPA 8260B, EPA 8260C
2-Chloroethyl vinyl ether	EPA 8260B
2-Chlorotoluene	EPA 8260B, EPA 8260C
2-Hexanone	EPA 8260B, EPA 8260C
2-Methylnaphthalene	EPA 8270C SIM
2-Naphthylamine	EPA 8270C, EPA 8270D
2,2-Dichloropropane	EPA 8260B, EPA 8260C
4-Chlorotoluene	EPA 8260B, EPA 8260C
4-Methyl-2-pentanone	EPA 8260B, EPA 8260C

Analyte	Method
Acenaphthene	EPA 8270C SIM
Acenaphthylene	EPA 8270C SIM
Acetone	EPA 8260B, EPA 8260C
Acrolein	EPA 8260B, EPA 8260C
Acrylamide	EPA 8316M
Acrylonitrile	EPA 8260B, EPA 8260C
Anthracene	EPA 8270C SIM
Benzene	EPA 8260B, EPA 8260C
Benzo (a) anthracene	EPA 8270C SIM
Benzo (a) pyrene	EPA 8270C SIM
Benzo (b) fluoranthene	EPA 8270C SIM
Benzo (ghi) perylene	EPA 8270C SIM
Benzo (k) fluoranthene	EPA 8270C SIM
Bis(2-chloroethyl)ether	EPA 8270C, EPA 8270D
Bis(2-ethylhexyl)phthalate	EPA 8270C, EPA 8270D
Bromobenzene	EPA 8260B, EPA 8260C
Bromochloromethane	EPA 8260B, EPA 8260C
Bromodichloromethane	EPA 8260B, EPA 8260C
Bromoform	EPA 8260B, EPA 8260C
Bromomethane	EPA 8260B, EPA 8260C
Carbazole	EPA 8270C, EPA 8270D
Carbon tetrachloride	EPA 8260B, EPA 8260C
Chlorobenzene	EPA 8260B, EPA 8260C
Chloroethane	EPA 8260B, EPA 8260C
Chloroform	EPA 8260B, EPA 8260C
Chloromethane	EPA 8260B, EPA 8260C
Chrysene	EPA 8270C SIM
cis-1,2-Dichloroethene	EPA 8260B, EPA 8260C
cis-1,3-Dichloropropene	EPA 8260B, EPA 8260C

Analyte	Method
Dibenzo (a,h) anthracene	EPA 8270C SIM
Dibromochloromethane	EPA 8260B, EPA 8260C
Dibromomethane	EPA 8260B, EPA 8260C
Dichlorodifluoromethane (Freon 12)	EPA 8260B, EPA 8260C
Ethyl acetate	EPA 8260B
Ethylbenzene	EPA 8260B, EPA 8260C
Fluoranthene	EPA 8270C SIM
Fluorene	EPA 8270C SIM
Hexachlorobutadiene	EPA 8260B, EPA 8260C
Indeno (1,2,3-cd) pyrene	EPA 8270C SIM
Isopropyl alcohol	EPA 8015B
Isopropylbenzene	EPA 8260B, EPA 8260C
m-Dichlorobenzene	EPA 8260B, EPA 8260C
m,p-Xylene	EPA 8260B
Methanol	EPA 8015D, EPA 8015B
Methyl tert-butyl ether (MTBE)	EPA 8260B, EPA 8260C
Methylene chloride	EPA 8260B, EPA 8260C
n-Butylbenzene	EPA 8260B, EPA 8260C
n-Propylbenzene	EPA 8260B, EPA 8260C
Naphthalene	EPA 8270C SIM, EPA 8260B, EPA 8260C
o-Dichlorobenzene	EPA 8260B, EPA 8260C
o-Xylene	EPA 8260B, EPA 8260C
p-Dichlorobenzene	EPA 8260B, EPA 8260C
p-Isopropyltoluene	EPA 8260B, EPA 8260C
Phenanthrene	EPA 8270C SIM
Phenol	EPA 8270C, EPA 8270D
Pyrene	EPA 8270C SIM
Pyridine	EPA 8270C, EPA 8270D
sec-Butylbenzene	EPA 8260B, EPA 8260C

Analyte	Method
Styrene	EPA 8260B, EPA 8260C
tert-Butylbenzene	EPA 8260B, EPA 8260C
Tetrachloroethene	EPA 8260B, EPA 8260C
Toluene	EPA 8260B, EPA 8260C
trans-1,2-Dichloroethene	EPA 8260B, EPA 8260C
trans-1,3-Dichloropropene	EPA 8260B, EPA 8260C
Trichloroethene	EPA 8260B, EPA 8260C
Trichlorofluoromethane	EPA 8260B, EPA 8260C
Vinyl chloride	EPA 8260B, EPA 8260C

Table 3: Full list of metals measured in crops and methods of analysis, as reported in certified laboratory reports

Analyte	Method
Antimony, Total	EPA 6020B, EPA 6020, EPA 6020A
Arsenic, Total	EPA 6020B, EPA 6020, EPA 6020A
Barium, Total	EPA 6020B, EPA 6020, EPA 6020A
Beryllium, Total	EPA 6020B, EPA 6020, EPA 6020A
Cadmium, Total	EPA 6020B, EPA 6020, EPA 6020A
Chromium, Total	EPA 6020B, EPA 6020, EPA 6020A
Cobalt, Total	EPA 6020B, EPA 6020, EPA 6020A
Copper, Total	EPA 6020B, EPA 6020, EPA 6020A
Lead, Total	EPA 6020B, EPA 6020, EPA 6020A
Lithium, Total	EPA 6010B, EPA 6010C
Molybdenum, Total	EPA 6020B, EPA 6020, EPA 6020A
Nickel, Total	EPA 6020B, EPA 6020, EPA 6020A
Selenium, Total	EPA 6020B, EPA 6020, EPA 6020A
Silver, Total	EPA 6020B, EPA 6020, EPA 6020A
Strontium, Total	EPA 6020B, EPA 6020, EPA 6020A
Thallium, Total	EPA 6020B, EPA 6020, EPA 6020A
Vanadium, Total	EPA 6020B, EPA 6020, EPA 6020A
Zinc, Total	EPA 6020B, EPA 6020, EPA 6020A

Table 4: 89 Crop/Chemical datasets with at least one detect in a treatment group[1]

Type of Chemical	Target Analytes	Carrots (3/6)	Potato (3/3)	Garlic (6/5)	Tomato (5/6)	Lemon (9/10)	Mandarin (10/10)	Navel (13/15)	Valencia (3/3)	Apples (0/4)	Cherries (2/2)	Grapes (19/21)	Almonds (20/20)	Pistachios (20/21)	Total
Metal	Strontium	X	X	X	X	X	X	X	X	X	X	X	X	X	13
Metal	Copper	X	X	X	X	X	X	X		X	X	X	X	X	12
Metal	Barium	X		X			X	X	X				X	X	7
Metal	Zinc			X									X	X	3
Metal	Antimony			X									X		2
Metal	Cadmium	X									X				2
Metal	Molybdenum			X									X		2
Metal	Nickel												X	X	2
Metal	Arsenic	X													1
Metal	Chromium	X													1
Metal	Lead	X													1
Organic Compounds	Acetone	X	X	X	X	X	X	X	X			X	X	X	11
Organic Compounds	Acrolein	X	X	X	X					X		X	X		7
Organic Compounds	Ethyl acetate	X	X		X					X		X	X		6
Organic Compounds	p-Isopropyltoluene	X				X	X	X	X						5
Organic Compounds	Bis(2-ethylhexyl)phthalate											X		X	2
Organic Compounds	2-Butanone													X	1
Organic Compounds	2-Chloroethyl vinyl ether							X							1
Organic Compounds	2-Hexanone											X			1
Organic Compounds	Bromomethane										X				1
Organic Compounds	Chloromethane										X				1
Organic Compounds	Methyl tert-butyl ether (MTBE)													X	1
Organic Compounds	sec-Butylbenzene						X								1
Alcohols	Methanol				X		X	X	X				X		5
---	**Total**	11	5	8	6	4	7	7	5	4	5	7	11	9	89

[1] Values below crop names are (sample size of Control group / sample size of Treatment group) for the majority of chemicals measured in samples collected from the crop.

Table 5a: Summary statistics for almonds

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total number samples	Percent Frequency of Detects	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Arithmetic-Standard Deviation[5]	Coefficient of Variation (Standard deviation/mean)	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to Step 3?[6]
Alcohols (Method 8015D)	Methanol	Control	0 / 20	0%	44	800	NA	NA	NA	NA	NA	NA	NA	NA	No
Alcohols (Method 8015D)	Methanol	Treatment	1 / 20	5%	41	790	49.1	31.2	0.64	7.0	87.5	110	140	790	No
Metals (Method 6020B)	Antimony	Control	2 / 20	10%	0.20	0.20	0.25	0.15	0.59	0.03	0.20	0.20	0.20	0.73	No
Metals (Method 6020B)	Antimony	Treatment	7 / 20	35%	0.20	0.20	0.44	0.41	0.92	0.09	0.20	0.20	0.61	1.80	No
Metals (Method 6020B)	Barium	Control	17 / 20	85%	0.10	0.10	0.96	0.55	0.58	0.12	0.56	1.04	1.40	1.90	**Yes**
Metals (Method 6020B)	Barium	Treatment	20 / 20	100%	0.10	0.10	1.45	0.45	0.31	0.10	1.10	1.35	1.90	2.40	**Yes**
Metals (Method 6020B)	Copper	Control	20 / 20	100%	0.29	0.29	8.01	1.48	0.18	0.33	6.78	8.20	9.13	10.0	**Yes**
Metals (Method 6020B)	Copper	Treatment	20 / 20	100%	0.29	0.29	8.02	1.66	0.21	0.37	6.88	7.70	8.80	12.0	**Yes**
Metals (Method 6020B)	Molybdenum	Control	1 / 20	5%	0.30	0.30	0.31	0.04	0.14	0.01	0.30	0.30	0.30	0.50	No
Metals (Method 6020B)	Molybdenum	Treatment	1 / 20	5%	0.30	0.30	0.31	0.06	0.18	0.01	0.30	0.30	0.30	0.56	No
Metals (Method 6020B)	Nickel	Control	5 / 20	25%	0.45	0.45	0.63	0.32	0.50	0.07	0.45	0.45	0.61	1.30	No
Metals (Method 6020B)	Nickel	Treatment	1 / 20	5%	0.45	0.45	0.48	0.12	0.25	0.03	0.45	0.45	0.45	1.00	No
Metals (Method 6020B)	Strontium	Control	20 / 20	100%	0.25	0.25	6.62	2.34	0.35	0.52	3.88	6.90	8.55	9.60	**Yes**
Metals (Method 6020B)	Strontium	Treatment	20 / 20	100%	0.25	0.25	6.61	1.64	0.25	0.37	5.30	6.50	8.03	9.50	**Yes**
Metals (Method 6020B)	Zinc	Control	20 / 20	100%	2.3	2.3	22.2	6.35	0.29	1.42	17.5	21.5	25.3	36.0	**Yes**
Metals (Method 6020B)	Zinc	Treatment	20 / 20	100%	2.3	2.3	26.5	6.30	0.24	1.41	23.0	26.5	31.0	39.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	6 / 20	30%	0.074	0.62	0.41	0.41	0.99	0.09	0.52	0.60	0.62	1.50	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	7 / 20	35%	0.074	0.62	0.33	0.27	0.81	0.06	0.42	0.59	0.61	1.10	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein[7]	Control	2 / 20	10%	0.053	0.45	0.27	0.02	0.06	0.00	0.34	0.39	0.43	0.45	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein[8]	Treatment	6 / 20	30%	0.053	0.45	0.29	0.06	0.20	0.01	0.34	0.39	0.43	0.45	No
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl acetate	Control	9 / 10	90%	0	0.086	0.81	0.84	1.04	0.27	0.29	0.37	1.15	3.00	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl acetate	Treatment	7 / 10	70%	0	0.087	0.70	1.32	1.88	0.42	0.11	0.21	0.43	4.60	**Yes**

