

# **Results of Proficiency Test**

## **Mono Ethylene Glycol (MEG)**

### **October 2012**

Organised by: Institute for Interlaboratory Studies (iis)  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 1994, the Institute for Interlaboratory Studies organised a proficiency test for the analysis of Mono Ethylene Glycol every year. As part of the annual proficiency test program of 2012/2013, the Institute decided to continue this proficiency test on Mono Ethylene Glycol. In this interlaboratory study 60 laboratories in 23 different countries have participated. See appendix 2 for the number of participants per country. In this report the results of the 2012 proficiency test are presented and discussed.

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. On request of several participants it was decided to send an Off-specification Mono Ethylene Glycol. This is in contrast to previous years in which a high purity MEG-fg was used each time. To get maximum information from this study it was decided to send 2 different samples:

|               | Bottle type              | Tests requested                       |
|---------------|--------------------------|---------------------------------------|
| Sample #12130 | 1.0 L amber glass bottle | for all regular determinations on MEG |
| Sample #12131 | 0.1 L amber glass bottle | for UV transmittance and Water only   |

table 1: type of samples

The participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

### 2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). 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 organisation 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 January 2010 (iis-protocol, version 3.2).

### 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

The necessary 80 litre bulk material of MEG polyester grade was obtained from a local production plant. The bulk material was transferred to a precleaned 200 litre drum and spiked with 0.095 mg/kg Chloride. After homogenisation, the batch was transferred to 80 amber glass bottles of 1.0 liter and labelled #12130. The homogeneity of the subsamples was checked by determination of Density in accordance with ASTM D4052:02e1, Chloride in accordance with ASTM E2469:08 and Iron in accordance with ASTM E394:09 on 8 stratified randomly selected samples.

|                 | Density @ 15°C<br>in kg/l | Chloride<br>in mg/kg | Iron<br>in mg/kg |
|-----------------|---------------------------|----------------------|------------------|
| Sample #12130-1 | 1.11662                   | 0.08                 | 0.32             |
| Sample #12130-2 | 1.11662                   | 0.09                 | 0.33             |
| Sample #12130-3 | 1.11662                   | 0.08                 | 0.33             |
| Sample #12130-4 | 1.11662                   | 0.08                 | 0.34             |
| Sample #12130-5 | 1.11662                   | 0.09                 | 0.32             |
| Sample #12130-6 | 1.11662                   | 0.09                 | 0.35             |
| Sample #12130-7 | 1.11662                   | 0.09                 | 0.33             |
| Sample #12130-8 | 1.11662                   | 0.09                 | 0.33             |

table 2: homogeneity test results of subsamples #12130

The second batch of approx. 30 litre was transferred to a precleaned can. After homogenisation, from the batch 83 amber glass bottles of 100mL were filled. The bottles were closed with special screw caps with Teflon inner layer, and labelled #12131. The homogeneity of the subsamples #12131 was checked by determination of UV-Transmittance without nitrogen sparging at 220, 250, 275 and 350 nm in accordance with ASTM E2193:08 and Water in accordance with ASTM E1064:12 on 8 stratified randomly selected samples.

|                 | UV(220nm)<br>in T% | UV(250nm)<br>in T% | UV(275nm)<br>in T% | UV(350nm)<br>in T% | Water<br>in mg/kg |
|-----------------|--------------------|--------------------|--------------------|--------------------|-------------------|
| Sample #12131-1 | 53.3               | 78.0               | 85.2               | 92.2               | 870               |
| Sample #12131-2 | 53.3               | 78.1               | 86.0               | 92.2               | 910               |
| Sample #12131-3 | 53.4               | 78.2               | 86.0               | 92.3               | 980               |
| Sample #12131-4 | 53.4               | 78.1               | 86.0               | 92.3               | 840               |
| Sample #12131-5 | 53.4               | 78.2               | 86.0               | 92.3               | 860               |
| Sample #12131-6 | 53.4               | 78.2               | 86.0               | 92.3               | 880               |
| Sample #12131-7 | 53.3               | 78.1               | 85.9               | 92.3               | 870               |
| Sample #12131-8 | 53.5               | 78.3               | 86.0               | 92.3               | 890               |

table 3: homogeneity test results of subsamples #12131

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods in agreement with the procedure of ISO 13528, Annex B2 in the next table:

|                               | Density | Cl    | Iron  | UV<br>220nm | UV<br>250nm | UV<br>275nm | UV<br>350nm | Water |
|-------------------------------|---------|-------|-------|-------------|-------------|-------------|-------------|-------|
| r (observed)                  | 0.00000 | 0.014 | 0.028 | 0.20        | 0.26        | 0.11        | 0.13        | 120   |
| 0.3xR <sub>(ASTM D4052)</sub> | 0.00015 | --    | --    | --          | --          | --          | --          | --    |
| 0.3xR <sub>(ASTM E2469)</sub> | --      | 0.028 | --    | --          | --          | --          | --          | --    |
| 0.3xR <sub>(ASTM E394)</sub>  | --      | --    | 0.054 | --          | --          | --          | --          | --    |
| 0.3xR <sub>(ASTM E2193)</sub> | --      | --    | --    | 2.90        | 0.62        | 0.33        | 0.28        | --    |
| 0.3xR <sub>(ASTM E1064)</sub> | --      | --    | --    | --          | --          | --          | --          | 220   |

table 4: homogeneity evaluation of subsamples #12130, #12131

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 2 bottles (1\*1.0L bottle labelled as #12130 and 1\*100 mL bottle, labelled as #12131), were sent on October 10, 2012.

## 2.5 STABILITY OF THE SAMPLES

The stability of the Mono Ethylene Glycol, packed in amber glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

## 2.6 ANALYSES

The participants were requested to determine Acidity as Acetic Acid, Aldehydes as Acetaldehyde, Appearance, Ash, Chloride as Cl, Colour (D5386), Colour Pt/Co (D1209) Density @ 20°C, Diethylene Glycol, Distillation (Initial Boiling Point, 50%recovered and Dry Point), Iron, Purity and Specific Gravity @ 20/20°C on sample #12130.

On sample #12131 was requested to determine UV Transmittance (at 350, 275, 250 and 220 nm) and Water.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website ([www.iisnl.com](http://www.iisnl.com)).

A SDS and a form to confirm receipt of the samples were added to the sample package.

## 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported.

Shortly after the deadline, the available results were screened for suspect data. A 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 (raw data of the) reported results.

Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the conclusions of statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

### 3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14).

### 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. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of

the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

$$z_{(\text{target})} = (\text{result} - \text{average of PT}) / \text{target standard deviation}$$

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

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 the fit-for-useness of the reported test result.

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

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

## 4 EVALUATION

In this interlaboratory study several problems were encountered with customs clearance or with the courier during dispatch of the samples to participants in Brazil, India and Saudi Arabia.

Six participants did not report any results and another twelve participants reported the results after the final reporting date. Finally, 54 laboratories did report 838 numerical results. Observed were 48 outlying results, which is 5.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

### 4.1 EVALUATION PER TEST

In this section, the results are discussed per sample and per test.

Not all data sets proved to have a normal distribution. Not normal distributions were found with the following determinations: Ash, Colour Pt/Co, Colour, Chloride, Density @15°C, Distillation (50% recovered), Purity and Specific Gravity. For these determinations the statistical evaluation should be used with due care.

Since 2010 a new version of ASTM E202 (and renewed in 2012) was published.

Regretfully, the new version is not always clear about the precision data in certain test methods, in those cases the precision data of ASTM E202: 2005 or specific test method was used.

Some of the used reference test methods provide precision data related to a specific concentration. For these tests, the target reproducibility is estimated.

- Acidity: This determination was problematic for a number of participants in accordance with ASTM E2679 and/or ASTM D1613. In total four statistical outliers were observed. Only the calculated reproducibility for the ASTM D1613 results after rejection of the statistical outliers is in full agreement with the requirements of the standard. The calculated reproducibility of the ASTM E2679:09 results after rejection of the statistical outlier is not in agreement with the requirements of the standard.
- Aldehydes: This determination may be problematic for a number of participants. Seven statistical outliers were observed. As the application range of ASTM E2313:08 is 0.5 – 50 mg/kg (see scope of test method) no significant conclusions were drawn. Four false negative test results and two false positive test results were observed.
- Appearance: No analytical problems were observed. Almost all participants agreed about the appearance of sample #12130, which was bright, clear and free of suspended matter. Only one participant reported “fail”.
- Ash: Only one statistical outlier (false positive result) was observed. Regretfully, the consensus value is below the application range (0.001 – 0.180 %M/M) of ASTM D482:07. Therefore no significant conclusions were drawn.
- Colour Pt/Co: The determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D1209:11.
- Colour D5386: The determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D5386:10.
- Chloride: This determination was very problematic. Two statistical outliers and one false negative test results were observed. The calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM E2469:08a. The average recovery of Chloride (theoretical increment of 0.095 mg Cl/kg) may be good: “less than 144%” (the actual blank Chloride content is unknown).
- Density: The determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers, is in agreement with the requirements of ASTM D4052:02e1.
- DEG: This determination was problematic at a low level of 52 mg/kg. Two statistical outliers were observed and the calculated reproducibility, after

rejection of the statistical outliers, is at all not in agreement the estimated requirements of ASTM E2409:08.

Distillation: The Initial Boiling Point was problematic. In total five statistical outliers were observed. The calculated reproducibilities after rejection of the statistical outliers are in good agreement with the requirements of ASTM D1078:11 for 50% recovered and Dry Point. The calculated reproducibility of the Initial Boiling Point does not at all meet the requirements of ASTM D1078:11.

Iron: This determination may not be problematic. Five statistical outliers and two false negatives test results were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated requirements of ASTM E1615:08 and ASTM D394:09.

Purity: Regretfully, no reproducibility data for purity are mentioned in ASTM E2409. Therefore no significant conclusions were drawn. However, the reported test results appear to be divided trimodally and the range of reported test results is large: from 99.08 – 99.974%M/M.. Obviously the variety of calculation methods for purity was used. Several laboratories subtracted only water, acidity and DEG (and aldehydes) from 100%, while other laboratories subtracted more items. This may be caused by the use of the ambiguous term “glycol impurity” in ASTM D2409 / E202.

Specific Gravity: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D4052:02e1.

UV: This determination was very problematic. In total eleven statistical outliers were observed. All calculated reproducibilities, after rejection of the statistical outliers, are not in agreement with the requirements of ASTM E2193:08 (unsparged and sparged), except for the calculated reproducibility for UV at 220 nm which is in agreement with the requirements of ASTM E2193:08 (sparged). Five participants reported to have used nitrogen to purge the sample prior to the measurement of UV and nine other participants reported to have used a 50mm cuvette prior to the measurement of UV.

Water: This determination was problematic for a number of participants. Seven statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM E1064:12.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM standards) are compared in the next table:

| Parameter                      | unit  | n  | average | $2.8 * s_{dR}$ | R (lit.) |
|--------------------------------|-------|----|---------|----------------|----------|
| Acidity as Acetic Acid (E2679) | mg/kg | 12 | 24.8    | 14.2           | 12.6     |
| Acidity as Acetic Acid (D1613) | mg/kg | 42 | 28.2    | 12.7           | 14.0     |
| Aldehydes as Acetaldehyde      | mg/kg | 23 | 247     | 79             | (212)    |
| Appearance                     |       | 49 | pass    | --             | --       |
| Ash                            | %M/M  | 26 | 0.0005  | 0.0011         | (0.0050) |
| Colour Pt/Co                   | ---   | 39 | 4.8     | 5.6            | 7.0      |
| Colour ASTM D5386              | ---   | 30 | 5.6     | 4.8            | 5.5      |
| Chloride as Cl                 | mg/kg | 25 | 0.14    | 0.25           | 0.11     |
| Density at 20°C                | kg/L  | 48 | 1.1132  | 0.0004         | 0.0005   |
| Diethylene Glycol              | mg/kg | 40 | 51.6    | 32.8           | 13.1     |
| Initial Boiling Point          | °C    | 37 | 193.4   | 5.6            | 3.0      |
| 50% recovered                  | °C    | 37 | 197.6   | 0.7            | 1.4      |
| Dry Point                      | °C    | 39 | 198.1   | 1.2            | 2.1      |
| Iron as Fe                     | mg/kg | 37 | 0.28    | 0.07           | 0.30     |
| Purity                         | %M/M  | 43 | 99.598  | 0.694          | unknown  |
| Specific Gravity 20/20°C       | ---   | 48 | 1.1152  | 0.0004         | 0.0005   |
| UV Transmittance at 350 nm     | %T    | 46 | 93.0    | 2.3            | 1.2      |
| UV Transmittance at 275 nm     | %T    | 45 | 86.6    | 3.7            | 2.1      |
| UV Transmittance at 250 nm     | %T    | 43 | 79.2    | 4.2            | 1.1      |
| UV Transmittance at 220 nm     | %T    | 42 | 54.2    | 4.9            | 4.1      |
| Water                          | mg/kg | 42 | 884     | 149            | 151      |

table 5: reproducibilities of samples #12130 and #12131

Results between brackets were below the application range of the method, therefore results should be evaluated with care

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2012 WITH PREVIOUS PTS

|                            | October 2012 | October 2011 | October 2010 | October 2009 |
|----------------------------|--------------|--------------|--------------|--------------|
| Number of reporting labs   | 54           | 63           | 62           | 56           |
| Number of results reported | 838          | 927          | 907          | 763          |
| Statistical outliers       | 48           | 42           | 36           | 36           |
| Percentage outliers        | 5.7%         | 4.5%         | 4.0%         | 4.7%         |

table 6: comparison of statistical summary parameters with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given in the following table:

|                            | October 2012 | October 2011 | October 2010 | October 2009 |
|----------------------------|--------------|--------------|--------------|--------------|
| Acidity as Acetic Acid     | - +          | --           | ++           | ++           |
| Aldehydes as Acetaldehyde  | (++)         | ++           | ++           | ++           |
| Ash                        | (++)         | (++)         | (++)         | (++)         |
| Colour Pt/Co               | ++           | -            | ++           | ++           |
| Colour ASTM D5368          | +            | --           | ++           | ++           |
| Chloride as Cl             | --           | +/-          | --           | ++           |
| Density at 20°C            | +            | ++           | ++           | ++           |
| Diethylene Glycol          | --           | +/-          | --           | --           |
| Initial Boiling Point      | --           | ++           | ++           | ++           |
| 50% recovered              | ++           | ++           | ++           | ++           |
| Dry Point                  | ++           | ++           | ++           | ++           |
| Iron as Fe                 | ++           | ++           | ++           | ++           |
| Purity                     | --           | ++           | ++           | ++           |
| Specific Gravity 20/20°C   | +            | ++           | ++           | ++           |
| UV Transmittance at 350 nm | --           | --           | ++           | ++           |
| UV Transmittance at 275 nm | --           | ++           | ++           | ++           |
| UV Transmittance at 250 nm | --           | --           | --           | --           |
| UV Transmittance at 220 nm | -            | ++           | +            | +/-          |
| Water                      | +/-          | ++           | ++           | +            |

table 7: comparison of determinations against the standard

Results between brackets were below the application range of the method, therefore results should be evaluated with care

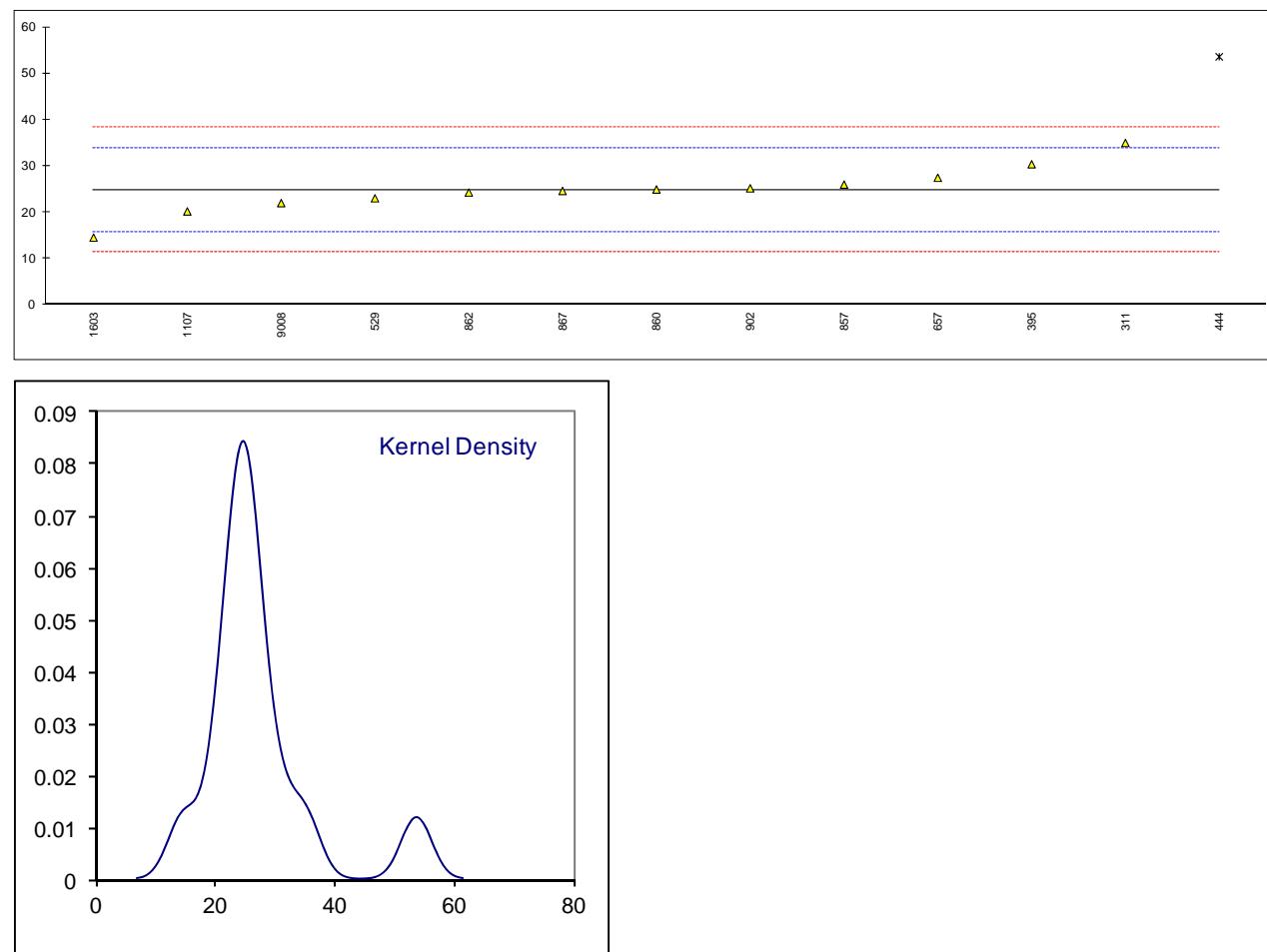
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard

