# Results of Proficiency Test
# Organotin Compounds in Textile
# December 2019


Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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# 1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather, there are some Ecolabelling schemes imposing environmental requirements for textile & leather products on a voluntary basis. Well known organizations are for instance: Bluesign® (Switzerland), which has created a Bluesign® system substances list (BSSL) and Oeko-Tex Standard 100 (Switzerland).

Since 2016, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Organotin components in textile every year. During the annual proficiency testing program of 2019/2020, it was decided to continue the proficiency test for the analysis of Organotin components in textile.
In this interlaboratory study 89 laboratories in 24 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

# 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands was the organizer of the proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each, both positive on Organotin and respectively labelled #19660 and #19661. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

## 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

## 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

## 2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

Two different batches of textile were enriched with a number of Organotin components. The first batch was a lilac colored cotton. The second batch was a pink colored cotton.
Both batches were cut finely, well mixed and divided over respectively 140 and 197 subsamples of 3 grams each and labelled #19660 and #19661 respectively.
The homogeneity of sample #19660 was checked by the determination of Dibutyltin (DBT) and the homogeneity of sample #19661 was checked by determination of Dimethyltin (DMT) in accordance with an in house test method on respectively 9 and 10 stratified randomly selected subsamples of each set.

| | Sample #19660 | Sample #19661 |
|---|---|---|
| | DBT in mg/kg | DMT in mg/kg |
| Sample 1 | 5.14 | 5.11 |
| Sample 2 | 5.19 | 6.19 |
| Sample 3 | 4.84 | 6.35 |
| Sample 4 | 4.29 | 5.19 |
| Sample 5 | 5.37 | 6.24 |
| Sample 6 | 4.97 | 5.15 |
| Sample 7 | 5.31 | 5.10 |
| Sample 8 | 4.24 | 5.97 |
| Sample 9 | 4.83 | 5.60 |
| Sample 10 | -- | 6.14 |

Table 1: homogeneity test results of subsamples #19660 and #19661

From the above test results the relative between sample standard deviations $RSD_r$ were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | Sample #19660 | Sample #19661 |
|---|---|---|
| | DBT in mg/kg | DMT in mg/kg |
| $RSD_r$ (observed) | 8.4% | 9.2% |
| Reference test method | ISO/TS16179:12 | ISO/TS16179:12 |
| 0.3 x $RSD_R$ (ref. test method) | 6.9% | 6.9% |
| 0.3 x $RSD_R$ (previous PTs) | 11.4% | 11.4% |

Table 2: evaluation of the relative standard deviations of subsamples #19660 and #19661

The calculated relative standard deviations $RSD_r$ for both samples were not in agreement with 0.3 times the $RSD_R$ of the reference test method, but they did meet 0.3 times the $RSD_R$ from previous proficiency tests (see chapter 4.3, table 5). Therefore, the homogeneities of #19660 and #19661 were assumed.

To each of the participating laboratories, one subsample of #19660 and one subsample of #19661 were sent on November 13, 2019.

## 2.5 ANALYZES

The participants were requested to determine Monomethyltin (MMT), Dimethyltin (DMT), Trimethyltin (TMT), Tripropyltin (TPT), Monobutyltin (MBT), Dibutyltin (DBT), Tributyltin (TBT), Tetrabutyltin (TeBT), Monooctyltin (MOT), Dioctyltin (DOT), Trioctyltin (TOT), Diphenyltin (DPhT), Triphenyltin (TPhT) and Tricyclohexyltin (TCyHT) on samples #19660 and #19661 applying the analysis procedure that is routinely used in the laboratory.
It was also requested to report if the laboratory was accredited for the requested components that were determined and to report some analytical details of the test method used.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but to report as much significant figures as possible. It was also requested not to report “less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

# 3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'.
After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test results are fit-for-use.

The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of Proficiency Test) / target standard deviation

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

| | | | |
|---|---|---|---|
| | $\lvert z \rvert$ | < 1 | good |
| 1 | < $\lvert z \rvert$ | < 2 | satisfactory |
| 2 | < $\lvert z \rvert$ | < 3 | questionable |
| 3 | < $\lvert z \rvert$ | | unsatisfactory |

## 4 EVALUATION

During the execution of this proficiency test no problems occurred with the dispatch of the samples. Four laboratories did not report any test results and two other laboratories reported the test results after the final reporting date. Not all laboratories were able to report all analyses requested.

In total 85 participants reported 317 numerical test results. Observed were 12 statistical outliers, which is 3.8% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred as “not OK” or “suspect”. The statistical evaluation of these data should be used with due care, see also paragraph 3.1.

### 4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the reported test results are discussed per sample and per component. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables in appendix 1 together with the reported test results. The abbreviations used in these tables are explained in appendix 5.

For the determination of Organotin in textile ISO/TS16179:12 is recommended to be the test method (see Bluesign v6.0, July 2016 and Oekotex standard, January 2019). In ISO/TS16179 not for all listed Organotin components precision data are available (see table B.1 of ISO/TS16179:12), but only for three Organotin components. Regretfully, the given RSD% for all three components deviate much from each other. Therefore, the RSD% in ISO/TS16179 were compared with the RSD% of the Organotin components in this PT for laboratories that followed ISO/TS16179 by using Methanol/Ethanol as extraction solvent and 60°C as extraction temperature for 60 minutes (see table 6 and appendix 1). The RSD% of the detected Organotin components are close to the RSD% of 23% for MBT. Therefore, it was decided to use the RSD of 23% for MBT for all Organotin components from ISO/TS16179:12 as reference.
The target reproducibility for each Organotin component will be 2.8 * 23 * mean PT /100.

**Sample #19660**

Monobutyltin (MBT): This determination was problematic at a concentration level of 0.78 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirements of ISO/TS16179:12.

Dibutyltin (DBT): This determination was not problematic at a concentration level of 4.9 mg/kg. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the reproducibility requirements of ISO/TS16179:12.

Other Organotin components: The concentrations reported for all other Organotin components were near or below the detection limit. Therefore, no z-scores were calculated. See appendix 2 for the reported test results.

**Sample #19661**

Monomethyltin (MMT): This determination was problematic at a concentration level of 0.13 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirements of ISO/TS16179:12.

Dimethyltin (DMT): This determination was not problematic at a concentration level of 4.9 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the reproducibility requirements of ISO/TS16179:12.

Trimethyltin (TMT): This determination was problematic at a concentration level of 0.084 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirements of ISO/TS16179:12.

Other Organotin components: The concentrations reported for all other Organotin components were near or below the detection limit. Therefore, no z-scores were calculated. See appendix 2 for the reported test results.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average test result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility are compared in the next table.

| Component | unit | n | average | 2.8 * sd | R(target) |
|---|---|---|---|---|---|
| Monobutyltin (MBT) | mg/kg | 76 | 0.78 | 0.72 | 0.50 |
| Dibutyltin (DBT) | mg/kg | 81 | 4.92 | 3.00 | 3.17 |

Table 3: reproducibilities of Organotin components in sample #19660

| Component | unit | n | average | 2.8 * sd | R(target) |
|---|---|---|---|---|---|
| Monomethyltin (MMT) | mg/kg | 46 | 0.13 | 0.14 | 0.08 |
| Dimethyltin (DMT) | mg/kg | 72 | 4.90 | 3.01 | 3.15 |
| Trimethyltin (TMT) | mg/kg | 30 | 0.08 | 0.06 | 0.05 |

Table 4: reproducibilities of Organotin components in sample #19661

Without further statistical calculations, it could be concluded that for a number of the observed Organotin components, especially with concentrations below 1 mg/kg the group of participating laboratories may have difficulties with the analysis. See also the discussion in paragraph 5.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2019 WITH THE PREVIOUS PTS

The performance of the determinations of the proficiency test was compared, expressed as the relative standard deviation (RSD) of the PT, see next table.

| Component | December 2019 | December 2018 | December 2017 | December 2016 | Reference ISO16179 |
|---|---|---|---|---|---|
| Monomethyltin (MMT) | 37% | -- | -- | -- | 23% |
| Dimethyltin (DMT) | 22% | 25 – 40% | 38% | -- | 23% |
| Trimethyltin (TMT) | 26% | | | | |
| Monobutyltin (MBT) | 33% | -- | -- | 37% | 23% |
| Dibutyltin (DBT) | 22% | 21% | 35% | -- | 23% |
| Tributyltin (TBT) | -- | 29 – 31% | 29% | -- | 23% |

Table 5: uncertainties comparison with previous proficiency tests

The uncertainties observed in this PT are in line in comparison with the uncertainties observed in previous PTs.

### 4.4 EVALUATION OF ANALYTICAL DETAILS

For this PT some analysis details were requested (see appendix 3). From the answers given the following can be summarized;

- The majority of the participants (81%) is accredited according to ISO/IEC17025 for the determination of Organotin components in textile.
- About 52% of the participants used as intake 1 gram and 36% mentioned to have used 0.5 gram or less.
- 53% of the participants used a mixture of Methanol and Ethanol and 15% used Acetone as extraction solvent. Twenty other participants reported to have used different solvents (e.g. Hexane or iso-Octane).
- The majority of the participants (86%) used ultrasonic bath for the extraction. Almost all participants (82%) used an extraction time of 60 minutes.
- About 56% of the participants reported to extract at 60°C and 19% to extract at 40°C. Furthermore, 44% of the group reported to observe a pH of 4.5 / 4.6 and 12 participants reported to observe a pH 5 or higher. About 46% have adjusted the pH.