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] MDLs inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

[7] Maximum value is the MDL of non-detects. The maximum detect is 0.280 mg/kg.

[8] Maximum value is the MDL of non-detects. The maximum detect is 0.350 mg/kg.

Table 5b: Exploratory data analysis for almonds

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Metals (Method 6020B)	Barium	Control	17 / 20	85%	0	0	0.96	**1.52**	1.04	1.30	Normal	Normal	Not applicable	**Yes**
Metals (Method 6020B)	Barium	Treatment	20 / 20	100%	0	0	1.45	**1.52**	1.35	1.30	Normal	Normal	Not applicable	**Yes**
Metals (Method 6020B)	Copper	Control	20 / 20	100%	0	0	8.01	**1.00**	8.20	0.94	Normal	Normal	Not applicable	**Yes**
Metals (Method 6020B)	Copper	Treatment	20 / 20	100%	0	0	8.02	**1.00**	7.70	0.94	Normal	Normal	Not applicable	**Yes**
Metals (Method 6020B)	Strontium	Control	20 / 20	100%	0	0	6.62	1.00	6.90	**0.94**	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Metals (Method 6020B)	Strontium	Treatment	20 / 20	100%	0	0	6.61	1.00	6.50	**0.94**	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Zinc	Control	20 / 20	100%	0	0	22.2	**1.19**	21.5	1.23	Normal	Normal	Not applicable	**Yes**
Metals (Method 6020B)	Zinc	Treatment	20 / 20	100%	0	0	26.5	**1.19**	26.5	1.23	Normal	Normal	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260B)	Acetone	Control	6 / 20	30%	4	0	0.41	0.81	0.60	0.98	Nonparametric hypothesis test	Nonparametric hypothesis test	Nonparametric hypothesis test	**Yes**
Volatile Organic Compounds (Method 8260B)	Acetone	Treatment	7 / 20	35%	0	0	0.33	0.81	0.59	0.98	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260B)	Ethyl acetate	Control	9 / 10	90%	0	0	0.81	0.87	0.37	0.58	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Volatile Organic Compounds (Method 8260B)	Ethyl acetate	Treatment	7 / 10	70%	1	0	0.70	0.87	0.21	0.58	Nonparametric hypothesis test	Nonparametric hypothesis test	Nonparametric hypothesis test	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 5c: Statistical analysis for almonds

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Minimum Detection Limits of Non-detects[2] (mg/kg)	Maximum Detection Limits of Non-detects (mg/kg)	Hypothesis Test: Mean or Median (mg/kg)	Hypothesis Test: Ratio of Means or Medians[3]	Hypothesis Test: Difference in Means or Medians (mg/kg)	Test	Standard Deviation[4]	Ratio of Standard Deviations[5]	Form 1[6]: p-value	Form 1 Result	Form 2[7]: S[8] (mg/kg)	Form 2: Pooled Standard Deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[9]	Form 2: p-value	Form 2: Result	Statistically Significant?
Metals (Method 6020B)	Barium	Control	17 / 20	85%	0.1	0.1	1.04	1.30	0.31	Wilcoxon-Mann-Whitney Test[10]	0.55	1.2	0.01	Treatment group > Control group	0.43	0.50	20	0.68	Treatment Group > Control group	**Yes**
Metals (Method 6020B)	Barium	Treatment	20 / 20	100%	Not Applicable	Not Applicable	1.35	medians	medians	Wilcoxon-Mann-Whitney Test	0.45	1.2	0.01	Treatment group > Control group	0.31	0.50	38	0.87	Treatment Group > Control group	**Yes**
Metals (Method 6020B)	Copper	Control	20 / 20	100%	Not Applicable	Not Applicable	8.01	1.00	0.01	*t*-test[11]	1.48	1.1	0.49	Treatment group ≤ Control group	1.25	1.6	20	0.01	Treatment Group ≤ Control group	No[12]
Metals (Method 6020B)	Copper	Treatment	20 / 20	100%	Not Applicable	Not Applicable	8.02	means	means	*t*-test	1.66	1.1	0.49	Treatment group ≤ Control group	0.01	1.6	304,012	0.50	Treatment Group > Control group	No
Metals (Method 6020B)	Zinc	Control	20 / 20	100%	Not Applicable	Not Applicable	22.2	1.19	4.3	*t*-test	6.4	1.0	0.02	Treatment group > Control group	5.1	6.4	20	0.34	Treatment Group > Control group	**Yes**
Metals (Method 6020B)	Zinc	Treatment	20 / 20	100%	Not Applicable	Not Applicable	26.5	means	means	*t*-test	6.3	1.0	0.02	Treatment group > Control group	4.3	6.4	28	0.49	Treatment Group > Control group	**Yes**
Volatile Organic Compounds (Method 8260B)	Acetone	Control	6 / 20	30%	0.460	0.620	Not Applicable	Not Applicable	Not Applicable	Fisher's Exact [13]	Not Applicable	Not Applicable	1.0	Treatment group = Control group	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	No
Volatile Organic Compounds (Method 8260B)	Acetone	Treatment	7 / 20	35%	0.074	0.620	Not Applicable	Not Applicable	Not Applicable	Fisher's Exact	Not Applicable	Not Applicable	1.0	Treatment group = Control group	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Differences in Method Detection Limits for non-detects for pooled datasets (Control and Treatment combined) informs selection of hypothesis test.

[3] Hypothesis test is based on sample medians for barium because the Control dataset includes non-detects. For copper and zinc, datasets are normally distributed with frequency of detects equal to100%, so hypothesis test is based on sample means.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Ratio is the higher Standard Deviation divided by the lower Standard Deviation. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[6] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[7] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[8] S equals the difference in mean or median that is tested with Form 2

[9] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample Standard Deviation and error rates (α=0.05, β=0.20)

[10] Wilcoxon-Mann-Whitney test is used to evaluate differences in medians for censored data with consistent MDLs.

[11] Students' t-test is used to evaluate differences in arithmetic means for dataset that are approximately normally distributed with equal variance.

[12] A difference in means of 0.01 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (n=20) and the pooled Standard Deviations are sufficient to conclude the means are not different by more than 1.25 mg/kg. Sample sizes of at least 300,000 are needed to evaluate the observed difference of 0.01 mg/kg at specified error rates given the pooled sample Standard Deviation.

[13] Fisher's Exact test is used to evaluate differences in the proportion of detects. This test is applied if Frequency of Detection < 50% in either dataset.

Table 6: Summary statistics for apples

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to Step 3?[6]
Metals (Method 6020B)	Copper	Control	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No
Metals (Method 6020B)	Copper	Treatment	1 / 4	25%	0.29	0.29	0.43	0.27	0.64	0.14	0.29	0.29	0.43	0.83	No
Metals (Method 6020B)	Strontium	Control	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No
Metals (Method 6020B)	Strontium	Treatment	4 / 4	100%	Not Applicable	Not Applicable	0.60	0.06	0.10	0.03	0.59	0.62	0.63	0.63	No
Volatile Organic Compounds (Method 8260B)	Acrolein	Control	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No
Volatile Organic Compounds (Method 8260B)	Acrolein	Treatment	3 / 4	75%	0.035	0.035	0.11	0.05	0.47	0.03	0.09	0.12	0.14	0.15	No
Volatile Organic Compounds (Method 8260B)	Ethyl acetate	Control	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No
Volatile Organic Compounds (Method 8260B)	Ethyl acetate	Treatment	4 / 4	100%	0.25	0.25	2.04	1.29	0.63	0.64	1.37	2.30	2.98	3.20	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 7: Summary statistics for carrots

