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ULUSAL METROLOJİ ENSTİTÜSÜ

Certification Report

Multiparameter in Diesel
UME CRM 1501

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ABREVIATIONS

ANOVA	Analysis of variance
CFPP	Cold filter plugging point
ISO	International standards organization
$MS_{between}$	Between-units mean square (ANOVA)
MS_{within}	Within-unit mean square (ANOVA)
RSD	Relative standard deviation
CRM	Certified reference material
SI	Standards internationale
TRaNS	Stratified random sample selection software

SYMBOLS

α	Significance level
n	Number of replicates per unit
s	Standard deviation
s_{bb}	Between-units standard deviation (ANOVA)
$s_{bb,rel}$	Between-units relative standard deviation
s_{wb}	Within-bottle standard deviation (ANOVA)
$s_{wb,rel}$	Within-bottle relative standard deviation
u_{bb}	Standard uncertainty related to possible between-bottle heterogeneity
$u_{\mathrm{bb,rel}}$	Relative standard uncertainty related to possible between-bottle heterogeneity
u^*_{bb}	Standard uncertainty of heterogeneity that can be hidden by method repeatability
$u^*_{bb,rel}$	Relative standard uncertainty of heterogeneity that can be hidden by method repeatability
u_{rect}	Standard uncertainty related to possible between-bottle heterogeneity modelled as rectangular distribution
$u_{\mathrm{rect,rel}}$	Relative standard uncertainty related to possible between-bottle heterogeneity modelled as rectangular distribution
u_{char}	Standard uncertainty related to characterisation
$u_{char,rel}$	Relative standard uncertainty related to characterisation
u_{lts}	Standard uncertainty related to long term stability
$u_{lts,rel}$	Relative standard uncertainty related to long term stability
u_{sts}	Standard uncertainty related to short term stability
$u_{sts,rel}$	Relative standard uncertainty related to short term stability
$\bar{t}$	Mean of the time points
t_α	Two-tail critical *t* value (t test)
t_i	Time point for each replicate

ABSTRACT

Diesel sold by petrochemical industy should have specifications which are identified in EN 590 and Directive 98/70/EC in Europe, United States Environmental Protection Agency (EPA), Republic of Turkey Energy Market Regulatory and Turkish Standards Institution considering quality [1-2].

The diesel producers and controlling laboratories have to check whether the produced fuel meets the specifications defined in the standards and directives. In order to be sure about the quality of the produced fuel, the equipment and methods used to measure the properties of fuel must be controlled. This control can be done by using certified reference materials.

In this study, it is aimed to produce of a certified reference material intended to be used for the verification of the methods for cold filter plugging point (CFPP), kinematic viscosity and density of diesel according to the ISO Guide 34 [3] and ISO Guide 35 [4].

INTRODUCTION

Diesel should have some specifications considering both performance and protection of the environment. These specifications are identified by TS EN 590 in Turkey and by EN 590 and directive 98/70/EC in Europe. The petrochemical industry companies are responsible to meet these specifications in their diesel products [1-2, 5].

In order to determine whether the produced diesel meets the specifications, standard methods are used. Although standard methods help comparability of measurement results between laboratories, they don't guarantee that the laboratories are applying them completely the same. Thus, certified reference materials are necessary to verify the correct use of standard methods performed by the laboratories. For this purpose, TÜBİTAK National Metrology Institute produced a certified reference material which can be used to verify the methods used for measurement of cold filter plugging point (CFPP), kinematic viscosity and density of diesel. Details for the production of the reference material are presented in this report.

Cold filter plugging point is defined as the lowest temperature that a diesel fuel sample can pass through a standard filter at a certain time interval when it is cooled under specified conditions and its unit is centigrade Celsius (°C). Density is defined as the mass of a unit volume of material which is homogenous and its unit is kg/m^3, kinematic viscosity is the resistance of a fluid against flowing and its unit is mm^2/s.

PARTICIPANTS

Activity	Laboratory
Sampling and processing	• TÜBİTAK UME, Ulusal Metroloji Enstitüsü (National Metrology Institute), Gebze Yerleşkesi, Barış Mah. Dr. Zeki Acar Cad. 41470, Gebze, Kocaeli, TURKEY (Processing) • TÜPRAŞ-İzmit Rafinerisi, Güney Mah. Petrol Cad. No:25/1 41780 Körfez/Kocaeli, TURKEY (TS EN/ISO/IEC 17025, Accreditation Certificate No: AB-0113-T) (Sampling)
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Stability study	• TÜBİTAK UME, Ulusal Metroloji Enstitüsü (National Metrology Institute), Gebze Yerleşkesi, Barış Mah. Dr. Zeki Acar Cad. 41470, Gebze, Kocaeli, TURKEY (Storage of Samples) • OMV-POAŞ-Haramidere, OMV POAŞ Haramidere Terminali, Avcılar/İstanbul, TURKEY (TS EN/ISO/IEC 17025, Accreditation Certificate No: AB-0066-T) (Short Term Stability Measurements)

Activity	Laboratory
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MATERIAL PROCESSING

The first step in the processing of the multiparameter in diesel reference material is the cleaning of the 300 L high density polyethylene homogenization tank, filtering equipment and 0.5 L amber glass bottles and their plastic caps. The homogenization tank was first cleaned with detergent and then rinsed with tap water and followed by high purity water (Milli-Q,18.2 MΩ·cm^{-1}) and dried. The filtering equipment and 0.5 L amber glass bottles and their plastic caps were also cleaned first with detergent and then they were rinsed with tap water and high purity water (Milli-Q,18.2 MΩ·cm^{-1}) and dried at 40 °C in a conventional oven equipped with hepa filter. Drying of the tank, filtering equipment and 0.5 L amber glass bottles and their plastic caps were completed in five days.

Raw diesel material meeting the target CFPP, density and kinematic viscosity specifications for the CRM, was obtained from TÜPRAŞ İzmit Petroleum Refinery. Diesel was filtered through 0.45 μm pore size filter (Whatman, 10370019 GF 6) and filled into 300 L high-density polyethylene tank. Material was homogenized by circulating with repetitive pouring and filling of the content. After homogenization, diesel sample was filled into cleaned and dried bottles as approximately 0.5 L. Total 528 units were labeled according to the filling order and placed into the reference temperature +4 °C storage room.

HOMOGENEITY

10 units covering the whole batch were selected with stratified sampling technique. From each selected unit, 2 parallel measurements were performed for each parameter. Measurements were performed under repeatability conditions, using validated methods with a random sequence to distinguish between the possible trends in analytical sequence and filling order. Measurements were performed by the OMV Petrol Ofisi Haramidere Laboratory.

In order to evaluate the homogeneity of the produced material, statistical calculations were performed for the measurement results on density, kinematic viscosity and cold filter plugging point parameters. One-way analysis of variance (ANOVA) was applied for each parameter on total of 20 measurement results.The unimodal distribution of data is an important prerequisite to apply the ANOVA statistical evaluation. Therefore, the distributions of sample averages as well as individual results were checked both for normality using normal probability plots and for unimodality using histograms. For all parameters, the individual results and bottle averages showed approximately normal and unimodal distribution.

The data were statistically checked to see any existence of a trend and/or outlier value. No significant trend was detected for filling order and analytical sequence. One outlying result was found for one of the two measurements results of kinematic viscosity (Grubbs' single test at α = 0.05). Since no technical reasons were identified for the outlying result, data was retained for statistical evaluation.