The effect of the reported analytical details on the determination of Dibutyltin (DBT) in sample #19660 was further investigated, see summary in below table.

| Analytical Details | unit | n | average | RSD |
|---|---|---|---|---|
| ISO/IEC17025 accredited | mg/kg | 65 | 4.94 | 22% |
| Not ISO/IEC17025 accredited | mg/kg | 11 | 4.84 | 24% |
| <1g sample intake | mg/kg | 30 | 4.96 | 18% |
| 1g sample intake | mg/kg | 41 | 4.99 | 24% |
| >1g sample intake | mg/kg | 3 | 4.82 | 4% |
| Methanol-Ethanol solvent | mg/kg | 42 | 4.89 | 22% |
| Acetone | mg/kg | 13 | 5.51 | 18% |

Table 6: effect of analytical details on DBT in textile sample #19660

It appeared that the effect of the analytical details on the determination of DBT is small and not statistically significant.

## 5 DISCUSSION

In this proficiency test for the determination of Organotin in textile, it was noticed that the majority of the participants was able to detect and quantify correctly the Organotin components Dibutyltin in sample #19660 and Dimethyltin in sample #19661.

When the test results of this interlaboratory study were compared to the Oeko-Tex Standard 100 (see table 7), it could be noted that some laboratories would make a different decision about the acceptability of the textile. Three reporting laboratories would accept sample #19660 based on MMT+DMT for classes 2, 3 and 4 (less than 1 mg/kg). All other of the reporting laboratories would have rejected sample #19660. Sample #19661 was rejected by all reporting laboratories, except one for too high level of Organotin present (more than 2 mg/kg).

| Oeko-Tex Standard 100 | Class 1 Baby clothes (mg/kg) | Class 2 Clothes direct skin contact (mg/kg) | Class 3 Clothes, no direct contact with skin (mg/kg) | Class 4 Decoration material (mg/kg) |
|---|---|---|---|---|
| TBT, TPhT | 0.5 | 1.0 | 1.0 | 1.0 |
| Other Organotin compounds | 1.0 | 2.0 | 2.0 | 2.0 |

Table 7: Ecolabelling Standard and Requirements for Textiles in EU

## 6 CONCLUSION

Although it is clear that not all laboratories followed the reported test method completely, it can be concluded that the observed variation in this interlaboratory study may not be caused by just one critical point in the analysis.
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

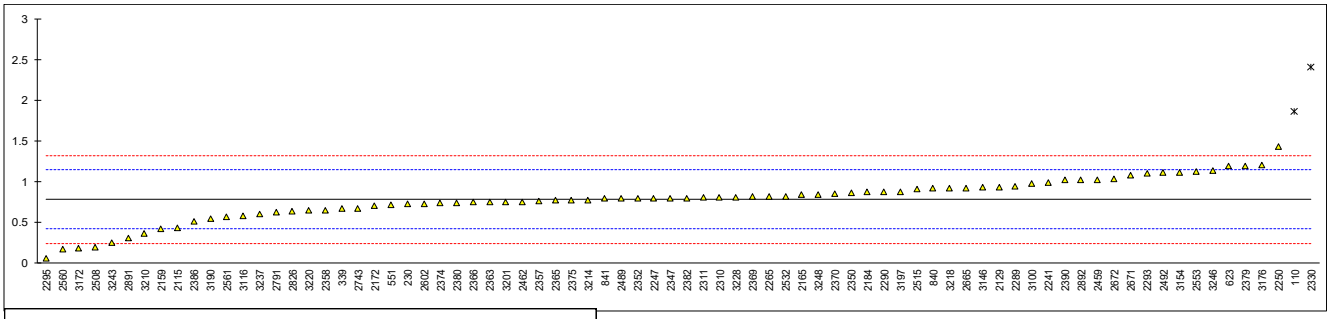
**APPENDIX 1**

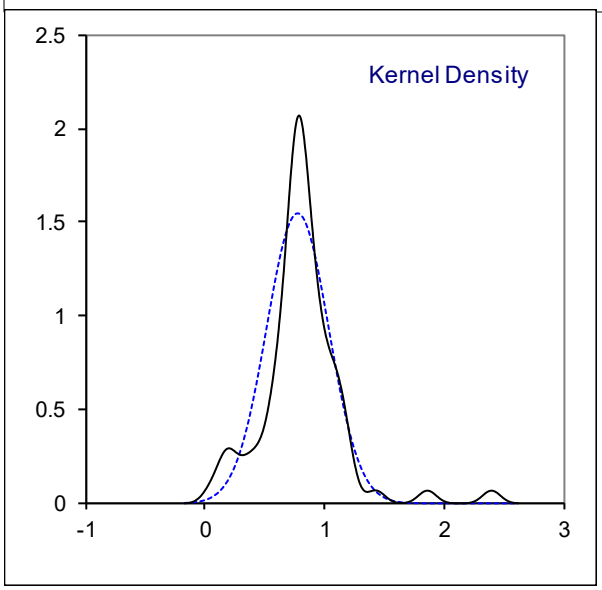
Determination of Monobutyltin (MBT) on sample #19660; results in mg/kg

| lab | Method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 110 | In house | 1.8655 | R(0.01) | 6.03 | |
| 210 | | ----- | | ----- | |
| 230 | ISO17353 | 0.723 | | -0.33 | |
| 339 | In house | 0.670 | | -0.62 | |
| 551 | In house | 0.715 | | -0.37 | |
| 623 | ISO TS 16179 | 1.19 | | 2.27 | |
| 840 | ISO TS 16179 | 0.92 | | 0.77 | |
| 841 | ISO TS 16179 | 0.79 | | 0.05 | |
| 2115 | In house | 0.437 | | -1.92 | |
| 2129 | ISO TS 16179 | 0.936 | | 0.86 | |
| 2159 | ISO17353 | 0.416 | | -2.03 | |
| 2165 | ISO TS 16179 | 0.84 | | 0.32 | |
| 2172 | | 0.701 | | -0.45 | |
| 2184 | ISO TS 16179 | 0.87 | | 0.49 | |
| 2241 | ISO TS 16179 | 0.987 | | 1.14 | |
| 2247 | ISO17353 | 0.80 | | 0.10 | |
| 2250 | ISO TS 16179 | 1.435 | | 3.63 | |
| 2265 | ISO TS 16179 | 0.82 | | 0.21 | |
| 2289 | ISO TS 16179 | 0.94 | | 0.88 | |
| 2290 | ISO TS 16179 | 0.87 | C | 0.49 | First reported 0.121 |
| 2293 | ISO TS 16179 | 1.10 | | 1.77 | |
| 2295 | ISO TS 16179 | 0.06 | | -4.01 | |
| 2310 | ISO17353 | 0.81 | | 0.16 | |
| 2311 | ISO17353 | 0.802 | | 0.11 | |
| 2330 | ISO17353 | 2.401 | R(0.01) | 9.00 | |
| 2347 | ISO TS 16179 | 0.80 | | 0.10 | |
| 2350 | ISO TS 16179 | 0.869 | | 0.48 | |
| 2352 | ISO TS 16179 | 0.796 | | 0.08 | |
| 2357 | ISO TS 16179 | 0.760 | | -0.12 | |
| 2358 | ISO17353 | 0.6526 | | -0.72 | |
| 2363 | ISO TS 16179 | 0.75 | | -0.18 | |
| 2365 | ISO TS 16179 | 0.77 | | -0.07 | |
| 2366 | ISO17353 | 0.75 | | -0.18 | |
| 2369 | ISO TS 16179 | 0.82 | | 0.21 | |
| 2370 | ISO17353 | 0.851 | | 0.38 | |
| 2374 | ISO17353 | 0.74 | | -0.23 | |
| 2375 | ISO17353 | 0.77 | | -0.07 | |
| 2379 | ISO TS 16179 | 1.197 | C | 2.31 | First reported 1.523 |
| 2380 | ISO17353 | 0.7407 | | -0.23 | |
| 2382 | ISO17353 | 0.800 | | 0.10 | |
| 2386 | ISO17353 | 0.508 | | -1.52 | |
| 2390 | | 1.02 | C | 1.32 | First reported 1.53 |
| 2459 | ISO TS 16179 | 1.021 | | 1.33 | |
| 2462 | ISO TS 16179 | 0.755 | | -0.15 | |
| 2489 | ISO17353 | 0.79 | | 0.05 | |
| 2492 | In house | 1.1126 | | 1.84 | |
| 2495 | | ----- | | ----- | |
| 2497 | | ----- | | ----- | |
| 2508 | ISO17353 | 0.19 | | -3.29 | |
| 2515 | ISO TS 16179 | 0.904 | | 0.68 | |
| 2522 | ISO17353 | NA | | ----- | |
| 2532 | ISO TS 16179 | 0.82 | | 0.21 | |
| 2553 | In house | 1.12 | | 1.88 | |
| 2560 | ISO17353 | 0.171 | | -3.40 | |
| 2561 | ISO TS 16179 | 0.5684 | | -1.19 | |
| 2590 | | ----- | | ----- | |
| 2602 | In house | 0.732 | | -0.28 | |
| 2644 | | ----- | | ----- | |
| 2665 | | 0.924 | | 0.79 | |
| 2671 | ISO TS 16179 | 1.08 | | 1.66 | |
| 2672 | ISO TS 16179 | 1.035 | | 1.41 | |
| 2743 | ISO TS 16179 | 0.6709 | | -0.62 | |
| 2758 | | ----- | | ----- | |
| 2791 | ISO TS 16179 | 0.63 | | -0.84 | |
| 2812 | | ----- | | ----- | |
| 2826 | ISO TS 16179 | 0.635 | | -0.82 | |
| 2864 | | ----- | | ----- | |
| 2891 | ISO TS 16179 | 0.31 | C | -2.62 | First reported <0.2 |
| 2892 | ISO TS 16179 | 1.020 | | 1.32 | |
| 2895 | | ----- | | ----- | |
| 3100 | ISO TS 16179 | 0.979 | | 1.10 | |
| 3116 | ISO TS 16179 | 0.5749 | | -1.15 | |
| 3146 | ISO TS 16179 | 0.93 | | 0.82 | |
| 3154 | ISO TS 16179 | 1.114 | | 1.85 | |