**APPENDIX 1**

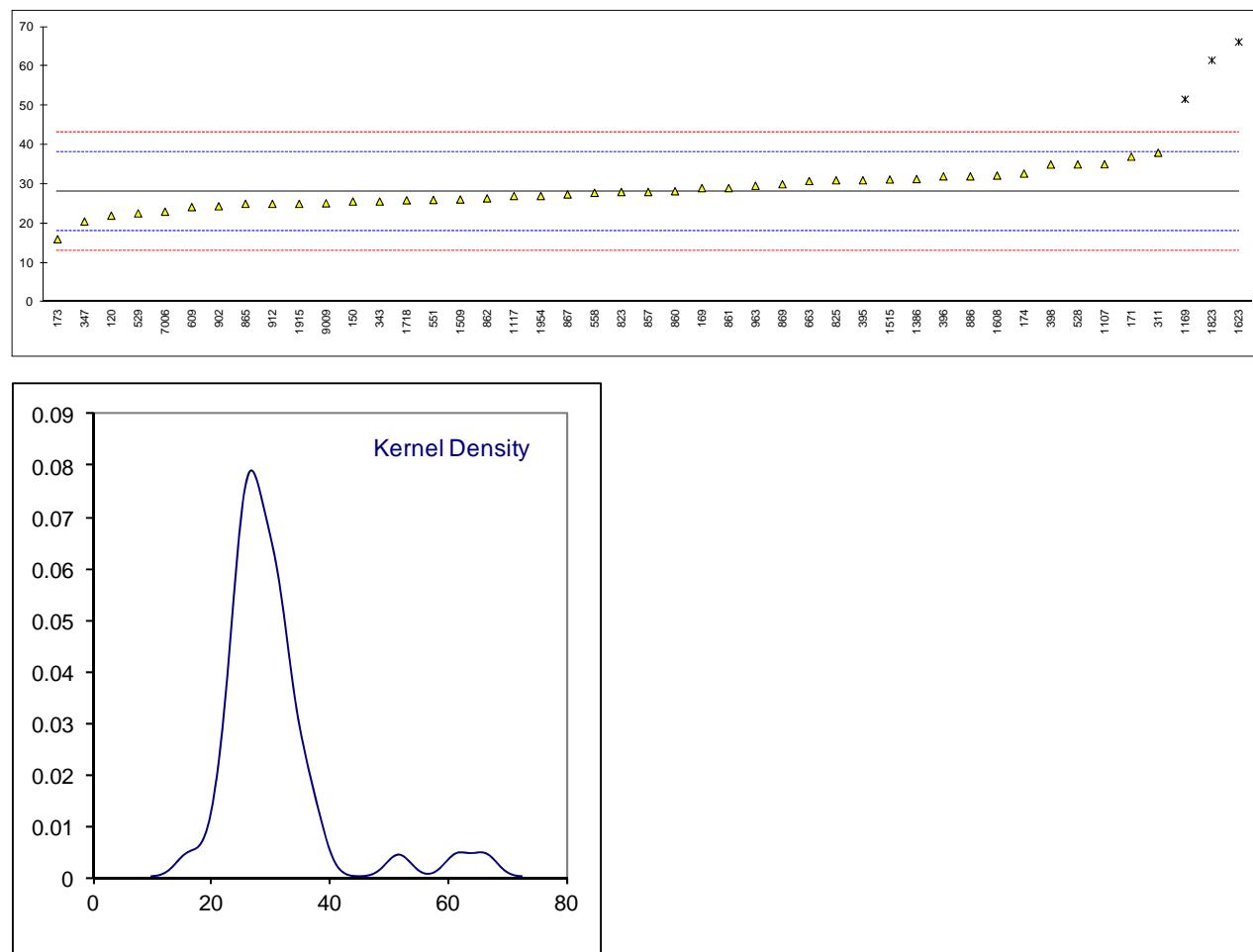
Determination of Acidity as Acetic Acid (E2679) on sample #12130; results in mg/kg

| lab  | method      | value  | mark      | z(targ) | remarks             |
|------|-------------|--------|-----------|---------|---------------------|
| 120  |             | ----   |           | ----    |                     |
| 150  |             | ----   |           | ----    |                     |
| 169  |             | ----   |           | ----    |                     |
| 171  |             | ----   |           | ----    |                     |
| 173  |             | ----   |           | ----    |                     |
| 174  |             | ----   |           | ----    |                     |
| 311  | E2679       | 35.0   |           | 2.27    |                     |
| 322  |             | ----   |           | ----    |                     |
| 323  |             | ----   |           | ----    |                     |
| 343  |             | ----   |           | ----    |                     |
| 347  |             | ----   |           | ----    |                     |
| 395  | E2679       | 30.4   |           | 1.25    |                     |
| 396  |             | ----   |           | ----    |                     |
| 398  |             | ----   |           | ----    |                     |
| 444  | E2679       | 53.6   | C,G(0.01) | 6.42    | First reported 1.3  |
| 528  |             | ----   |           | ----    |                     |
| 529  | E2679       | 23.053 |           | -0.39   |                     |
| 551  |             | ----   |           | ----    |                     |
| 557  |             | ----   |           | ----    |                     |
| 558  |             | ----   |           | ----    |                     |
| 609  |             | ----   |           | ----    |                     |
| 657  | E2679       | 27.48  | C         | 0.59    | First reported 31.5 |
| 663  |             | ----   |           | ----    |                     |
| 823  |             | ----   |           | ----    |                     |
| 825  |             | ----   |           | ----    |                     |
| 857  | E2679       | 26.01  |           | 0.27    |                     |
| 860  | E2679       | 24.94  |           | 0.03    |                     |
| 861  |             | ----   |           | ----    |                     |
| 862  | E2679       | 24.3   |           | -0.11   |                     |
| 865  |             | ----   |           | ----    |                     |
| 867  | E2679       | 24.63  |           | -0.04   |                     |
| 869  |             | ----   |           | ----    |                     |
| 886  |             | ----   |           | ----    |                     |
| 902  | E2679       | 25.2   |           | 0.09    |                     |
| 912  |             | ----   |           | ----    |                     |
| 913  |             | ----   |           | ----    |                     |
| 962  |             | ----   |           | ----    |                     |
| 963  |             | ----   |           | ----    |                     |
| 1107 | E2679       | 20.2   |           | -1.03   |                     |
| 1117 |             | ----   |           | ----    |                     |
| 1151 |             | ----   |           | ----    |                     |
| 1169 |             | ----   |           | ----    |                     |
| 1217 |             | ----   |           | ----    |                     |
| 1386 |             | ----   |           | ----    |                     |
| 1467 |             | ----   |           | ----    |                     |
| 1492 |             | ----   |           | ----    |                     |
| 1509 |             | ----   |           | ----    |                     |
| 1515 |             | ----   |           | ----    |                     |
| 1603 | in house    | 14.56  |           | -2.29   |                     |
| 1608 |             | ----   |           | ----    |                     |
| 1623 |             | ----   |           | ----    |                     |
| 1701 |             | ----   |           | ----    |                     |
| 1718 |             | ----   |           | ----    |                     |
| 1823 |             | ----   |           | ----    |                     |
| 1866 |             | ----   |           | ----    |                     |
| 1915 |             | ----   |           | ----    |                     |
| 1954 |             | ----   |           | ----    |                     |
| 7006 |             | ----   |           | ----    |                     |
| 9008 | E2679       | 22     |           | -0.63   |                     |
| 9009 |             | ----   |           | ----    |                     |
|      | normality   | OK     |           |         |                     |
|      | n           | 12     |           |         |                     |
|      | outliers    | 1      |           |         |                     |
|      | mean (n)    | 24.81  |           |         |                     |
|      | st.dev. (n) | 5.058  |           |         |                     |
|      | R(calc.)    | 14.16  |           |         |                     |
|      | R(E2679:09) | 12.56  |           |         |                     |



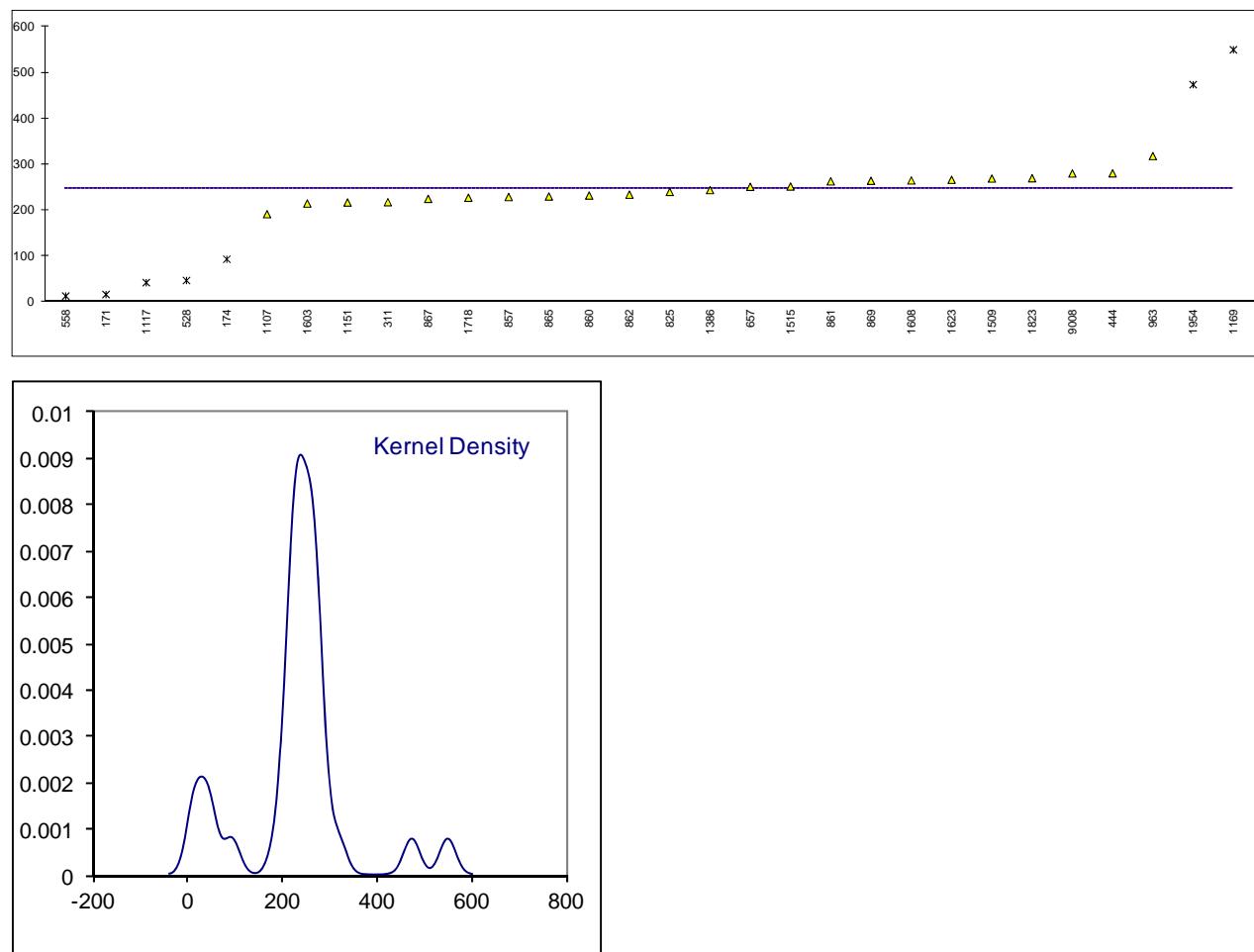
## Determination of Acidity as Acetic Acid (D1613) on sample #12130; results in mg/kg

| lab  | method      | value | mark      | z(targ) | remarks   |
|------|-------------|-------|-----------|---------|---|
| 120  | D1613       | 22    |           | -1.24   |   |
| 150  | D1613       | 25.6  |           | -0.52   |   |
| 169  | D1613       | 29    |           | 0.16    |   |
| 171  | D1613       | 37    |           | 1.76    |   |
| 173  | D1613       | 16    | C         | -2.44   | Reported 0.0016 (unit error, %M/M instead of mg/kg) |
| 174  | D1613       | 32.70 | C         | 0.90    | First reported 46                                   |
| 311  | D1613       | 38    |           | 1.96    |   |
| 322  |             | ----  |           | ----    |   |
| 323  |             | ----  |           | ----    |   |
| 343  | D1613       | 25.6  |           | -0.52   |   |
| 347  | D1613       | 20.51 |           | -1.53   |   |
| 395  | D1613       | 31.0  |           | 0.56    |   |
| 396  | D1613       | 32    |           | 0.76    |   |
| 398  | D1613       | 35    |           | 1.36    |   |
| 444  |             | ----  |           | ----    |   |
| 528  | D1613       | 35.03 |           | 1.37    |   |
| 529  | D1613       | 22.59 |           | -1.12   |   |
| 551  | D1613       | 26    |           | -0.44   |   |
| 557  |             | ----  |           | ----    |   |
| 558  | D1613       | 27.8  |           | -0.08   |   |
| 609  | D1613       | 24.17 |           | -0.80   |   |
| 657  |             | ----  |           | ----    |   |
| 663  | D1613       | 30.8  |           | 0.52    |   |
| 823  | D1613       | 28    |           | -0.04   |   |
| 825  | D1613       | 31    |           | 0.56    |   |
| 857  | D1613       | 28.0  |           | -0.04   |   |
| 860  | D1613       | 28.2  |           | 0.00    |   |
| 861  | D1613       | 29.0  |           | 0.16    |   |
| 862  | D1613       | 26.4  |           | -0.36   |   |
| 865  | D1613       | 25.0  |           | -0.64   |   |
| 867  | D1613       | 27.4  |           | -0.16   |   |
| 869  | D1613       | 30.0  |           | 0.36    |   |
| 886  | D1613       | 32    |           | 0.76    |   |
| 902  | D1613       | 24.4  |           | -0.76   |   |
| 912  | D1613       | 25    |           | -0.64   |   |
| 913  |             | ----  |           | ----    |   |
| 962  |             | ----  |           | ----    |   |
| 963  | D1613       | 29.6  |           | 0.28    |   |
| 1107 | D1613       | 35.07 |           | 1.38    |   |
| 1117 | D1613       | 27.0  |           | -0.24   |   |
| 1151 |             | ----  |           | ----    |   |
| 1169 | D1613       | 51.6  | C,G(0.01) | 4.68    | First reported 42.93                                |
| 1217 |             | ----  |           | ----    |   |
| 1386 | D1613       | 31.3  |           | 0.62    |   |
| 1467 |             | ----  |           | ----    |   |
| 1492 |             | ----  |           | ----    |   |
| 1509 | D1613       | 26.1  | C         | -0.42   | First reported 14.6                                 |
| 1515 | D1613       | 31.2  | C         | 0.60    | First reported 3.12                                 |
| 1603 |             | ----  |           | ----    |   |
| 1608 | D1613       | 32.2  |           | 0.80    |   |
| 1623 | D1613       | 66.18 | C,G(0.01) | 7.60    | First reported 59.33                                |
| 1701 |             | ----  |           | ----    |   |
| 1718 | D1613       | 25.90 |           | -0.46   |   |
| 1823 | D1613       | 61.5  | G(0.01)   | 6.66    |   |
| 1866 |             | ----  |           | ----    |   |
| 1915 | D1613       | 25    |           | -0.64   |   |
| 1954 | D1613       | 27    |           | -0.24   |   |
| 7006 | D1613       | 23    |           | -1.04   |   |
| 9008 |             | ----  |           | ----    |   |
| 9009 | D1613       | 25.15 |           | -0.61   |   |
|      | normality   | OK    |           |         |   |
|      | n           | 42    |           |         |   |
|      | outliers    | 3     |           |         |   |
|      | mean (n)    | 28.18 |           |         |   |
|      | st.dev. (n) | 4.518 |           |         |   |
|      | R(calc.)    | 12.65 |           |         |   |
|      | R(D1613:12) | 14.00 |           |         |   |



## Determination of Aldehydes as Acetaldehyde on sample #12130; results in mg/kg

| lab  | method      | value    | mark     | z(targ) | remarks                           |
|------|-------------|----------|----------|---------|-----------------------------------|
| 120  |             | ----     |          | ----    |                                   |
| 150  |             | ----     |          | ----    |                                   |
| 169  |             | ----     |          | ----    |                                   |
| 171  | E2313       | 16       | DG(0.05) | ----    | False negative result?            |
| 173  |             | ----     |          | ----    |                                   |
| 174  | E2313       | 93       | G(0.05)  | ----    |                                   |
| 311  | E2313       | 217.5    |          | ----    |                                   |
| 322  | E2313       | >50      |          | ----    |                                   |
| 323  | E2313       | >50      |          | ----    |                                   |
| 343  |             | ----     |          | ----    |                                   |
| 347  |             | ----     |          | ----    |                                   |
| 395  | E2313       | >50      |          | ----    |                                   |
| 396  |             | ----     |          | ----    |                                   |
| 398  | E2313       | >50      |          | ----    |                                   |
| 444  | E2313       | 280.35   |          | ----    |                                   |
| 528  | INH-86      | 46.635   | DG(0.05) | ----    | False negative result?            |
| 529  |             | ----     |          | ----    |                                   |
| 551  |             | ----     |          | ----    |                                   |
| 557  |             | ----     |          | ----    |                                   |
| 558  | NBR-5769    | 12.6     | DG(0.05) | ----    | False negative result?            |
| 609  |             | ----     |          | ----    |                                   |
| 657  | E2313       | 251      |          | ----    |                                   |
| 663  |             | ----     |          | ----    |                                   |
| 823  |             | ----     |          | ----    |                                   |
| 825  | E2313       | 240      |          | ----    |                                   |
| 857  | E2313       | 228.8    |          | ----    |                                   |
| 860  | E2313       | 232      |          | ----    |                                   |
| 861  | E2313       | 263.0    |          | ----    |                                   |
| 862  | E2313       | 234      |          | ----    |                                   |
| 865  | E2313       | 230.0    |          | ----    |                                   |
| 867  | E2313       | 224.7    |          | ----    |                                   |
| 869  | E2313       | 264.1    |          | ----    |                                   |
| 886  |             | ----     |          | ----    |                                   |
| 902  | E2313       | >50      |          | ----    |                                   |
| 912  |             | ----     |          | ----    |                                   |
| 913  |             | ----     |          | ----    |                                   |
| 962  |             | ----     |          | ----    |                                   |
| 963  | E2313       | 318.2    |          | ----    |                                   |
| 1107 | E2313       | 191.4    |          | ----    |                                   |
| 1117 | INH-2812    | 41.9     | DG(0.05) | ----    | False negative result?            |
| 1151 | E2313       | 216.9    |          | ----    |                                   |
| 1169 | E2313       | 550.07   | DG(0.05) | ----    | False positive result?            |
| 1217 |             | ----     |          | ----    |                                   |
| 1386 | E2313       | 243.89   |          | ----    |                                   |
| 1467 |             | ----     |          | ----    |                                   |
| 1492 |             | ----     |          | ----    |                                   |
| 1509 | E2313       | 269.17   |          | ----    |                                   |
| 1515 | E2313       | 251.76   |          | ----    |                                   |
| 1603 | in house    | 214.6    |          | ----    |                                   |
| 1608 | E2313       | 265      |          | ----    |                                   |
| 1623 | INH-012     | 266.36   |          | ----    |                                   |
| 1701 |             | ----     |          | ----    |                                   |
| 1718 | E2313       | 227.21   |          | ----    |                                   |
| 1823 | E2313       | 269.8    |          | ----    |                                   |
| 1866 |             | ----     |          | ----    |                                   |
| 1915 |             | ----     |          | ----    |                                   |
| 1954 | E2313       | 474      | DG(0.05) | ----    | False positive result?            |
| 7006 |             | ----     |          | ----    |                                   |
| 9008 | E2313       | 280.2    |          | ----    |                                   |
| 9009 |             | ----     |          | ----    |                                   |
|      | normality   | OK       |          |         |                                   |
|      | n           | 23       |          |         |                                   |
|      | outliers    | 7        |          |         |                                   |
|      | mean (n)    | 246.95   |          |         |                                   |
|      | st.dev. (n) | 28.172   |          |         |                                   |
|      | R(calc.)    | 78.88    |          |         |                                   |
|      | R(E2313:08) | (212.43) |          |         | Application range: 0.5 – 50 mg/kg |



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## Determination of Appearance on sample #12130;

| lab  | method        | value   | mark | z(targ) | remarks         |
|------|---------------|---------|------|---------|-----------------|
| 120  | E2680         | Fail    |      | ----    |                 |
| 150  | E2680         | Pass    |      | ----    |                 |
| 169  | E2680         | C&B     |      | ----    |                 |
| 171  | E2680         | C&F     |      | ----    |                 |
| 173  | E2680         | CFSM    |      | ----    |                 |
| 174  | E2680         | Pass    |      | ----    |                 |
| 311  | E2680         | Pass    |      | ----    |                 |
| 322  | E2680         | Pass    |      | ----    |                 |
| 323  | E2680         | Pass    |      | ----    |                 |
| 343  | E2680         | Pass    |      | ----    |                 |
| 347  | E2680         | Pass    |      | ----    |                 |
| 395  | E2680         | Pass    |      | ----    |                 |
| 396  | E2680         | Pass    |      | ----    |                 |
| 398  | E2680         | Pass    |      | ----    |                 |
| 444  | E2680         | Pass    |      | ----    |                 |
| 528  | E2680         | CFSM    |      | ----    |                 |
| 529  | E2680         | Pass    |      | ----    |                 |
| 551  | E2680         | Pass    |      | ----    |                 |
| 557  |               | ----    |      | ----    |                 |
| 558  | E2680         | Pass    |      | ----    |                 |
| 609  | E2680         | Pass    |      | ----    |                 |
| 657  | E2680         | Pass    |      | ----    |                 |
| 663  | E2680         | Pass    |      | ----    |                 |
| 823  | E2680         | Pass    |      | ----    |                 |
| 825  | E2680         | Pass    |      | ----    |                 |
| 857  | E2680         | Pass    |      | ----    |                 |
| 860  | E2680         | Pass    |      | ----    |                 |
| 861  | E2680         | Pass    |      | ----    |                 |
| 862  | E2680         | Pass    |      | ----    |                 |
| 865  | E2680         | Pass    |      | ----    |                 |
| 867  | E2680         | Pass    |      | ----    |                 |
| 869  | E2680         | Pass    |      | ----    |                 |
| 886  | E2680         | Pass    |      | ----    |                 |
| 902  | E2680         | Pass    |      | ----    |                 |
| 912  | E2680         | Pass    |      | ----    |                 |
| 913  | E2680         | CFFSM   |      | ----    |                 |
| 962  |               | ----    |      | ----    |                 |
| 963  | E2680         | Pass    |      | ----    |                 |
| 1107 | Visual        | C&B     |      | ----    |                 |
| 1117 | D4176         | On-spec |      | ----    |                 |
| 1151 |               | -----   |      | -----   |                 |
| 1169 | D4176         | Pass    |      | ----    |                 |
| 1217 |               | -----   |      | -----   |                 |
| 1386 | E2680         | CFFSM   |      | ----    |                 |
| 1467 |               | -----   |      | -----   |                 |
| 1492 |               | -----   |      | -----   |                 |
| 1509 | D4176         | CFFSM   |      | ----    |                 |
| 1515 | D4176         | Pass    |      | ----    |                 |
| 1603 | in house      | CFP     |      | ----    |                 |
| 1608 | D4176         | Pass    |      | ----    |                 |
| 1623 | D2090         | Clear   |      | ----    |                 |
| 1701 |               | -----   |      | -----   |                 |
| 1718 | D4176         | CFFSM   |      | ----    |                 |
| 1823 | E2680         | Pass    |      | ----    |                 |
| 1866 |               | -----   |      | -----   |                 |
| 1915 | E2680         | C&B     |      | ----    |                 |
| 1954 | Visual        | Clear   |      | ----    |                 |
| 7006 |               | -----   |      | -----   |                 |
| 9008 | E2680         | Pass    |      | ----    |                 |
| 9009 |               | -----   |      | -----   |                 |
|      | normality     | n.a.    |      |         |                 |
|      | n             | 49      |      |         |                 |
|      | outliers      | 1       |      |         |                 |
|      | mean (n)      | Pass    |      |         | 1 reported fail |
|      | st.dev. (n)   | n.a.    |      |         |                 |
|      | R(calc.)      | n.a.    |      |         |                 |
|      | R(E2680:09e1) | n.a.    |      |         |                 |