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Arsenic	Control	0 / 3	0%	0.07	0.20	0.16	0.08	0.48	0.04	0.14	0.20	0.20	0.20	No
Metals (Method 6020B)	Arsenic	Treatment	1 / 6	17%	0.20	0.20	0.18	0.04	0.24	0.02	0.20	0.20	0.20	0.20	No
Metals (Method 6020B)	Barium	Control	3 / 3	100%	0.07	0.10	1.93	0.25	0.13	0.15	1.80	1.90	2.05	2.20	No
Metals (Method 6020B)	Barium	Treatment	6 / 6	100%	0.07	0.10	2.46	1.33	0.54	0.54	1.83	2.20	3.03	4.60	No
Metals (Method 6020B)	Cadmium	Control	0 / 3	0%	0.03	0.06	0.05	0.02	0.35	0.01	0.05	0.06	0.06	0.06	No
Metals (Method 6020B)	Cadmium	Treatment	1 / 6	17%	0.06	0.06	0.06	0.00	0.08	0.00	0.06	0.06	0.06	0.06	No
Metals (Method 6020B)	Chromium	Control	1 / 3	33%	0.23	0.23	0.19	0.08	0.42	0.04	0.16	0.23	0.23	0.23	No
Metals (Method 6020B)	Chromium	Treatment	1 / 6	17%	0.23	0.23	0.23	0.00	0.02	0.00	0.23	0.23	0.23	0.23	No
Metals (Method 6020B)	Copper	Control	3 / 3	100%	0.04	0.29	0.79	0.03	0.04	0.02	0.77	0.78	0.80	0.82	No
Metals (Method 6020B)	Copper	Treatment	6 / 6	100%	0.04	0.29	1.01	0.25	0.24	0.10	0.90	1.05	1.18	1.30	No
Metals (Method 6020B)	Lead	Control	0 / 3	0%	0.02	0.21	0.15	0.00	0.00	0.00	0.12	0.21	0.21	0.21	No
Metals (Method 6020B)	Lead	Treatment	1 / 6	17%	0.21	0.21	0.18	0.00	0.00	0.00	0.21	0.21	0.21	0.21	No
Metals (Method 6020B)	Strontium	Control	3 / 3	100%	0.04	0.25	4.57	0.68	0.15	0.39	4.30	4.80	4.95	5.10	No
Metals (Method 6020B)	Strontium	Treatment	6 / 6	100%	0.04	0.25	4.67	0.52	0.11	0.21	4.63	4.75	5.03	5.10	No
Volatile Organic Compounds (Method 8260B and 8260C	Acetone	Control	2 / 3	67%	0.0025	0.048	1.81	2.94	1.62	1.70	0.12	0.23	2.72	5.20	No
Volatile Organic Compounds (Method 8260B and 8260C	Acetone	Treatment	5 / 6	83%	0.0025	0.049	0.39	0.20	0.52	0.08	0.38	0.43	0.50	0.57	No
Volatile Organic Compounds (Method 8260B and 8260C	Acrolein	Control	2 / 3	67%	0.050	0.055	0.27	0.19	0.70	0.11	0.17	0.27	0.36	0.45	No
Volatile Organic Compounds (Method 8260B and 8260C	Acrolein	Treatment	5 / 6	83%	0.052	0.057	0.50	0.43	0.87	0.18	0.25	0.42	0.53	1.30	No
Volatile Organic Compounds (Method 8260B and 8260C	Ethyl acetate	Control	2 / 2	100%	0.050	0.055	0.47	0.11	0.23	0.07	0.43	0.47	0.50	0.54	No
Volatile Organic Compounds (Method 8260B and 8260C	Ethyl acetate	Treatment	5 / 5	100%	0.052	0.057	0.64	0.28	0.45	0.13	0.50	0.56	0.67	1.10	No
Volatile Organic Compounds (Method 8260B and 8260C	p-Isopropyltoluene	Control	3 / 3	100%	0.001	0.010	0.31	0.23	0.75	0.14	0.18	0.22	0.40	0.58	No
Volatile Organic Compounds (Method 8260B and 8260C	p-Isopropyltoluene	Treatment	6 / 6	100%	0.001	0.011	0.46	0.52	1.15	0.21	0.20	0.26	0.42	1.50	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 8: Summary statistics for cherries

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Cadmium	Control	0 / 2	0%	0.06	0.06	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Metals (Method 6020B)	Cadmium	Treatment	1 / 2	50%	0.06	0.06	0.16	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.26	No
Metals (Method 6020B)	Copper	Control	2 / 2	100%	0.29	0.29	0.70	0.01	0.01	0.01	0.69	0.70	0.70	0.70	No
Metals (Method 6020B)	Copper	Treatment	2 / 2	100%	0.29	0.29	1.15	0.21	0.18	0.15	1.08	1.15	1.23	1.30	No
Metals (Method 6020B)	Strontium	Control	2 / 2	100%	0.25	0.25	0.67	0.00	0.00	0.00	0.67	0.67	0.67	0.67	No
Metals (Method 6020B)	Strontium	Treatment	2 / 2	100%	0.25	0.25	1.00	0.15	0.15	0.10	0.94	1.00	1.05	1.10	No
Volatile Organic Compounds (Method 8260B and 8260C)	Bromomethane	Control	0 / 2	0%	0.10	0.10	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	Bromomethane	Treatment	1 / 2	50%	0.10	0.10	0.10	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.10	No
Volatile Organic Compounds (Method 8260B and 8260C)	Chloromethane	Control	1 / 2	50%	0.050	0.050	0.084	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.084	No
Volatile Organic Compounds (Method 8260B and 8260C)	Chloromethane	Treatment	1 / 2	50%	0.050	0.050	0.080	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.080	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Inclusive of non-detects and detects, for metals is the range of method detection limits (MDLs), and for Volatile Organic Compounds is the range of method reporting limits (MRLs).

[3] Inclusive of non-detects and detects, for metals is the range of method detection limits (MDLs), and for Volatile Organic Compounds is the range of method reporting limits (MRLs).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 9a: Summary statistics for garlic

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Antimony	Control	2 / 6	33%	0.20	0.20	0.33	0.20	0.60	0.08	0.20	0.20	0.46	0.61	No
Metals (Method 6020B)	Antimony	Treatment	1 / 5	20%	0.20	0.20	0.28	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.61	No
Metals (Method 6020B)	Barium	Control	4 / 6	67%	0.10	0.10	0.68	0.62	0.91	0.25	0.21	0.55	0.97	1.70	No
Metals (Method 6020B)	Barium	Treatment	2 / 5	40%	0.10	0.10	0.76	0.91	1.20	0.41	0.10	0.10	1.60	1.90	No
Metals (Method 6020B)	Copper	Control	6 / 6	100%	0.29	0.29	2.32	0.29	0.13	0.12	2.10	2.45	2.50	2.60	**Yes**
Metals (Method 6020B)	Copper	Treatment	5 / 5	100%	0.29	0.29	2.38	0.11	0.05	0.05	2.40	2.40	2.40	2.50	**Yes**
Metals (Method 6020B)	Molybdenum	Control	0 / 6	0%	0.30	0.30	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Metals (Method 6020B)	Molybdenum	Treatment	1 / 5	20%	0.30	0.30	0.36	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.61	No
Metals (Method 6020B)	Strontium	Control	6 / 6	100%	0.25	0.25	1.57	0.583	0.37	0.238	1.23	1.65	1.70	2.50	**Yes**
Metals (Method 6020B)	Strontium	Treatment	5 / 5	100%	0.25	0.25	2.20	0.543	0.25	0.243	1.80	2.00	2.50	3.00	**Yes**
Metals (Method 6020B)	Zinc	Control	6 / 6	100%	2.30	2.30	11.5	1.22	0.11	0.50	10.5	12.0	12.0	13.0	**Yes**
Metals (Method 6020B)	Zinc	Treatment	5 / 5	100%	2.30	2.30	11.6	0.89	0.08	0.40	11.0	11.0	12.0	13.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	2 / 6	33%	0.12	1.00	0.74	0.73	0.99	0.30	0.16	0.60	0.98	2.00	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	4 / 5	80%	0.12	1.00	1.29	1.07	0.83	0.48	0.36	1.00	2.10	2.70	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein[7]	Control	6 / 6	100%	0.09	0.73	18.1	20.2	1.11	8.23	2.10	9.55	36.5	43.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein[8]	Treatment	5 / 5	100%	0.09	0.73	3.80	1.82	0.48	0.81	2.20	3.70	4.50	6.50	**Yes**

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

[7] Maximum values for both data groups are MDLs of non-detects. The maximum detect is 0.280 mg/kg.

[8] Maximum values for both data groups are MDLs of non-detects. The maximum detect is 0.350 mg/kg.

Table 9b: Exploratory data analysis for garlic

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Metals (Method 6020B)	Copper	Control	6 / 6	100%	0	0	2.32	No Data	2.45	No Data	Normal	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Copper	Treatment	5 / 5	100%	1	0	2.38	**1.03**	2.40	0.98	Nonparametric hypothesis test	Normal	Nonparametric hypothesis test	**Yes**
Metals (Method 6020B)	Strontium	Control	6 / 6	100%	0	0	1.57	No Data	1.65	No Data	Normal	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Strontium	Treatment	5 / 5	100%	0	0	2.20	**1.40**	2.00	1.21	Normal	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Zinc	Control	6 / 6	100%	0	0	11.50	No Data	12.00	No Data	Normal	Normal	Not Applicable	No
Metals (Method 6020B)	Zinc	Treatment	5 / 5	100%	0	0	11.60	1.01	11.00	0.92	Normal	Nonparametric hypothesis test	Not Applicable	No
Volatile Organic Compounds (Method 8260B)	Acrolein	Control	6 / 6	100%	0	0	18.10	No Data	9.55	No Data	Normal	Nonparametric hypothesis test	Not Applicable	No
Volatile Organic Compounds (Method 8260B)	Acrolein	Treatment	5 / 5	100%	0	0	3.80	0.21	3.70	0.39	Normal	Normal	Not Applicable	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 9c: Statistical analysis for garlic

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Mean (mg/kg)	Ratio of Means[2]	Difference in Means[3] (mg/kg)	Hypothesis Test[4]	Standard Deviation (mg/kg)	Ratio of Standard of Deviations[5]	Form 1[6]: p-value	Form 1: Result	Form 2[7]: S[8] (mg/kg)	Form 2: Pooled Standard Deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[9]	Form 2: p-value	Form 2: Result	Statistically Significant?
Metals (Method 6020B)	Copper	Control	6 / 6	100%	2.32	1.03	0.06	*t*-test	0.29	2.7	0.32	Treatment group ≤ Control group	0.40	0.23	5	0.02	Treatment group≤ Control group	No[10]
Metals (Method 6020B)	Copper	Treatment	5 / 5	100%	2.38	1.03	0.06	*t*-test	0.11	2.7	0.32	Treatment group ≤ ontrol group	0.06	0.23	182	0.40	Treatment group > Control group	No[11]
Metals (Method 6020B)	Strontium	Control	6 / 6	100%	1.57	1.40	0.63	*t*-test	0.58	1.1	0.05	Treatment group > ontrol group	1.00	0.57	5	0.23	Treatment group > Control group	Yes
Metals (Method 6020B)	Strontium	Treatment	5 / 5	100%	2.20	1.40	0.63	*t*-test	0.54	1.1	0.05	Treatment group > ontrol group	0.63	0.57	11	0.50	Treatment group > Control group	Yes

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[3] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[4] Students' t-test is used to evaluate differences in arithmetic means for dataset that are approximately normally distributed with equal variance.

[5] Ratio is the higher SD divided by the lower SD. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[6] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[7] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[8] S = difference in mean or median that is tested with Form 2. The choice of S is guided by the Power of the test to detect differences of at least S given the sample sizes and pooled sample SD.