| lab | Method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 3160 | | ----- | | ----- | |
| 3172 | ISO TS 16179 | 0.18 | C | -3.35 | First reported 0.059 |
| 3176 | ISO17353 | 1.20 | | 2.33 | |
| 3190 | ISO17353 | 0.542 | | -1.33 | |
| 3197 | ISO17353 | 0.88 | | 0.55 | |
| 3201 | In house | 0.7548 | C | -0.15 | First reported 7.549 |
| 3210 | In house | 0.369 | | -2.30 | |
| 3214 | ISO TS 16179 | 0.773 | | -0.05 | |
| 3218 | ISO TS 16179 | 0.923 | | 0.79 | |
| 3220 | ISO TS 16179 | 0.65 | C | -0.73 | First reported 0.122 |
| 3228 | ISO TS 16179 | 0.81 | | 0.16 | |
| 3237 | ISO TS 16179 | 0.6 | | -1.01 | |
| 3243 | In house | 0.25 | | -2.96 | |
| 3246 | ISO TS 16179 | 1.132 | | 1.95 | |
| 3248 | In house | 0.840 | | 0.32 | |
| | | | | | Only ISO16179:12 *) |
| | normality | OK | | | suspect |
| | n | 76 | | | 39 |
| | outliers | 2 | | | 0 |
| | mean (n) | 0.7818 | | | 0.8237 |
| | st.dev. (n) | 0.25865 | RSD = 33% | | 0.25736 RSD = 31% |
| | R(calc.) | 0.7242 | | | 0.7206 |
| | st.dev.(ISO/TS16179:12) | 0.17981 | | | 0.18945 |
| | R(ISO/TS16179:12) | 0.5035 | | | 0.5305 |

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes



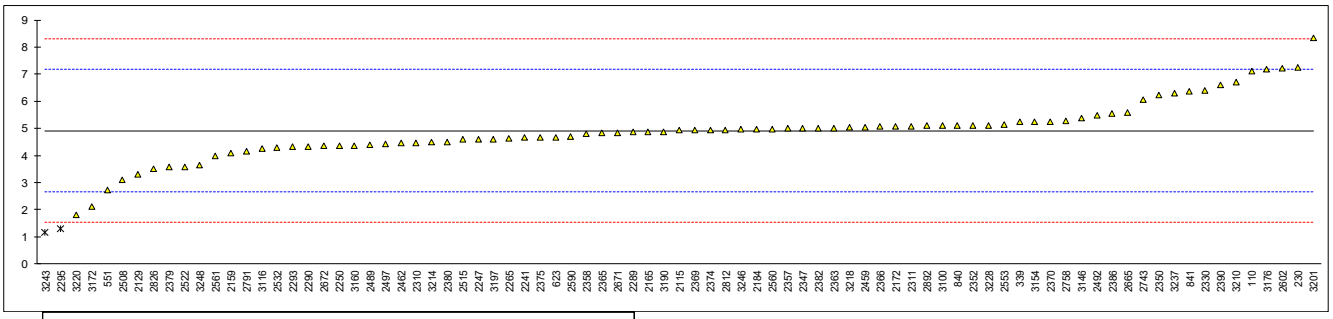


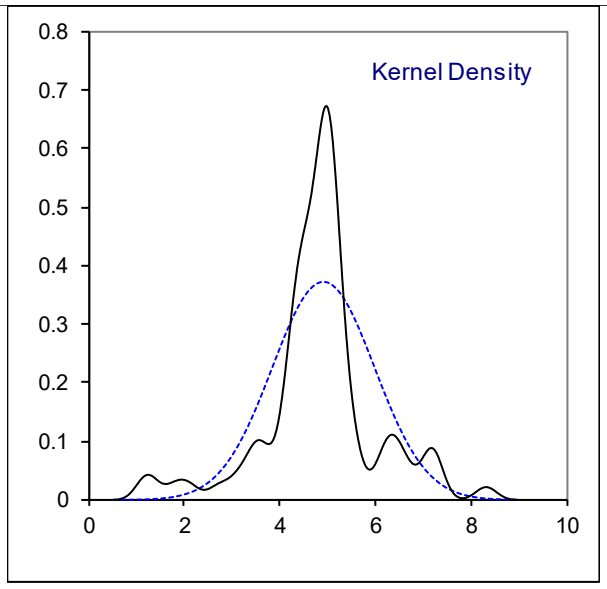
Determination of Dibutyltin (DBT) on sample #19660; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | In house | 7.1285 | | 1.96 | |
| 210 | | ----- | | ----- | |
| 230 | ISO17353 | 7.235 | | 2.05 | |
| 339 | In house | 5.235 | | 0.28 | |
| 551 | In house | 2.715 | | -1.95 | |
| 623 | ISO TS 16179 | 4.68 | | -0.21 | |
| 840 | ISO TS 16179 | 5.11 | | 0.17 | |
| 841 | ISO TS 16179 | 6.36 | | 1.28 | |
| 2115 | In house | 4.931 | | 0.01 | |
| 2129 | ISO TS 16179 | 3.29 | | -1.44 | |
| 2159 | ISO17353 | 4.087 | | -0.73 | |
| 2165 | ISO TS 16179 | 4.86 | | -0.05 | |
| 2172 | | 5.065 | | 0.13 | |
| 2184 | ISO TS 16179 | 4.98 | | 0.06 | |
| 2241 | ISO TS 16179 | 4.653 | | -0.23 | |
| 2247 | ISO17353 | 4.60 | | -0.28 | |
| 2250 | ISO TS 16179 | 4.365 | | -0.49 | |
| 2265 | ISO TS 16179 | 4.63 | | -0.25 | |
| 2289 | ISO TS 16179 | 4.86 | | -0.05 | |
| 2290 | ISO TS 16179 | 4.31 | C | -0.54 | First reported 1.072 |
| 2293 | ISO TS 16179 | 4.31 | | -0.54 | |
| 2295 | ISO TS 16179 | 1.30 | DG(0.05) | -3.20 | |
| 2310 | ISO17353 | 4.46 | | -0.40 | |
| 2311 | ISO17353 | 5.074 | | 0.14 | |
| 2330 | ISO17353 | 6.399 | | 1.31 | |
| 2347 | ISO TS 16179 | 5.00 | | 0.07 | |
| 2350 | ISO TS 16179 | 6.220 | | 1.15 | |
| 2352 | ISO TS 16179 | 5.112 | | 0.17 | |
| 2357 | ISO TS 16179 | 4.990 | | 0.07 | |
| 2358 | ISO17353 | 4.8145 | | -0.09 | |
| 2363 | ISO TS 16179 | 5.02 | | 0.09 | |
| 2365 | ISO TS 16179 | 4.85 | | -0.06 | |
| 2366 | ISO17353 | 5.06 | | 0.13 | |
| 2369 | ISO TS 16179 | 4.94 | | 0.02 | |
| 2370 | ISO17353 | 5.25 | | 0.30 | |
| 2374 | ISO17353 | 4.95 | | 0.03 | |
| 2375 | ISO17353 | 4.67 | | -0.22 | |
| 2379 | ISO TS 16179 | 3.575 | | -1.19 | |
| 2380 | ISO17353 | 4.506 | | -0.36 | |
| 2382 | ISO17353 | 5.000 | | 0.07 | |
| 2386 | ISO17353 | 5.56 | | 0.57 | |
| 2390 | | 6.59 | | 1.48 | |
| 2459 | ISO TS 16179 | 5.053 | | 0.12 | |
| 2462 | ISO TS 16179 | 4.451 | | -0.41 | |
| 2489 | ISO17353 | 4.39 | | -0.46 | |
| 2492 | In house | 5.4948 | | 0.51 | |
| 2495 | | ----- | | ----- | |
| 2497 | ISO TS 16179 | 4.418 | | -0.44 | |
| 2508 | ISO17353 | 3.09 | | -1.61 | |
| 2515 | ISO TS 16179 | 4.593 | | -0.29 | |
| 2522 | ISO17353 | 3.59 | | -1.17 | |
| 2532 | ISO TS 16179 | 4.3 | | -0.54 | |
| 2553 | In house | 5.15 | | 0.21 | |
| 2560 | ISO17353 | 4.987 | | 0.06 | |
| 2561 | ISO TS 16179 | 4.000 | | -0.81 | |
| 2590 | ISO TS 16179 | 4.694 | | -0.20 | |
| 2602 | In house | 7.226 | | 2.04 | |
| 2644 | | ----- | | ----- | |
| 2665 | | 5.578 | | 0.59 | |
| 2671 | ISO TS 16179 | 4.85 | | -0.06 | |
| 2672 | ISO TS 16179 | 4.345 | | -0.50 | |
| 2743 | ISO TS 16179 | 6.0742 | C | 1.02 | First reported 9.2589 |
| 2758 | ISO TS 16179 | 5.277 | | 0.32 | |
| 2791 | ISO TS 16179 | 4.15 | | -0.68 | |
| 2812 | ISO17353 | 4.95 | | 0.03 | |
| 2826 | ISO TS 16179 | 3.50 | | -1.25 | |
| 2864 | | ----- | | ----- | |
| 2891 | ISO TS 16179 | <0.2 | C | <-4.17 | Possibly a false negative test result? First reported <0.31 |
| 2892 | ISO TS 16179 | 5.100 | | 0.16 | |
| 2895 | | ----- | | ----- | |
| 3100 | ISO TS 16179 | 5.104 | | 0.17 | |
| 3116 | ISO TS 16179 | 4.240 | | -0.60 | |
| 3146 | ISO TS 16179 | 5.37 | | 0.40 | |
| 3154 | ISO TS 16179 | 5.241 | | 0.29 | |