B&amp;C = Bright and clear

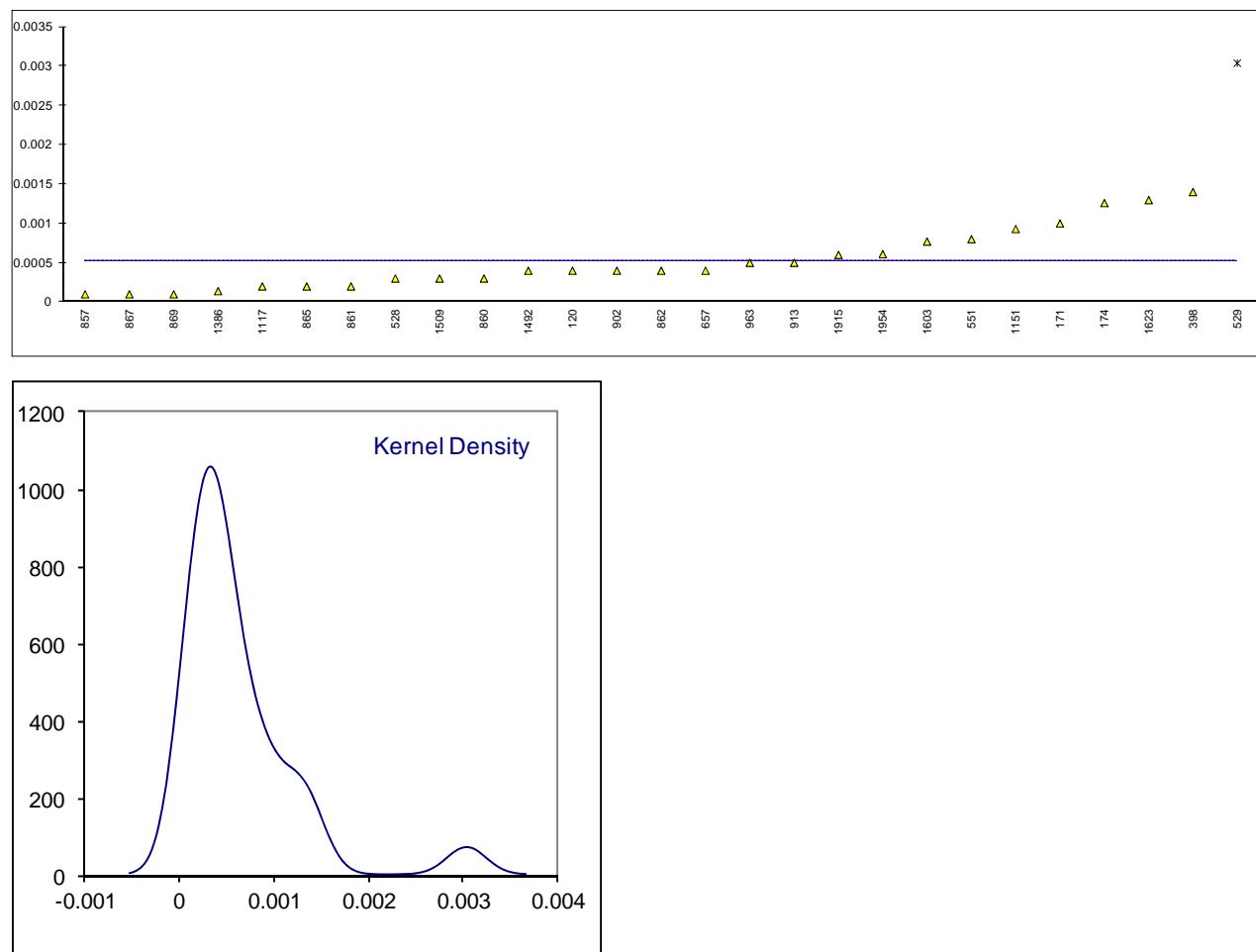
C&amp;F = Clear and Free

CFFSM = Clear and free from suspended matter

SF = Suspended Free

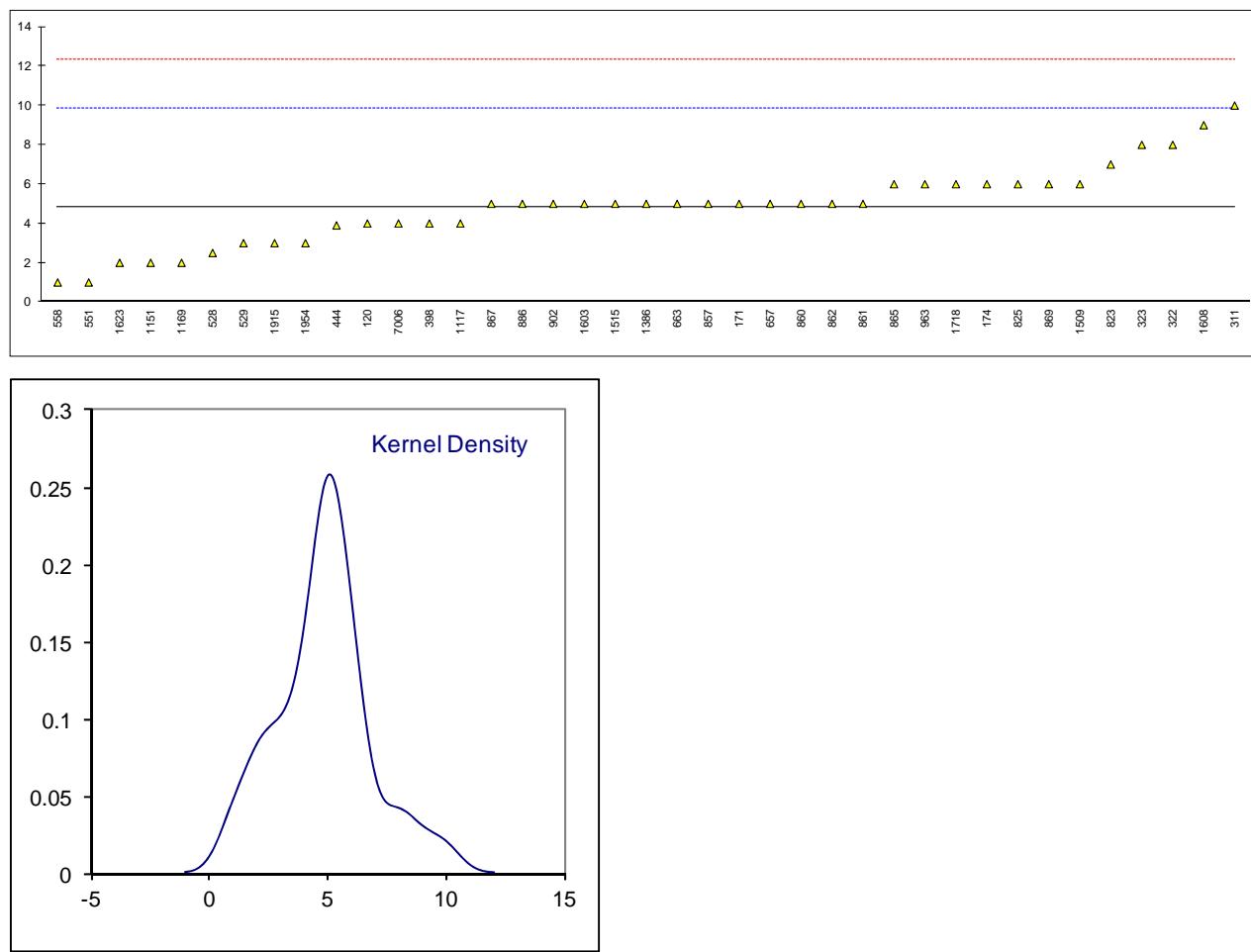
## Determination of Ash on sample #12130; results in %M/M

| lab  | method      | value     | mark    | z(targ) | remarks                              |
|------|-------------|-----------|---------|---------|--------------------------------------|
| 120  | D482        | 0.0004    | ----    |         |                                      |
| 150  | D482        | <0.001    | ----    |         |                                      |
| 169  | D482        | <0.001    | ----    |         |                                      |
| 171  | D482        | 0.001     | ----    |         |                                      |
| 173  | D482        | <0.0001   | ----    |         |                                      |
| 174  | D482        | 0.00126   | ----    |         |                                      |
| 311  | D482        | <0.01     | ----    |         |                                      |
| 322  |             | ----      |         |         |                                      |
| 323  | D482        | <0.001    | ----    |         |                                      |
| 343  | D482        | <0.001    | ----    |         |                                      |
| 347  |             | ----      |         |         |                                      |
| 395  |             | ----      |         |         |                                      |
| 396  |             | ----      |         |         |                                      |
| 398  | D482        | 0.0014    | ----    |         |                                      |
| 444  |             | ----      |         |         |                                      |
| 528  | D482        | 0.0003    | ----    |         |                                      |
| 529  | D482        | 0.003039  | G(0.01) |         | False positive result?               |
| 551  | D482        | 0.0008    | ----    |         |                                      |
| 557  |             | ----      |         |         |                                      |
| 558  |             | ----      |         |         |                                      |
| 609  |             | ----      |         |         |                                      |
| 657  | D482        | 0.0004    | ----    |         |                                      |
| 663  | D482        | <0.001    | ----    |         |                                      |
| 823  |             | ----      |         |         |                                      |
| 825  |             | ----      |         |         |                                      |
| 857  | D482        | 0.0001    | ----    |         |                                      |
| 860  | D482        | 0.0003    | ----    |         |                                      |
| 861  | D482        | 0.0002    | ----    |         |                                      |
| 862  | D482        | 0.0004    | ----    |         |                                      |
| 865  | D482        | 0.0002    | ----    |         |                                      |
| 867  | D482        | 0.0001    | ----    |         |                                      |
| 869  | D482        | 0.0001    | ----    |         |                                      |
| 886  | D482        | <0.001    | ----    |         |                                      |
| 902  | D482        | 0.0004    | ----    |         |                                      |
| 912  | D482        | <0.001    | ----    |         |                                      |
| 913  | D482        | 0.00050   | ----    |         |                                      |
| 962  |             | ----      |         |         |                                      |
| 963  | D482        | 0.0005    | ----    |         |                                      |
| 1107 | D482        | <0.001    | ----    |         |                                      |
| 1117 | D482        | 0.0002    | ----    |         |                                      |
| 1151 | D482        | 0.00093   | ----    |         |                                      |
| 1169 |             | ----      |         |         |                                      |
| 1217 |             | ----      |         |         |                                      |
| 1386 | D482        | 0.00014   | ----    |         |                                      |
| 1467 |             | ----      |         |         |                                      |
| 1492 | D482        | 0.0004    | ----    |         |                                      |
| 1509 | D482        | 0.0003    | ----    |         |                                      |
| 1515 |             | ----      |         |         |                                      |
| 1603 | in house    | 0.00077   | ----    |         |                                      |
| 1608 | D482        | <0.001    | ----    |         |                                      |
| 1623 | D482        | 0.001297  | ----    |         |                                      |
| 1701 |             | ----      |         |         |                                      |
| 1718 | D482        | <0.001    | ----    |         |                                      |
| 1823 | D482        | <0.0010   | ----    |         |                                      |
| 1866 |             | ----      |         |         |                                      |
| 1915 | D482        | 0.0006    | ----    |         |                                      |
| 1954 | D482        | 0.00061   | ----    |         |                                      |
| 7006 |             | ----      |         |         |                                      |
| 9008 |             | ----      |         |         |                                      |
| 9009 |             | ----      |         |         |                                      |
|      | normality   | not OK    |         |         |                                      |
|      | n           | 26        |         |         |                                      |
|      | outliers    | 1         |         |         |                                      |
|      | mean (n)    | 0.00052   |         |         |                                      |
|      | st.dev. (n) | 0.000384  |         |         |                                      |
|      | R(calc.)    | 0.00107   |         |         |                                      |
|      | R(D482:07)  | (0.00500) |         |         | Application range 0.001 – 0.180 %M/M |



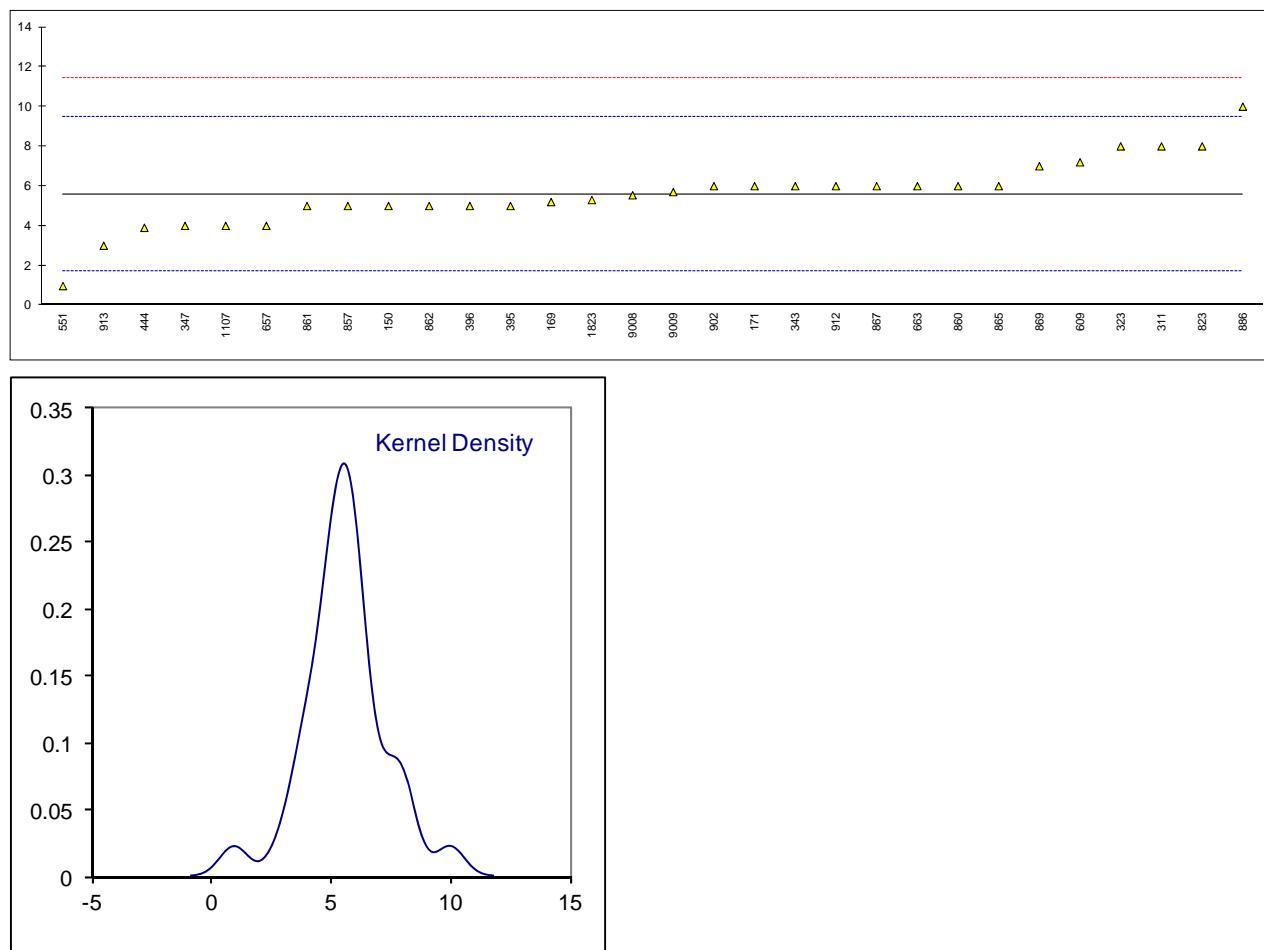
## Determination of Colour Pt/Co on sample #12130;

| lab  | method      | value  | mark | z(targ) | remarks           |
|------|-------------|--------|------|---------|-------------------|
| 120  | D1209       | 4      |      | -0.33   |                   |
| 150  |             | ----   |      | ----    |                   |
| 169  |             | ----   |      | ----    |                   |
| 171  | D1209       | 5      |      | 0.07    |                   |
| 173  | D1209       | <5     |      | ----    |                   |
| 174  | D1209       | 6      |      | 0.47    |                   |
| 311  | D1209       | 10     |      | 2.07    |                   |
| 322  | D1209       | 8      |      | 1.27    |                   |
| 323  | D1209       | 8      |      | 1.27    |                   |
| 343  |             | ----   |      | ----    |                   |
| 347  |             | ----   |      | ----    |                   |
| 395  |             | ----   |      | ----    |                   |
| 396  |             | ----   |      | ----    |                   |
| 398  | D1209       | 4      |      | -0.33   |                   |
| 444  | D5386       | 3.9    |      | -0.37   |                   |
| 528  | D1209       | 2.5    |      | -0.93   |                   |
| 529  | D1209       | 3      |      | -0.73   |                   |
| 551  | D1209       | 1      |      | -1.53   |                   |
| 557  |             | ----   |      | ----    |                   |
| 558  | NBR-5769    | 1      |      | -1.53   |                   |
| 609  |             | ----   |      | ----    |                   |
| 657  | D1209       | 5      |      | 0.07    |                   |
| 663  | D1209       | 5      |      | 0.07    |                   |
| 823  | D1209       | 7      |      | 0.87    |                   |
| 825  | D1209       | 6      |      | 0.47    |                   |
| 857  | D1209       | 5      |      | 0.07    |                   |
| 860  | D1209       | 5      |      | 0.07    |                   |
| 861  | D1209       | 5      |      | 0.07    |                   |
| 862  | D1209       | 5      |      | 0.07    |                   |
| 865  | D1209       | 6      |      | 0.47    |                   |
| 867  | D1209       | 5      |      | 0.07    |                   |
| 869  | D1209       | 6      |      | 0.47    |                   |
| 886  | D1209       | 5      | C    | 0.07    | First reported 10 |
| 902  | D1209       | 5      |      | 0.07    |                   |
| 912  |             | ----   |      | ----    |                   |
| 913  |             | ----   |      | ----    |                   |
| 962  |             | ----   |      | ----    |                   |
| 963  | D1209       | 6      |      | 0.47    |                   |
| 1107 | D1209       | <5     |      | ----    |                   |
| 1117 | D1209       | 4      |      | -0.33   |                   |
| 1151 | D1209       | 2      |      | -1.13   |                   |
| 1169 | D1209       | 2      |      | -1.13   |                   |
| 1217 |             | ----   |      | ----    |                   |
| 1386 | D1209       | 5      |      | 0.07    |                   |
| 1467 |             | ----   |      | ----    |                   |
| 1492 |             | ----   |      | ----    |                   |
| 1509 | D1209       | 6      |      | 0.47    |                   |
| 1515 | D1209       | 5      |      | 0.07    |                   |
| 1603 | in house    | 5      |      | 0.07    |                   |
| 1608 | D1209       | 9      |      | 1.67    |                   |
| 1623 | D1209       | 2.0    |      | -1.13   |                   |
| 1701 |             | ----   |      | ----    |                   |
| 1718 | D1209       | 6      |      | 0.47    |                   |
| 1823 |             | ----   |      | ----    |                   |
| 1866 |             | ----   |      | ----    |                   |
| 1915 | D1209       | 3      |      | -0.73   |                   |
| 1954 | D1209       | 3      |      | -0.73   |                   |
| 7006 | D1209       | 4      |      | -0.33   |                   |
| 9008 |             | ----   |      | ----    |                   |
| 9009 |             | ----   |      | ----    |                   |
|      | normality   | not OK |      |         |                   |
|      | n           | 39     |      |         |                   |
|      | outliers    | 0      |      |         |                   |
|      | mean (n)    | 4.8    |      |         |                   |
|      | st.dev. (n) | 2.00   |      |         |                   |
|      | R(calc.)    | 5.6    |      |         |                   |
|      | R(D1209:11) | 7.0    |      |         |                   |