[9] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample SD and error rates (α=0.05, β=0.20).

[10] A difference in means of 0.06 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (number=5 and 6) and the pooled standard deviations are sufficient to conclude the means are not different by more than 0.32 mg/kg. Sample sizes of at least 182 are needed to evaluate the observed difference of 0.06 mg/kg at specified error rates given the pooled sample standard deviation.

[11] A difference in means of 0.06 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (number=5 and 6) and the pooled standard deviations are sufficient to conclude the means are not different by more than 0.32 mg/kg. Sample sizes of at least 182 are needed to evaluate the observed difference of 0.06 mg/kg at specified error rates given the pooled sample standard deviation.

Table 10a: Summary statistics for grapes

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Copper	Control	19 / 20	95%	0.29	0.29	2.30	2.09	0.91	0.47	0.79	1.00	3.53	7.60	**Yes**
Metals (Method 6020B)	Copper	Treatment	18 / 21	86%	0.29	0.29	1.20	0.91	0.76	0.20	0.62	0.85	1.40	4.00	**Yes**
Metals (Method 6020B)	Strontium	Control	10 / 20	50%	0.25	0.25	0.48	0.25	0.53	0.06	0.25	0.39	0.66	0.95	**Yes**
Metals (Method 6020B)	Strontium	Treatment	18 / 21	86%	0.25	0.25	0.73	0.35	0.48	0.08	0.58	0.66	0.85	1.60	**Yes**
Semivolatile Organic Compounds (Method 8270C)	Bis(2-ethylhexyl) phthalate	Control	1 / 19	5%	0.08	0.50	0.37	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	2.00	No
Semivolatile Organic Compounds (Method 8270C)	Bis(2-ethylhexyl) phthalate	Treatment	1 / 21	5%	0.09	0.49	0.34	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	1.30	No
Volatile Organic Compounds (Method 8260B and 8260C)	2-Hexanone	Control	2 / 19	11%	0.01	0.06	0.08	0.15	1.82	0.03	0.02	0.06	0.06	0.64	No
Volatile Organic Compounds (Method 8260B and 8260C)	2-Hexanone	Treatment	4 / 21	19%	0.01	0.06	0.13	0.21	1.67	0.05	0.01	0.02	0.06	0.71	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	9 / 19	47%	0.12	0.12	0.32	0.47	1.50	0.11	0.12	0.12	0.22	2.00	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	9 / 21	43%	0.03	0.12	0.18	0.31	1.68	0.07	0.08	0.12	0.12	1.50	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Control	11 / 19	58%	0.09	0.09	0.14	0.10	0.68	0.02	0.09	0.09	0.14	0.40	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Treatment	10 / 21	48%	0.03	0.09	0.11	0.03	0.30	0.01	0.09	0.09	0.13	0.17	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Control	9 / 9	100%	0.029	0.045	0.58	0.67	1.16	0.22	0.27	0.31	0.4	2.3	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Treatment	11 / 11	100%	0.029	0.048	0.29	0.09	0.31	0.03	0.23	0.26	0.33	0.46	**Yes**

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 10b: Exploratory data analysis for grapes

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Metals (Method 6020B)	Copper	Control	19 / 20	95%	0	0	2.30	No Data	1.00	No Data	Normal	nonparametric hypothesis test	Not applicable	No
Metals (Method 6020B)	Copper	Treatment	18 / 21	86%	1	0	1.20	0.52	0.85	**0.85**	nonparametric hypothesis test	nonparametric hypothesis test	Normal	No
Metals (Method 6020B)	Strontium	Control	10 / 20	50%	0	0	0.48	No Data	0.39	No Data	nonparametric hypothesis test	nonparametric hypothesis test	Not applicable	**Yes**
Metals (Method 6020B)	Strontium	Treatment	18 / 21	86%	0	0	0.73	1.52	0.66	**1.71**	nonparametric hypothesis test	nonparametric hypothesis test	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260B)	Acetone	Control	9 / 19	47%	3	3	0.32	No Data	0.12	No Data	nonparametric hypothesis test	nonparametric hypothesis test	nonparametric hypothesis test	No
Volatile Organic Compounds (Method 8260B)	Acetone	Treatment	9 / 21	43%	3	1	0.18	**0.58**	0.12	1.00	nonparametric hypothesis test	nonparametric hypothesis test	nonparametric hypothesis test	No
Volatile Organic Compounds (Method 8260B)	Acrolein	Control	11 / 19	58%	2	0	0.14	No Data	0.09	No Data	nonparametric hypothesis test	nonparametric hypothesis test	nonparametric hypothesis test	No
Volatile Organic Compounds (Method 8260B)	Acrolein	Treatment	10 / 21	48%	0	0	0.11	**0.75**	0.09	1.00	nonparametric hypothesis test	nonparametric hypothesis test	Not applicable	No
Volatile Organic Compounds (Method 8260B)	Ethyl Acetate	Control	9 / 9	100%	1	1	0.58	No Data	0.31	No Data	nonparametric hypothesis test	nonparametric hypothesis test	nonparametric hypothesis test	No
Volatile Organic Compounds (Method 8260B)	Ethyl Acetate	Treatment	11 / 11	100%	0	0	0.29	0.50	0.26	**0.84**	Normal	Normal	Not applicable	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 10c: Statistical analysis for grapes

Type of Analytes and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Minimum Detection limits of non-detects[2] (mg/kg)	Maximum Detection limits of non-detects[3] (mg/kg)	Median (mg/kg)	Ratio of Medians[4]	Difference in Medians[5]	Hypothesis Test[6]	Standard Deviation (mg/kg)	Ratio of Standard Deviations[7]	Form 1[8]: p-value	Form 1: Results	Form 2[9]: S[10] (mg/kg)	Form 2: Pooled Standard deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[11]	Form 2: p-value	Form 2: Result	Statistically significant?
Metals (Method 6020B)	Strontium	Control	10 / 20	50%	0.25	0.25	0.39	1.7	0.28	Wilcoxon-Mann-Whitney Test	0.25	1.4	0.02	Treatment group > Control group	0.28	0.30	17	0.57	Treatment group > Control group	**Yes**[12]
Metals (Method 6020B)	Strontium	Treatment	18 / 21	86%	0.25	0.25	0.66	1.7	0.28	Wilcoxon-Mann-Whitney Test	0.35	1.4	0.02	Treatment group > Control group	0.28	0.30	17	0.57	Treatment group > Control group	**Yes**[13]

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[3] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[4] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[5] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[6] Wilcoxon-Mann-Whitney test is used to evaluate differences in medians for censored data with consistent Method detection limits.

[7] Ratio is the higher standard deviation divided by the lower standard deviation. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[8] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[9] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[10] S equals the difference in mean or median that is tested with Form 2. The choice of S is guided by the Power of the test to detect differences of at least S given the sample sizes and pooled sample standard deviation.

[11] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample SD and error rates (α=0.05, β=0.20).

[12] The difference in medians of 0.28 mg/kg is statistically significant using Form 1. With Form 2, the sample sizes provide sufficient power to evaluate the observed difference in medians at specified error rates.

[13] The difference in medians of 0.28 mg/kg is statistically significant using Form 1. With Form 2, the sample sizes provide sufficient power to evaluate the observed difference in medians at specified error rates.

Table 11a: Summary statistics for lemons

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Copper	Control	2 / 9	22%	0.29	0.29	0.44	0.37	0.84	0.12	0.29	0.29	0.29	1.40	No
Metals (Method 6020B)	Copper	Treatment	5 / 10	50%	0.29	0.29	0.49	0.23	0.46	0.07	0.29	0.43	0.61	0.85	No
Metals (Method 6020B)	Strontium	Control	9 / 9	100%	0.25	0.25	1.71	0.60	0.35	0.20	1.30	1.80	2.10	2.70	**Yes**
Metals (Method 6020B)	Strontium	Treatment	10 / 10	100%	0.25	0.25	2.31	0.51	0.22	0.16	2.05	2.35	2.75	2.90	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Limonene	Control	2 / 9	22%	0.02	0.10	0.11	0.15	1.28	0.05	0.02	0.02	0.10	0.42	No
Volatile Organic Compounds (Method 8260B and 8260C)	Limonene	Treatment	1 / 10	10%	0.02	0.11	0.08	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	0.38	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	2 / 9	22%	0.12	0.49	0.27	0.18	0.66	0.06	0.12	0.12	0.46	0.49	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	3 / 10	30%	0.12	0.57	0.37	0.21	0.59	0.07	0.12	0.49	0.53	0.59	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Control	3 / 9	33%	0.03	0.11	4.10	6.83	1.66	2.28	0.03	0.10	5.60	18.0	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Treatment	3 / 10	30%	0.03	0.13	5.19	9.90	1.91	3.13	0.03	0.11	5.58	30.0	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 11b: Exploratory data analysis for lemons

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Metals (Method 6020B)	Strontium	Control	9 / 9	100%	0	0	1.71	No Data	1.80	No Data	Normal	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Strontium	Treatment	10 / 10	100%	0	0	2.31	**1.4**	2.35	1.3	Normal	Normal	Not Applicable	**Yes**

[1] Subset of target analytes with at least four detects in both the Control and Treatment Datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 11c: Statistical analysis for lemons

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Mean (mg/kg)	Ratio of Means[2]	Difference in Means[3] (mg/kg)	Hypothesis Test[4]	Standard Deviation (mg/kg)	Ratio of Standard of Deviations[5]	Form 1[6]: p-value	Form 1: Result	Form 2[7]: S[8] (mg/kg)	Form 2: Pooled Standard Deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[9]	Form 2: p-value	Form 2: Result	Statistically Significant?
Metals (Method 6020B)	Strontium	Control	9 / 9	100%	1.71	1.3	0.60	*t*-test	0.60	1.2	0.02	Treatment group > Control group	0.60	0.55	11	0.55	Treatment group > Control group	**Yes[10]**
Metals (Method 6020B)	Strontium	Treatment	10 / 10	100%	2.31	1.3	0.60	*t*-test	0.51	1.2	0.02	Treatment group > Control group	0.60	0.55	11	0.55	Treatment group > Control group	**Yes[11]**

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[3] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[4] Students' t-test is used to evaluate differences in arithmetic means for dataset that are approximately normally distributed with equal variance.

[5] Ratio is the higher standard deviation divided by the lower standard deviation. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[6] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[7] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[8] S equals the difference in mean or median that is tested with Form 2. The choice of S is guided by the Power of the test to detect differences of at least S given the sample sizes and pooled sample SD.