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 3160 | ISO TS 16179 | 4.365 | | -0.49 | |
| 3172 | ISO TS 16179 | 2.11 | C | -2.48 | First reported 1.327 |
| 3176 | ISO17353 | 7.17 | | 1.99 | |
| 3190 | ISO17353 | 4.865 | | -0.04 | |
| 3197 | ISO17353 | 4.60 | | -0.28 | |
| 3201 | In house | 8.321 | C | 3.01 | First reported 83.21 |
| 3210 | In house | 6.698 | | 1.58 | |
| 3214 | ISO TS 16179 | 4.499 | | -0.37 | |
| 3218 | ISO TS 16179 | 5.027 | | 0.10 | |
| 3220 | ISO TS 16179 | 1.804 | | -2.75 | |
| 3228 | ISO TS 16179 | 5.12 | | 0.18 | |
| 3237 | ISO TS 16179 | 6.3 | | 1.22 | |
| 3243 | In house | 1.1725 | C,DG(0.05) | -3.31 | First reported 1.58 |
| 3246 | ISO TS 16179 | 4.969 | | 0.05 | |
| 3248 | In house | 3.66 | | -1.11 | |
| | | | | | Only ISO16179:12 *) |
| | normality | suspect | | | not OK |
| | n | 81 | | | 43 |
| | outliers | 2 | | | 1 |
| | mean (n) | 4.9157 | | | 4.7977 |
| | st.dev. (n) | 1.07127 | RSD = 22% | | 1.20850 RSD = 25% |
| | R(calc.) | 2.9995 | | | 3.3838 |
| | st.dev.(ISO/TS16179:12) | 1.13060 | | | 1.10346 |
| | R(ISO/TS16179:12) | 3.1657 | | | 3.0897 |

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes



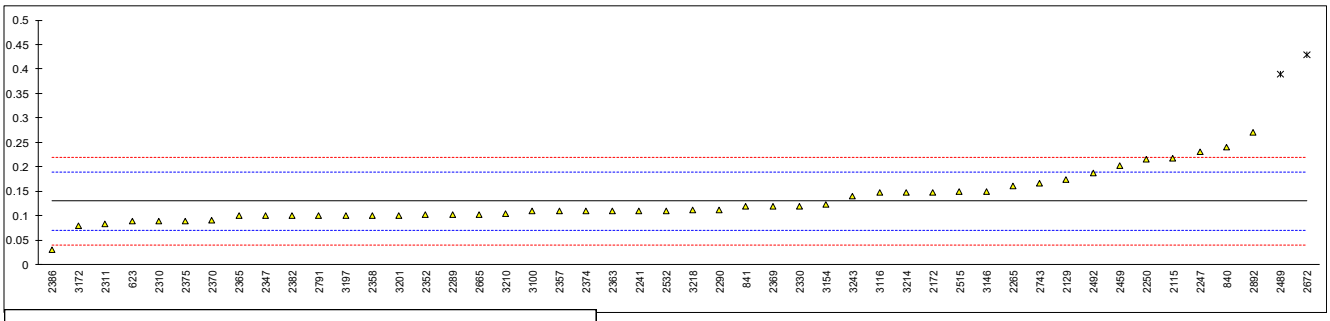


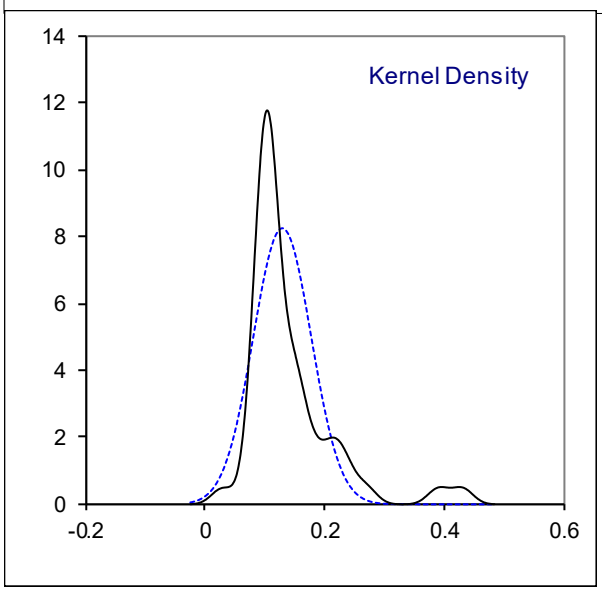
Determination of Monomethyltin (MMT) on sample #19661; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | | ----- | | ----- | |
| 210 | | ----- | | ----- | |
| 230 | | ----- | | ----- | |
| 339 | In house | < 0.1 | | ----- | |
| 551 | In house | N.D. | | ----- | |
| 623 | ISO TS 16179 | 0.09 | | -1.34 | |
| 840 | ISO TS 16179 | 0.24 | | 3.68 | |
| 841 | ISO TS 16179 | 0.12 | | -0.33 | |
| 2115 | In house | 0.217 | | 2.91 | |
| 2129 | ISO TS 16179 | 0.175 | | 1.51 | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2172 | | 0.1484 | | 0.62 | |
| 2184 | | ----- | | ----- | |
| 2241 | ISO TS 16179 | 0.110 | | -0.67 | |
| 2247 | ISO17353 | 0.23 | | 3.35 | |
| 2250 | ISO TS 16179 | 0.215 | | 2.85 | |
| 2265 | ISO TS 16179 | 0.16 | | 1.01 | |
| 2289 | ISO TS 16179 | 0.102 | | -0.93 | |
| 2290 | ISO TS 16179 | 0.112 | | -0.60 | |
| 2293 | | ----- | | ----- | |
| 2295 | | ----- | | ----- | |
| 2310 | ISO17353 | 0.09 | | -1.34 | |
| 2311 | ISO17353 | 0.0833 | | -1.56 | |
| 2330 | ISO17353 | 0.120 | | -0.33 | |
| 2347 | ISO TS 16179 | 0.10 | | -1.00 | |
| 2350 | | ----- | | ----- | |
| 2352 | ISO TS 16179 | 0.102 | | -0.93 | |
| 2357 | ISO TS 16179 | 0.110 | | -0.67 | |
| 2358 | ISO17353 | 0.1005 | | -0.98 | |
| 2363 | ISO TS 16179 | 0.11 | | -0.67 | |
| 2365 | ISO TS 16179 | 0.10 | | -1.00 | |
| 2366 | ISO17353 | <0.50 | | ----- | |
| 2369 | ISO TS 16179 | 0.12 | | -0.33 | |
| 2370 | ISO17353 | 0.0911 | | -1.30 | |
| 2374 | ISO17353 | 0.11 | | -0.67 | |
| 2375 | ISO17353 | 0.09 | | -1.34 | |
| 2379 | ISO TS 16179 | Not detected | | ----- | |
| 2380 | ISO17353 | ND | | ----- | |
| 2382 | ISO17353 | 0.100 | | -1.00 | |
| 2386 | ISO17353 | 0.03 | | -3.34 | |
| 2390 | | ----- | | ----- | |
| 2459 | ISO TS 16179 | 0.202 | | 2.41 | |
| 2462 | | ----- | | ----- | |
| 2489 | ISO17353 | 0.39 | R(0.01) | 8.71 | |
| 2492 | In house | 0.1875 | | 1.93 | |
| 2495 | | ----- | | ----- | |
| 2497 | | ----- | | ----- | |
| 2508 | | ----- | | ----- | |
| 2515 | ISO TS 16179 | 0.149 | | 0.64 | |
| 2522 | ISO17353 | NA | | ----- | |
| 2532 | ISO TS 16179 | 0.11 | | -0.67 | |
| 2553 | In house | ND | | ----- | |
| 2560 | | ----- | | ----- | |
| 2561 | ISO TS 16179 | <0.05 | | ----- | |
| 2590 | | ----- | | ----- | |
| 2602 | | ----- | | ----- | |
| 2644 | | ----- | | ----- | |
| 2665 | | 0.102 | | -0.93 | |
| 2671 | | ----- | | ----- | |
| 2672 | ISO TS 16179 | 0.430 | R(0.01) | 10.04 | |
| 2743 | ISO TS 16179 | 0.1664 | | 1.22 | |
| 2758 | | ----- | | ----- | |
| 2791 | ISO TS 16179 | 0.10 | | -1.00 | |
| 2812 | | ----- | | ----- | |
| 2826 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2891 | | ----- | | ----- | |
| 2892 | ISO TS 16179 | 0.270 | | 4.69 | |
| 2895 | | ----- | | ----- | |
| 3100 | ISO TS 16179 | 0.109 | | -0.70 | |
| 3116 | ISO TS 16179 | 0.1475 | | 0.59 | |
| 3146 | ISO TS 16179 | 0.15 | | 0.67 | |
| 3154 | ISO TS 16179 | 0.123 | | -0.23 | |