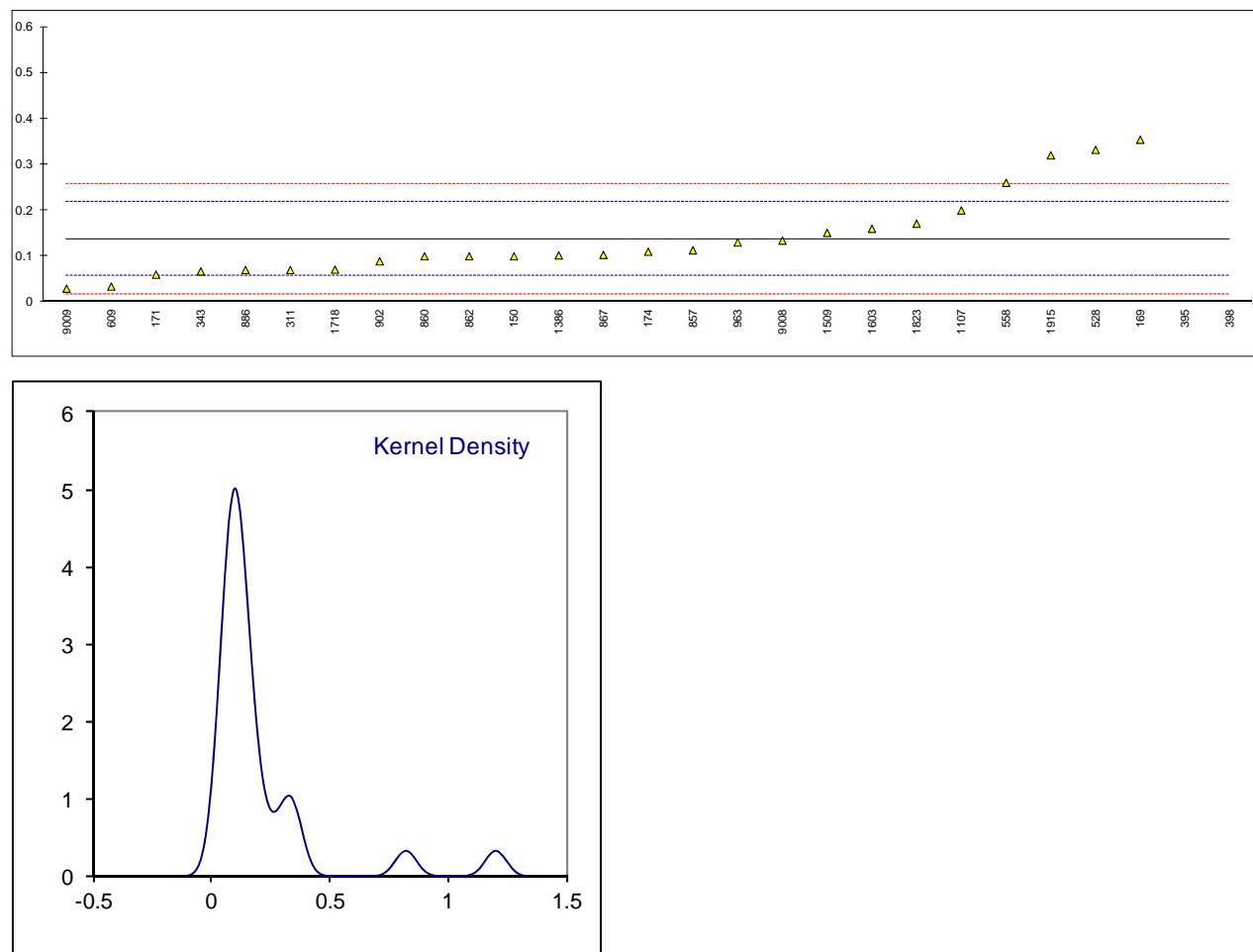
## Determination of Colour (D5386) on sample #12130;

| lab  | method      | value  | mark | z(targ) | remarks |
|------|-------------|--------|------|---------|---------|
| 120  |             | ----   |      | ----    |         |
| 150  | D5386       | 5      |      | -0.30   |         |
| 169  | D5386       | 5.2    |      | -0.20   |         |
| 171  | D5386       | 6      |      | 0.21    |         |
| 173  |             | ----   |      | ----    |         |
| 174  |             | ----   |      | ----    |         |
| 311  | D5386       | 8      |      | 1.23    |         |
| 322  |             | ----   |      | ----    |         |
| 323  | D5386       | 8      |      | 1.23    |         |
| 343  | D5386       | 6      |      | 0.21    |         |
| 347  | D5386       | 4      |      | -0.82   |         |
| 395  | D5386       | 5      |      | -0.30   |         |
| 396  | D5386       | 5      |      | -0.30   |         |
| 398  |             | ----   |      | ----    |         |
| 444  | D5386       | 3.9    |      | -0.87   |         |
| 528  |             | ----   |      | ----    |         |
| 529  |             | ----   |      | ----    |         |
| 551  | D5386       | 0.97   |      | -2.37   |         |
| 557  |             | ----   |      | ----    |         |
| 558  |             | ----   |      | ----    |         |
| 609  | D5386       | 7.2    |      | 0.82    |         |
| 657  | D5386       | 4      |      | -0.82   |         |
| 663  | D5386       | 6      |      | 0.21    |         |
| 823  | D5386       | 8      |      | 1.23    |         |
| 825  |             | ----   |      | ----    |         |
| 857  | D5386       | 5      |      | -0.30   |         |
| 860  | D5386       | 6      |      | 0.21    |         |
| 861  | D5386       | 5      |      | -0.30   |         |
| 862  | D5386       | 5      |      | -0.30   |         |
| 865  | D5386       | 6      |      | 0.21    |         |
| 867  | D5386       | 6      |      | 0.21    |         |
| 869  | D5386       | 7      |      | 0.72    |         |
| 886  | D5386       | 10     |      | 2.26    |         |
| 902  | D5386       | 6      |      | 0.21    |         |
| 912  | D5386       | 6      |      | 0.21    |         |
| 913  | D5386       | 3      |      | -1.33   |         |
| 962  |             | ----   |      | ----    |         |
| 963  |             | ----   |      | ----    |         |
| 1107 | D5386       | 4      |      | -0.82   |         |
| 1117 |             | ----   |      | ----    |         |
| 1151 |             | ----   |      | ----    |         |
| 1169 |             | ----   |      | ----    |         |
| 1217 |             | ----   |      | ----    |         |
| 1386 |             | ----   |      | ----    |         |
| 1467 |             | ----   |      | ----    |         |
| 1492 |             | ----   |      | ----    |         |
| 1509 |             | ----   |      | ----    |         |
| 1515 |             | ----   |      | ----    |         |
| 1603 |             | ----   |      | ----    |         |
| 1608 |             | ----   |      | ----    |         |
| 1623 |             | ----   |      | ----    |         |
| 1701 |             | ----   |      | ----    |         |
| 1718 |             | ----   |      | ----    |         |
| 1823 | D5386       | 5.3    |      | -0.15   |         |
| 1866 |             | ----   |      | ----    |         |
| 1915 |             | ----   |      | ----    |         |
| 1954 |             | ----   |      | ----    |         |
| 7006 |             | ----   |      | ----    |         |
| 9008 | D5386       | 5.54   |      | -0.03   |         |
| 9009 | D5386       | 5.7    |      | 0.05    |         |
|      | normality   | not OK |      |         |         |
|      | n           | 30     |      |         |         |
|      | outliers    | 0      |      |         |         |
|      | mean (n)    | 5.6    |      |         |         |
|      | st.dev. (n) | 1.70   |      |         |         |
|      | R(calc.)    | 4.8    |      |         |         |
|      | R(D5386:10) | 5.5    |      |         |         |



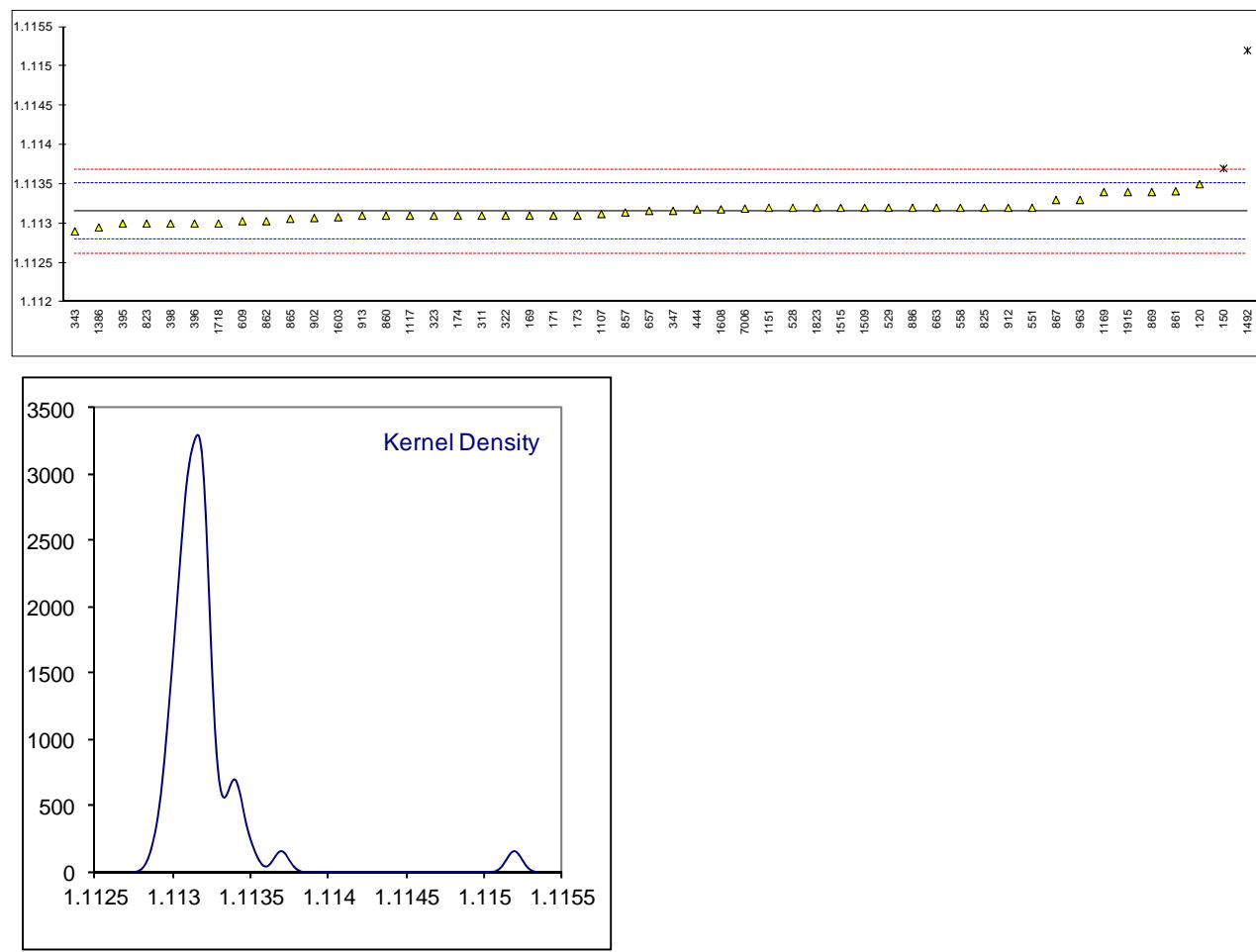
## Determination of Chloride as Cl on sample #12130; results in mg/kg

| lab  | method       | value  | mark      | z(targ) | remarks                |
|------|--------------|--------|-----------|---------|------------------------|
| 120  | INH-221      | <0.5   |           | ----    |                        |
| 150  | INH-2367     | 0.1    |           | -0.93   |                        |
| 169  | E2469        | 0.354  |           | 5.40    |                        |
| 171  | E2469        | 0.06   |           | -1.93   |                        |
| 173  | INH-221      | <0.5   |           | ----    |                        |
| 174  | E2469        | 0.11   |           | -0.68   |                        |
| 311  | E2469        | 0.07   |           | -1.68   |                        |
| 322  |              | ----   |           | ----    |                        |
| 323  |              | ----   |           | ----    |                        |
| 343  | E2469        | 0.067  |           | -1.75   |                        |
| 347  |              | ----   |           | ----    |                        |
| 395  | E2469        | 0.82   | G(0.01)   | 17.02   |                        |
| 396  |              | ----   |           | ----    |                        |
| 398  | E2469        | 1.2    | C,G(0.01) | 26.50   | First reported 0.802   |
| 444  | E2469        | <0.01  |           | <-3.17  | False negative result? |
| 528  | E2469        | 0.332  |           | 4.86    |                        |
| 529  |              | ----   |           | ----    |                        |
| 551  |              | ----   |           | ----    |                        |
| 557  |              | ----   |           | ----    |                        |
| 558  | NBR-7342     | 0.26   |           | 3.06    |                        |
| 609  | E2469        | 0.034  |           | -2.57   |                        |
| 657  | INH-0055     | <0.5   |           | ----    |                        |
| 663  |              | ----   |           | ----    |                        |
| 823  |              | ----   |           | ----    |                        |
| 825  |              | ----   |           | ----    |                        |
| 857  | E2469        | 0.113  |           | -0.60   |                        |
| 860  | IMPCA002     | 0.1    |           | -0.93   |                        |
| 861  |              | ----   |           | ----    |                        |
| 862  | E2469        | 0.10   |           | -0.93   |                        |
| 865  | INH-001      | <0.5   |           | ----    |                        |
| 867  | E2469        | 0.103  |           | -0.85   |                        |
| 869  |              | ----   |           | ----    |                        |
| 886  | INH-082      | 0.07   |           | -1.68   |                        |
| 902  | E2469        | 0.089  |           | -1.20   |                        |
| 912  |              | ----   |           | ----    |                        |
| 913  |              | ----   |           | ----    |                        |
| 962  |              | ----   |           | ----    |                        |
| 963  | E2469        | 0.13   |           | -0.18   |                        |
| 1107 | in house     | 0.2    |           | 1.57    |                        |
| 1117 |              | ----   |           | ----    |                        |
| 1151 |              | ----   |           | ----    |                        |
| 1169 | E2901        | <0.1   |           | ----    |                        |
| 1217 |              | ----   |           | ----    |                        |
| 1386 | E2469        | 0.102  |           | -0.88   |                        |
| 1467 |              | ----   |           | ----    |                        |
| 1492 |              | ----   |           | ----    |                        |
| 1509 | E2469        | 0.151  |           | 0.34    |                        |
| 1515 | E2469        | <0.05  |           | <-2.17  |                        |
| 1603 | in house     | 0.16   |           | 0.57    |                        |
| 1608 |              | ----   |           | ----    |                        |
| 1623 |              | ----   |           | ----    |                        |
| 1701 |              | ----   |           | ----    |                        |
| 1718 | E2469        | 0.071  |           | -1.65   |                        |
| 1823 | INH-2901     | 0.171  |           | 0.84    |                        |
| 1866 |              | ----   |           | ----    |                        |
| 1915 | E2469        | 0.32   |           | 4.56    |                        |
| 1954 | INH-5295     | <0.1   |           | ----    |                        |
| 7006 |              | ----   |           | ----    |                        |
| 9008 | E2469        | 0.134  |           | -0.08   |                        |
| 9009 | E2469        | 0.0294 | C         | -2.69   | First reported 235     |
|      | normality    | not OK |           |         |                        |
|      | n            | 25     |           |         |                        |
|      | outliers     | 2      | Spike:    |         |                        |
|      | mean (n)     | 0.137  | 0.095     |         | Recovery <144%         |
|      | st.dev. (n)  | 0.0904 |           |         |                        |
|      | R(calc.)     | 0.253  |           |         |                        |
|      | R(E2469:08a) | 0.112  |           |         |                        |



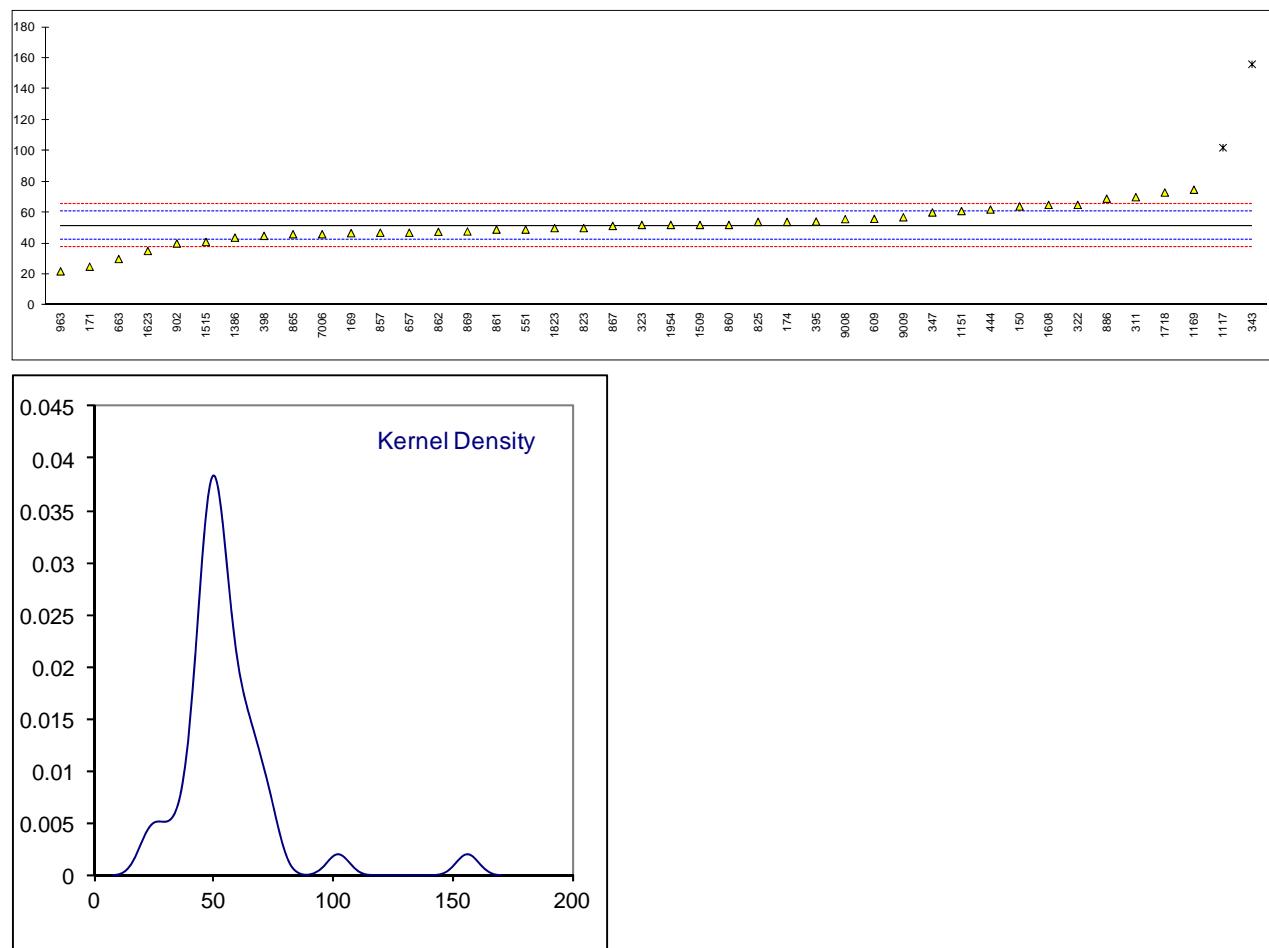
## Determination of Density at 20 °C on sample #12130; results in kg/L

| lab  | method        | value    | mark      | z(targ) | remarks   |
|------|---------------|----------|-----------|---------|---|
| 120  | D4052         | 1.1135   |           | 1.94    |   |
| 150  | D4052         | 1.1137   | C,G(0.01) | 3.06    | First reported 1.1124                           |
| 169  | D4052         | 1.1131   |           | -0.30   |   |
| 171  | D4052         | 1.1131   |           | -0.30   |   |
| 173  | D4052         | 1.1131   |           | -0.30   |   |
| 174  | D4052         | 1.1131   |           | -0.30   |   |
| 311  | D4052         | 1.1131   |           | -0.30   |   |
| 322  | D4052         | 1.1131   |           | -0.30   |   |
| 323  | D4052         | 1.1131   |           | -0.30   |   |
| 343  | D4052         | 1.1129   |           | -1.42   |   |
| 347  | D4052         | 1.11316  |           | 0.04    |   |
| 395  | D4052         | 1.1130   |           | -0.86   |   |
| 396  | D4052         | 1.1130   |           | -0.86   |   |
| 398  | D4052         | 1.1130   |           | -0.86   |   |
| 444  | D4052         | 1.11318  |           | 0.15    |   |
| 528  | D4052         | 1.1132   | C         | 0.26    | Reported 1.1152 (swiched with Specific Gravity) |
| 529  | D4052         | 1.1132   |           | 0.26    |   |
| 551  | D4052         | 1.1132   |           | 0.26    |   |
| 557  |               | -----    |           | -----   |   |
| 558  | D4052         | 1.1132   |           | 0.26    |   |
| 609  | D4052         | 1.11303  |           | -0.69   |   |
| 657  | D4052         | 1.11316  |           | 0.04    |   |
| 663  | D4052         | 1.1132   |           | 0.26    |   |
| 823  | D4052         | 1.1130   |           | -0.86   |   |
| 825  | D4052         | 1.1132   |           | 0.26    |   |
| 857  | D4052         | 1.11314  |           | -0.08   |   |
| 860  | D4052         | 1.1131   |           | -0.30   |   |
| 861  | D4052         | 1.11341  |           | 1.44    |   |
| 862  | D4052         | 1.11303  |           | -0.69   |   |
| 865  | D4052         | 1.11306  |           | -0.52   |   |
| 867  | D4052         | 1.1133   |           | 0.82    |   |
| 869  | D4052         | 1.1134   |           | 1.38    |   |
| 886  | D4052         | 1.1132   |           | 0.26    |   |
| 902  | D4052         | 1.11307  |           | -0.47   |   |
| 912  | D4052         | 1.1132   |           | 0.26    |   |
| 913  | D4052         | 1.1131   |           | -0.30   |   |
| 962  |               | -----    |           | -----   |   |
| 963  | D4052         | 1.1133   |           | 0.82    |   |
| 1107 | D4052         | 1.11312  |           | -0.19   |   |
| 1117 | D4052         | 1.1131   |           | -0.30   |   |
| 1151 | D4052         | 1.1132   |           | 0.26    |   |
| 1169 | D4052         | 1.1134   |           | 1.38    |   |
| 1217 |               | -----    |           | -----   |   |
| 1386 | D4052         | 1.11295  |           | -1.14   |   |
| 1467 |               | -----    |           | -----   |   |
| 1492 | D4052         | 1.1152   | G(0.01)   | 11.46   |   |
| 1509 | D4052         | 1.1132   |           | 0.26    |   |
| 1515 | D4052         | 1.1132   |           | 0.26    |   |
| 1603 | in house      | 1.11308  |           | -0.41   |   |
| 1608 | D4052         | 1.11318  |           | 0.15    |   |
| 1623 |               | -----    |           | -----   |   |
| 1701 |               | -----    |           | -----   |   |
| 1718 | D4052         | 1.1130   |           | -0.86   |   |
| 1823 | D4052         | 1.1132   |           | 0.26    |   |
| 1866 |               | -----    |           | -----   |   |
| 1915 | D4052         | 1.1134   |           | 1.38    |   |
| 1954 |               | -----    |           | -----   |   |
| 7006 | D4052         | 1.11319  |           | 0.20    |   |
| 9008 |               | -----    |           | -----   |   |
| 9009 |               | -----    |           | -----   |   |
|      | normality     | not OK   |           |         |   |
|      | n             | 48       |           |         |   |
|      | outliers      | 2        |           |         |   |
|      | mean (n)      | 1.11315  |           |         |   |
|      | st.dev. (n)   | 0.000126 |           |         |   |
|      | R(calc.)      | 0.00035  |           |         |   |
|      | R(D4052:02e1) | 0.00050  |           |         |   |