[9] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample standard deviation and error rates (α=0.05, β=0.20).

[10] The difference in means of 0.60 mg/kg is statistically significant using Form 1. With Form 2, the sample sizes provide nearly the sufficient power to evaluate the observed difference in means at specified error rates.

[11] The difference in means of 0.60 mg/kg is statistically significant using Form 1. With Form 2, the sample sizes provide nearly the sufficient power to evaluate the observed difference in means at specified error rates.

Table 12a: Summary statistic for mandarins

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Alcohols (Method 8015D)	Methanol	Control	4 / 10	40%	45	240	176	96	0.55	30	74	220	240	280	**Yes**
Alcohols (Method 8015D)	Methanol	Treatment	4 / 10	40%	45	250	213	123	0.58	39	93	245	280	380	**Yes**
Metals (Method 6020B)	Barium	Control	3 / 10	30%	0.10	0.10	0.22	0.20	0.89	0.06	0.10	0.10	0.40	0.52	No
Metals (Method 6020B)	Barium	Treatment	3 / 10	30%	0.10	0.10	0.29	0.32	1.09	0.10	0.10	0.10	0.52	0.91	No
Metals (Method 6020B)	Copper	Control	6 / 10	60%	0.29	0.29	0.66	0.63	0.96	0.20	0.29	0.55	0.61	2.40	**Yes**
Metals (Method 6020B)	Copper	Treatment	9 / 10	90%	0.29	0.29	0.57	0.13	0.22	0.04	0.53	0.56	0.64	0.78	**Yes**
Metals (Method 6020B)	Strontium	Control	10 / 10	100%	0.25	0.25	1.93	0.84	0.43	0.27	1.23	1.65	2.65	3.30	**Yes**
Metals (Method 6020B)	Strontium	Treatment	10 / 10	100%	0.25	0.25	2.21	0.71	0.32	0.23	1.58	2.05	2.78	3.30	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	3 / 10	30%	0.12	0.53	0.29	0.17	0.58	0.05	0.12	0.29	0.45	0.53	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	3 / 10	30%	0.12	0.59	0.33	0.19	0.58	0.06	0.12	0.39	0.48	0.59	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Control	3 / 10	30%	0.03	0.12	1.80	3.05	1.69	0.97	0.03	0.11	3.11	8.8	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Treatment	7 / 10	70%	0.11	0.13	2.83	3.64	1.29	1.15	0.32	1.20	4.23	10.0	No
Volatile Organic Compounds (Method 8260B and 8260C)	sec-Butylbenzene	Control	0 / 10	0%	0.02	0.10	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	sec-Butylbenzene	Treatment	1 / 10	10%	0.02	0.11	0.07	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.25	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 12b: Exploratory analysis for mandarins

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Alcohols (Method 8015D)	Methanol	Control	4 / 10	40%	0	0	176	No Data	220	No Data	Nonparametric Hypothesis test	Nonparametric Hypothesis test	Not Applicable	**Yes**
Alcohols (Method 8015D)	Methanol	Treatment	4 / 10	40%	0	0	213	1.21	245	**1.11**	Nonparametric Hypothesis test	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Copper	Control	6 / 10	60%	1	1	0.66	No Data	0.55	No Data	Normal	Nonparametric Hypothesis test	Nonparametric Hypothesis test	**Yes**
Metals (Method 6020B)	Copper	Treatment	9 / 10	90%	0	0	0.57	0.86	0.56	**1.02**	Normal	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Strontium	Control	10 / 10	100%	0	0	1.93	No Data	1.65	No Data	Normal	Normal	Not Applicable	**Yes**
Metals (Method 6020B)	Strontium	Treatment	10 / 10	100%	0	0	2.21	**1.15**	2.05	1.24	Normal	Normal	Not Applicable	**Yes**

[1] Subset of target analytes with at least four detects in both the Control and Treatment Datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 12c: Statistical analysis for mandarins

Type of Analytes and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Minimum Detection limits of non-detects[2] (mg/kg)	Maximum Detection limits of non-detects[3] (mg/kg)	Median (mg/kg)	Ratio of Medians[4]	Difference in Medians[5]	Hypothesis Test[6]	Standard Deviation (mg/kg)	Ratio of Standard Deviations[7]	Form 1[8]: p-value	Form 1: Results	Form 2[9]: S[10] (mg/kg)	Form 2: Pooled Standard deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[11]	Form 2: p-value	Form 2: Result	Statistically significant?
Alcohols (Method 8015D)	Methanol	Control	4 / 10	40%	45	240	Not applicable	Not applicable	Not applicable	Fisher's Exact[12]	Not applicable	Not applicable	1.00	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Alcohols (Method 8015D)	Methanol	Treatment	4 / 10	40%	45	250	Not applicable	Not applicable	Not applicable	Fisher's Exact[13]	Not applicable	Not applicable	1.00	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Metals (Method 6020B)	Copper	Control	6 / 10	60%	0.29	0.29	0.55	1.0	0.01	Wilcoxon-Mann-Whitney Test	0.63	5.0	0.32	Treatment group ≤ Control group	0.47	0.38	10	< 0.01	Treatment group ≤ Control group	No[14]
Metals (Method 6020B)	Copper	Treatment	9 / 10	90%	0.29	0.29	0.56	medians	0.01	Wilcoxon-Mann-Whitney Test	0.13	5.0	0.32	Treatment group ≤ Control group	0.01	0.38	20,658	0.56	Treatment group > Control group	No[15]
Metals (Method 6020B)	Strontium	Control	10 / 10	100%	N/A	N/A	1.93	1.2	0.28	*t*-test	0.84	1.2	0.22	Treatment group ≤ Control group	0.90	0.78	10	0.046	Treatment group ≤ Control group	No[16]
Metals (Method 6020B)	Strontium	Treatment	10 / 10	100%	N/A	N/A	2.21	means	0.28	*t*-test	0.71	1.2	0.22	Treatment group ≤ Control group	0.28	0.78	96	0.50	Treatment group > Control group	No[17]

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[3] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[4] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[5] Hypothesis test is based on difference in sample means because both datasets are normal distributions with Frequency of Detection equal to 100%.

[6] Students' *t*-test is used to evaluate differences in arithmetic means for dataset that are approximately normally distributed.

[7] Ratio is the higher standard deviation divided by the lower standard deviation. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[8] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[9] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[10] S equals the difference in mean or median that is tested with Form 2. The choice of S is guided by the Power of the test to detect differences of at least S given the sample sizes and pooled sample standard deviation.

[11] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample standard deviation and error rates (α=0.05, β=0.20).

[12] Fisher's Exact test is used to evaluate differences in proportions of detects. This test is applied if frequency of detection < 50% in either dataset.

[13] Fisher's Exact test is used to evaluate differences in proportions of detects. This test is applied if frequency of detection < 50% in either dataset.

[14] A difference in medians of 0.01 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (n=10) and the pooled standard deviation are sufficient to conclude the medians are not different by more than 0.47 mg/kg. Sample sizes of at least 20,000 are needed to evaluate the observed difference of 0.01 mg/kg at specified error rates given the pooled sample standard deviation.

[15] A difference in medians of 0.01 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (n=10) and the pooled standard deviation are sufficient to conclude the medians are not different by more than 0.47 mg/kg. Sample sizes of at least 20,000 are needed to evaluate the observed difference of 0.01 mg/kg at specified error rates given the pooled sample standard deviation.

[16] A difference in means of 0.28 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (n=10) and the pooled standard deviation are sufficient to conclude the means are not different by more than 0.90 mg/kg. Sample sizes of at least 96 are needed to evaluate the observed difference of 0.28 mg/kg at specified error rates given the pooled sample standard deviation.

[17] A difference in means of 0.28 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (n=10) and the pooled standard deviation are sufficient to conclude the means are not different by more than 0.90 mg/kg. Sample sizes of at least 96 are needed to evaluate the observed difference of 0.28 mg/kg at specified error rates given the pooled sample standard deviation.

Table 13a: Summary statistics for navel oranges

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Alcohols (Method 8015D)	Methanol	Control	5 / 13	38%	45	240	146	83	0.57	23	52	140	230	240	**Yes**
Alcohols (Method 8015D)	Methanol	Treatment	7 / 15	47%	46	270	208	103	0.50	27	132	250	265	340	**Yes**
Metals (Method 6020B)	Barium	Control	9 / 13	69%	0.10	0.10	0.66	0.46	0.70	0.13	0.10	0.60	0.98	1.30	**Yes**
Metals (Method 6020B)	Barium	Treatment	8 / 15	53%	0.10	0.10	0.42	0.33	0.77	0.08	0.10	0.55	0.68	0.98	**Yes**
Metals (Method 6020B)	Copper	Control	6 / 13	46%	0.29	0.29	0.44	0.18	0.41	0.05	0.29	0.29	0.54	0.77	**Yes**
Metals (Method 6020B)	Copper	Treatment	4 / 15	27%	0.29	0.29	0.35	0.11	0.32	0.03	0.29	0.29	0.40	0.57	**Yes**
Metals (Method 6020B)	Strontium	Control	15 / 15	100%	0.25	0.25	2.37	0.57	0.24	0.15	2.10	2.20	2.40	3.90	**Yes**
Metals (Method 6020B)	Strontium	Treatment	13 / 13	100%	0.25	0.25	2.25	1.06	0.47	0.29	1.60	2.00	2.50	5.10	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Limonene	Control	2 / 13	15%	0.02	0.10	0.11	0.15	1.36	0.04	0.02	0.02	0.10	0.49	No
Volatile Organic Compounds (Method 8260B and 8260C)	Limonene	Treatment	2 / 15	13%	0.02	0.11	0.11	0.18	1.63	0.05	0.02	0.02	0.10	0.68	No
Volatile Organic Compounds (Method 8260B and 8260C)	2-Chloroethyl vinyl ether	Control	0 / 13	0%	0.05	0.20	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	2-Chloroethyl vinyl ether	Treatment	1 / 15	7%	0.05	0.22	0.15	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	1.10	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	4 / 13	31%	0.12	0.51	0.35	0.19	0.56	0.05	0.12	0.41	0.50	0.58	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	3 / 15	20%	0.12	0.55	0.32	0.23	0.72	0.06	0.12	0.12	0.53	0.75	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Control	4 / 13	31%	0.03	0.11	4.92	8.8	1.79	2.45	0.03	0.11	6.40	28.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Treatment	4 / 15	27%	0.03	0.12	5.84	11.2	1.92	2.89	0.03	0.11	5.56	35.0	**Yes**