Using ANOVA, within unit (s_{wb}) and between units (s_{bb}) homogeneity standard deviation values were calculated with the following equations:

$$s_{wb} = \sqrt{MS_{within}} \quad (1)$$

MS_{within}: Within-unit mean square (ANOVA)

s_{wb}, is equivalent to the *s* of the method, provided that subsamples are representative for the whole unit.

$$s_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n}} \quad (2)$$

$MS_{between}$: Mean square between-units

n : Number of replicates per unit

When $MS_{between} < MS_{within}$, s_{bb} cannot be calculated. Instead, u^*_{bb}, the heterogeneity that can be hidden by the method repeatability, is calculated, according to the equation 3 [6]:

$$u^*_{bb} = \frac{s_{wb}}{\sqrt{n}} \sqrt[4]{\frac{2}{\nu_{MSwithin}}} \quad (3)$$

$\nu_{MSwithin}$: Degrees of freedom of MS_{within}

For the variables that ANOVA were applied, the larger value of s_{bb} or u^*_{bb} is taken as uncertainty contribution for homogeneity, u_{bb} (Table 1).

Table 1. Results of the homogeneity study

Parameter	$s_{wb,rel}$ (%)	$s_{bb,rel}$ (%)	$u^*_{bb,rel}$ (%)	$u_{bb,rel}$ (%)
Cold Filter Plugging Point (°C)	*	*	*	*
Kinematic Viscosity (at 40 °C) (mm^2/s)	0.009	0.011	0.008	0.011
Density (at 15 °C) (kg/m^3)	0.0004	0.0005	0.0003	0.0005

*The uncertainty arised for the homogenity study of cold filter plugging point was found to be negligible.

Even with retention of outliers, the between-unit variation for all parameters is generally low (maximum 0.015 %). The same result was obtained from the measurement of all units for cold filter plugging point parameter and it was observed that there is no contribution to the uncertainty due to homogeneity. Data used for the evaluation of homogeneity are given as graphs in Annex 1.

STABILITY

Stability studies were performed by simulating similar conditions in laboratory as the transfer conditions during the transportation of the material to the customers (short term stability) and storage conditions (long term stability).

18 units for the short term stability and 4 units for long term stability tests were selected using TRaNS (stratified random sampling software).

For short term stability study, test temperatures were selected as +20 °C and +50 °C, for time periods of 1, 2, 3 and 4 weeks. For each temperature and test period, 2 bottles were placed at test cabinets; total 8 units were placed at +20 °C and 8 units were placed at +50 °C. As the reference point, 2 units were directly placed at +4 °C. Right after each test time period, 2 units from each test temperature were transferred to the reference temperature, +4 °C. At the end of 4 weeks, the analysis of all transferred and reference units was performed isochronously. Measurements were performed by the OMV Petrol Ofisi Haramidere Laboratory.

For long term stability study, 4 units were kept at room temperature (20 ± 4) °C for 0 and 21 weeks. Right after 21 weeks at room temperature, units were transferred to +4 °C, reference temperature and analysis of all units was performed isochronously. Measurements were performed by the İnönü University Petroleum Research Laboratory.

Short Term Stability Study Results:

The results obtained from isochronous measurements were first grouped according to the time period and then evaluated for each time point. These evaluations were carried out for both temperatures, separately.

The results were screened for single outliers by applying the Grubbs' test at confidence levels of 95% and 99%. The measured concentration values were plotted against time and the regression lines were calculated to check for significant trends indicating possible changes in the values of the parameters by time. The calculated slope values were tested for significance using a *t*-test, with $t_{\alpha,df}$ being the critical *t*-value (two-tailed) for a significance level α = 0.05 (95% confidence level). Related graphs are given in Annex 2.

One outlying value for kinematic viscosity was identified in the statistical evaluation (Grubbs' test) of the data; nevertheless, as there was no technical reason to exclude this value from evaluation, it remained in the data set. The data points were plotted against storage time at the test temperature and the regression line was calculated. In all cases the slope of the regression line was not found to be significantly different from zero. The data evaluation results for the short-term stability at +20 °C and +50 °C are summarised in Table 2.

Table 2. Results of short term stability tests

Parameter	Is the slope significantly different from zero at test temperature +20 °C at a level of 95% and 99% confidence?	Outlier*	Is the slope significantly different from zero at test temperature +50 °C at a level of 95% and 99% confidence?	Outlier *
Cold Filter Plugging Point (°C)	No	-	No	-
Kinematic Viscosity (at 40 °C) (mm^2/s)	No	One (SGT, 95%)	No	-
Density (at 15 °C) (kg/m^3)	No	-	No	-

*SGT: Single Grubbs' Test

The material was observed to be stable at +20 °C and +50 °C for up to 4 weeks. Thus, it was decided that unless the transfer temperature does not exceed 50 °C and the time does not exceed 4 weeks, the material can be shipped to the customers without any cooling precautions.

Long Term Stability Results:

Shelf life of the produced CRM is determined with the results of the long term stability studies. For evaluation of the long term stability, two bottles were placed at +20 °C for 21 weeks and transferred to reference temperature after the test period. Then, these units were measured isochronously with the reference units which were stored at +4 °C.

The data for each time point was obtained by 2 replicate measurements for each of the two units. Results of the average of 4 measurements for each time point is given in Annex 3. The error bars on each time point is calculated as the standard deviation of the 4 measurement results.

Grubbs' test did not yield any outlier data for cold filter plugging point, density and kinematic viscosity.

Obtained values were plotted against time and the regression line was calculated. The long term stability uncertainty, u_{lts}, of the material is then calculated with the following equation 4 [7]

$$u_{lts} = \frac{RSD}{\sqrt{\sum (t_i - \bar{t})^2}} \times t \qquad (4)$$

where

RSD : The relative standard deviation of all results of the stability study

t_i : Being the time point for each replicate

$\bar{t}$: Being the average of all time points

t : Being the proposed shelf life at +20 °C

The uncertainty contribution, u_{lts} was calculated for 12 months (t) at +20 °C. This uncertainty was one of the three parameters of the overall uncertainty budget of the certified values. Results are given in Table 3. The graphs for long term stability are given in Annex 3.

Table 3. Results of the long-term stability tests of 12 months

Parameter	Is slope significantly different from zero at +20 °C*?	$u_{lts,rel}$[%] for shelf-life of 12 months at +20 °C
Cold Filter Plugging Point (°C)	No	3.49
Kinematic Viscosity (at 40 °C) (mm^2/s)	No	0.12
Density (at 15 °C) (kg/m^3)	No	0.0011

* Data are evaluated at confidence level of 95 %

Based on the obtained results, material was found to be stable to store at +20 °C for 12 months. In addition, to ensure stability beyond the initial shelf life, it will be re-evaluated in certain periods, based on the results of regular post-certification monitoring.

CHARACTERISATION

According to ISO Guide 34, the characterization and the value assignment can be carried out in different ways. In this project, the characterisation of cold filter plugging point (CFPP), kinematic viscosity (at 40 °C) and density (at 15 °C) parameters of material was carried out by an interlaboratory comparison exercise. The selection criterias for the participating laboratories included their expertise in the analysis of diesel samples for the target parameters, accreditation for the same or similar samples and successful participation in proficiency testing schemes in the relevant field and the presence of laboratory quality management system. National laboratories were contacted for this study. All selected laboratories had accreditation according to ISO/IEC 17025 [8] for analysis of the target parameters in diesel. Laboratories were asked to use validated/verified methods.

