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Filter loss study: loss of hydrocarbons from filter when sampling and storing oil mist and oil vapour samples

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Safety, Health and Environment
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Problem description

The purpose of the study is to characterize the loss of aerosol and vapour from sampler used when sampling oil mist and oil vapour for 3 different base oils used for drilling.
Main points to be examined:
- Give an overview of and discuss known methods for sampling oil aerosol and oil vapour, methods for separation of particle sizes and the most common methods of analysis for oil aerosols.
- Give an overview of known health effects caused by oil mist and oil vapour.
- Give a characterization of the different oils
- Carry out tests with 3 oils. The is first to be added to the filter and air the drawn through the filter in a cassette.
- Carry out storing tests through 40 days. Main method of analysis is to be gravimetry.
- Following variations in storing is to be tested:
  - With or without plug when storing (in exicator)
  - Filter in sampling cassette or in bowl in exicator
  - Cassette and filter stored at different temperatures (e.g. room temperature, fridge temperature)
- Qualitative and quantitative analysis of base oils of components evaporated during storage.
- Present conclusions about loss of hydrocarbons from sampler during storage

Trondheim – June 2011

Summary

The main purpose of the thesis was to find the best way of storing samples of aerosol from drilling mud on filter.

The patterns of loss depend on the properties of the base oil and the concentration of the sample. Most sample is lost during aspiration for high volatility samples, but the degree of loss during storage has similar patterns. Lower concentration samples loose more than higher concentration samples during aspiration.

Filters should be taken out of the cassette before weighing rather than weighing the whole cassette with filter.

Storage on cellulose acetate plus glass fibre filter combination and on double glass fibre filter have similar patterns, but more sample is lost during aspiration with a double glass fibre filter than with a cellulose acetate plus glass fibre filter combination for EDC Pearl and Sipdrill 2.0.

Samples should be stored at refrigerator temperature for higher concentrations (ca. 70 mg/m^3) and at room temperature for low concentrations (ca. 20 mg/m^3). Plugs do not make much difference.

How long a sample can be stored depends on the volatility of the sample, as high volatility (low viscosity) samples tend to lose more during aspiration.

Definitions

Aerosol: Finely distributed particles of solids, liquid or a mix of solids and liquid in air.

Aerodynamic diameter: Defines the size of the particle, and is dependent on the particle's density, shape and size. It corresponds to the diameter of a spherical particle with density of $1gcm^{-3}$ and with the same rate of fall as the particle of interest.

Inhalable fraction: The mass fraction of the total number of airborne particles that can be inhaled through nose and mouth. These particles have an aerodynamic diameter ≤ 100 μm.

Thoracal fraction: The mass fraction of inhaled particles that can pass the nose and mouth. These particles have an aerodynamic diameter < 30 μm.

Respirable fraction: The mass fraction of inhaled particles that can force its way down to the terminal bronchioli and pulmonary vesicles. These particles have an aerodynamic diameter < 10 μm.

Quantitative analysis: Indicates the *amount* of each substance in a sample.

Qualitative analysis: reveals the *identity* of the elements and compounds in a sample.

Analytes: The components of a sample that are to be determined.

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Introduction

1 Drilling mud and exposure in oil exploitation

Drilling mud is used for purposes like lubricating and cooling the drill stem and bit, to provide pressure support in the well and for transporting cuttings to the surface (**Feil! Finner ikke referansekilden.**) (Steinsvåg et al., 2006).