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 3160 | | ----- | | ----- | |
| 3172 | ISO TS 16179 | 0.079 | | -1.70 | |
| 3176 | | ----- | | ----- | |
| 3190 | | ----- | | ----- | |
| 3197 | ISO17353 | 0.10 | | -1.00 | |
| 3201 | In house | 0.101 | C | -0.97 | First reported 1.014 |
| 3210 | In house | 0.104 | | -0.87 | |
| 3214 | ISO TS 16179 | 0.148 | | 0.61 | |
| 3218 | ISO TS 16179 | 0.111 | | -0.63 | |
| 3220 | ISO TS 16179 | ND | | ----- | |
| 3228 | | ----- | | ----- | |
| 3237 | | ----- | | ----- | |
| 3243 | In house | 0.14 | | 0.34 | |
| 3246 | ISO TS 16179 | NA | | ----- | |
| 3248 | | ----- | | ----- | |
| | | | | | Only ISO16179:12 *) |
| | normality | not OK | | | not OK |
| | n | 46 | | | 24 |
| | outliers | 2 | | | 1 |
| | mean (n) | 0.1299 | | | 0.1392 |
| | st.dev. (n) | 0.04828 | RSD = 37% | | 0.04861 RSD = 35% |
| | R(calc.) | 0.1352 | | | 0.1361 |
| | st.dev.(ISO/TS16179:12) | 0.02988 | | | 0.03201 |
| | R(ISO/TS16179:12) | 0.0837 | | | 0.0896 |

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes



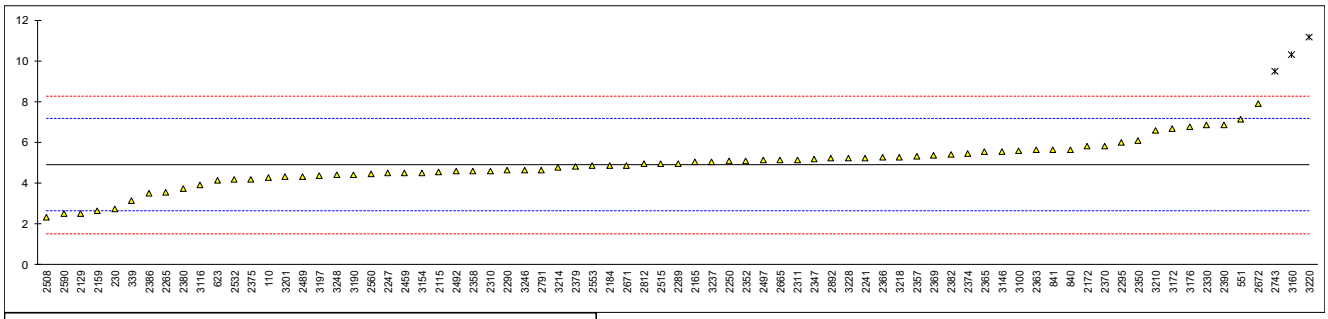



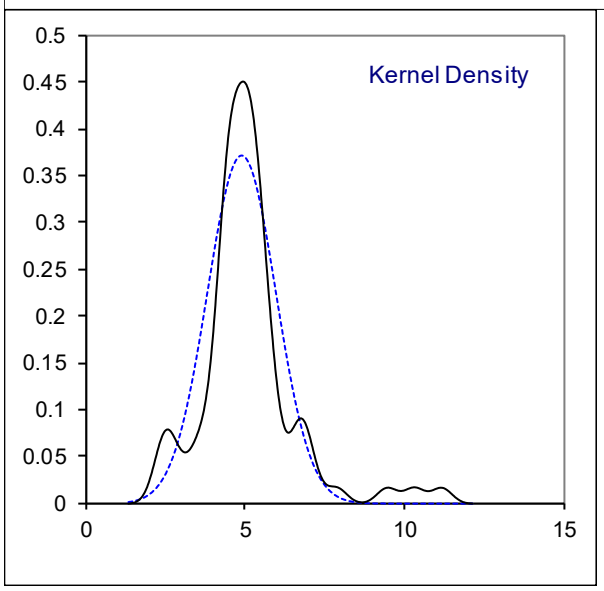
Determination of Dimethyltin (DMT) on sample #19661; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | In house | 4.2695 | | -0.56 | |
| 210 | | ----- | | ----- | |
| 230 | ISO17353 | 2.739 | | -1.92 | |
| 339 | In house | 3.115 | | -1.58 | |
| 551 | In house | 7.115 | | 1.97 | |
| 623 | ISO TS 16179 | 4.15 | | -0.66 | |
| 840 | ISO TS 16179 | 5.63 | | 0.65 | |
| 841 | ISO TS 16179 | 5.62 | | 0.64 | |
| 2115 | In house | 4.563 | | -0.30 | |
| 2129 | ISO TS 16179 | 2.504 | | -2.13 | |
| 2159 | ISO17353 | 2.617 | | -2.03 | |
| 2165 | ISO TS 16179 | 5.02 | | 0.11 | |
| 2172 | | 5.790 | | 0.79 | |
| 2184 | ISO TS 16179 | 4.88 | | -0.02 | |
| 2241 | ISO TS 16179 | 5.220 | | 0.29 | |
| 2247 | ISO17353 | 4.50 | | -0.35 | |
| 2250 | ISO TS 16179 | 5.070 | | 0.15 | |
| 2265 | ISO TS 16179 | 3.55 | | -1.20 | |
| 2289 | ISO TS 16179 | 4.96 | | 0.05 | |
| 2290 | ISO TS 16179 | 4.611 | | -0.26 | |
| 2293 | | ----- | | ----- | |
| 2295 | ISO TS 16179 | 6.0 | | 0.98 | |
| 2310 | ISO17353 | 4.6 | | -0.26 | |
| 2311 | ISO17353 | 5.144 | | 0.22 | |
| 2330 | ISO17353 | 6.845 | | 1.73 | |
| 2347 | ISO TS 16179 | 5.19 | | 0.26 | |
| 2350 | ISO TS 16179 | 6.072 | | 1.04 | |
| 2352 | ISO TS 16179 | 5.109 | | 0.19 | |
| 2357 | ISO TS 16179 | 5.330 | | 0.38 | |
| 2358 | ISO17353 | 4.5921 | | -0.27 | |
| 2363 | ISO TS 16179 | 5.61 | | 0.63 | |
| 2365 | ISO TS 16179 | 5.52 | | 0.55 | |
| 2366 | ISO17353 | 5.25 | | 0.31 | |
| 2369 | ISO TS 16179 | 5.38 | | 0.43 | |
| 2370 | ISO17353 | 5.81 | | 0.81 | |
| 2374 | ISO17353 | 5.46 | | 0.50 | |
| 2375 | ISO17353 | 4.20 | | -0.62 | |
| 2379 | ISO TS 16179 | 4.810 | | -0.08 | |
| 2380 | ISO17353 | 3.71 | | -1.05 | |
| 2382 | ISO17353 | 5.410 | | 0.45 | |
| 2386 | ISO17353 | 3.49 | | -1.25 | |
| 2390 | | 6.849 | | 1.73 | |
| 2459 | ISO TS 16179 | 4.503 | | -0.35 | |
| 2462 | | ----- | | ----- | |
| 2489 | ISO17353 | 4.33 | | -0.50 | |
| 2492 | In house | 4.5713 | | -0.29 | |
| 2495 | | ----- | | ----- | |
| 2497 | ISO TS 16179 | 5.124 | C | 0.20 | First reported 0.058 |
| 2508 | ISO17353 | 2.30 | | -2.31 | |
| 2515 | ISO TS 16179 | 4.957 | | 0.05 | |
| 2522 | ISO17353 | NA | | ----- | |
| 2532 | ISO TS 16179 | 4.18 | | -0.64 | |
| 2553 | In house | 4.84 | | -0.05 | |
| 2560 | ISO17353 | 4.456 | | -0.39 | |
| 2561 | ISO TS 16179 | <0.05 | | <-4.30 | Possibly a false negative test result? |
| 2590 | ISO TS 16179 | 2.485 | | -2.14 | |
| 2602 | | ----- | | ----- | |
| 2644 | | ----- | | ----- | |
| 2665 | | 5.137 | | 0.21 | |
| 2671 | ISO TS 16179 | 4.88 | | -0.02 | |
| 2672 | ISO TS 16179 | 7.880 | | 2.65 | |
| 2743 | ISO TS 16179 | 9.4674 | R(0.01) | 4.06 | |
| 2758 | | ----- | | ----- | |
| 2791 | ISO TS 16179 | 4.63 | | -0.24 | |
| 2812 | ISO17353 | 4.93 | | 0.03 | |
| 2826 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2891 | | ----- | | ----- | |
| 2892 | ISO TS 16179 | 5.200 | | 0.27 | |
| 2895 | | ----- | | ----- | |
| 3100 | ISO TS 16179 | 5.592 | | 0.62 | |
| 3116 | ISO TS 16179 | 3.886 | | -0.90 | |
| 3146 | ISO TS 16179 | 5.55 | | 0.58 | |
| 3154 | ISO TS 16179 | 4.515 | | -0.34 | |