Each laboratory were sent two units of samples which were selected by TRaNS from the whole batch of samples to represent the whole set. Laboratories were requested to treat each unit as two separate samples. Each laboratory was asked to report 2 independent measurement results. Each laboratory reported 4 independent measurement results for two sample units, together their associated measurement uncertainty values and the approach used for the estimation of measurement uncertainty.

The reference materials used for the calibration were also requested from the laboratories in order to assure the traceability of reported results. A list of laboratories with their lab codes and their corresponding methodology used in the assignment is summarized in Table 4.

The uncertainty arising from the characterization study, u_{char} was also taken into account for the calculation of uncertainty on the property value for each parameter. The characterization uncertainty, u_{char}, is calculated according to the equation 5:

$$u_{char}=\frac{SD}{\sqrt{n}} \tag{5}$$

u_{char} : The uncertainty arising from the characterisation

SD : The standard deviation of the mean of the accepted results from participant laboratories

n : The number of accepted results and laboratories participated to the interlaboratory study

Table 4. Methods used by the laboratories

Parameter	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB 10	LAB 11
Cold Filter Plugging Point (°C)	M1	M1	M1	M1	M1	M1	M1	M1	M1	M1	
Kinematic Viscosity (at 40 °C) (mm^2/s)	M2	M2	M2	M2	M2	M2	M2	M2	M2	M2	M2
Density (at 15 °C) (kg/m^3)	M3	M3	M3	M3	M3	M3	M3	M3	M3	M3	M3

M1: EN 116
M2: EN ISO 3104/T1
M3: EN ISO 12185

PROPERTY VALUE AND UNCERTAINTY ASSIGNMENT

Datasets from laboratories were received and technically evaluated. A set of data were rejected if a laboratory reported a technical problem about their measurement. Data were also rejected if both of the following criteria were fulfilled at the same time:

- Presence of outlier in Grubbs' test (99 %)
- If the relative standard deviation of 4 independent results from the lab is greater than the relative standard deviation of the results by all laboratories

Datasets were technically evaluated for each parameter and RSD of results reported by all laboratories were obtained as 4% for cold fitler plugging point, 0.14 % for kinematic viscosity (at 40 °C) and 0.01 % for density (at 15 °C). After technical evaluation, it was decided to include all reported data for the final calculation. Graphs for characterisation data are given in Annex 4.

The certified values calculated from the characterisation study are given in Table 5. All the certified values reported are calculated as the mean of the laboratory means of all the accepted data sets.

The uncertainty of the certified values contains contribution of the characterisation u_{char}, the homogeneity u_{bb} and the long-term stability u_{lts}.

These different parameters were combined using equation 6 to calculate the CRM uncertainty:

$$U_{CRM} = k \cdot \sqrt{u_{char}^2 + u_{bb}^2 + u_{lts}^2} \quad (6)$$

The expanded uncertainty of the certified value U_{CRM} is calculated with a coverage factor of k = 2, representing a confidence level of approximately 95%. Expanded uncertainties reported by the laboratories whose results were accepted are given with the graphs in Appendix 4. The certified values and uncertainties are summarised in Table 5 and the % contribution of each parameter to the uncertainties are given in Table 6.

Table 5. The certified values and their uncertainties

Parameter	Certified Value	U_{CRM}	$u_{bb,rel}$ (%)	$u_{lts,rel}$ (%)	$u_{char,rel}$ (%)	n_{char}	$U_{CRM,rel}$ (%)
Cold Filter Plugging Point	-21.1 °C	1.6 °C	0.00	3.49	1.26	10	7.6
Kinematic Viscosity (at 40 °C)	2.506 mm^2/s	0.007 mm^2/s	0.011	0.124	0.043	11	0.264
Density (at 15 °C)	825.82 kg/m^3	0.05 kg/m^3	0.001	0.001	0.003	11	0.007

Table 6. Percent contribution of each uncertainty parameter to U_{CRM}.

Parameter	$u_{bb,rel}$ (%)	$u_{lts,rel}$ (%)	$u_{char,rel}$ (%)
Cold Filter Plugging Point (°C)	0.0	73.4	26.6
Kinematic Viscosity (at 40 °C)	6.3	69.6	24.2
Density (at 15 °C)	11.2	24.5	64.2

COMMUTABILITY

Commutability is the mathematical relationship of the equation between the reference material and the results produced by the different measurement methods that can be used to measure the routine samples it represents [9]. In this study the measurement results for all parameters are method dependent obtained different measurement methods were not applied for any of the measured parameters.

The diesel used for production of the reference material was obtained from common fuel supplier therefore it can be accepted as representative for similar routine diesel samples.

TRACEABILITY

Cold filter plugging point, density and kinematic viscosity are method dependent parameters and can be obtained by strict application of method EN 116 (Cold filter plugging point) [10], EN ISO 3104/T1 (kinematic viscosity) [11] and EN ISO 12185 (density) [12]. The certificate values assigned to the CRM are method dependent and therefore they are operationally defined.

The metrological traceability of the certified values were ensured by using SI traceable tools (calibrators, reference materials) for the calibration of their instruments and equipments by all the laboratories participating to the interlaboratory study.

INSTRUCTIONS FOR USE

Storage conditions

The material should be stored at (20 ± 4) °C, in a dark and clean environment. In order to prevent contamination, it is recommended that the bottle should be opened in a clean environment, pipette or any other material should not be inserted into the bottle and bottle should not be kept as opened for a long time.

Minimum sample intake

Measurements should be performed by sampling the amounts specified in the standard methods. EN 116 [10] for cold filter clogging point, EN ISO 3104/T1 [11] for kinematic viscosity and EN ISO 12185 [12] for density must be taken into account.

Safety precautions

Material contains diesel. It is strongly recommended that the material must be handled and disposed according to the safety guidelines where applicable. All safety precautions, e.g. working in a fume hood and or using suitable masks, must be taken. All precautions for flammable materials are also valid for this material. Please refer to the Safety Datasheet before any use of the material.

REFERENCES

[1] CEN EN 590, Automotive fuels - Diesel - Requirements and test methods, 2013

[2] Directive 98/70/EC, Quality of petrol and diesel fuels, 1998.

[3] ISO Guide 34, General requirements for the competence of reference material producers, 2009

[4] ISO Guide 35, Reference materials - General and statistical principles for certification, 2006

[5] TS EN 590, Automotive fuels - Diesel - Requirements and test methods, 2014.

[6] T. P. J. Linsinger, J. Pauwels, A. M. H. Van der Veen, H. Schimmel, A. Lamberty, Homogeneity and stability of reference materials, Accred. Qual. Assur. 6, 20–25, 2001.

[7] T. P. J. Linsinger, J. Pauwels, A. Lamberty, H. Schimmel, A. M. H. van der Veen, L. Siekmann, Estimating the uncertainty of stability for matrix CRMs, Fresenius J Anal. Chem. 370, 183–188, 2001.

[8] ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories, 2005

[9] H. Vesper, H. Emons, M. Gnezda, C. P. Jain, W. G. Miller, R. Rej, G. Schumann, J. Tate, L. Thienpont, J. E. Vaks, Characterization and Qualification of Commutable Reference Materials for Laboratory Medicine; Approved Guideline, CLSI document C53-A, Clinical and Laboratory Standards Institute, Wayne, PA, USA, 2010.