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 13b: Exploratory data for navels oranges

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Alcohols (Method 8015D)	Methanol	Control	5 / 13	38%	0	see plot	146	No Data	140	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Alcohols (Method 8015D)	Methanol	Treatment	7 / 15	47%	0	see plot	208	1.42	250	**1.79**	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Metals (Method 6020B)	Barium	Control	9 / 13	69%	0	0	0.66	No Data	0.60	No Data	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Barium	Treatment	8 / 15	53%	0	0	0.42	0.64	0.55	**0.92**	Normal	Nonparametric hypothesis test	Not applicable	No
Metals (Method 6020B)	Copper	Control	6 / 13	46%	0	0	0.44	No Data	0.29	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Metals (Method 6020B)	Copper	Treatment	4 / 15	27%	0	0	0.35	**0.81**	0.29	1.00	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Metals (Method 6020B)	Strontium	Control	15 / 15	100%	2	1	2.37	No Data	2.20	No Data	Normal	Nonparametric hypothesis test	Normal	No
Metals (Method 6020B)	Strontium	Treatment	13 / 13	100%	0	0	2.25	**1.05**	2.00	1.10	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Volatile Organic Compounds (Method 8250B)	p-Isopropyl toluene	Control	4 / 13	31%	1	1	4.92	No Data	0.11	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Nonparametric hypothesis test	**Yes**
Volatile Organic Compounds (Method 8250B)	p-Isopropyl toluene	Treatment	4 / 15	27%	2	2	5.84	1.19	0.11	**1.00**	Nonparametric hypothesis test	Nonparametric hypothesis test	Nonparametric hypothesis test	**Yes**

[1] Subset of target analytes with at least four detects in both the Control and Treatment Datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 13c: Statistical analysis for navel oranges

Type of Analyte and Method	Target Analytes[1]	Group	Number of detects out of number of samples	Percent Frequency of Detection	Minimum Detection Limits of Non-detects[2]	Maximum Detection Limits of Non-detects[3]	Hypothesis Test[4]	p-value	Result	Statistically Significant?
Alcohols (Method 8015D)	Methanol	Control	5 / 13	38%	19	240	Fisher's Exact	0.72	Treatment group = Control group	No
Alcohols (Method 8015D)	Methanol	Treatment	7 / 15	47%	19	270	Fisher's Exact	0.72	Treatment group = Control group	No
Volatile Organic Compounds (Method 8260B)	p-Isopropyltoluene	Control	4 / 13	31%	0.028	0.11	Fisher's Exact	1.00	Treatment group = Control group	No
Volatile Organic Compounds (Method 8260B)	p-Isopropyltoluene	Treatment	4 / 15	27%	0.028	0.11	Fisher's Exact	1.00	Treatment group = Control group	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Multiple detection limits for methanol and p-isopropyltoluene is indicative of variability in dilutions applied to samples. However, the frequency of dilution factors across samples was approximately the same for control and treatment groups; therefore, dilution frequency is not a source of bias for these two-sample statistical tests.

[3] Multiple detection limits for methanol and p-isopropyltoluene is indicative of variability in dilutions applied to samples. However, the frequency of dilution factors across samples was approximately the same for control and treatment groups; therefore, dilution frequency is not a source of bias for these two-sample statistical tests.

[4] Fisher's Exact test is used to evaluate differences in the proportion of detects. This test is applied if FOD < 50% in either dataset.

Table 14: Summary statistics for Valencia oranges

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Alcohols (Method 8015D)	Methanol	Control	1 / 3	33%	46	240	175	N/A	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	240	No
Alcohols (Method 8015D)	Methanol	Treatment	1 / 3	33%	50	250	187	N/A	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	260	No
Metals (Method 6020B)	Barium	Control	2 / 3	67%	0.10	0.10	0.52	0.38	0.74	0.22	0.36	0.61	0.73	0.85	No
Metals (Method 6020B)	Barium	Treatment	3 / 3	100%	0.10	0.10	1.03	0.15	0.15	0.09	0.95	1.00	1.10	1.20	No
Metals (Method 6020B)	Strontium	Control	3 / 3	100%	0.25	0.25	2.97	1.05	0.35	0.61	2.45	3.00	3.50	4.00	No
Metals (Method 6020B)	Strontium	Treatment	3 / 3	100%	0.25	0.25	4.00	0.78	0.20	0.45	3.75	4.40	4.45	4.50	No
Volatile Organic Compounds (Method 8260B and 8260C)	Limonene	Control	1 / 3	33%	0.02	0.10	0.54	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	1.50	No
Volatile Organic Compounds (Method 8260B and 8260C)	Limonene	Treatment	1 / 3	33%	0.02	0.10	0.26	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.67	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	1 / 3	33%	0.12	0.50	0.45	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.73	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	1 / 3	33%	0.12	0.54	0.44	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	0.67	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Control	1 / 3	33%	0.03	0.11	12.4	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	37.0	No
Volatile Organic Compounds (Method 8260B and 8260C)	p-Isopropyltoluene	Treatment	1 / 3	33%	0.03	0.12	6.4	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	19.0	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 15a: Summary statistics for pistachios

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Barium	Control	7 / 20	35%	0.10	0.10	0.29	0.28	0.95	0.06	0.10	0.10	0.56	0.87	**Yes**
Metals (Method 6020B)	Barium	Treatment	10 / 21	48%	0.10	0.10	0.39	0.35	0.88	0.08	0.10	0.10	0.62	1.1	**Yes**
Metals (Method 6020B)	Copper	Control	20 / 20	100%	0.29	0.29	5.99	1.32	0.22	0.29	5.03	5.95	6.70	8.60	**Yes**
Metals (Method 6020B)	Copper	Treatment	21 / 21	100%	0.29	0.29	5.68	1.63	0.29	0.36	4.60	5.30	6.50	9.80	**Yes**
Metals (Method 6020B)	Nickel	Control	1 / 20	5%	0.45	0.45	0.48	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	1.00	No
Metals (Method 6020B)	Nickel	Treatment	1 / 21	5%	0.45	0.45	0.50	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	1.60	No
Metals (Method 6020B)	Strontium	Control	20 / 20	100%	0.25	0.25	1.87	0.54	0.29	0.12	1.50	1.80	2.10	3.30	**Yes**
Metals (Method 6020B)	Strontium	Treatment	21 / 21	100%	0.25	0.25	2.06	0.76	0.37	0.17	1.60	1.90	2.20	3.80	**Yes**
Metals (Method 6020B)	Zinc	Control	20 / 20	100%	2.3	2.3	11.90	2.28	0.19	0.51	10.75	11.5	13.3	16.0	**Yes**
Metals (Method 6020B)	Zinc	Treatment	21 / 21	100%	2.3	2.3	11.31	2.05	0.18	0.45	9.70	11.0	13.0	15.0	**Yes**
Semivolatile Organic Compounds (Method 8270C)	Bis(2-ethylhexyl) phthalate	Control	0 / 20	0%	0.10	9.6	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Semivolatile Organic Compounds (Method 8270C)	Bis(2-ethylhexyl) phthalate	Treatment	1 / 21	5%	0.10	9.6	4.51	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	9.6	No
Volatile Organic Compounds (Method 8260B and 8260C)	2-Butanone	Control	10 / 21	48%	0.13	0.37	4.61	4.87	1.06	1.06	0.36	0.37	8.1	13.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	2-Butanone	Treatment	10 / 21	48%	0.14	0.37	4.25	4.25	1.00	0.93	0.36	0.37	8.1	11.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	10 / 21	48%	0.22	0.62	2.18	1.93	0.88	0.42	0.60	0.62	3.1	6.0	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	12 / 21	57%	0.23	0.62	2.03	1.35	0.67	0.29	0.61	2.1	3.1	4.4	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Methyl tert-butyl ether (MTBE)	Control	10 / 21	48%	0.09	0.25	0.46	0.24	0.52	0.05	0.24	0.25	0.68	0.78	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Methyl tert-butyl ether (MTBE)	Treatment	10 / 21	48%	0.09	0.25	0.50	0.28	0.56	0.06	0.24	0.25	0.77	0.89	**Yes**

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 15b: Exploratory data analysis for pistachios

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Metals (Method 6020B)	Barium	Control	7 / 20	35%	0	0	0.29	No Data	0.10	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Metals (Method 6020B)	Barium	Treatment	10 / 21	48%	0	0	0.39	**1.36**	0.10	1.00	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Metals (Method 6020B)	Copper	Control	20 / 20	100%	0	0	5.99	No Data	5.95	No Data	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Copper	Treatment	21 / 21	100%	0	0	5.68	**0.95**	5.30	0.89	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Strontium	Control	20 / 20	100%	0	0	1.87	No Data	1.80	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Metals (Method 6020B)	Strontium	Treatment	21 / 21	100%	0	0	2.06	1.10	1.90	**1.06**	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Metals (Method 6020B)	Zinc	Control	20 / 20	100%	0	0	11.90	No Data	11.50	No Data	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Zinc	Treatment	21 / 21	100%	0	0	11.31	**0.95**	11.00	0.96	Normal	Normal	Not applicable	No
Volatile Organic Compounds (Method 8260)	2-Butanone	Control	10 / 21	48%	0	0	4.61	No Data	0.37	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Volatile Organic Compounds (Method 8260)	2-Butanone	Treatment	10 / 21	48%	0	0	4.25	**0.92**	0.37	1.00	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	No
Volatile Organic Compounds (Method 8260)	Acetone	Control	10 / 21	48%	0	0	2.18	No Data	0.62	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260)	Acetone	Treatment	12 / 21	57%	0	0	2.03	0.93	2.10	**3.39**	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260)	Methyl tert-butyl ether (MTBE)	Control	10 / 21	48%	0	0	0.46	No Data	0.25	No Data	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260)	Methyl tert-butyl ether (MTBE)	Treatment	10 / 21	48%	0	0	0.50	**1.10**	0.25	1.00	Nonparametric hypothesis test	Nonparametric hypothesis test	Not applicable	**Yes**

[1] Subset of target analytes with at least four detects in both the Control and Treatment Datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 15c: Statistical analysis for pistachios