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 3160 | ISO TS 16179 | 10.321 | R(0.01) | 4.81 | |
| 3172 | ISO TS 16179 | 6.663 | | 1.57 | |
| 3176 | ISO17353 | 6.78 | | 1.67 | |
| 3190 | ISO17353 | 4.420 | | -0.42 | |
| 3197 | ISO17353 | 4.38 | | -0.46 | |
| 3201 | In house | 4.296 | C | -0.53 | First reported 42.96 |
| 3210 | In house | 6.586 | | 1.50 | |
| 3214 | ISO TS 16179 | 4.757 | | -0.13 | |
| 3218 | ISO TS 16179 | 5.279 | | 0.34 | |
| 3220 | ISO TS 16179 | 11.182 | R(0.01) | 5.58 | |
| 3228 | ISO TS 16179 | 5.21 | | 0.28 | |
| 3237 | ISO TS 16179 | 5.03 | | 0.12 | |
| 3243 | | ----- | | ----- | |
| 3246 | ISO TS 16179 | 4.627 | | -0.24 | |
| 3248 | In house | 4.41 | | -0.43 | |
| | | | | | Only ISO16179:12 *) |
| | normality | OK | | | not OK |
| | n | 72 | | | 35 |
| | outliers | 3 | | | 0 |
| | mean (n) | 4.8985 | | | 5.0770 |
| | st.dev. (n) | 1.07593 | RSD = 22% | | 0.95051 RSD = 19% |
| | R(calc.) | 3.0126 | | | 2.6614 |
| | st.dev.(ISO/TS16179:12) | 1.12665 | | | 1.16770 |
| | R(ISO/TS16179:12) | 3.1546 | | | 3.2696 |

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes



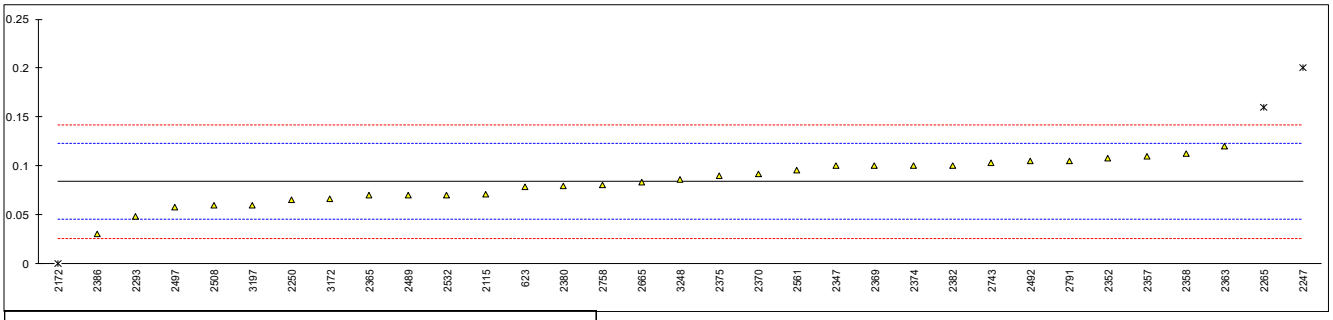


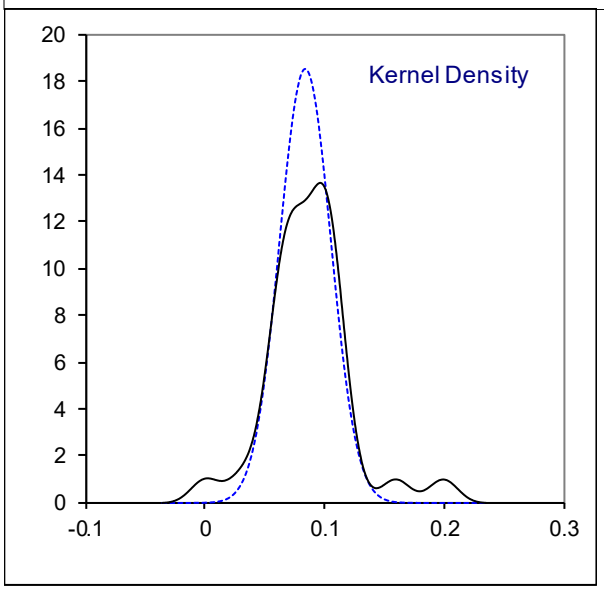
Determination of Trimethyltin (TMT) on sample #19661; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | In house | ND | | ----- | |
| 210 | | ----- | | ----- | |
| 230 | | ----- | | ----- | |
| 339 | | ----- | | ----- | |
| 551 | In house | N.D. | | ----- | |
| 623 | ISO TS 16179 | 0.079 | | -0.26 | |
| 840 | ISO TS 16179 | not detected | | ----- | |
| 841 | ISO TS 16179 | ND | | ----- | |
| 2115 | In house | 0.071 | | -0.67 | |
| 2129 | ISO TS 16179 | <0,1 | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2172 | | 0 | R(0.05) | -4.35 | |
| 2184 | | ----- | | ----- | |
| 2241 | | ----- | | ----- | |
| 2247 | ISO17353 | 0.2 | R(0.05) | 6.01 | |
| 2250 | ISO TS 16179 | 0.065 | | -0.98 | |
| 2265 | ISO TS 16179 | 0.16 | R(0.05) | 3.94 | |
| 2289 | ISO TS 16179 | <0.05 | | ----- | |
| 2290 | ISO TS 16179 | <0.05 | | ----- | |
| 2293 | ISO TS 16179 | 0.0488 | | -1.82 | |
| 2295 | | ----- | | ----- | |
| 2310 | ISO17353 | Not Detected | | ----- | |
| 2311 | ISO17353 | Not Detected | | ----- | |
| 2330 | ISO17353 | ND | | ----- | |
| 2347 | ISO TS 16179 | 0.10 | | 0.83 | |
| 2350 | | ----- | | ----- | |
| 2352 | ISO TS 16179 | 0.108 | | 1.25 | |
| 2357 | ISO TS 16179 | 0.110 | | 1.35 | |
| 2358 | ISO17353 | 0.1128 | | 1.49 | |
| 2363 | ISO TS 16179 | 0.12 | | 1.87 | |
| 2365 | ISO TS 16179 | 0.07 | | -0.72 | |
| 2366 | ISO17353 | <0.50 | | ----- | |
| 2369 | ISO TS 16179 | 0.1 | | 0.83 | |
| 2370 | ISO17353 | 0.0920 | | 0.42 | |
| 2374 | ISO17353 | 0.10 | | 0.83 | |
| 2375 | ISO17353 | 0.09 | | 0.31 | |
| 2379 | ISO TS 16179 | Not detected | | ----- | |
| 2380 | ISO17353 | 0.0798 | | -0.21 | |
| 2382 | ISO17353 | 0.100 | | 0.83 | |
| 2386 | ISO17353 | 0.03 | | -2.79 | |
| 2390 | | ----- | | ----- | |
| 2459 | | ----- | | ----- | |
| 2462 | | ----- | | ----- | |
| 2489 | ISO17353 | 0.07 | | -0.72 | |
| 2492 | In house | 0.1048 | | 1.08 | |
| 2495 | | ----- | | ----- | |
| 2497 | ISO TS 16179 | 0.058 | C | -1.34 | First reported 5.124 |
| 2508 | ISO17353 | 0.06 | | -1.24 | |
| 2515 | | ----- | | ----- | |
| 2522 | ISO17353 | NA | | ----- | |
| 2532 | ISO TS 16179 | 0.07 | | -0.72 | |
| 2553 | In house | ND | | ----- | |
| 2560 | | ----- | | ----- | |
| 2561 | ISO TS 16179 | 0.0959 | | 0.62 | |
| 2590 | | ----- | | ----- | |
| 2602 | | ----- | | ----- | |
| 2644 | | ----- | | ----- | |
| 2665 | | 0.083 | | -0.05 | |
| 2671 | | ----- | | ----- | |
| 2672 | | ----- | | ----- | |
| 2743 | ISO TS 16179 | 0.1034 | | 1.01 | |
| 2758 | ISO TS 16179 | 0.080 | | -0.20 | |
| 2791 | ISO TS 16179 | 0.105 | | 1.09 | |
| 2812 | | ----- | | ----- | |
| 2826 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2891 | ISO TS 16179 | < 0,2 | | ----- | |
| 2892 | | ----- | | ----- | |
| 2895 | | ----- | | ----- | |
| 3100 | ISO TS 16179 | <0.05 | | ----- | |
| 3116 | | ----- | | ----- | |
| 3146 | | ----- | | ----- | |
| 3154 | | ----- | | ----- | |