[10] TS EN 116, Diesel and domestic heating fuels- Determination of cold filter plugging point, 2015

[11] TS 1451 EN ISO 3104/T1, Petroleum products-Transparent and opague liquids- Determination of kinematic viscosity and calculation of dynamic viscosity, 2005

[12] TS EN ISO 12185, Crude petroleum and petroleum products - Determination of density - Oscillating U- tube method, 2007

REVISION HISTORY

Date	Remarks
23.03.2016	First issue.
23.11.2018	In Table 1 the average values were excluded and $u^*_{bb,rel}$ and $u_{bb,rel}$ values for kinematic viscosity and density were updated. In Table 5 $U_{bb,rel}$, and $U_{CRM,rel}$ values for kinematic viscosity and density and significant digits of certified value and uncertainty of cold fitler plugging point were updated. All values in Table 6 were updated.

Annex 1. Graphs for Homogeneity Study

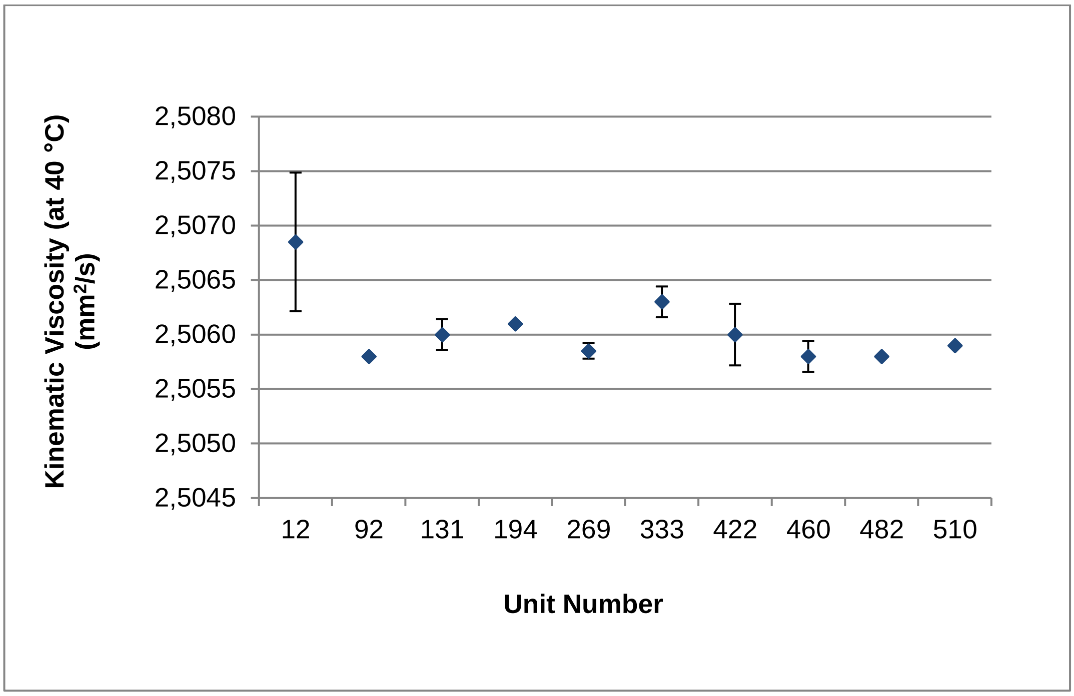

Figure 1. UME CRM 1501 Kinematic Viscosity (at 40 °C), Homogeneity

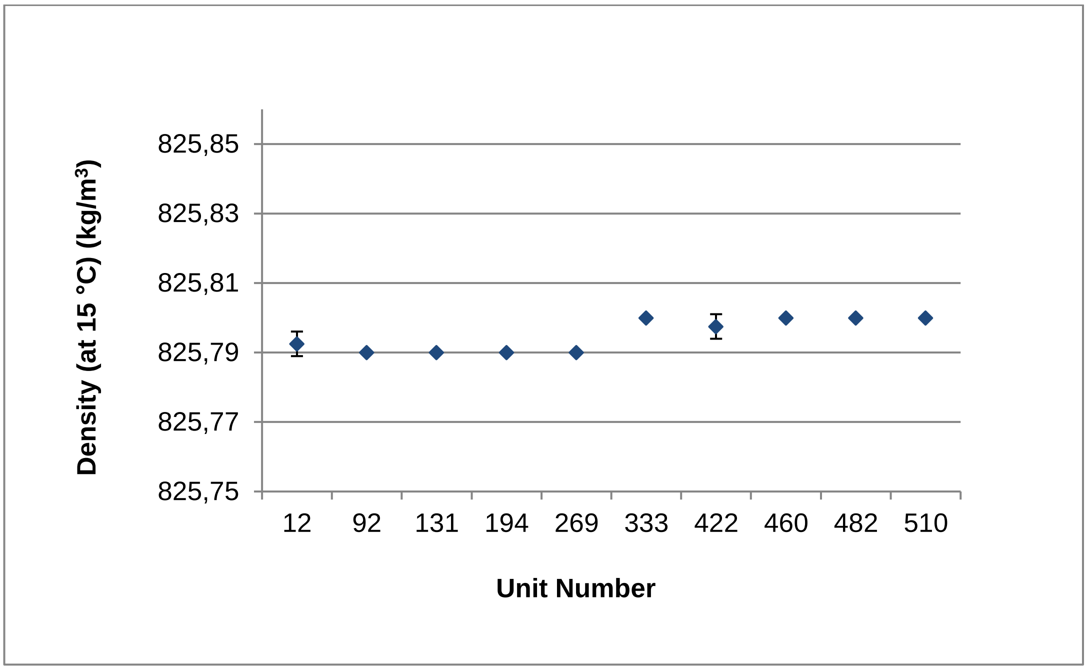

Figure 2. UME CRM 1501 Density (at 15 °C), Homogeneity

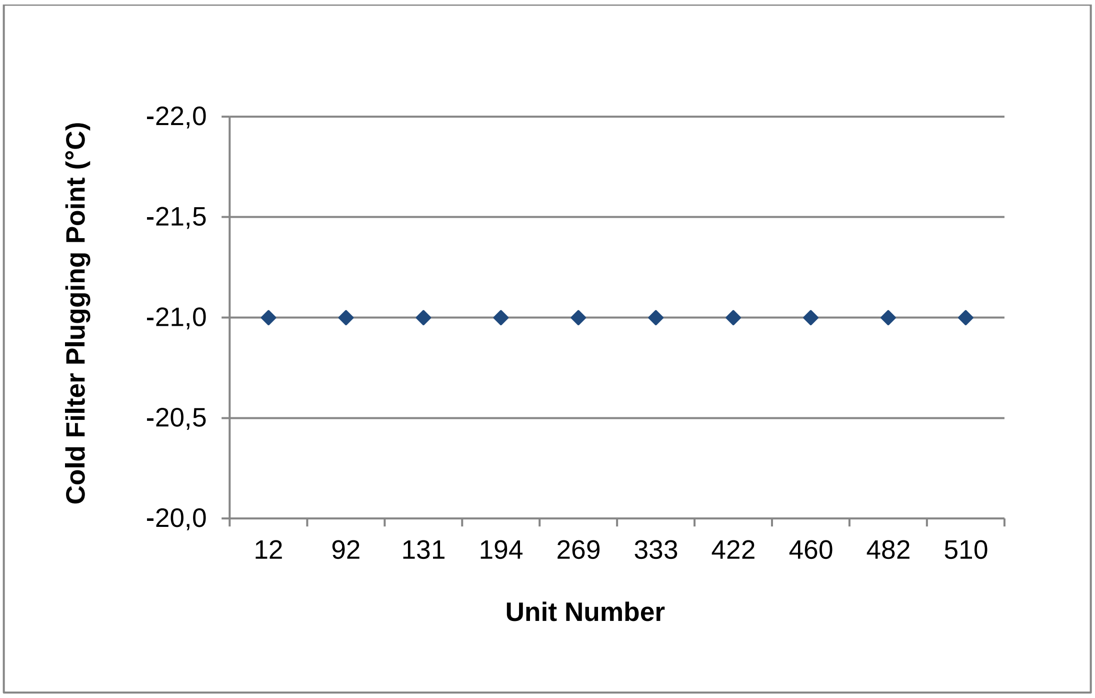

Figure 3. UME CRM 1501 Cold Filter Plugging Point, Homogeneity

Annex 2. Graphs for Short Term Stability Studies

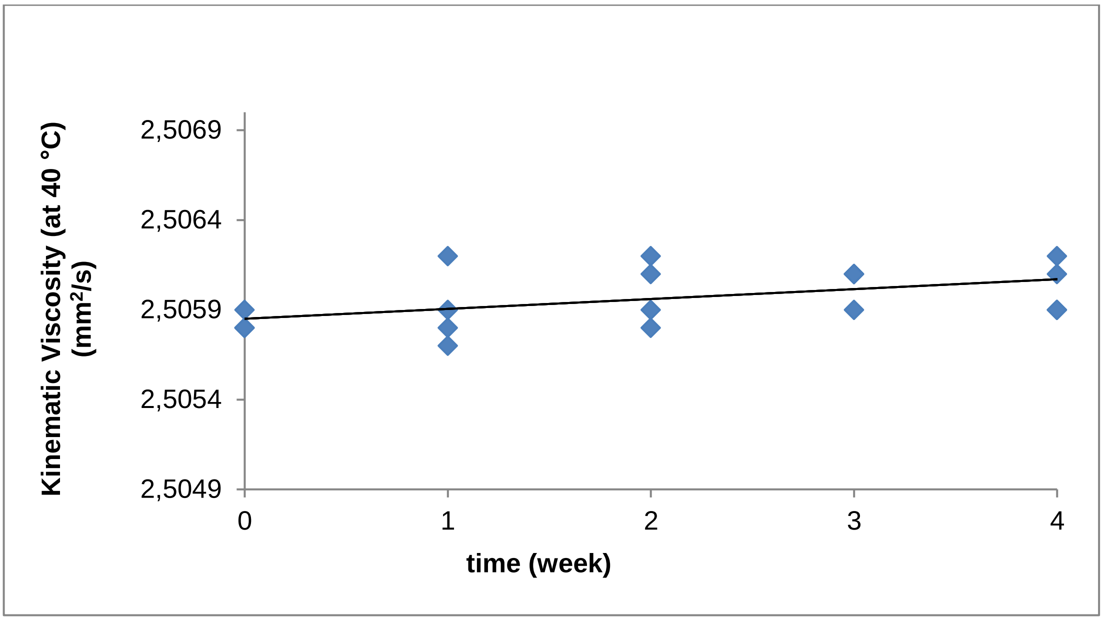

Figure 4. Short Term Stability of UME CRM 1501 for Kinematic Viscosity (at 40 °C) at +20 °C

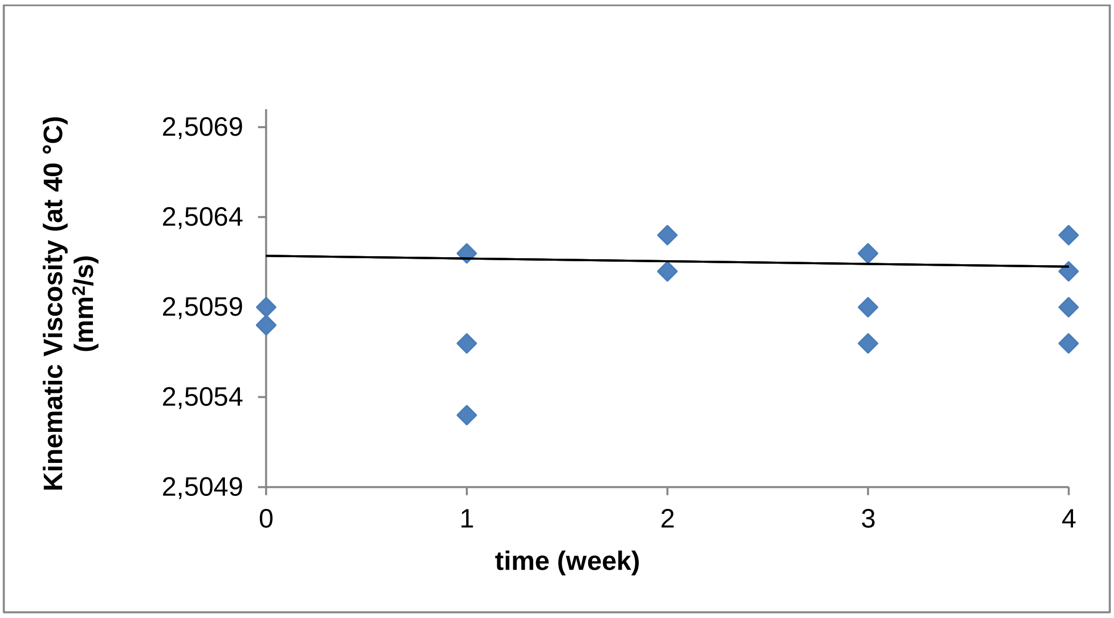

Figure 5. Short Term Stability of UME CRM 1501 for Kinematic Viscosity (at 40 °C) at +50 °C