Type of Analytes and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Minimum Detection Limits of non-detects[2] (mg/kg)	Maximum Detection Limits of non-detects (mg/kg)	Median (mg/kg)	Ratio of Medians	Difference in Medians	Hypothesis Test[3]	Standard Deviation (mg/kg)	Ratio of Standard Deviations[4]	Form 1[5]: p-value	Form 1: Results	Form 2[6]: S[7] (mg/kg)	Form 2: Pooled Standard deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[8]	Form 2: p-value	Form 2: Result	Statistically significant?
Metals (Method 6020B)	Barium	Control	7 / 20	35%	0.10	0.10	Not applicable	Not applicable	Not applicable	Fisher's Exact	Not applicable	Not applicable	1.0	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Metals (Method 6020B)	Barium	Treatment	10 / 21	48%	0.10	0.10	Not applicable	Not applicable	Not applicable	Fisher's Exact	Not applicable	Not applicable	1.0	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Metals (Method 6020B)	Strontium	Control	20 / 20	100%	N/A	N/A	1.80	1.1	0.10	Wilcoxon-Mann-Whitney Test[9]	0.54	1.4	0.24	Treatment group ≤ Control group	0.57	0.66	20	< 0.1	Treatment group≤ Control group	No[10]
Metals (Method 6020B)	Strontium	Treatment	21 / 21	100%	N/A	N/A	1.90	1.1	0.10	Wilcoxon-Mann-Whitney Test	0.76	1.4	0.24	Treatment group ≤ Control group	0.10	0.66	627	0.54	Treatment group > Control group	No
Volatile Organic Compounds (Method 8260B)	Acetone	Control	10 / 21	48%	0.220	0.620	Not applicable	Not applicable	Not applicable	Fisher's Exact	Not applicable	Not applicable	0.76	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Volatile Organic Compounds (Method 8260B)	Acetone	Treatment	12 / 21	57%	0.230	0.620	Not applicable	Not applicable	Not applicable	Fisher's Exact	Not applicable	Not applicable	0.76	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Volatile Organic Compounds (Method 8260B)	Methyl tert-butyl ether (MTBE)	Control	10 / 21	48%	0.086	0.250	Not applicable	Not applicable	Not applicable	Fisher's Exact	Not applicable	Not applicable	1.0	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Volatile Organic Compounds (Method 8260B)	Methyl tert-butyl ether (MTBE)	Treatment	10 / 21	48%	0.091	0.250	Not applicable	Not applicable	Not applicable	Fisher's Exact	Not applicable	Not applicable	1.0	Treatment group = Control group	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Multiple detection limits for Volatile Organic Compounds is indicative of variability in dilutions applied to samples. However, the frequency of dilution factors across samples was approximately the same for control and treatment groups; therefore, dilution frequency is not a source of bias for these two-sample statistical tests.

[3] Fisher's Exact test is used to evaluate differences in the proportion of detects. This test is applied if frequency of detection < 50% in either dataset

[4] Ratio is the higher standard deviation divided by the lower standard deviation. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[5] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[6] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[7] S equals the difference in mean or median that is tested with Form 2. The choice of S is guided by the Power of the test to detect differences of at least S given the sample sizes and pooled sample standard deviation.

[8] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample SD and error rates (α=0.05, β=0.20).

[9] Wilcoxon-Mann-Whitney test is used to evaluate differences in medians for censored data with consistent MDLs.

[10] A difference in medians of 0.10 mg/kg is not statistically significant using Form 1. Likewise, with Form 2, current sample sizes (n=20 and 21) and the pooled standard deviation are sufficient to conclude the medians are not different by more than 0.57 mg/kg. Sample sizes of at least 627 are needed to evaluate the observed difference of 0.10 mg/kg at specified error rates given the pooled sample standard deviation.

Table 16: Summary statistics for potatoes

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Metals (Method 6020B)	Copper	Control	3 / 3	100%	0.29	0.29	0.88	0.02	0.02	0.01	0.87	0.87	0.89	0.90	No
Metals (Method 6020B)	Copper	Treatment	3 / 3	100%	0.29	0.29	1.02	0.07	0.07	0.04	0.99	1.00	1.05	1.10	No
Metals (Method 6020B)	Strontium	Control	0 / 3	0%	0.25	0.25	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	No
Metals (Method 6020B)	Strontium	Treatment	3 / 3	100%	0.25	0.25	0.70	0.05	0.07	0.03	0.67	0.69	0.72	0.75	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	3 / 3	100%	0.047	0.048	2.44	3.69	1.51	2.13	0.32	0.33	3.52	6.70	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	2 / 3	67%	0.044	0.051	0.36	0.40	1.11	0.23	0.14	0.22	0.52	0.82	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Control	2 / 3	67%	0.034	0.035	0.10	0.06	0.57	0.03	0.08	0.12	0.13	0.14	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Treatment	3 / 3	100%	0.032	0.036	0.18	0.02	0.12	0.01	0.17	0.17	0.19	0.20	No
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Control	3 / 3	100%	0.055	0.056	6.87	0.49	0.07	0.28	6.70	7.10	7.15	7.20	No
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Treatment	3 / 3	100%	0.052	0.059	2.72	2.32	0.85	1.34	1.53	2.60	3.85	5.10	No

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 17a: Summary statistics for tomatoes

Type of Analyte and Method	Target Analytes[1]	Group	Number Detects out of Number of Samples	Percent Frequency of Detection	Minimum Detection Limits[2]	Maximum Detection Limits[3]	Mean[4]	Standard Deviation[5]	Coefficient of Variation	Standard Error for the Mean	25th Percentile	50th Percentile	75th Percentile	Maximum	Continue to step 3?[6]
Alcohols (Method 8015D)	Methanol	Control	5 / 5	100%	20	24	396	21	0.05	9.3	380	390	400	430	**Yes**
Alcohols (Method 8015D)	Methanol	Treatment	6 / 6	100%	24	26	385	21	0.05	8.5	373	385	390	420	**Yes**
Metals (Method 6020B)	Copper	Control	5 / 5	100%	0.29	0.29	0.64	0.05	0.08	0.02	0.59	0.67	0.68	0.68	**Yes**
Alcohols (Method 8015D)	Copper	Treatment	6 / 6	100%	0.29	0.29	0.63	0.07	0.11	0.03	0.61	0.63	0.66	0.74	**Yes**
Alcohols (Method 8015D)	Strontium	Control	2 / 5	40%	0.25	0.25	0.36	0.15	0.42	0.07	0.25	0.25	0.50	0.55	No
Alcohols (Method 8015D)	Strontium	Treatment	2 / 6	33%	0.25	0.25	0.35	0.15	0.43	0.06	0.25	0.25	0.45	0.56	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	5 / 5	100%	0.049	0.050	0.46	0.06	0.13	0.03	0.43	0.45	0.46	0.57	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	6 / 6	100%	0.024	0.050	0.63	0.29	0.47	0.12	0.51	0.67	0.68	1.10	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Control	5 / 5	100%	0.035	0.036	0.21	0.06	0.29	0.03	0.17	0.21	0.24	0.30	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Treatment	6 / 6	100%	0.017	0.036	0.15	0.06	0.41	0.03	0.13	0.17	0.19	0.23	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Control	5 / 5	100%	0.057	0.058	0.80	0.26	0.33	0.12	0.59	0.81	0.87	1.20	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Treatment	6 / 6	100%	0.027	0.058	0.25	0.09	0.34	0.03	0.18	0.26	0.32	0.34	**Yes**

[1] Subset of target analytes with at least one detect in the Treatment dataset. All units are mg/kg.

[2] Method detection limits (MDLs) inclusive of non-detects and detects.

[3] Method detection limits (MDLs) inclusive of non-detects and detects.

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[6] Analyte is included in Step 3 (Exploratory Data Analysis) if there are at least 4 detects in both Control and Treatment datasets.

Table 17b: Exploratory data analysis for tomatoes

Type of Analyte and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Potential Outliers: Count based on interquartile range[2]	Potential Outliers: Count based on Q-Q Plot[3]	Mean Ratio of Treatment or Control[4]	Ratio of Means of Treatment and Control	Median Ratio of Treatment or Control	Ratio of Medians of Treatment and Control	Goodness of fit evaluation for Normality: Q-Q Plot[5]	Goodness of fit evaluation for Normality: Test[6]	Goodness of fit evaluation for Normality: Sensitive to Outliers?[7]	Continue to Step 5?[8]
Alcohols (Method 8015D)	Methanol	Control	5 / 5	100%	0	0	396	No Data	390	No Data	Nonparametric hypothesis test	Normal	Not applicable	No
Alcohols (Method 8015D)	Methanol	Treatment	6 / 6	100%	0	0	385	**0.97**	385	0.99	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Copper	Control	5 / 5	100%	0	0	0.64	No Data	0.67	No Data	Normal	Normal	Not applicable	No
Metals (Method 6020B)	Copper	Treatment	6 / 6	100%	0	0	0.63	**0.99**	0.63	0.94	Nonparametric hypothesis test	Normal	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	5 / 5	100%	1	0	0.46	No Data	0.45	No Data	Nonparametric hypothesis test	Normal	Normal	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	6 / 6	100%	0	0	0.63	**1.36**	0.67	1.48	Nonparametric hypothesis test	Normal	Not applicable	**Yes**
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Control	5 / 5	100%	0	0	0.21	No Data	0.21	No Data	Normal	Normal	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	Acrolein	Treatment	6 / 6	100%	0	0	0.15	**0.72**	0.17	0.79	Normal	Normal	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Control	5 / 5	100%	0	0	0.80	No Data	0.81	No Data	Normal	Normal	Not applicable	No
Volatile Organic Compounds (Method 8260B and 8260C)	Ethyl Acetate	Treatment	6 / 6	100%	0	0	0.25	**0.31**	0.26	0.32	Normal	Normal	Not applicable	No

[1] Subset of target analytes with at least four detects in both the Control and Treatment Datasets. All units are mg/kg.

[2] Count of potential outliers screened using X > 75th percentile + 3.0 x Interquartile Range. Non-detects are excluded from the count of outliers.

[3] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[4] Parameter estimated with Kaplan-Meier methods if dataset includes one or more non-detects.

[5] Refer to Appendix B for one-page data summaries by Crops/Analyte groups (Control and Treatment).

[6] Refer to Appendix B for one-page summary of Goodness-of-Fit test for Normality by Crops/Analyte groups (Control and Treatment).

[7] Goodness-of-fit evaluation was repeated excluding potential outlier(s). Result are “Not applicable” if dataset has 0 potential outliers.

[8] Analyte is included in Step 5 (Statistical Analysis) if 1) both Control and Treatment have frequency of detects < 50%; or 2) both Control and Treatment have frequency of detects ≥ 50% and Ratio ≥ 1. Ratio is based on medians if either dataset exhibits non-normal distribution frequency of detects<100%.