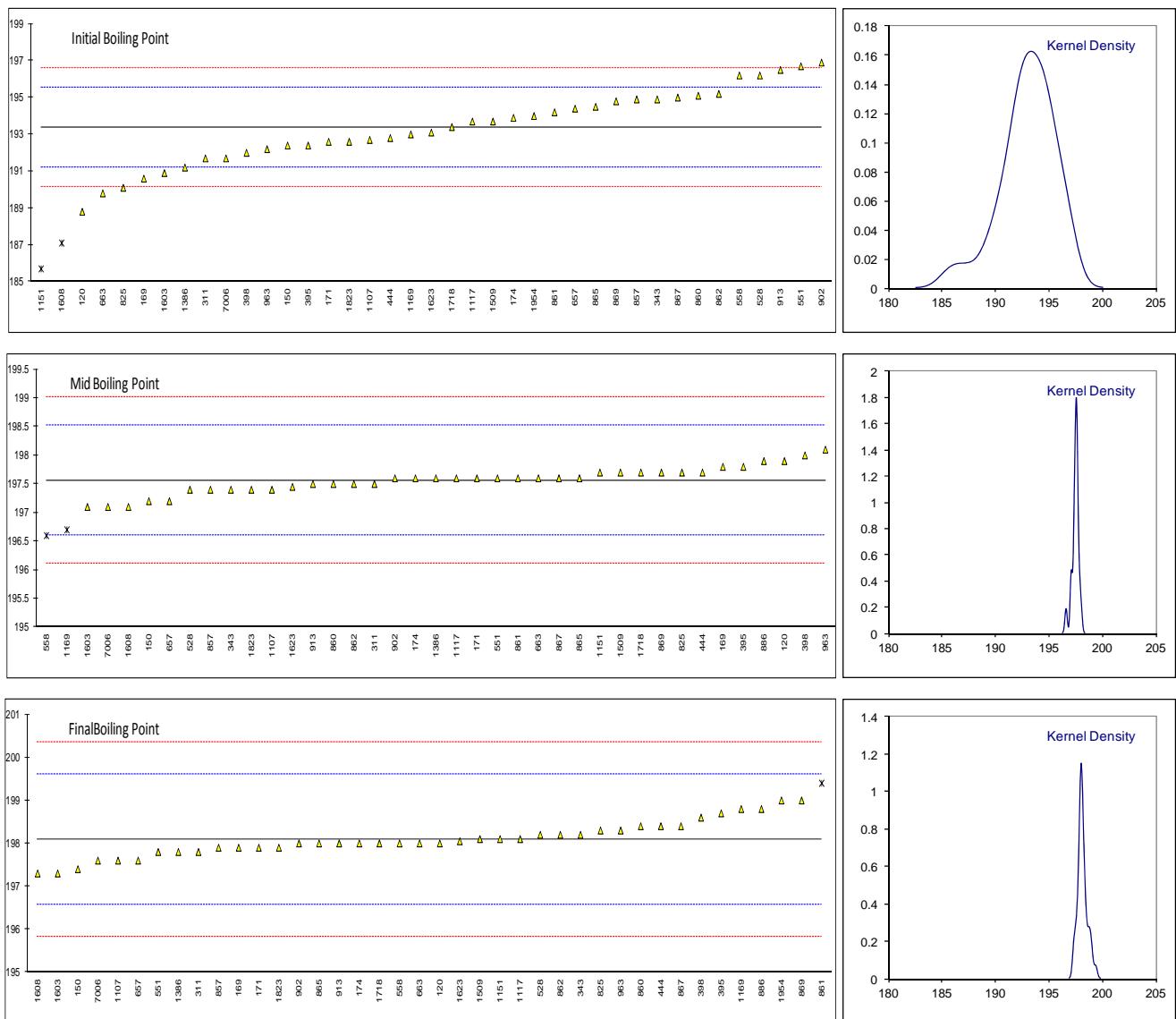
## Determination of Diethylene Glycol on sample #12130; results in mg/kg

| lab  | method      | value  | mark      | z(targ) | remarks  |
|------|-------------|--------|-----------|---------|--|
| 120  |             | ----   |           | ----    |  |
| 150  | E2409       | 64     |           | 2.66    |  |
| 169  | E2409       | 46.7   |           | -1.04   |  |
| 171  | E2409       | 25     |           | -5.67   |  |
| 173  | INH-540     | <100   | C         | ----    | Reported <0.01 (unit error? %M/M instead of mg/kg) |
| 174  | E2409       | 54     |           | 0.52    |  |
| 311  | E2409       | 70     |           | 3.94    |  |
| 322  | E2409       | 65     |           | 2.87    |  |
| 323  | E2409       | 52     |           | 0.09    |  |
| 343  | E2409       | 156    | C,G(0.01) | 22.29   | First reported <5                                  |
| 347  | E2409       | 60.1   |           | 1.82    |  |
| 395  | E2409       | 54.24  |           | 0.57    |  |
| 396  |             | ----   |           | ----    |  |
| 398  | E2409       | 45     |           | -1.40   |  |
| 444  | E2409       | 62     |           | 2.23    |  |
| 528  |             | ----   |           | ----    |  |
| 529  |             | ----   |           | ----    |  |
| 551  | E2409       | 49     |           | -0.55   |  |
| 557  |             | ----   |           | ----    |  |
| 558  |             | ----   |           | ----    |  |
| 609  | E2409       | 56     |           | 0.95    |  |
| 657  | E2409       | 47     |           | -0.97   |  |
| 663  | E2409       | 30     |           | -4.60   |  |
| 823  | E2409       | 50     |           | -0.33   |  |
| 825  | E2409       | 54     |           | 0.52    |  |
| 857  | E2409       | 47     |           | -0.97   |  |
| 860  | E2409       | 52     |           | 0.09    |  |
| 861  | E2409       | 49     |           | -0.55   |  |
| 862  | E2409       | 47.6   |           | -0.85   |  |
| 865  | E2409       | 46     |           | -1.19   |  |
| 867  | E2409       | 51.4   |           | -0.03   |  |
| 869  | E2409       | 47.8   |           | -0.80   |  |
| 886  | E2409       | 69.0   |           | 3.72    |  |
| 902  | E2409       | 40     |           | -2.47   |  |
| 912  |             | ----   |           | ----    |  |
| 913  |             | ----   |           | ----    |  |
| 962  |             | ----   |           | ----    |  |
| 963  | E2409       | 22     |           | -6.31   |  |
| 1107 | in house    | <100   |           | ----    |  |
| 1117 | E2409       | 102    | G(0.01)   | 10.77   |  |
| 1151 | E2409       | 61     |           | 2.01    |  |
| 1169 | E2409       | 74.8   |           | 4.96    |  |
| 1217 |             | ----   |           | ----    |  |
| 1386 | E2409       | 43.8   |           | -1.66   |  |
| 1467 |             | ----   |           | ----    |  |
| 1492 |             | ----   |           | ----    |  |
| 1509 | E2409       | 52.0   |           | 0.09    |  |
| 1515 | E2409       | 41     | C         | -2.25   | First reported 2450                                |
| 1603 | in house    | <50    |           | ----    |  |
| 1608 | E2409       | 65     |           | 2.87    |  |
| 1623 | E2409       | 35.18  |           | -3.50   |  |
| 1701 |             | ----   |           | ----    |  |
| 1718 | E2409       | 73.0   |           | 4.58    |  |
| 1823 | E2409       | 50     |           | -0.33   |  |
| 1866 |             | ----   |           | ----    |  |
| 1915 |             | ----   |           | ----    |  |
| 1954 | E2409       | 52     |           | 0.09    |  |
| 7006 | E2409       | 46     |           | -1.19   |  |
| 9008 | E2409       | 55.8   |           | 0.90    |  |
| 9009 | E2409       | 57     |           | 1.16    |  |
|      | normality   | OK     |           |         |  |
|      | n           | 40     |           |         |  |
|      | outliers    | 2      |           |         |  |
|      | mean (n)    | 51.56  |           |         |  |
|      | st.dev. (n) | 11.717 |           |         |  |
|      | R(calc.)    | 32.81  |           |         |  |
|      | R(E2409:08) | 13.12  |           |         |  |



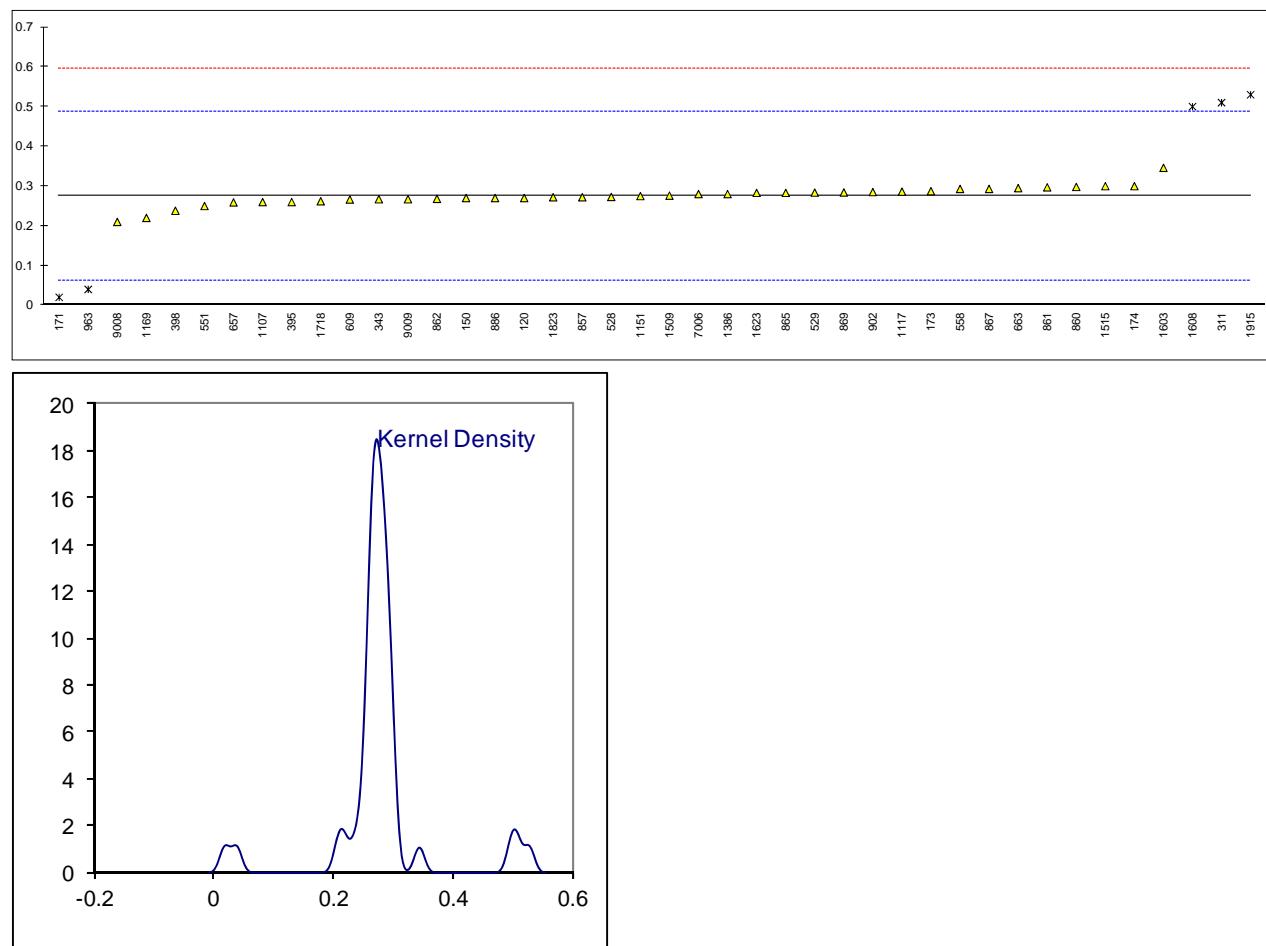
## Determination of Distillation: IBP, 50% recovered, Dry Point on sample #12130; results in °C

| lab         | method   | IBP    | mark     | z(targ) | 50%    | mark     | z(targ) | DP     | mark    | z(targ) | remarks |
|-------------|----------|--------|----------|---------|--------|----------|---------|--------|---------|---------|---------|
| 120         | D1078    | 188.8  |          | -4.24   | 197.9  |          | 0.70    | 198.0  |         | -0.12   |         |
| 150         | D1078    | 192.4  |          | -0.90   | 197.2  |          | -0.75   | 197.4  |         | -0.91   |         |
| 169         | D1078    | 190.6  |          | -2.57   | 197.8  |          | 0.49    | 197.9  |         | -0.25   |         |
| 171         | D1078    | 192.6  |          | -0.72   | 197.6  |          | 0.08    | 197.9  |         | -0.25   |         |
| 173         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 174         | D1078    | 193.9  |          | 0.49    | 197.6  |          | 0.08    | 198.0  |         | -0.12   |         |
| 311         | D1078    | 191.7  |          | -1.55   | 197.5  |          | -0.13   | 197.8  |         | -0.38   |         |
| 322         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 323         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 343         | D1078    | 194.9  |          | 1.42    | 197.4  |          | -0.34   | 198.2  |         | 0.15    |         |
| 347         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 395         | D1078    | 192.4  |          | -0.90   | 197.8  |          | 0.49    | 198.7  |         | 0.81    |         |
| 396         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 398         | D1078    | 192.0  |          | -1.27   | 198.0  |          | 0.91    | 198.6  |         | 0.68    |         |
| 444         | D1078    | 192.8  |          | -0.53   | 197.7  |          | 0.28    | 198.4  |         | 0.41    |         |
| 528         | D1078    | 196.2  |          | 2.62    | 197.4  |          | -0.34   | 198.2  |         | 0.15    |         |
| 529         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 551         | D1078    | 196.7  |          | 3.09    | 197.6  |          | 0.08    | 197.8  |         | -0.38   |         |
| 557         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 558         | NBR-7140 | 196.2  |          | 2.62    | 196.6  | DG(0.01) | -2.00   | 198.0  |         | -0.12   |         |
| 609         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 657         | D1078    | 194.4  |          | 0.95    | 197.2  |          | -0.75   | 197.6  |         | -0.65   |         |
| 663         | D1078    | 189.8  |          | -3.32   | 197.6  |          | 0.08    | 198.0  |         | -0.12   |         |
| 823         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 825         | D1078    | 190.1  |          | -3.04   | 197.7  |          | 0.28    | 198.3  |         | 0.28    |         |
| 857         | D1078    | 194.9  |          | 1.42    | 197.4  |          | -0.34   | 197.9  |         | -0.25   |         |
| 860         | D1078    | 195.1  |          | 1.60    | 197.5  |          | -0.13   | 198.4  |         | 0.41    |         |
| 861         | D1078    | 194.2  |          | 0.77    | 197.6  |          | 0.08    | 199.4  | G(0.05) | 1.73    |         |
| 862         | D1078    | 195.2  |          | 1.70    | 197.5  |          | -0.13   | 198.2  |         | 0.15    |         |
| 865         | D1078    | 194.5  |          | 1.05    | 197.6  |          | 0.08    | 198.0  |         | -0.12   |         |
| 867         | D1078    | 195.0  |          | 1.51    | 197.6  |          | 0.08    | 198.4  |         | 0.41    |         |
| 869         | D1078    | 194.8  |          | 1.32    | 197.7  |          | 0.28    | 199.0  |         | 1.20    |         |
| 886         |          | ----   |          | ----    | 197.9  |          | 0.70    | 198.8  |         | 0.94    |         |
| 902         | D1078    | 196.9  |          | 3.27    | 197.6  |          | 0.08    | 198.0  |         | -0.12   |         |
| 912         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 913         | D1078    | 196.5  |          | 2.90    | 197.5  |          | -0.13   | 198.0  |         | -0.12   |         |
| 962         |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 963         | D1078    | 192.2  |          | -1.09   | 198.1  |          | 1.11    | 198.3  |         | 0.28    |         |
| 1107        | D1078    | 192.7  |          | -0.62   | 197.4  |          | -0.34   | 197.6  |         | -0.65   |         |
| 1117        | D1078    | 193.7  |          | 0.30    | 197.6  |          | 0.08    | 198.1  |         | 0.02    |         |
| 1151        | D1078    | 185.7  | DG(0.05) | -7.12   | 197.7  |          | 0.28    | 198.1  |         | 0.02    |         |
| 1169        | D1078    | 193.0  |          | -0.35   | 196.7  | DG(0.01) | -1.79   | 198.8  |         | 0.94    |         |
| 1217        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1386        | D1078    | 191.2  |          | -2.02   | 197.6  |          | 0.08    | 197.8  |         | -0.38   |         |
| 1467        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1492        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1509        | D1078    | 193.7  |          | 0.30    | 197.7  |          | 0.28    | 198.1  |         | 0.02    |         |
| 1515        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1603        | in house | 190.9  | DG(0.05) | -2.30   | 197.1  |          | -0.96   | 197.3  |         | -1.04   |         |
| 1608        | D1078    | 187.1  |          | -5.82   | 197.1  |          | -0.96   | 197.3  |         | -1.04   |         |
| 1623        | D1078    | 193.10 |          | -0.25   | 197.45 |          | -0.24   | 198.05 |         | -0.05   |         |
| 1701        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1718        | D1078    | 193.4  |          | 0.03    | 197.7  |          | 0.28    | 198.0  |         | -0.12   |         |
| 1823        | D1078    | 192.6  |          | -0.72   | 197.4  |          | -0.34   | 197.9  |         | -0.25   |         |
| 1866        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1915        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 1954        | D1078    | 194    |          | 0.58    | -----  |          | -----   | 199    |         | 1.20    |         |
| 7006        | D1078    | 191.7  |          | -1.55   | 197.1  |          | -0.96   | 197.6  |         | -0.65   |         |
| 9008        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| 9009        |          | ----   |          | ----    | ----   |          | ----    | ----   |         | ----    |         |
| normality   |          | OK     | not OK   |         | OK     |          |         |        |         |         |         |
| n           |          | 37     |          | 37      |        |          |         | 39     |         |         |         |
| outliers    |          | 2      |          | 2       |        |          |         | 1      |         |         |         |
| mean (n)    |          | 193.37 |          | 197.56  |        |          |         | 198.09 |         |         |         |
| st.dev. (n) |          | 2.005  |          | 0.236   |        |          |         | 0.417  |         |         |         |
| R(calc.)    |          | 5.61   |          | 0.66    |        |          |         | 1.17   |         |         |         |
| R(D1078:11) |          | 3.02   |          | 1.35    |        |          |         | 2.12   |         |         |         |