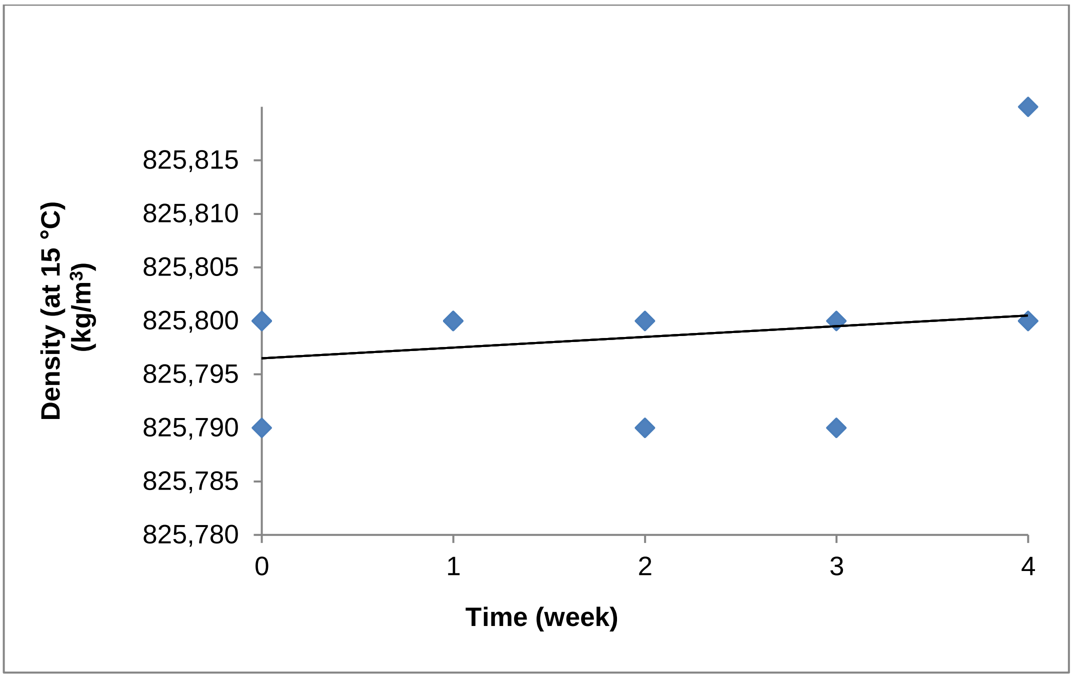

Figure 6. Short Term Stability of UME CRM 1501 for density (at 15 °C) at +20 °C

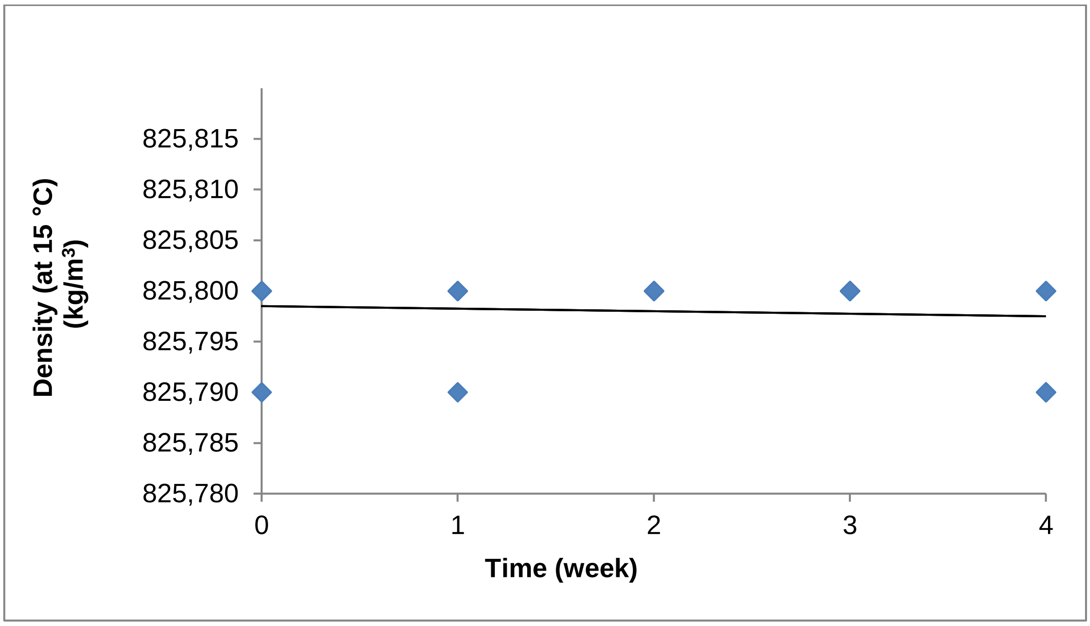

Figure 7. Short Term Stability of UME CRM 1501 for density (at 15 °C) at +50 °C

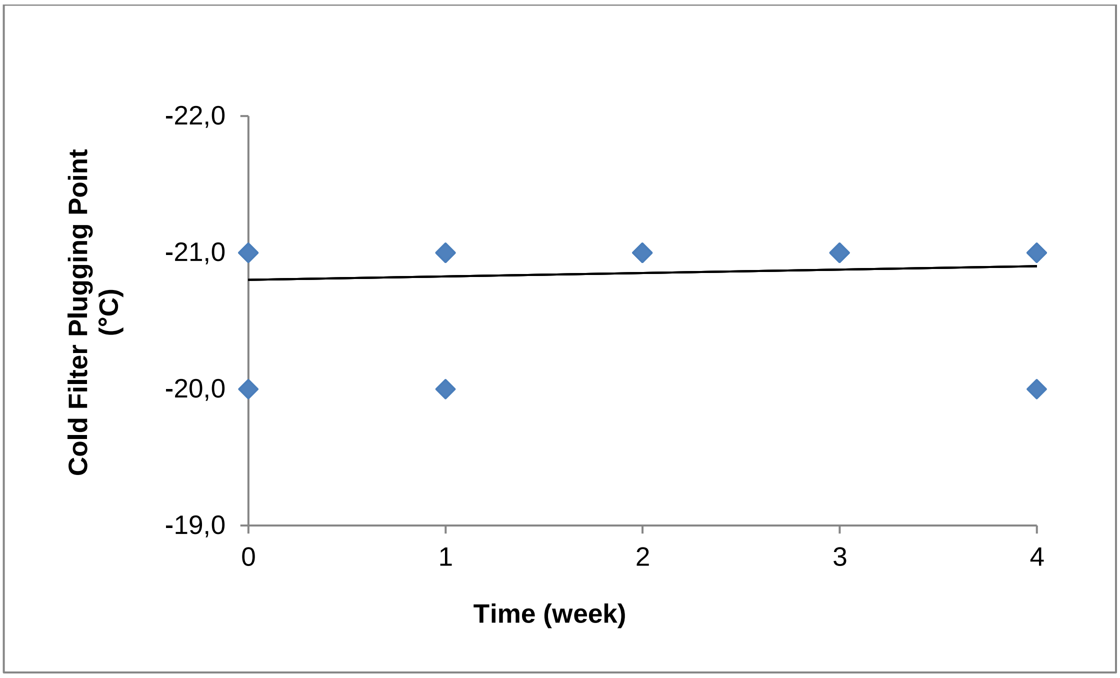

Figure 8. Short Term Stability of UME CRM 1501 for Cold Filter Plugging Point at +20 °C

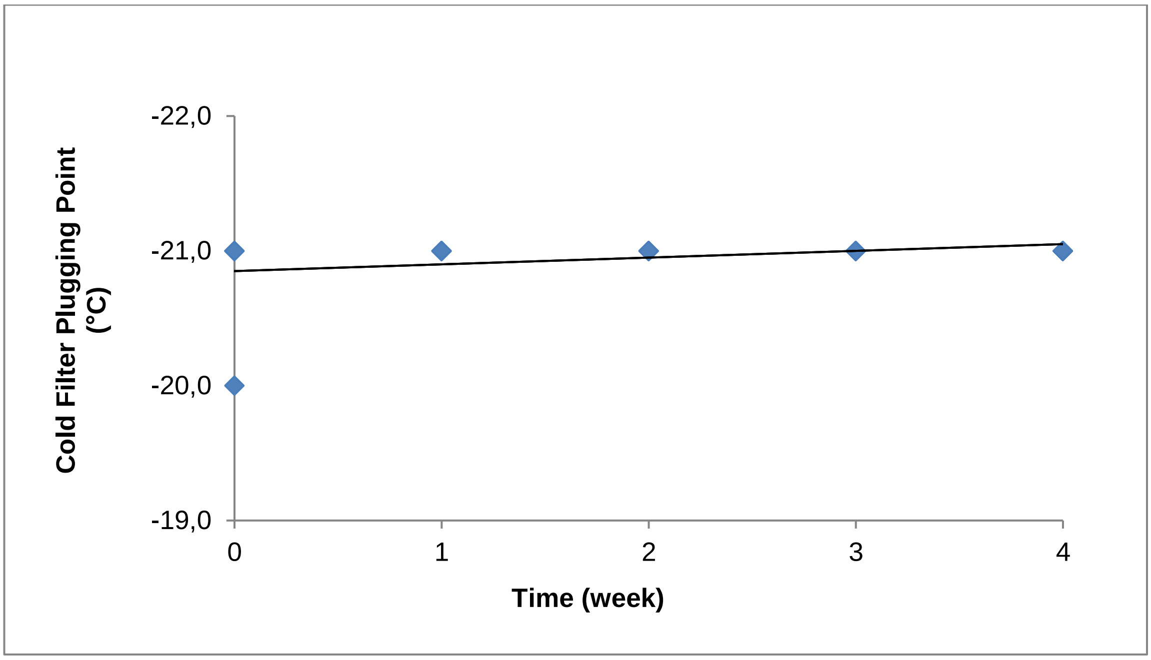

Figure 9. Short Term Stability of UME CRM 1501 for Cold Filter Plugging Point at +50 °C