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 3160 | | ----- | | ----- | |
| 3172 | ISO TS 16179 | 0.066 | | -0.93 | |
| 3176 | | ----- | | ----- | |
| 3190 | | ----- | | ----- | |
| 3197 | ISO17353 | 0.06 | | -1.24 | |
| 3201 | In house | n.a. | | ----- | |
| 3210 | | ----- | | ----- | |
| 3214 | ISO TS 16179 | <0.2 | | ----- | |
| 3218 | | ----- | | ----- | |
| 3220 | ISO TS 16179 | ND | | ----- | |
| 3228 | | ----- | | ----- | |
| 3237 | | ----- | | ----- | |
| 3243 | | ----- | | ----- | |
| 3246 | ISO TS 16179 | Not detected | | ----- | |
| 3248 | In house | 0.086 | | 0.11 | |
| | | | | | Only ISO16179:12 *) |
| | normality | OK | | | OK |
| | n | 30 | | | 12 |
| | outliers | 3 | | | 1 |
| | mean (n) | 0.0840 | | | 0.0836 |
| | st.dev. (n) | 0.02148 | RSD = 26% | | 0.02244 RSD = 27% |
| | R(calc.) | 0.0601 | | | 0.0628 |
| | st.dev.(ISO/TS16179:12) | 0.01931 | | | 0.01923 |
| | R(ISO/TS16179:12) | 0.0541 | | | 0.0538 |

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes



**APPENDIX 2**

Determination of other Organotin components on sample #19660; results in mg/kg

| lab | MMT | DMT | TMT | TPT | TBT | TeBT | MOT | DOT | TOT | DPhT | TPhT | TCyHT |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 110 | ----- | 0.01133 | 0.01033 | ----- | 0.02000 | ND | ND | ND | ND | ND | ND | ND |
| 210 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 230 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 339 | < 0.1 | < 0.1 | ----- | ----- | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 |
| 551 | N.D. | N.D. | N.D. | N.D. | 0.12 | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. |
| 623 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 840 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 841 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2115 | ----- | 0.027 | 0.027 | ----- | 0.028 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2129 | <0,1 | <0,1 | <0,1 | <0,1 | 0.027 | <0,1 | <0,1 | <0,1 | <0,1 | <0,1 | <0,025 | <0,1 |
| 2159 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2165 | ----- | ND | ----- | ----- | ND | ND | ND | ND | ----- | ----- | ND | ND |
| 2172 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2184 | ----- | ND | ----- | ----- | ND | ----- | ND | ND | ----- | ----- | ND | ND |
| 2241 | <0.05 | <0.05 | ----- | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | ----- | <0.05 | <0.05 | <0.05 |
| 2247 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2250 | ----- | 0.035 | ----- | ----- | 0.040 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2265 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 |
| 2289 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2290 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2293 | ----- | ----- | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2295 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2310 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2311 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2330 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2347 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 |
| 2350 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2352 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2357 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2358 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2363 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2365 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2366 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 |
| 2369 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2370 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | 0.0355 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 2374 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2375 | ----- | ----- | ----- | ----- | 0.15 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2379 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2380 | nd | nd | nd | nd | nd | nd | nd | nd | nd | nd | nd | nd |
| 2382 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 |
| 2386 | <0,01 | <0,01 | <0,01 | <0,01 | 0.03 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 |
| 2390 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2459 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ----- | ND |
| 2462 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2489 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2492 | ----- | 0.0340 | ----- | ----- | 0.0334 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2495 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2497 | ----- | ----- | 0.0149 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2508 | ----- | ----- | ----- | ----- | 0.03 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2515 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2522 | NA | NA | NA | NA | <0.1 | NA | NA | <0.1 | NA | NA | <0.1 | NA |
| 2532 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2553 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2560 | ----- | 0 | ----- | 0 | 0 | 0 | 0 | 0 | 0 | 0 | ----- | 0 |
| 2561 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2590 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2602 | ----- | ----- | ----- | ----- | 0.0378 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2644 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2665 | <0.001 | 0.033 | 0.026 | 0.014 | 0.036 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| 2671 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2672 | <0.1 | <0.1 | ----- | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 2743 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | 0.1064 | ----- |
| 2758 | ----- | ----- | 0.021 | < 0.010 | 0.032 | ----- | ----- | < 0.010 | < 0.010 | ----- | < 0.010 | < 0.010 |
| 2791 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2812 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2826 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2864 | ----- | ----- | ----- | ----- | <0.2 | ----- | ----- | ----- | ----- | ----- | <0.2 | ----- |
| 2891 | ----- | ----- | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 |
| 2892 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2895 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3100 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 3116 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3146 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3154 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |

| lab | MMT | DMT | TMT | TPT | TBT | TeBT | MOT | DOT | TOT | DPhT | TPhT | TCyHT |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3160 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3176 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3190 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3197 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 |
| 3201 | n.n. | 0.0411 | n.a. | n.a. | n.n. | n.n. | n.n. | n.n. | n.a. | n.n. | n.n. | n.a. |
| 3210 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3214 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 |
| 3218 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3220 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 3228 | ----- | ND | ----- | ----- | ND | ----- | ND | ND | ----- | ----- | ND | ND |
| 3237 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3243 | n.d. | ----- | ----- | ----- | n.d. | n.d. | n.d. | n.d. | ----- | n.d. | n.d. | n.d. |
| 3246 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3248 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |

MMT = Monomethyltin
DMT = Dimethyltin
TMT = Trimethyltin
TPT = Tripropyltin
TBT = Tributyltin
TeBT = Tetrabutyltin
MOT = Monooctyltin
DOT = Dioctyltin
TOT = Trioctyltin
DPhT = Diphenyltin
TPhT = Triphenyltin
TCyHT = Tricyclohexyltin

Determination of other Organotin components on sample #19661; results in mg/kg

| lab | TPT | MBT | DBT | TBT | TeBT | MOT | DOT | TOT | DPhT | TPhT | TCyHT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 110 | ----- | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 210 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 230 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 339 | ----- | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 |
| 551 | N.D. | N.D. | N.D. | 0.12 | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. |
| 623 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 840 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 841 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ----- |
| 2115 | ----- | 0.023 | ----- | 0.016 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2129 | <0,1 | <0,1 | <0,1 | <0,025 | <0,1 | <0,1 | <0,1 | <0,1 | <0,1 | <0,025 | <0,1 |
| 2159 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2165 | ----- | ND | ND | ND | ND | ND | ND | ----- | ----- | ND | ND |
| 2172 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2184 | ----- | ND | ND | ND | ----- | ND | ND | ----- | ----- | ND | ND |
| 2241 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | ----- | <0.05 | <0.05 | <0.05 |
| 2247 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2250 | ----- | 0.025 | ----- | 0.020 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2265 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 | < 0,05 |
| 2289 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2290 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2293 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2295 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2310 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2311 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2330 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2347 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 |
| 2350 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2352 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2357 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2358 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2363 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2365 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2366 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 |
| 2369 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2370 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 2374 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2375 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2379 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2380 | nd | nd | nd | nd | nd | nd | nd | nd | nd | nd | nd |
| 2382 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 |
| 2386 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 |
| 2390 | ----- | ----- | ----- | 0.024 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2459 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2462 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2489 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2492 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2495 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2497 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2508 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2515 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2522 | NA | NA | <0.1 | <0.1 | NA | NA | <0.1 | NA | NA | <0.1 | NA |
| 2532 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2553 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2560 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | ----- | 0 |
| 2561 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 2590 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2602 | ----- | 0.0274 | ----- | 0.0246 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2644 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2665 | <0.001 | 0.014 | 0.002 | 0.017 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| 2671 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2672 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 2743 | ----- | 0.0509 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | 0.0753 | ----- |
| 2758 | < 0.010 | ----- | < 0.010 | 0.018 | ----- | ----- | < 0.010 | < 0.010 | ----- | < 0.010 | < 0.010 |
| 2791 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2812 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2826 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2864 | ----- | ----- | ----- | <0.2 | ----- | ----- | ----- | ----- | ----- | <0.2 | ----- |
| 2891 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 | < 0,2 |
| 2892 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2895 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3100 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 3116 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3146 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3154 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |

| lab | TPT | MBT | DBT | TBT | TeBT | MOT | DOT | TOT | DPhT | TPhT | TCyHT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 3160 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3176 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3190 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3197 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 |
| 3201 | n.a. | n.n. | 0.0052 | n.a. | n.n. | 0.0421 | n.n. | 0.0041 | n.n. | 0.0237 | n.a. |
| 3210 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3214 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 |
| 3218 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3220 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 3228 | ND | ND | ----- | ----- | ND | ND | ----- | ND | ND | ND | ----- |
| 3237 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3243 | n.d. | n.d. | n.d. | ----- | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | ----- |
| 3246 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3248 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |

TPT = Tripropyltin
MBT = Monobutyltin
DBT = Dibutyltin
TBT = Tributyltin
TeBT = Tetrabutyltin
MOT = Monooctyltin
DOT = Dioctyltin
TOT = Trioctyltin
DPhT = Diphenyltin
TPhT = Triphenyltin
TCyHT = Tricyclohexyltin