Table 17c: Statistical analysis for tomatoes

Type of Analytes and Method	Target Analytes[1]	Group	Number of Detects out of Total Samples	Percent Frequency of Detects	Median (mg/kg)	Ratio of Medians[2]	Difference in Medians	Hypothesis Test[3]	Standard Deviation (mg/kg)	Ratio of Standard Deviations[4]	Form 1[5]: p-value	Form 1: Results	Form 2[6]: S[7] (mg/kg)	Form 2: Pooled Standard deviation (mg/kg)	Form 2: Number for α=0.05, β=0.20[8]	Form 2: p-value	Form 2: Result	Statistically Significant?
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Control	5 / 5	100%	0.46	1.4	0.17	*t*-test	0.06	4.7	0.12	Treatment group ≤ Control group	0.34	0.22	6	0.11	Treatment group > Control group	**Mixed[9]**
Volatile Organic Compounds (Method 8260B and 8260C)	Acetone	Treatment	6 / 6	100%	0.63	1.4	0.17	*t*-test	0.29	4.7	0.12	Treatment group ≤ Control group	0.17	0.22	22	0.49	Treatment group > Control group	**Mixed[10]**

[1] Subset of target analytes with at least four detects in both the Control and Treatment datasets and hypothesis testing is warranted. All units are mg/kg.

[2] Test is based on difference in sample means because both datasets are normal distributions with frequency of detection equal to 100%.

[3] Welch-Satterthwaite test is used (in lieu of Student's *t*-test) to evaluate differences in arithmetic means for dataset that are approximately normally distributed with unequal variances.

[4] Ratio is the higher standard deviation divided by the lower standard deviation. Ratio > 3.0 indicates that the assumption of equal variance that underlies parametric and nonparametric hypothesis tests is violated, which can introduce uncertainty in the findings.

[5] Form 1 denotes a null hypothesis of Treatment ≤ Control.

[6] Form 2 denotes a null hypothesis of Treatment > Control + S, where S is the statistically significant difference in means or medians.

[7] S equals the difference in mean or median that is tested with Form 2. The choice of S is guided by the Power of the test to detect differences of at least S given the sample sizes and pooled sample standard deviation.

[8] Check on the minimum sample size in each group (Control and Treatment) needed to detect a difference (S) in mean or medians, given pooled sample standard deviation and error rates (α=0.05, β=0.20).

[9] Difference in means of 0.17 mg/kg is not statistically significant using Form 1, however the t-test lacks sufficient power to detect a difference; at least n=22 in both Control and Treatment groups is needed to evaluate the observed difference in means given the pooled SD of 0.22 mg/kg and specified error rates. With Form 2, the sample sizes are sufficient to evaluate a difference in means of 0.34 mg/kg, and to conclude that the Treatment mean may be elevated (p=0.11).

[10] Difference in means of 0.17 mg/kg is not statistically significant using Form 1, however the t-test lacks sufficient power to detect a difference; at least n=22 in both Control and Treatment groups is needed to evaluate the observed difference in means given the pooled SD of 0.22 mg/kg and specified error rates. With Form 2, the sample sizes are sufficient to evaluate a difference in means of 0.34 mg/kg, and to conclude that the Treatment mean may be elevated (p=0.11).

Table 18: Summary of crops with similar chemical profiles for metals and organics

Commodity	Profiles for Metals	Notes	Profiles for Organics	Notes
Carrot	C	same profile as citrus, with 1.5x -2x higher Sr, Cu, Ba		
Potato	B	80% of cherries	D	matches tomatoes; 2x higher ethyl acetate Treatment and 6x higher ethyl acetate Control
Garlic	A	matches almonds with rank: Zn > Cu > Sr > Ba = Ni > Sb = Mo; 50% of almonds for Zn, Cu, Sr		
Tomato	B	50% of cherries	D	rank: ethyl acetate > acetone > acrolein
Lemon	C	citrus profile + carrots, rank order: Sr > Cu (no Ba)	E	citrus profile, rank: p-isopropyltoluene > acetone > limonene
Mandarin	C	citrus profile + carrots, rank order: Sr > Cu > Ba	E	50% of lemon for p-isopropyltoluene and acetone; no limonene
Navel orange	C	citrus profile + carrots, rank order: Sr > Ba > Cu	E	citrus profile, matches lemon
Valencia orange	C	citrus profile + carrots, rank order: Sr > Ba = Cu (no Cu in Treatment)	E	citrus profile, matches lemon
Apple				
Cherry	B	rank: Sr = Cu		
Grape	B	matches cherries for Control and 2x higher for Cu Treatment	D	matches tomatoes
Almond	A	rank: Zn > Cu > Sr > Ba > Sb = Mo = Ni	D	matches tomatoes
Pistachio	A	matches garlic profile closely with rank: Zn > Cu > Sr > Ba = Ni; 2x garlic for Cu; no Sb or Mo		

Table 19: Chemicals detected in crops irrigated with blended produced water with respective MCLs for Drinking Water

Analyte	Analyte Type	MCLs for Drinking Water [mg/L]
Methanol	Alcohol	NA
Antimony	Metal	0.006
Arsenic	Metal	0.01
Barium	Metal	1
Cadmium	Metal	0.005
Chromium	Metal	0.05
Copper	Metal	1.3
Lead	Metal	0.015
Molybdenum	Metal	NA
Nickel	Metal	0.1
Selenium	Metal	0.05
Strontium	Metal	NA
Zinc	Metal	NA
2-butanone	Volatile Organic Compound	NA
2-chloroethyl vinyl ether	Volatile Organic Compound	NA
2-hexanone	Volatile Organic Compound	NA
Acetone	Volatile Organic Compound	NA
Acrolein	Volatile Organic Compound	NA
Bromomethane	Volatile Organic Compound	NA
Chloromethane	Volatile Organic Compound	NA
Ethyl acetate	Volatile Organic Compound	NA
MTBE	Volatile Organic Compound	0.013
p-isopropyltoluene	Volatile Organic Compound	NA
sec-butylbenzene	Volatile Organic Compound	NA

Table 20: Summary of analytic results for treated produced water samples (effluent water)

Analyte	Minimum of Detected [mg/L]	Mean of Detected [mg/L]	Maximum of Detected [mg/L]	Total Number	Number Detected	Number Exceeding Standard	% Detected	% Exceeding Standard
2-butanone	0.0027	0.0071	0.0180	32	7	NA	21.9	NA
2-chloroethyl vinyl ether	ND	ND	ND	11	0	NA	0	NA
2-hexanone	0.0012	0.0037	0.0012	33	1	NA	3	NA
Acetone	0.0110	0.0236	0.1000	37	17	NA	45.9	NA
Acrolein	ND	ND	ND	18	0	NA	0	NA
Antimony	0.0001	0.8148	0.0060	63	27	0	42.9	0
Arsenic	0.0001	0.0331	0.0910	160	143	120	89.4	75
Barium	0.0000	0.6568	0.1200	82	54	0	65.9	0
Bromomethane	0.00046	0.00046	0.00046	92	1	NA	1.1	NA
Cadmium	ND	ND	ND	74	0	0	0	0
Chloromethane	ND	ND	ND	97	0	NA	0	NA
Chromium	0.0006	0.3477	0.0040	58	9	0	15.5	0
Copper	0.0001	0.0047	0.0045	65	25	NA	38.5	NA
Ethyl acetate	ND	ND	ND	11	0	NA	0	NA
Lead	0.0001	0.0041	0.0005	76	4	0	5.3	0
Methanol	ND	ND	ND	13	0	NA	0	NA
Molybdenum	0.0003	0.0076	0.0150	65	41	NA	63.1	NA
MTBE	ND	ND	ND	71	0	0	0	0
Nickel	0.0003	0.0086	0.0026	76	34	0	44.7	0
p-isopropyltoluene	0.0003	0.0001	0.0003	55	1	NA	1.8	NA
sec-butylbenzene	0.0004	0.0009	0.0027	83	33	NA	39.8	NA
Selenium	0.0003	0.0048	0.0028	69	21	0	30.4	0
Strontium	0.0790	0.1798	0.9100	66	56	NA	84.8	NA
Zinc	0.0018	1.1145	0.0360	69	32	NA	46.4	NA

Table 21: Summary of analytic results for blended produced water samples

Analyte	Minimum of Detected [mg/L]	Mean of Detected [mg/L]	Maximum of Detected [mg/L]	Total Number	Number Detected	Number Exceeding Standard	% Detected	% Exceeding Standard
2-butanone	NA	NA	NA	19	0	NA	0	NA
2-chloroethyl vinyl ether	NA	NA	NA	2	0	NA	0	NA
2-hexanone	NA	NA	NA	20	0	NA	0	NA
Acetone	0.0050	0.0095	0.0500	23	7	NA	30.4	NA
Acrolein	NA	NA	NA	10	0	NA	0	NA
Antimony	0.0001	0.0027	0.0110	54	24	1	44.4	1.9
Arsenic	0.0002	0.0119	0.0650	132	113	70	85.6	53
Barium	0.0043	0.0301	0.2000	61	53	0	86.9	0
Bromomethane	0.0014	0.0014	0.0014	69	1	NA	1.4	NA
Cadmium	0.0040	0.0003	0.0040	54	1	0	1.9	0
Chloromethane	NA	NA	NA	73	0	NA	0	NA
Chromium	0.0006	0.0026	0.0400	47	21	0	44.7	0
Copper	0.0006	0.0077	0.0870	54	40	NA	74.1	NA
Ethyl acetate	NA	NA	NA	4	0	NA	0	NA
Lead	0.0001	0.0012	0.0044	53	25	0	47.2	0
Methanol	1.6	0.4162	1.6	11	1	NA	9.1	NA
Molybdenum	0.0004	0.0030	0.0120	54	41	NA	75.9	NA
MTBE	NA	NA	NA	54	0	0	0	0
Nickel	0.0004	0.0025	0.0200	54	36	0	66.7	0
p-isopropyltoluene	0.0006	0.0001	0.0006	47	1	NA	2.1	NA
sec-butylbenzene	0.0002	0.0002	0.0004	69	11	NA	15.9	NA
Selenium	0.0002	0.0018	0.0075	55	21	0	38.2	0
Strontium	0.0180	0.1286	0.4600	52	45	NA	86.5	NA
Zinc	0.0018	0.0109	0.1010	55	34	NA	61.8	NA