Annex 3. Graphs for Long Term Stability Studies

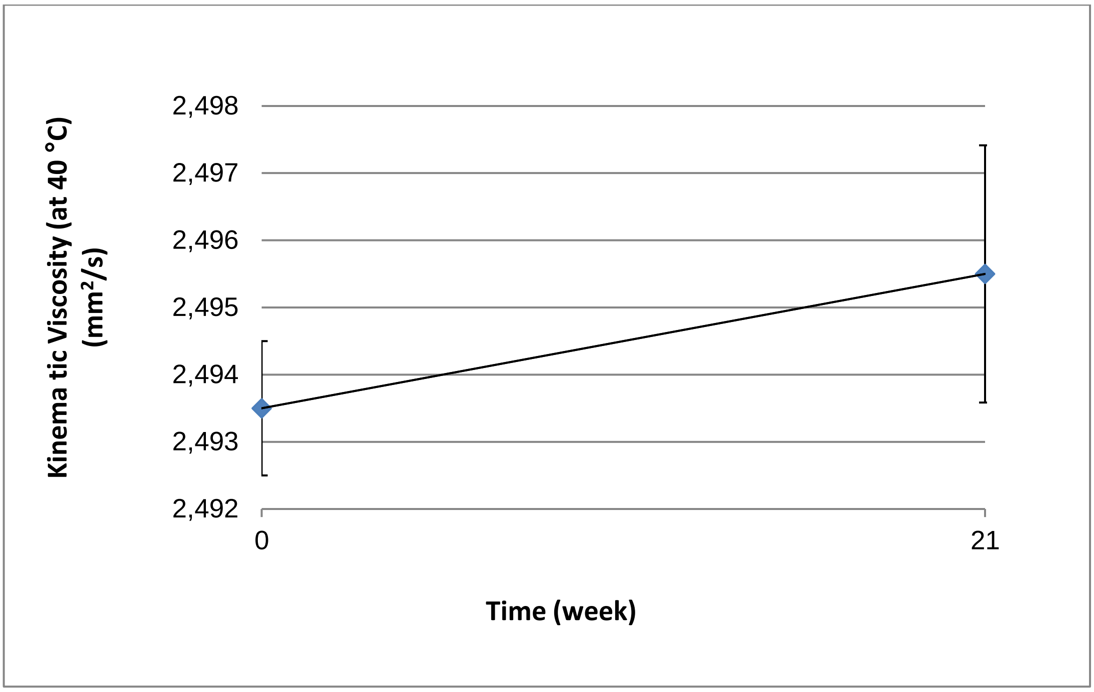

Figure 10. Long Term Stability of UME CRM 1501 of Kinematic Viscosity (at 40 °C) at +20 °C

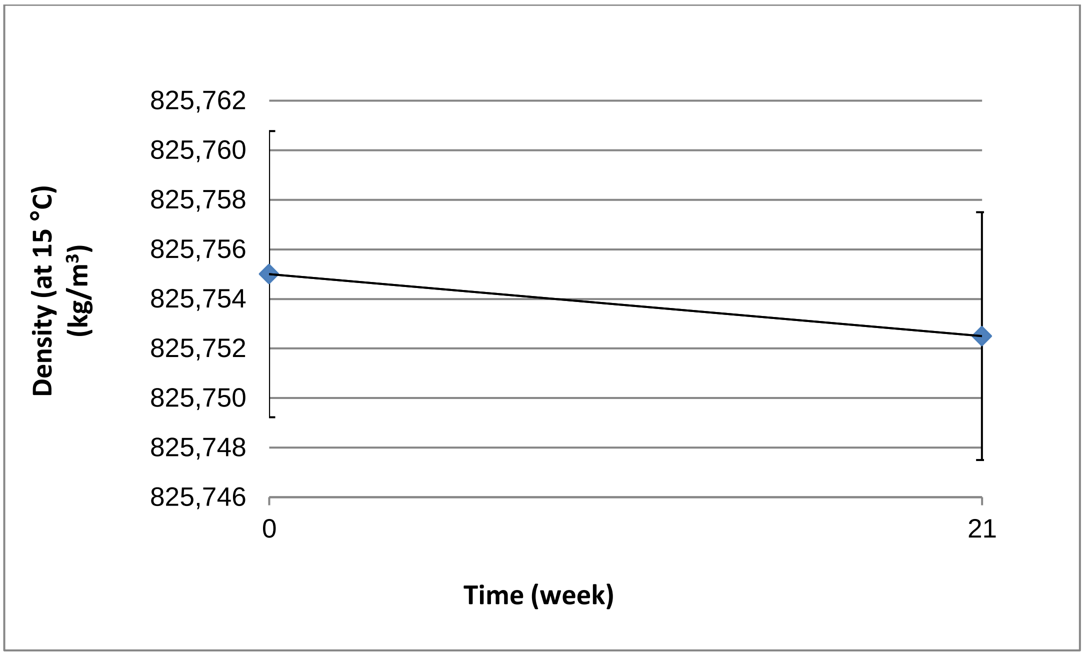

Figure 11. Long Term Stability of UME CRM 1501 of Density (at 15 °C) at +20 °C

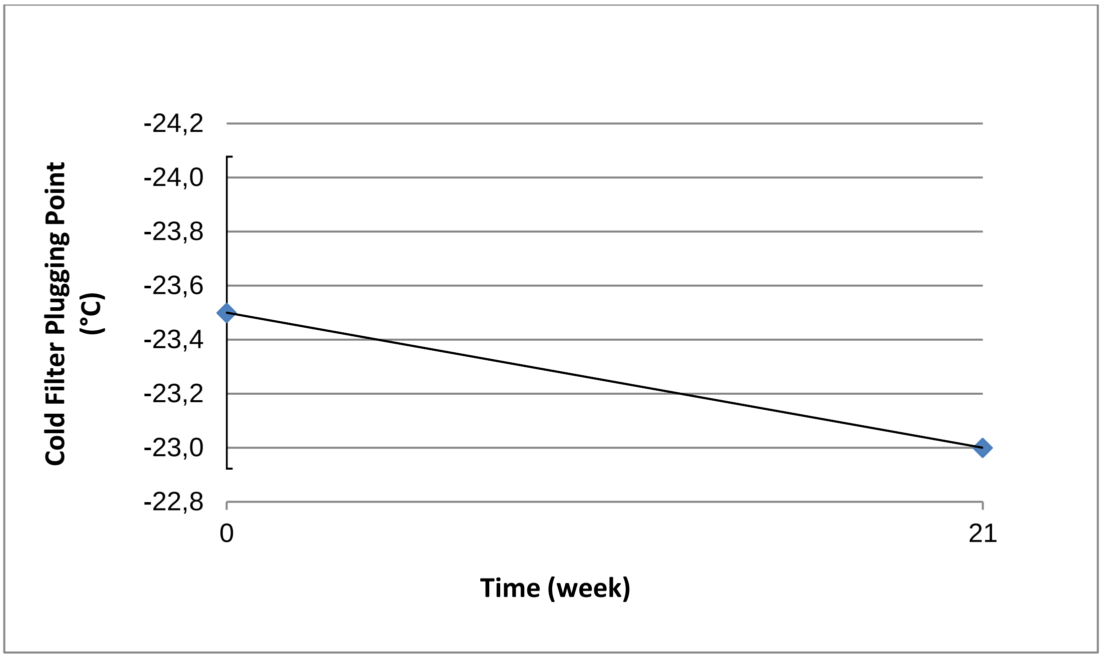

Figure 12. Long Term Stability of UME CRM 1501 for Cold Filter Plugging Point at +20 °C