## APPENDIX 3 Analytical details

| lab | ISO/IEC 17025 accredited | Sample intake (in g) | Type of extraction | Solvent used to extract/release | Extraction time/temp (min/°C) | pH after adding the buffer | Extraction solution acidified until pH 4.5 |
|---|---|---|---|---|---|---|---|
| 110 | Yes | 1 | Ultrasonic | Acetone | 60 / 40 | 4.70 | Yes |
| 210 | --- | | --- | | | | --- |
| 230 | Yes | 1 | Ultrasonic | Acetone | 60 | | Yes |
| 339 | No | 0.5 - 2.5 | Ultrasonic | Acetone | 60 / 40 | | Yes |
| 551 | No | 1.0 | Ultrasonic | Methanol-Ethanol | 60 / 60 | | No |
| 623 | Yes | 1 | Ultrasonic | Methanol-Ethanol | 60 / 60 | | No |
| 840 | Yes | 1 | Ultrasonic | Methanol-Ethanol (8:2) | 60 / 60 | 4.5 | No |
| 841 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol - 4:1 | 60 / 60 | 4.5 | Yes |
| 2115 | Yes | 1 | Ultrasonic | | 60 / 40 | | --- |
| 2129 | Yes | 0,6 | Ultrasonic | Acidic methanol (HCl) | 60 / 60 | not | No |
| 2159 | Yes | 1 | Ultrasonic | Sodium diethyldithiocarbamate | 60 / 70 | 4,5 | No |
| 2165 | Yes | 1 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 5.8 | No |
| 2172 | Yes | 0.5 | Ultrasonic | Methanol | 60 / 70 | 4.5 | Yes |
| 2184 | Yes | 2 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 5.8 | No |
| 2241 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol - 4:1 | 60 / 60 | 4.5. | No |
| 2247 | --- | | --- | | | | --- |
| 2250 | Yes | 0,5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 5,9 | No |
| 2265 | No | 0,5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | not | No |
| 2289 | Yes | 1.0 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.5 | Yes |
| 2290 | Yes | | --- | | | | --- |
| 2293 | No | 1.001 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | | No |
| 2295 | Yes | 1 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.5 | Yes |
| 2310 | Yes | 1 | Ultrasonic | Acetone | 60 / 40 | 4.5-4.8 | Yes |
| 2311 | Yes | 1 | Ultrasonic | Acetone | 60 / 40 | 5.4 | Yes |
| 2330 | No | 0.5 | Ultrasonic | Acetone | 60 / 40 | 8.533, 8.553 | Yes |
| 2347 | Yes | 1.0 | Other | | | | Yes |
| 2350 | Yes | 1 | Ultrasonic | Isooctane | 60 / 60 | | --- |
| 2352 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.51 | Yes |
| 2357 | --- | | --- | | | | --- |
| 2358 | Yes | 1 | Ultrasonic | Acetone | 60 / 40 | | Yes |
| 2363 | Yes | 1 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.5 | Yes |
| 2365 | Yes | 0.6 | Soxhlet / AES | Methanol-Ethanol – 4:1 | 60 / 60 | 4.5 | Yes |
| 2366 | No | 1 | Ultrasonic | Acetone | 60 / 40 | 4.8 | Yes |
| 2369 | --- | | --- | | | | --- |
| 2370 | Yes | 1 | Ultrasonic | Ethanol | 60 / 20 | 4.5 | Yes |
| 2374 | No | 1 | Ultrasonic | Acetone | 60 / 40 | 4.9 | Yes |
| 2375 | Yes | 0,5 | Ultrasonic | Acetone | 60 / 40 | 5.8 | Yes |
| 2379 | Yes | 1.0 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | Yes |
| 2380 | Yes | 1.0 | Ultrasonic | Acetone | 60 / 40 | 4.6 | No |
| 2382 | Yes | 0.5 | Ultrasonic | Hexane | 60 / 40 | 4.5 | Yes |
| 2386 | Yes | 0,5 | Ultrasonic | Acetone | 30 / 40 | 4,5 | No |
| 2390 | Yes | 1.0 | Ultrasonic | Acetone | 60 / 40 | 4.6 | Yes |
| 2459 | Yes | 1.0 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | Yes |
| 2462 | --- | | --- | | | | --- |
| 2489 | Yes | 0.6 | Ultrasonic | Ethanol | 60 / 20 | 4.5 | Yes |
| 2492 | Yes | 0.3 | Ultrasonic | Ethanol/Acetic Acid (95/5) | 60 / 40 | | Yes |
| 2495 | --- | | --- | | | | --- |
| 2497 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | | Yes |
| 2508 | Yes | 0.5 | Ultrasonic | Ethanol/Acetic Acid (95/5) | 60 / 40 | | --- |
| 2515 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | Yes |
| 2522 | No | 1 | Ultrasonic | Hexane | 120 / 70 | 4.5 | Yes |
| 2532 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol | 60 / 60 | No adjusted | No |
| 2553 | Yes | 1 | Ultrasonic | Methanol-Ethanol 1:1 | 60 / 60 | 8.0 | No |
| 2560 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 20 | 5.3 | Yes |
| 2561 | Yes | 0.9488 | --- | Methanol-Ethanol (80:20) | 60 / 60 | | --- |
| 2590 | Yes | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | No |
| 2602 | Yes | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4,5 | No |
| 2644 | --- | | --- | | | | --- |
| 2665 | Yes | 0.5 | Other | Ethanol, Hexane | 1000 / 20 | 4 | No |
| 2671 | Yes | 1 | Ultrasonic | Isooctane | 60 / 60 | 4.5 | Yes |
| 2672 | Yes | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | | No |
| 2743 | Yes | 1 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.5 | Yes |
| 2758 | No | 0.5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | | No |
| 2791 | Yes | 1.0 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | Yes |
| 2812 | Yes | 1.0023 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.5 | Yes |
| 2826 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | Yes |
| 2864 | Yes | 0.5 | Soxhlet / AES | Acidic methanol (HCl) | 30 / 65 | 2.0 | No |

| lab | ISO/IEC 17025 accredited | Sample intake (in g) | Type of extraction | Solvent used to extract/release | Extraction time/temp (min/°C) | pH after adding the buffer | Extraction solution acidified until pH 4.5 |
|---|---|---|---|---|---|---|---|
| 2891 | Yes | 1 | Ultrasonic | Methanol-Ethanol – 4:1 | 60 / 60 | 4,5 | Yes |
| 2892 | Yes | 1.0 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | No |
| 2895 | --- | | --- | | | | --- |
| 3100 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | | --- |
| 3116 | Yes | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4.5 | No |
| 3146 | Yes | 0,5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4,5 | No |
| 3154 | Yes | 0,5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | | --- |
| 3160 | No | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 6.2 | No |
| 3172 | Yes | | --- | | | | --- |
| 3176 | Yes | 0,5 | Ultrasonic | Acidic methanol (HCl) | 30 / 20 | | Yes |
| 3190 | Yes | 2 | Ultrasonic | Ethanol | 120 / 20 | | Yes |
| 3197 | Yes | 2 | Ultrasonic | Ethanol | 120 / 22 | 4,5 | No |
| 3201 | Yes | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | not tested | No |
| 3210 | No | 1 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | 4,5 | No |
| 3214 | Yes | 1 | Ultrasonic | Methanol-Ethanol – 4:1 | 60 / 60 | 4.5 | Yes |
| 3218 | Yes | 0.5 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 4.5 | No |
| 3220 | Yes | 1 | Ultrasonic | Methanol-Ethanol-Isooctane | 60 / 60 | 5.75 | No |
| 3228 | Yes | 1 | Ultrasonic | Methanol-Ethanol | 60 / 60 | 5.8 | No |
| 3237 | Yes | 0,5 | Ultrasonic | Methanol-Ethanol (80:20) | 60 / 60 | - | No |
| 3243 | Yes | 1 | Ultrasonic | Ethanol | 60 / 60 | | No |
| 3246 | --- | | --- | | | | --- |
| 3248 | Yes | 0.5 | Ultrasonic | Methanol | 60 / 70 | 4.5 | Yes |

## APPENDIX 4

### Number of participants per country

2 labs in BANGLADESH
2 lab in BRAZIL
1 lab in CAMBODIA
2 labs in FRANCE
12 labs in GERMANY
1 lab in GUATEMALA
6 labs in HONG KONG
8 labs in INDIA
1 labs in INDONESIA
7 labs in ITALY
1 lab in MAURITIUS
1 lab in MOROCCO
20 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in SRI LANKA
1 lab in SWITZERLAND
3 labs in TAIWAN R.O.C.
1 lab in THAILAND
7 labs in TURKEY
1 lab in U.S.A.
1 lab in UNITED KINGDOM
6 labs in VIETNAM

**APPENDIX 5**

**Abbreviations**

| | |
|---|---|
| C | = final test result after checking of first reported suspect test result |
| D(0.01) | = outlier in Dixon's outlier test |
| D(0.05) | = straggler in Dixon's outlier test |
| G(0.01) | = outlier in Grubbs' outlier test |
| G(0.05) | = straggler in Grubbs' outlier test |
| DG(0.01) | = outlier in Double Grubbs' outlier test |
| DG(0.05) | = straggler in Double Grubbs' outlier test |
| R(0.01) | = outlier in Rosner's outlier test |
| R(0.05) | = straggler in Rosner's outlier test |
| E | = possibly an error in calculations |
| W | = test result withdrawn on request of participant |
| ex | = test result excluded from statistical evaluation |
| n.a. | = not applicable |
| n.e. | = not evaluated |
| n.d. | = not detected |
| fr. | = first reported |

**Literature**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