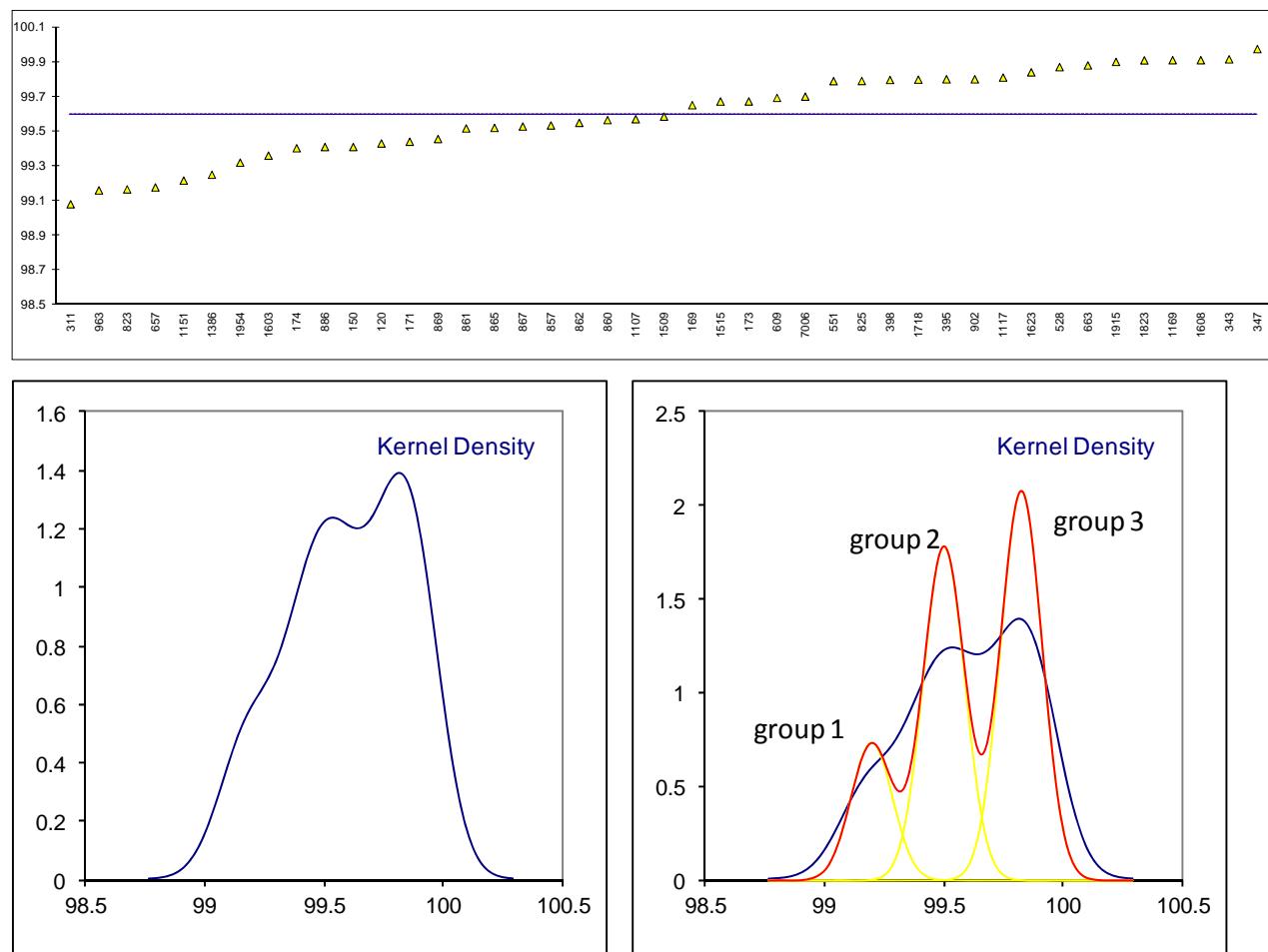
## Determination of Iron as Fe on sample #12130; results in mg/kg

| lab  | method      | value   | mark      | z(targ) | remarks                     |
|------|-------------|---------|-----------|---------|-----------------------------|
| 120  | INH-0290    | 0.27    |           | -0.05   |                             |
| 150  | E202        | 0.27    |           | -0.05   |                             |
| 169  |             | ----    |           | ----    |                             |
| 171  | E1615       | 0.02    | DG(0.01)  | -2.40   |                             |
| 173  | INH-290     | 0.2872  |           | 0.11    |                             |
| 174  | E1615       | 0.30    | C         | 0.23    | First reported 0.11         |
| 311  | E1615       | 0.51    | DG(0.05)  | 2.21    |                             |
| 322  | E1615       | >0.20   |           | ----    |                             |
| 323  | E1615       | >0.20   |           | ----    |                             |
| 343  | E1615       | 0.267   |           | -0.08   |                             |
| 347  |             | ----    |           | ----    |                             |
| 395  | E1615       | 0.26    |           | -0.14   |                             |
| 396  |             | ----    |           | ----    |                             |
| 398  | E1615       | 0.238   |           | -0.35   |                             |
| 444  | E1615       | <0.001  |           | <-2.57  | False negative?             |
| 528  | E1615       | 0.2727  |           | -0.02   |                             |
| 529  | E1615       | 0.2838  |           | 0.08    |                             |
| 551  | E394        | 0.25    |           | -0.24   |                             |
| 557  |             | ----    |           | ----    |                             |
| 558  | NBR7448     | 0.293   |           | 0.17    |                             |
| 609  | E1615       | 0.2664  |           | -0.08   |                             |
| 657  | E1615       | 0.259   |           | -0.15   |                             |
| 663  | E1615       | 0.295   |           | 0.19    |                             |
| 823  |             | ----    |           | ----    |                             |
| 825  |             | ----    |           | ----    |                             |
| 857  | E1615       | 0.272   |           | -0.03   |                             |
| 860  | E394        | 0.298   |           | 0.21    |                             |
| 861  | E394        | 0.297   |           | 0.21    |                             |
| 862  | E1615       | 0.268   |           | -0.07   |                             |
| 865  | E394        | 0.283   |           | 0.07    |                             |
| 867  | E1615       | 0.293   |           | 0.17    |                             |
| 869  | E1615       | 0.284   |           | 0.08    |                             |
| 886  | E202        | 0.27    |           | -0.05   |                             |
| 902  | E1615       | 0.285   |           | 0.09    |                             |
| 912  |             | ----    |           | ----    |                             |
| 913  |             | ----    |           | ----    |                             |
| 962  |             | ----    |           | ----    |                             |
| 963  | E202        | 0.04    | DG(0.01)  | -2.21   |                             |
| 1107 | E202        | 0.26    |           | -0.14   |                             |
| 1117 | E394        | 0.286   |           | 0.10    |                             |
| 1151 | E1615       | 0.275   |           | 0.00    |                             |
| 1169 | E394        | 0.22    |           | -0.52   |                             |
| 1217 |             | ----    |           | ----    |                             |
| 1386 | E394        | 0.280   |           | 0.05    |                             |
| 1467 |             | ----    |           | ----    |                             |
| 1492 |             | ----    |           | ----    |                             |
| 1509 | E394        | 0.276   |           | 0.01    |                             |
| 1515 | E394        | 0.30    |           | 0.23    |                             |
| 1603 | in house    | 0.346   |           | 0.67    |                             |
| 1608 | E394        | 0.50    | C,G(0.01) | 2.11    | First reported 0.54         |
| 1623 | E202        | 0.283   |           | 0.07    |                             |
| 1701 |             | ----    |           | ----    |                             |
| 1718 | E394        | 0.262   |           | -0.12   |                             |
| 1823 | E394        | 0.272   |           | -0.03   |                             |
| 1866 |             | ----    |           | ----    |                             |
| 1915 | E394        | 0.53    | DG(0.05)  | 2.39    |                             |
| 1954 | E394        | <0.05   |           | <-2.11  | False negative?             |
| 7006 | E1615       | 0.28    |           | 0.05    |                             |
| 9008 | E1615       | 0.21    |           | -0.61   |                             |
| 9009 | E1615       | 0.267   | C         | -0.08   | First reported 0.533        |
|      | normality   | OK      |           |         |                             |
|      | n           | 37      |           |         |                             |
|      | outliers    | 5       |           |         |                             |
|      | mean (n)    | 0.2751  |           |         |                             |
|      | st.dev. (n) | 0.02329 |           |         |                             |
|      | R(calc.)    | 0.0652  |           |         |                             |
|      | R(E1615:08) | 0.2982  |           |         | Compare R(E394:09) = 0.1484 |



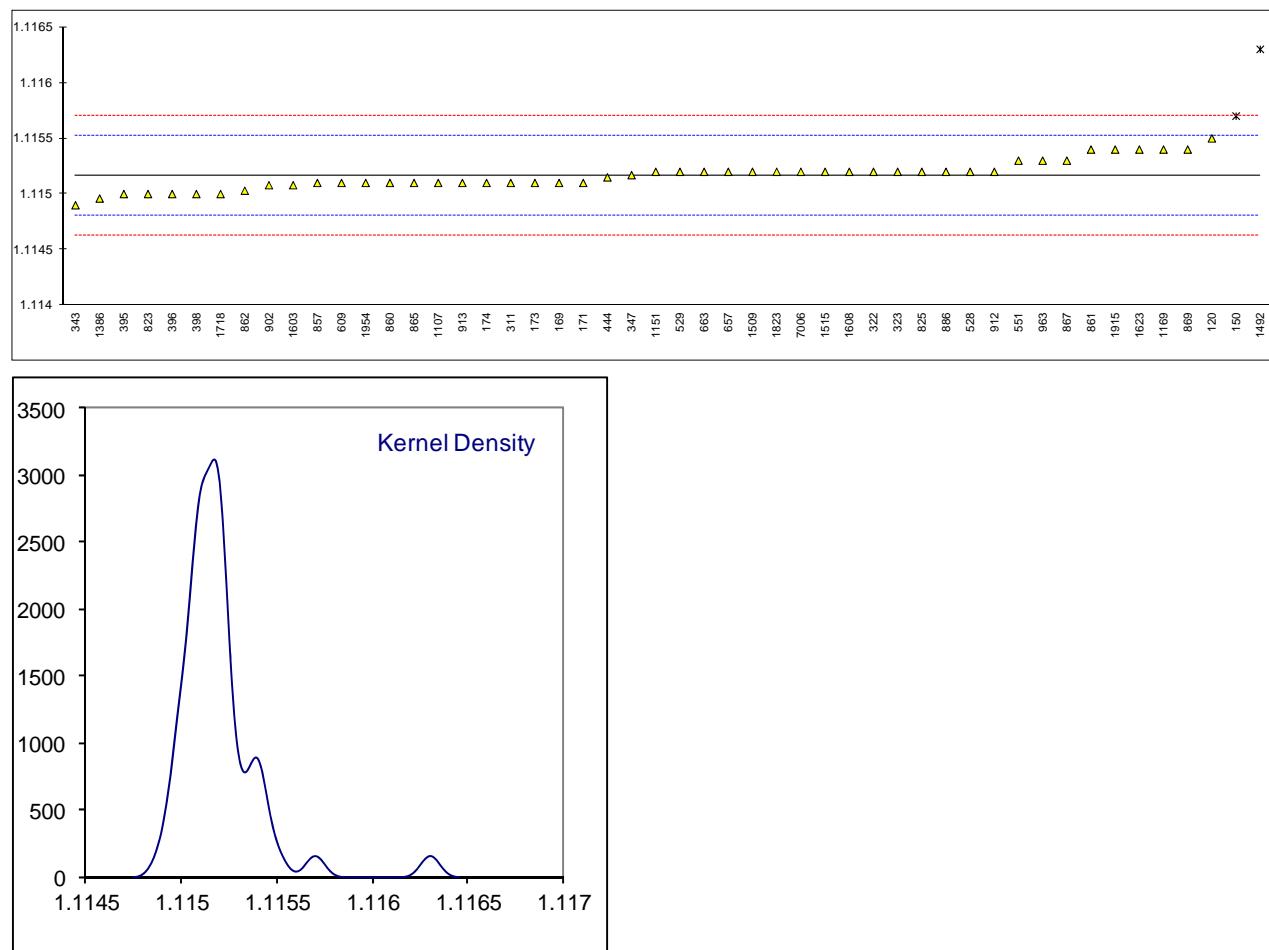
## Determination of Purity on sample #12130; results in %M/M

| lab  | method      | value     | mark    | z(targ) | remarks |
|------|-------------|-----------|---------|---------|---------|
| 120  | E202        | 99.43     | ----    |         |         |
| 150  | E2409       | 99.41     | ----    |         |         |
| 169  | E202        | 99.6508   | ----    |         |         |
| 171  | E2409       | 99.44     | ----    |         |         |
| 173  | INH-540     | 99.672197 | ----    |         |         |
| 174  | E202        | 99.403    | ----    |         |         |
| 311  | E2409       | 99.08     | ----    |         |         |
| 322  | E202        | <99.80    | ----    |         |         |
| 323  | E202        | <99.80    | ----    |         |         |
| 343  | E202        | 99.915    | ----    |         |         |
| 347  | E2409       | 99.974    | ----    |         |         |
| 395  | E202        | 99.80     | ----    |         |         |
| 396  |             | ----      | ----    |         |         |
| 398  | E202        | 99.796    | ----    |         |         |
| 444  |             | ----      | ----    |         |         |
| 528  | E202        | 99.87     | ----    |         |         |
| 529  |             | ----      | ----    |         |         |
| 551  | E2409       | 99.7893   | ----    |         |         |
| 557  |             | ----      | ----    |         |         |
| 558  |             | ----      | ----    |         |         |
| 609  | E2409       | 99.693    | ----    |         |         |
| 657  | E2409       | 99.1773   | ----    |         |         |
| 663  | E2409       | 99.88     | ----    |         |         |
| 823  | E2409       | 99.166    | ----    |         |         |
| 825  | E202        | 99.79     | ----    |         |         |
| 857  | E2409       | 99.534    | ----    |         |         |
| 860  | E202        | 99.565    | ----    |         |         |
| 861  | E2409       | 99.517    | ----    |         |         |
| 862  | E202        | 99.549    | ----    |         |         |
| 865  | E2409       | 99.520    | ----    |         |         |
| 867  | E202        | 99.528    | ----    |         |         |
| 869  | E202        | 99.456    | ----    |         |         |
| 886  | E2409       | 99.41     | ----    |         |         |
| 902  | E2409       | 99.80     | ----    |         |         |
| 912  |             | ----      | ----    |         |         |
| 913  |             | ----      | ----    |         |         |
| 962  |             | ----      | ----    |         |         |
| 963  | E2409       | 99.16     | ----    |         |         |
| 1107 | in house    | 99.57     | ----    |         |         |
| 1117 | E202        | 99.81     | ----    |         |         |
| 1151 | E202        | 99.2169   | ----    |         |         |
| 1169 | E2409       | 99.91     | ----    |         |         |
| 1217 |             | ----      | ----    |         |         |
| 1386 | E2409       | 99.250    | ----    |         |         |
| 1467 |             | ----      | ----    |         |         |
| 1492 |             | ----      | ----    |         |         |
| 1509 | E2409       | 99.585    | ----    |         |         |
| 1515 | E2409       | 99.672    | ----    |         |         |
| 1603 | in house    | 99.36     | ----    |         |         |
| 1608 | E2409       | 99.91     | ----    |         |         |
| 1623 | E2409       | 99.84     | ----    |         |         |
| 1701 |             | ----      | ----    |         |         |
| 1718 | E2409       | 99.798    | ----    |         |         |
| 1823 | E2409       | 99.909    | ----    |         |         |
| 1866 |             | ----      | ----    |         |         |
| 1915 | E202        | 99.9      | ----    |         |         |
| 1954 | E2409       | 99.32     | ----    |         |         |
| 7006 | E202        | 99.7      | ----    |         |         |
| 9008 |             | ----      | ----    |         |         |
| 9009 |             | ----      | ----    |         |         |
|      | normality   | not OK    |         | Group 1 | Group 2 |
|      | n           | 43        | 7       | OK      | OK      |
|      | outliers    | 0         | 0       | 0       | 0       |
|      | mean (n)    | 99.5983   | 99.1957 | 99.5332 | 99.8557 |
|      | st.dev. (n) | 0.24790   | 0.07608 | 0.10616 | 0.05954 |
|      | R(calc.)    | 0.6941    | 0.2130  | 0.2973  | 0.1667  |
|      | R(lit)      | unknown   | unknown | unknown | unknown |



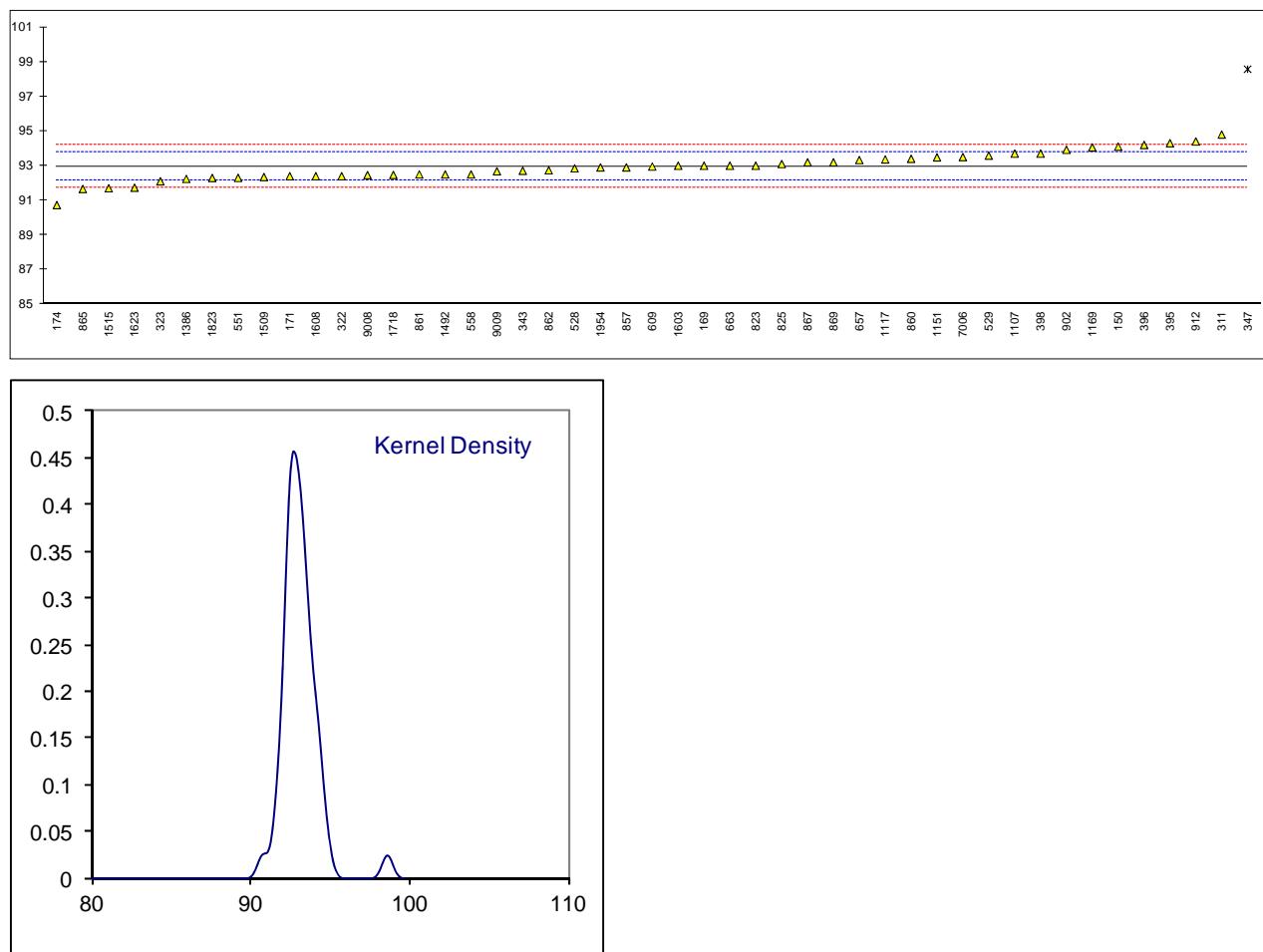
## Determination of Specific Gravity 20/20°C on sample #12130;

| lab  | method        | Value    | mark      | z(targ) | remarks                                 |
|------|---------------|----------|-----------|---------|---|
| 120  | D4052         | 1.1155   |           | 1.87    |   |
| 150  | D4052         | 1.1157   | C,G(0.01) | 2.99    | First reported 0.1144                   |
| 169  | D4052         | 1.1151   |           | -0.37   |   |
| 171  | D4052         | 1.1151   |           | -0.37   |   |
| 173  | D4052         | 1.1151   |           | -0.37   |   |
| 174  | E202          | 1.1151   |           | -0.37   |   |
| 311  | E202          | 1.1151   |           | -0.37   |   |
| 322  | E202          | 1.1152   |           | 0.19    |   |
| 323  | E202          | 1.1152   |           | 0.19    |   |
| 343  | D4052         | 1.1149   |           | -1.49   |   |
| 347  | D4052         | 1.11517  |           | 0.02    |   |
| 395  | E202          | 1.1150   |           | -0.93   |   |
| 396  | E202          | 1.1150   |           | -0.93   |   |
| 398  | E202          | 1.1150   |           | -0.93   |   |
| 444  | D4052         | 1.11515  |           | -0.09   |   |
| 528  | D4052         | 1.1152   | C         | 0.19    | Reported 1.1132 (switched with density) |
| 529  | D4052         | 1.1152   |           | 0.19    |   |
| 551  | D4052         | 1.1153   |           | 0.75    |   |
| 557  |               | -----    |           | -----   |   |
| 558  |               | -----    |           | -----   |   |
| 609  | D4052         | 1.1151   |           | -0.37   |   |
| 657  | D4052         | 1.1152   |           | 0.19    |   |
| 663  | D4052         | 1.1152   |           | 0.19    |   |
| 823  | E202          | 1.1150   |           | -0.93   |   |
| 825  | E202          | 1.1152   |           | 0.19    |   |
| 857  | D4052         | 1.1151   |           | -0.37   |   |
| 860  | E202          | 1.1151   |           | -0.37   |   |
| 861  | D4052         | 1.1154   |           | 1.31    |   |
| 862  | E202          | 1.11503  |           | -0.76   |   |
| 865  | D4052         | 1.1151   |           | -0.37   |   |
| 867  | E202          | 1.1153   |           | 0.75    |   |
| 869  | E202          | 1.1154   |           | 1.31    |   |
| 886  | D4052         | 1.1152   |           | 0.19    |   |
| 902  | D4052         | 1.11508  |           | -0.48   |   |
| 912  | D4052         | 1.1152   |           | 0.19    |   |
| 913  | D4052         | 1.1151   |           | -0.37   |   |
| 962  |               | -----    |           | -----   |   |
| 963  | D4052         | 1.1153   |           | 0.75    |   |
| 1107 | D4052         | 1.1151   |           | -0.37   |   |
| 1117 |               | -----    |           | -----   |   |
| 1151 | D4052         | 1.1152   |           | 0.19    |   |
| 1169 | D4052         | 1.1154   |           | 1.31    |   |
| 1217 |               | -----    |           | -----   |   |
| 1386 | D4052         | 1.11496  |           | -1.15   |   |
| 1467 |               | -----    |           | -----   |   |
| 1492 | E202          | 1.1163   | G(0.01)   | 6.35    |   |
| 1509 | D4052         | 1.1152   |           | 0.19    |   |
| 1515 | D4052         | 1.1152   |           | 0.19    |   |
| 1603 | in house      | 1.11508  |           | -0.48   |   |
| 1608 | D4052         | 1.1152   |           | 0.19    |   |
| 1623 | D891          | 1.1154   |           | 1.31    |   |
| 1701 |               | -----    |           | -----   |   |
| 1718 | D4052         | 1.1150   |           | -0.93   |   |
| 1823 | D4052         | 1.1152   |           | 0.19    |   |
| 1866 |               | -----    |           | -----   |   |
| 1915 | E202          | 1.1154   |           | 1.31    |   |
| 1954 | D4052         | 1.1151   |           | -0.37   |   |
| 7006 | D4052         | 1.1152   |           | 0.19    |   |
| 9008 |               | -----    |           | -----   |   |
| 9009 |               | -----    |           | -----   |   |
|      | normality     | not OK   |           |         |   |
|      | n             | 48       |           |         |   |
|      | outliers      | 2        |           |         |   |
|      | mean (n)      | 1.11517  |           |         |   |
|      | st.dev. (n)   | 0.000130 |           |         |   |
|      | R(calc.)      | 0.00036  |           |         |   |
|      | R(D4052:02e1) | 0.00050  |           |         |   |