Annex 4. Graphs for Characterisation Studies

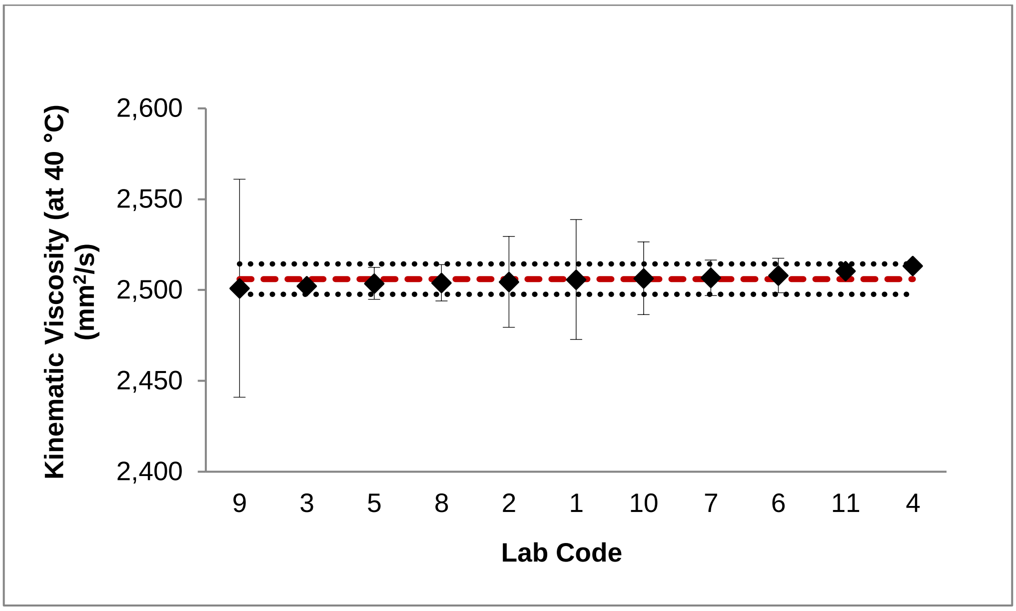

Figure 13. UME CRM 1501 Kinematic Viscosity (at 40 °C), Characterisation

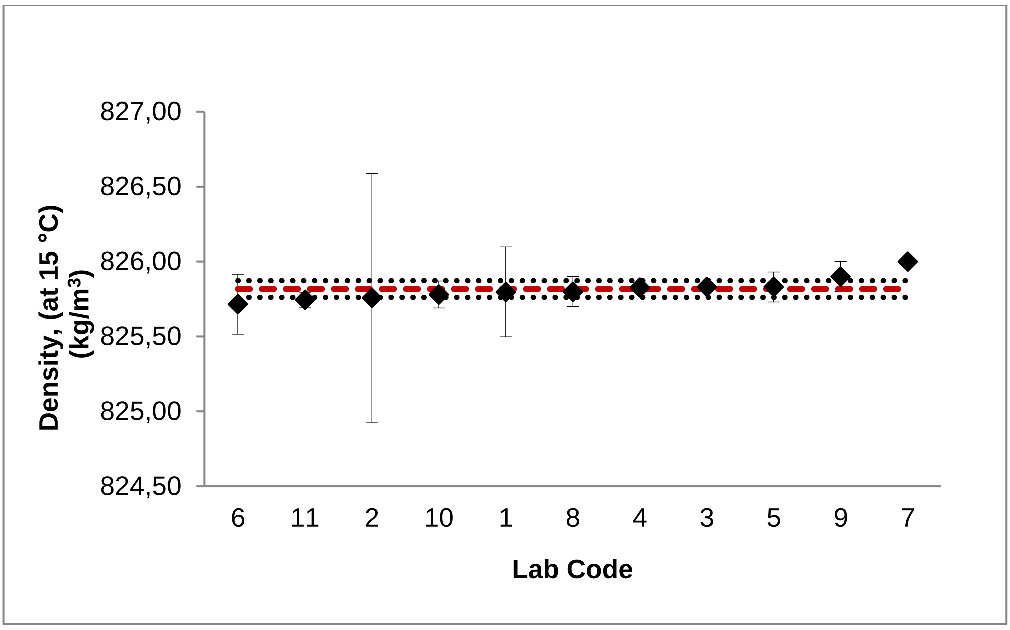

Figure 14. UME CRM 1501 Density (at 15 °C), Characterisation

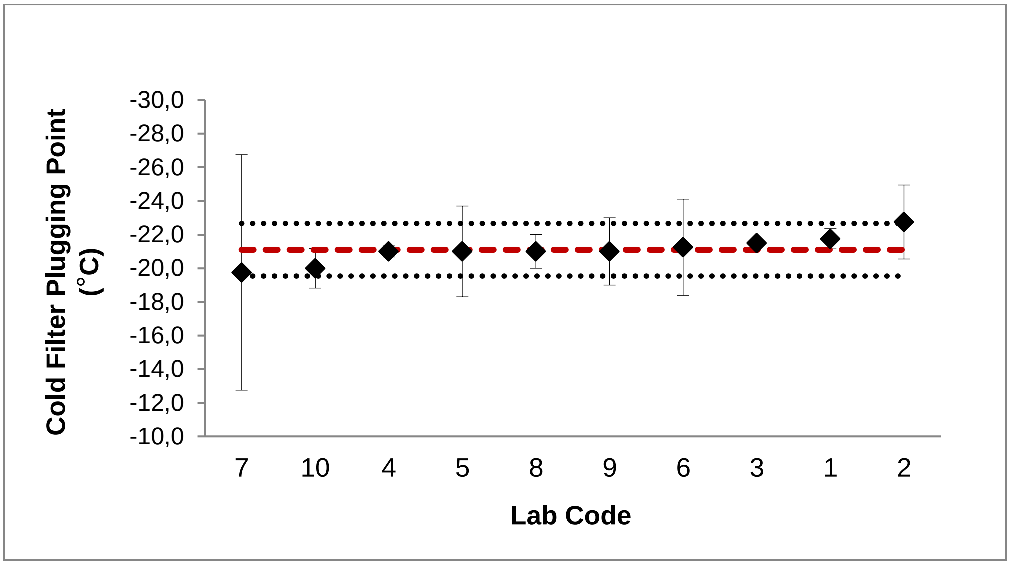

Figure 15. UME CRM 1501 Cold Filter Plugging Point, Characterisation