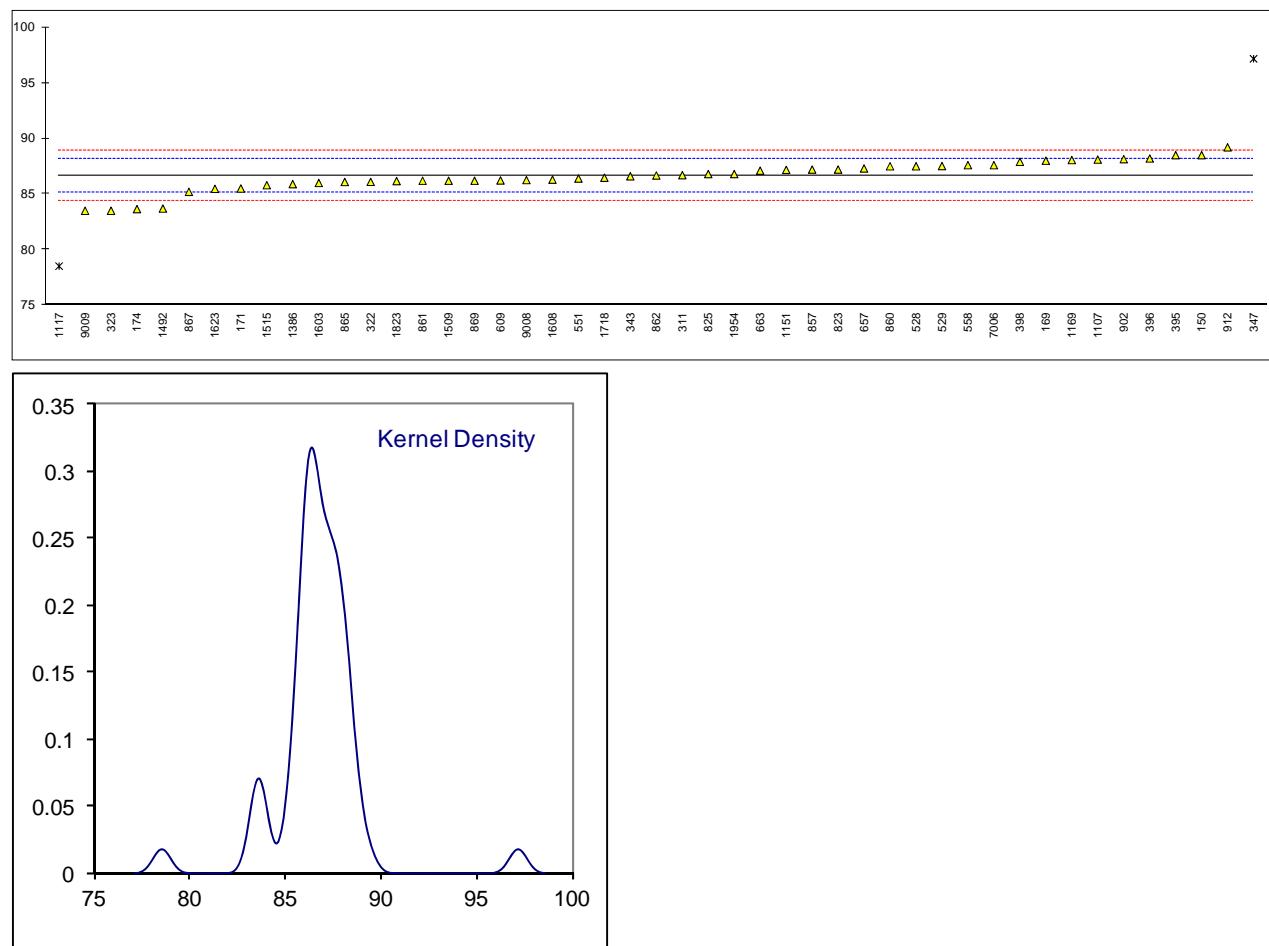
## Determination of UV Transmittance at 350 nm on sample #12131; results in %Transmittance

| lab         | method | value  | mark      | z(targ) | sparged | Cuvet | remarks                              |
|-------------|--------|--------|-----------|---------|---------|-------|--------------------------------------|
| 120         |        | ----   |           | ----    |         |       |                                      |
| 150         | E2193  | 94.1   |           | 2.78    | --      | --    |                                      |
| 169         | E2193  | 93.0   |           | 0.12    | No      | 10    |                                      |
| 171         | E2193  | 92.4   |           | -1.34   | No      | 10    |                                      |
| 173         |        | ----   |           | ----    |         |       |                                      |
| 174         | E2193  | 90.73  |           | -5.39   | No      | 10    |                                      |
| 311         | E2193  | 94.8   |           | 4.48    | No      | 50    |                                      |
| 322         | E2193  | 92.4   |           | -1.34   | No      | 10    |                                      |
| 323         | E2193  | 92.1   |           | -2.07   | No      | 10    |                                      |
| 343         | E2193  | 92.71  |           | -0.59   | No      | 10    |                                      |
| 347         | E2193  | 98.571 | G(0.01)   | 13.63   | No      | 10    |                                      |
| 395         | E2193  | 94.3   |           | 3.27    | No      | 10    |                                      |
| 396         | E2193  | 94.2   |           | 3.03    | No      | 10    |                                      |
| 398         | E2193  | 93.7   |           | 1.81    | No      | 10    |                                      |
| 444         |        | ----   |           | ----    |         |       |                                      |
| 528         | E2193  | 92.85  | C         | -0.25   | --      | --    | Reported 55.05 (switched with 220nm) |
| 529         | E2193  | 93.583 |           | 1.53    | --      | --    |                                      |
| 551         | E2193  | 92.3   |           | -1.58   | No      | 10    |                                      |
| 557         |        | ----   |           | ----    |         |       |                                      |
| 558         | E2193  | 92.5   |           | -1.10   | --      | --    |                                      |
| 609         | E2193  | 92.953 |           | 0.00    | Yes     | 10    |                                      |
| 657         | E2193  | 93.33  |           | 0.92    | No      | 10    |                                      |
| 663         | E2193  | 93.0   | C         | 0.12    | Yes     | 10    | First reported 64.8                  |
| 823         | E2193  | 93.0   |           | 0.12    | No      | 10    |                                      |
| 825         | E2193  | 93.1   |           | 0.36    | --      | --    |                                      |
| 857         | E2193  | 92.9   |           | -0.13   | No      | 10    |                                      |
| 860         | E2193  | 93.4   |           | 1.09    | No      | 10    |                                      |
| 861         | E2193  | 92.5   |           | -1.10   | No      | 10    |                                      |
| 862         | E2193  | 92.74  |           | -0.52   | No      | 50    |                                      |
| 865         | E2193  | 91.66  |           | -3.14   | No      | 10    |                                      |
| 867         | E2193  | 93.2   |           | 0.60    | No      | 10    |                                      |
| 869         | E2193  | 93.2   |           | 0.60    | No      | 10    |                                      |
| 886         |        | ----   |           | ----    |         |       |                                      |
| 902         | E2193  | 93.92  |           | 2.35    | No      | 10    |                                      |
| 912         | E2193  | 94.4   |           | 3.51    | No      | 10    |                                      |
| 913         |        | ----   |           | ----    |         |       |                                      |
| 962         |        | ----   |           | ----    |         |       |                                      |
| 963         |        | ----   |           | ----    |         |       |                                      |
| 1107        | E2193  | 93.7   |           | 1.81    | No      | 10    |                                      |
| 1117        | E2193  | 93.36  |           | 0.99    | No      | 50    |                                      |
| 1151        | E2193  | 93.48  |           | 1.28    | No      | 10    |                                      |
| 1169        | E2193  | 94.06  |           | 2.69    | Yes     | 50    |                                      |
| 1217        |        | ----   |           | ----    |         |       |                                      |
| 1386        | E2193  | 92.242 |           | -1.72   | Yes     | 10    |                                      |
| 1467        |        | ----   |           | ----    |         |       |                                      |
| 1492        | E2193  | 92.50  |           | -1.10   | Yes     | 10    |                                      |
| 1509        | E2193  | 92.35  |           | -1.46   | No      | 50    |                                      |
| 1515        | E2193  | 91.7   |           | -3.04   | No      | 50    |                                      |
| 1603        | E2193  | 93     |           | 0.12    | No      | 10    |                                      |
| 1608        | E2193  | 92.4   |           | -1.34   | No      | 50    |                                      |
| 1623        | E2193  | 91.73  |           | -2.97   | No      | 10    |                                      |
| 1701        |        | ----   |           | ----    |         |       |                                      |
| 1718        | E2193  | 92.47  |           | -1.17   | No      | 50    |                                      |
| 1823        | E2193  | 92.293 |           | -1.60   | No      | 50    |                                      |
| 1866        |        | ----   |           | ----    |         |       |                                      |
| 1915        |        | ----   |           | ----    |         |       |                                      |
| 1954        | E2193  | 92.9   |           | -0.13   | --      | --    |                                      |
| 7006        | E2193  | 93.5   |           | 1.33    | No      | 10    |                                      |
| 9008        | E2193  | 92.46  |           | -1.19   | No      | 10    |                                      |
| 9009        | E2193  | 92.683 |           | -0.65   | No      | 10    |                                      |
| normality   |        | OK     |           |         |         |       |                                      |
| n           |        | 46     |           |         |         |       |                                      |
| outliers    |        | 1      |           |         |         |       |                                      |
| mean (n)    |        | 92.95  |           |         |         |       |                                      |
| st.dev. (n) |        | 0.809  |           |         |         |       |                                      |
| R(calc.)    |        | 2.26   |           |         |         |       |                                      |
| R(E2193:08) |        | 1.15   | unsparged |         |         |       | Compare R(E2193: sparged) = 0.94     |



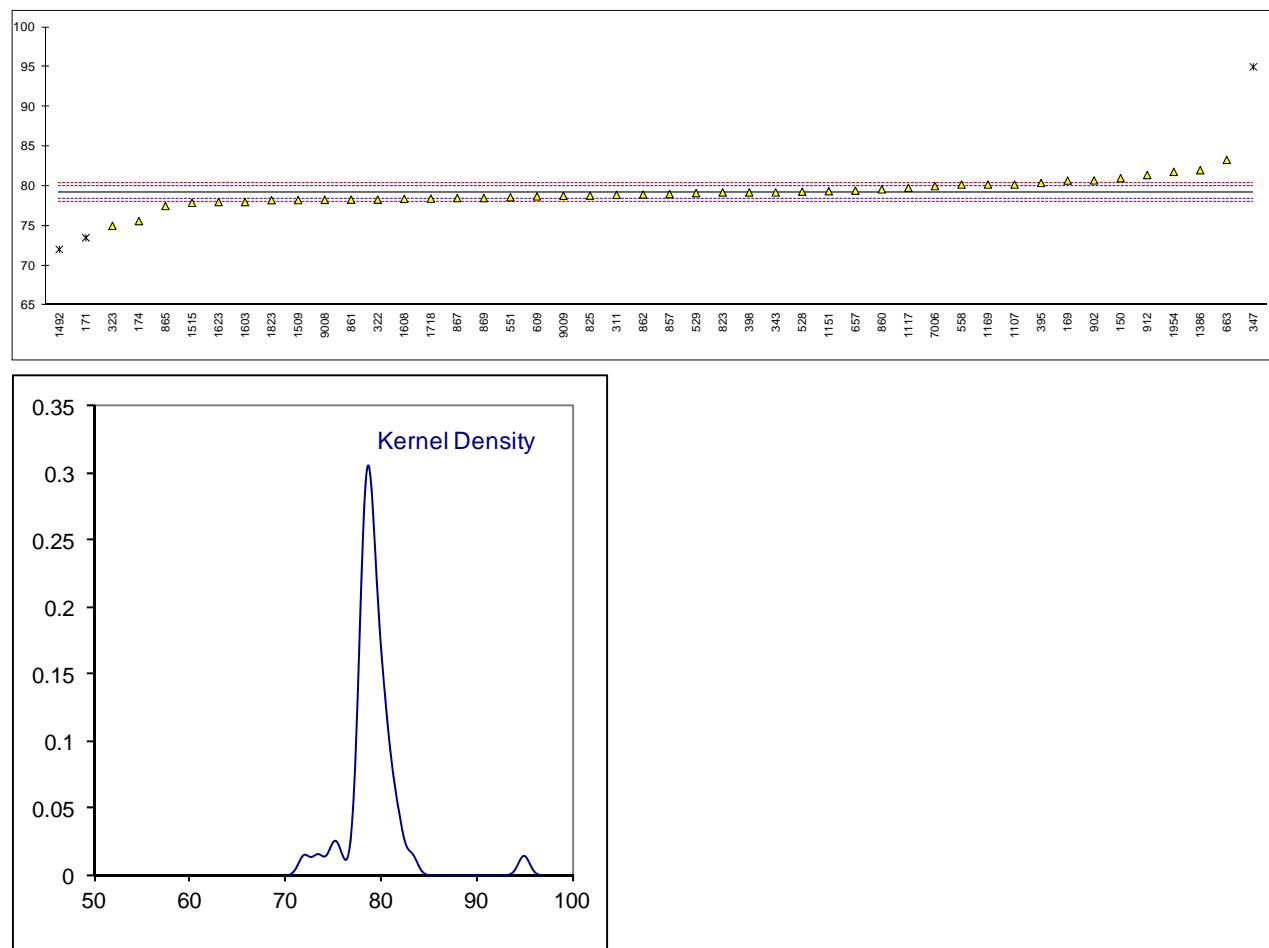
## Determination of UV Transmittance at 275 nm on sample #12131; results in %Transmittance

| lab   | method | value  | mark    | z(targ) | remarks                              |
|---|--------|--------|---------|---------|--------------------------------------|
| 120   |        | ----   |         | ----    |                                      |
| 150   | E2193  | 88.5   |         | 2.48    |                                      |
| 169   | E2193  | 88.0   |         | 1.81    |                                      |
| 171   | E2193  | 85.5   |         | -1.51   |                                      |
| 173   |        | ----   |         | ----    |                                      |
| 174   | E2193  | 83.65  |         | -3.97   |                                      |
| 311   | E2193  | 86.7   |         | 0.08    |                                      |
| 322   | E2193  | 86.1   |         | -0.71   |                                      |
| 323   | E2193  | 83.5   |         | -4.17   |                                      |
| 343   | E2193  | 86.60  |         | -0.05   |                                      |
| 347   | E2193  | 97.162 | G(0.01) | 14.00   |                                      |
| 395   | E2193  | 88.5   |         | 2.48    |                                      |
| 396   | E2193  | 88.2   |         | 2.08    |                                      |
| 398   | E2193  | 87.9   |         | 1.68    |                                      |
| 444   |        | ----   |         | ----    |                                      |
| 528   | E2193  | 87.50  | C       | 1.15    | Reported 79.30 (switched with 250nm) |
| 529   | E2193  | 87.52  |         | 1.17    |                                      |
| 551   | E2193  | 86.4   |         | -0.32   |                                      |
| 557   |        | ----   |         | ----    |                                      |
| 558   | E2193  | 87.6   |         | 1.28    |                                      |
| 609   | E2193  | 86.227 |         | -0.55   |                                      |
| 657   | E2193  | 87.30  |         | 0.88    |                                      |
| 663   | E2193  | 87.1   | C       | 0.62    | First reported 83.3                  |
| 823   | E2193  | 87.2   |         | 0.75    |                                      |
| 825   | E2193  | 86.8   |         | 0.22    |                                      |
| 857   | E2193  | 87.2   |         | 0.75    |                                      |
| 860   | E2193  | 87.5   |         | 1.15    |                                      |
| 861   | E2193  | 86.2   |         | -0.58   |                                      |
| 862   | E2193  | 86.67  |         | 0.04    |                                      |
| 865   | E2193  | 86.09  |         | -0.73   |                                      |
| 867   | E2193  | 85.2   |         | -1.91   |                                      |
| 869   | E2193  | 86.2   |         | -0.58   |                                      |
| 886   |        | ----   |         | ----    |                                      |
| 902   | E2193  | 88.14  |         | 2.00    |                                      |
| 912   | E2193  | 89.2   |         | 3.41    |                                      |
| 913   |        | ----   |         | ----    |                                      |
| 962   |        | ----   |         | ----    |                                      |
| 963   |        | ----   |         | ----    |                                      |
| 1107  | E2193  | 88.1   |         | 1.95    |                                      |
| 1117  | E2193  | 78.52  | G(0.01) | -10.80  |                                      |
| 1151  | E2193  | 87.17  |         | 0.71    |                                      |
| 1169  | E2193  | 88.06  |         | 1.89    |                                      |
| 1217  |        | ----   |         | ----    |                                      |
| 1386  | E2193  | 85.896 |         | -0.99   |                                      |
| 1467  |        | ----   |         | ----    |                                      |
| 1492  | E2193  | 83.70  |         | -3.91   |                                      |
| 1509  | E2193  | 86.20  |         | -0.58   |                                      |
| 1515  | E2193  | 85.8   |         | -1.11   |                                      |
| 1603  | E2193  | 86     |         | -0.85   |                                      |
| 1608  | E2193  | 86.3   |         | -0.45   |                                      |
| 1623  | E2193  | 85.47  |         | -1.55   |                                      |
| 1701  |        | ----   |         | ----    |                                      |
| 1718  | E2193  | 86.47  |         | -0.22   |                                      |
| 1823  | E2193  | 86.166 |         | -0.63   |                                      |
| 1866  |        | ----   |         | ----    |                                      |
| 1915  |        | ----   |         | ----    |                                      |
| 1954  | E2193  | 86.8   |         | 0.22    |                                      |
| 7006  | E2193  | 87.6   |         | 1.28    |                                      |
| 9008  | E2193  | 86.26  |         | -0.50   |                                      |
| 9009  | E2193  | 83.499 |         | -4.17   |                                      |
|   |        |        |         |         |                                      |
| normality   |        |        |         |         |                                      |
| n   |        |        |         |         |                                      |
| outliers  |        |        |         |         |                                      |
| mean (n)  |        |        |         |         |                                      |
| st.dev. (n)   |        |        |         |         |                                      |
| R(calc.)  |        |        |         |         |                                      |
| R(E2193:08)   |        |        |         |         |                                      |
| 2.11                      unsparged                      Compare R(E2193: sparged) = 1.10 |        |        |         |         |                                      |



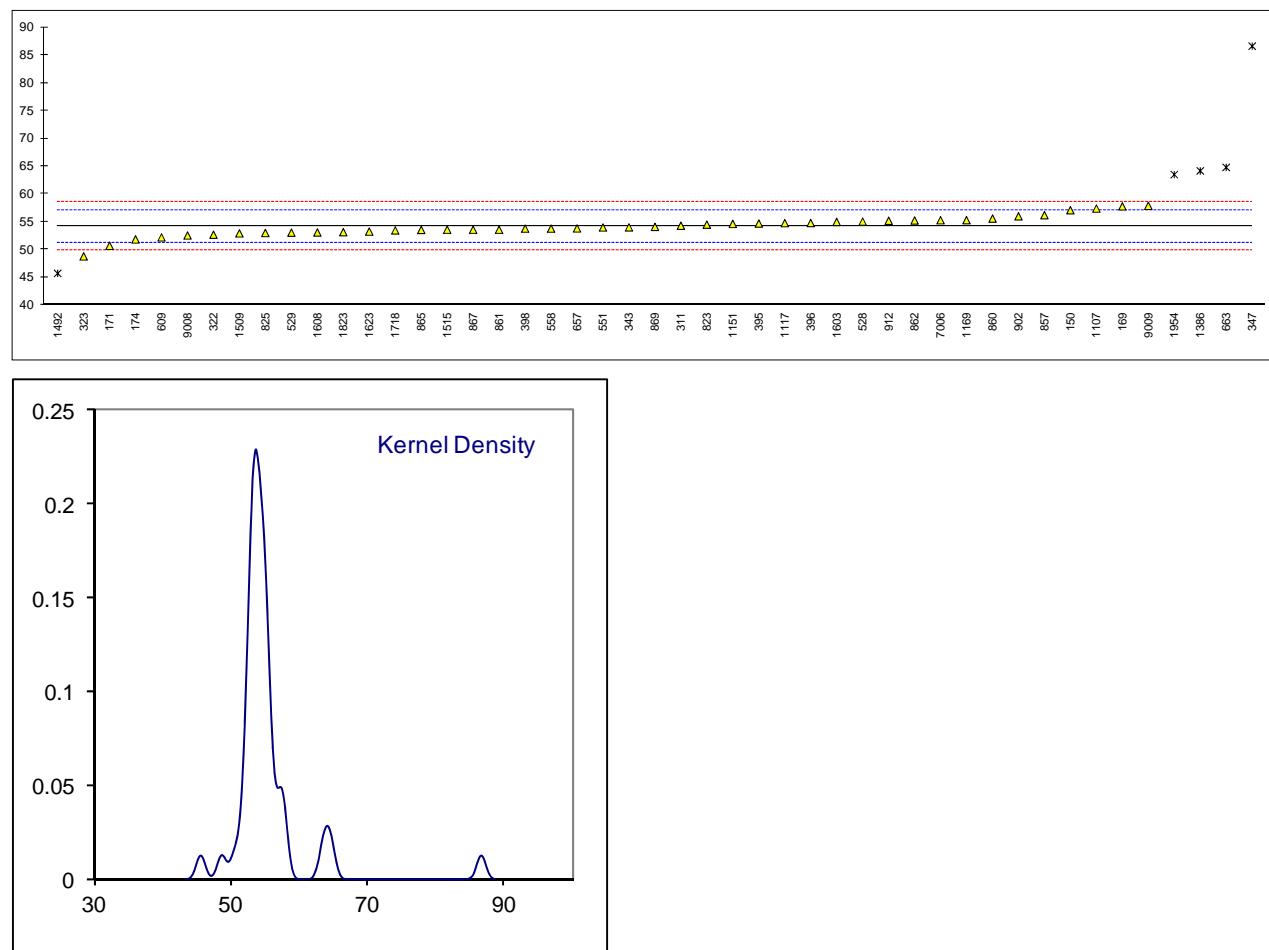
## Determination of UV Transmittance at 250 nm on sample #12131; results in %Transmittance

| lab                              | method | value  | mark    | z(targ) | remarks                              |
|----------------------------------|--------|--------|---------|---------|--------------------------------------|
| 120                              |        | ----   |         | ----    |                                      |
| 150                              | E2193  | 81.0   |         | 4.61    |                                      |
| 169                              | E2193  | 80.7   |         | 3.84    |                                      |
| 171                              | E2193  | 73.5   | G(0.05) | -14.45  |                                      |
| 173                              |        | ----   |         | ----    |                                      |
| 174                              | E2193  | 75.60  |         | -9.11   |                                      |
| 311                              | E2193  | 78.9   |         | -0.73   |                                      |
| 322                              | E2193  | 78.3   |         | -2.25   |                                      |
| 323                              | E2193  | 75.0   |         | -10.64  |                                      |
| 343                              | E2193  | 79.21  |         | 0.06    |                                      |
| 347                              | E2193  | 95.005 | G(0.01) | 40.19   |                                      |
| 395                              | E2193  | 80.4   |         | 3.08    |                                      |
| 396                              | E2193  | ----   |         | ----    |                                      |
| 398                              | E2193  | 79.2   |         | 0.03    |                                      |
| 444                              |        | ----   |         | ----    |                                      |
| 528                              | E2193  | 79.30  | C       | 0.29    | Reported 87.50 (switched with 275nm) |
| 529                              | E2193  | 79.117 |         | -0.18   |                                      |
| 551                              | E2193  | 78.6   |         | -1.49   |                                      |
| 557                              |        | ----   |         | ----    |                                      |
| 558                              | E2193  | 80.2   |         | 2.57    |                                      |
| 609                              | E2193  | 78.706 |         | -1.22   |                                      |
| 657                              | E2193  | 79.43  |         | 0.62    |                                      |
| 663                              | E2193  | 83.3   | C       | 10.45   | First reported 87.1                  |
| 823                              | E2193  | 79.2   |         | 0.03    |                                      |
| 825                              | E2193  | 78.8   |         | -0.98   |                                      |
| 857                              | E2193  | 79.0   |         | -0.47   |                                      |
| 860                              | E2193  | 79.6   |         | 1.05    |                                      |
| 861                              | E2193  | 78.3   |         | -2.25   |                                      |
| 862                              | E2193  | 78.94  |         | -0.63   |                                      |
| 865                              | E2193  | 77.50  |         | -4.29   |                                      |
| 867                              | E2193  | 78.5   |         | -1.75   |                                      |
| 869                              | E2193  | 78.5   |         | -1.75   |                                      |
| 886                              |        | ----   |         | ----    |                                      |
| 902                              | E2193  | 80.72  |         | 3.90    |                                      |
| 912                              | E2193  | 81.4   |         | 5.62    |                                      |
| 913                              |        | ----   |         | ----    |                                      |
| 962                              |        | ----   |         | ----    |                                      |
| 963                              |        | ----   |         | ----    |                                      |
| 1107                             | E2193  | 80.2   |         | 2.57    |                                      |
| 1117                             | E2193  | 79.79  |         | 1.53    |                                      |
| 1151                             | E2193  | 79.36  |         | 0.44    |                                      |
| 1169                             | E2193  | 80.20  |         | 2.57    |                                      |
| 1217                             |        | ----   |         | ----    |                                      |
| 1386                             | E2193  | 82.002 |         | 7.15    |                                      |
| 1467                             |        | ----   |         | ----    |                                      |
| 1492                             | E2193  | 72.04  | G(0.05) | -18.16  |                                      |
| 1509                             | E2193  | 78.25  |         | -2.38   |                                      |
| 1515                             | E2193  | 77.9   |         | -3.27   |                                      |
| 1603                             | E2193  | 78     |         | -3.02   |                                      |
| 1608                             | E2193  | 78.4   |         | -2.00   |                                      |
| 1623                             | E2193  | 77.99  |         | -3.04   |                                      |
| 1701                             |        | ----   |         | ----    |                                      |
| 1718                             | E2193  | 78.43  |         | -1.92   |                                      |
| 1823                             | E2193  | 78.234 |         | -2.42   |                                      |
| 1866                             |        | ----   |         | ----    |                                      |
| 1915                             |        | ----   |         | ----    |                                      |
| 1954                             | E2193  | 81.8   |         | 6.64    |                                      |
| 7006                             | E2193  | 80.0   |         | 2.07    |                                      |
| 9008                             | E2193  | 78.28  |         | -2.30   |                                      |
| 9009                             | E2193  | 78.775 |         | -1.05   |                                      |
| <br>                             |        |        |         |         |                                      |
| normality                        |        |        |         |         |                                      |
| n                                |        |        |         |         |                                      |
| outliers                         |        |        |         |         |                                      |
| mean (n)                         |        |        |         |         |                                      |
| st.dev. (n)                      |        |        |         |         |                                      |
| R(calc.)                         |        |        |         |         |                                      |
| R(E2193:08)                      |        |        |         |         |                                      |
| unsparged                        |        |        |         |         |                                      |
| Compare R(E2193: sparged) = 2.06 |        |        |         |         |                                      |



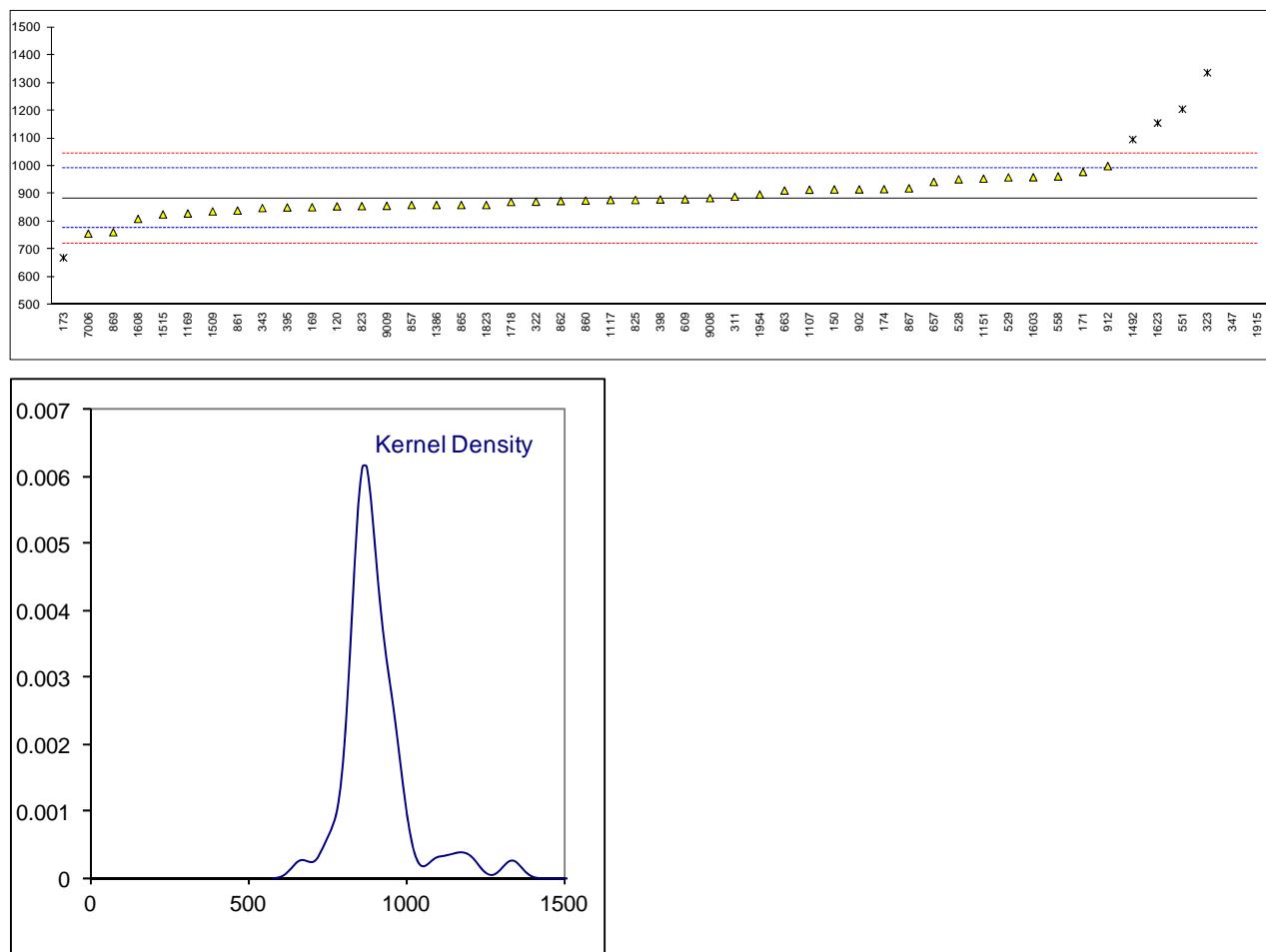
## Determination of UV Transmittance at 220 nm on sample #12131; results in %Transmittance

| lab  | method      | value  | mark      | z(targ) | remarks                              |
|------|-------------|--------|-----------|---------|--------------------------------------|
| 120  |             | ----   |           | ----    |                                      |
| 150  | E2193       | 57.1   |           | 2.02    |                                      |
| 169  | E2193       | 57.8   |           | 2.50    |                                      |
| 171  | E2193       | 50.7   |           | -2.41   |                                      |
| 173  |             | ----   |           | ----    |                                      |
| 174  | E2193       | 51.85  |           | -1.61   |                                      |
| 311  | E2193       | 54.3   |           | 0.08    |                                      |
| 322  | E2193       | 52.7   |           | -1.02   |                                      |
| 323  | E2193       | 48.8   |           | -3.72   |                                      |
| 343  | E2193       | 54.01  |           | -0.12   |                                      |
| 347  | E2193       | 86.596 | G(0.01)   | 22.43   |                                      |
| 395  | E2193       | 54.7   |           | 0.36    |                                      |
| 396  | E2193       | 54.8   |           | 0.43    |                                      |
| 398  | E2193       | 53.8   | C         | -0.26   | First reported 93.8                  |
| 444  |             | ----   |           | ----    |                                      |
| 528  | E2193       | 55.05  | C         | 0.60    | Reported 92.85 (switched with 350nm) |
| 529  | E2193       | 53.063 |           | -0.77   |                                      |
| 551  | E2193       | 54.0   |           | -0.12   |                                      |
| 557  |             | ----   |           | ----    |                                      |
| 558  | E2193       | 53.8   |           | -0.26   |                                      |
| 609  | E2193       | 52.218 |           | -1.36   |                                      |
| 657  | E2193       | 53.83  |           | -0.24   |                                      |
| 663  | E2193       | 64.8   | DG(0.01)  | 7.35    | First reported 93.0                  |
| 823  | E2193       | 54.5   |           | 0.22    |                                      |
| 825  | E2193       | 53.0   |           | -0.82   |                                      |
| 857  | E2193       | 56.2   |           | 1.40    |                                      |
| 860  | E2193       | 55.6   |           | 0.98    |                                      |
| 861  | E2193       | 53.6   |           | -0.40   |                                      |
| 862  | E2193       | 55.26  |           | 0.75    |                                      |
| 865  | E2193       | 53.55  |           | -0.44   |                                      |
| 867  | E2193       | 53.6   |           | -0.40   |                                      |
| 869  | E2193       | 54.1   |           | -0.06   |                                      |
| 886  |             | ----   |           | ----    |                                      |
| 902  | E2193       | 56.01  |           | 1.27    |                                      |
| 912  | E2193       | 55.2   |           | 0.71    |                                      |
| 913  |             | ----   |           | ----    |                                      |
| 962  |             | ----   |           | ----    |                                      |
| 963  |             | ----   |           | ----    |                                      |
| 1107 | E2193       | 57.4   |           | 2.23    |                                      |
| 1117 | E2193       | 54.78  |           | 0.42    |                                      |
| 1151 | E2193       | 54.64  |           | 0.32    |                                      |
| 1169 | E2193       | 55.31  |           | 0.78    |                                      |
| 1217 |             | ----   |           | ----    |                                      |
| 1386 | E2193       | 64.167 | DG(0.01)  | 6.91    |                                      |
| 1467 |             | ----   |           | ----    |                                      |
| 1492 | E2193       | 45.74  | G(0.05)   | -5.84   |                                      |
| 1509 | E2193       | 52.94  |           | -0.86   |                                      |
| 1515 | E2193       | 53.6   |           | -0.40   |                                      |
| 1603 | E2193       | 55     |           | 0.57    |                                      |
| 1608 | E2193       | 53.1   |           | -0.75   |                                      |
| 1623 | E2193       | 53.26  |           | -0.64   |                                      |
| 1701 |             | ----   |           | ----    |                                      |
| 1718 | E2193       | 53.47  |           | -0.49   |                                      |
| 1823 | E2193       | 53.161 |           | -0.70   |                                      |
| 1866 |             | ----   |           | ----    |                                      |
| 1915 |             | ----   |           | ----    |                                      |
| 1954 | E2193       | 63.5   | G(0.01)   | 6.45    |                                      |
| 7006 | E2193       | 55.3   |           | 0.77    |                                      |
| 9008 | E2193       | 52.56  |           | -1.12   |                                      |
| 9009 | E2193       | 57.892 |           | 2.57    |                                      |
|      | normality   | OK     |           |         |                                      |
|      | n           | 42     |           |         |                                      |
|      | outliers    | 5      |           |         |                                      |
|      | mean (n)    | 54.18  |           |         |                                      |
|      | st.dev. (n) | 1.763  |           |         |                                      |
|      | R(calc.)    | 4.94   |           |         |                                      |
|      | R(E2193:08) | 4.05   | unsparged |         | Compare R(E2193: sparged) = 9.68     |



## Determination of Water on sample #12131; results in mg/kg

| lab  | method      | value    | mark      | z(targ) | remarks   |
|------|-------------|----------|-----------|---------|---|
| 120  | E1064       | 855      |           | -0.53   |   |
| 150  | E1064       | 916      |           | 0.60    |   |
| 169  | E1064       | 852      |           | -0.59   |   |
| 171  | E1064       | 979      |           | 1.77    |   |
| 173  | E1064       | 670      | CG(0.05)  | -3.96   | Reported 0.0670 (unit error? %M/M instead of mg/kg) |
| 174  | E1064       | 917      | C         | 0.62    | First reported 682                                  |
| 311  | E1064       | 890      |           | 0.12    |   |
| 322  | E1064       | 872      |           | -0.22   |   |
| 323  | E1064       | 1336     | G(0.01)   | 8.38    |   |
| 343  | E1064       | 849      |           | -0.64   |   |
| 347  | E1064       | 1885     | G(0.01)   | 18.56   |   |
| 395  | E1064       | 851.19   |           | -0.60   |   |
| 396  |             | ----     |           | ----    |   |
| 398  | E1064       | 880      |           | -0.07   |   |
| 444  |             | ----     |           | ----    |   |
| 528  | E1064       | 952.38   |           | 1.27    |   |
| 529  | E1064       | 959.56   |           | 1.41    |   |
| 551  | E1064       | 1204.73  | G(0.05)   | 5.95    |   |
| 557  |             | ----     |           | ----    |   |
| 558  | E1064       | 962.7    |           | 1.47    |   |
| 609  | E1064       | 880.61   |           | -0.06   |   |
| 657  | E1064       | 943      |           | 1.10    |   |
| 663  | E1064       | 912      |           | 0.53    |   |
| 823  | E1064       | 856      |           | -0.51   |   |
| 825  | E1064       | 878      |           | -0.10   |   |
| 857  | E1064       | 860      |           | -0.44   |   |
| 860  | E1064       | 876      |           | -0.14   |   |
| 861  | E1064       | 840      |           | -0.81   |   |
| 862  | E1064       | 874      |           | -0.18   |   |
| 865  | E1064       | 860      |           | -0.44   |   |
| 867  | E1064       | 920      |           | 0.67    |   |
| 869  | E1064       | 762.2    |           | -2.25   |   |
| 886  |             | ----     |           | ----    |   |
| 902  | E1064       | 916.2    |           | 0.60    |   |
| 912  | E1064       | 1000     |           | 2.16    |   |
| 913  |             | ----     |           | ----    |   |
| 962  |             | ----     |           | ----    |   |
| 963  |             | ----     |           | ----    |   |
| 1107 | E1064       | 915      |           | 0.58    |   |
| 1117 | D4672       | 878      |           | -0.10   |   |
| 1151 | E1064       | 955      |           | 1.32    |   |
| 1169 | E1064       | 829.4    |           | -1.00   |   |
| 1217 |             | ----     |           | ----    |   |
| 1386 | E1064       | 860      |           | -0.44   |   |
| 1467 |             | ----     |           | ----    |   |
| 1492 | E1064       | 1095.695 | G(0.05)   | 3.93    |   |
| 1509 | E1064       | 837      |           | -0.86   |   |
| 1515 | E1064       | 826      |           | -1.07   |   |
| 1603 | E1064       | 960      |           | 1.42    |   |
| 1608 | E1064       | 810      |           | -1.36   |   |
| 1623 | E203        | 1155.00  | C,G(0.05) | 5.03    | First reported 1181.33.                             |
| 1701 |             | ----     |           | ----    |   |
| 1718 | E1064       | 871.0    |           | -0.23   |   |
| 1823 | E1064       | 860      |           | -0.44   |   |
| 1866 |             | ----     |           | ----    |   |
| 1915 | E1064       | 3777     | G(0.01)   | 53.62   |   |
| 1954 | E203        | 898      |           | 0.27    |   |
| 7006 | E203        | 756.93   |           | -2.35   |   |
| 9008 | E1064       | 884.8    |           | 0.02    |   |
| 9009 | E1064       | 857      |           | -0.49   |   |
|      | normality   | OK       |           |         |   |
|      | n           | 42       |           |         |   |
|      | outliers    | 7        |           |         |   |
|      | mean (n)    | 883.6    |           |         |   |
|      | st.dev. (n) | 53.03    |           |         |   |
|      | R(calc.)    | 148.5    |           |         |   |
|      | R(E1064:12) | 151.1    |           |         | Compare R(E202:12) = 729.5                          |



**APPENDIX 2****Number of participants per country**

2 laboratories in BELGIUM  
3 laboratories in BRAZIL  
1 laboratory in CANADA  
1 laboratory in GERMANY  
4 laboratories in INDIA  
1 laboratory in IRAN  
3 laboratories in ITALY  
2 laboratories in KOREA  
1 laboratory in KUWAIT  
2 laboratories in MALAYSIA  
2 laboratories in MEXICO  
9 laboratories in P.R. of CHINA  
1 laboratory in PAKISTAN  
7 laboratories in SAUDI ARABIA  
3 laboratories in SINGAPORE  
2 laboratories in SPAIN  
1 laboratory in TAIWAN R.O.C.  
1 laboratory in THAILAND  
3 laboratories in THE NETHERLANDS  
1 laboratory in TURKEY  
8 laboratories in U.S.A.  
1 laboratory in UNITED KINGDOM  
1 laboratory in VENEZUELA

## APPENDIX 3

### Abbreviations:

|          |  |
|----------|--|
| C        | = final result after checking of first reported suspect 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                     |
| on db    | = on dry basis   |
| ex       | = excluded from calculations                                   |
| E        | = probably error in calculations                               |
| U        | = probably reported in different unit                          |
| n.a.     | = not applicable   |
| fr.      | = first reported   |
| W        | = withdrawn on request of the participant                      |

### Literature:

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