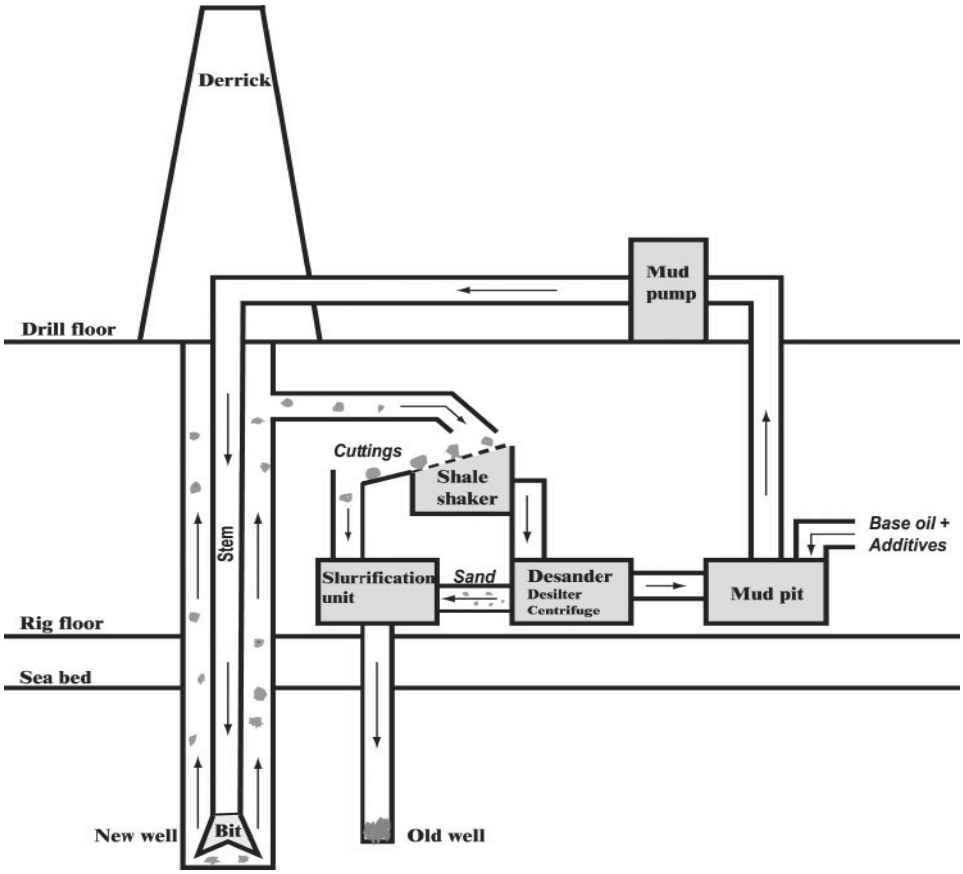

Figure 1-1 The Drilling mud process (Steinsvåg et al., 2006)

First the base oil and additives are mixed in the mud pit. The mud then leaves the pumps at high pressure, flows inside the drill stem, passes through the nozzles of the drill bit, return to the outside of the stem and transports rock cuttings to the platform surface. Vibrating screens and other cleaning equipment separates the solids and liquids. The mud returns to the mud pit and is recycled, while the cuttings and sand are crushed in slurrification units, blended with water and pumped to an old well for storage. (Gardner, 2003; Steinsvåg et al., 2006)

Depending on the system used the mud is a mixture of either oil- or water-based fluids and a large number of additives (Hudgins, 1991,as cited in (Steinsvåg et al., 2006). When using oil-based mud both mist and vapour will form and contaminate the working atmosphere (Malvik & Børresen, 1988). The exposure levels in the working atmosphere is expected to be influenced by factors such as mud temperature, mud flow rate, well length, well section and viscosity of the base oil (Steinsvåg et al., 2006).

According to Steinsvåg et al. (2006) the main area of exposure to oil mist and vapour in oil exploitation are the vibrating screens in the shale shaker area in the drilling mud process. Gardner (2003) suggests that oil droplets are generated by both the vibrating screens of the shale shakers and condensation of vapour.

Steinsvåg et al. (2006) described personal exposure to oil mist and oil vapour from 1979 to 2004 in the mud-handling areas of offshore drilling facilities when drilling with oil-based muds. They found that both oil mist and oil vapour had downward time trends, of 6% per year and 8% per year respectively. Exposure levels were associated with rig type, mud temperature, technical control measures, base oil, viscosity of the base oil, work area and season.

Galea et al. (2010) suggests that cooling the drilling fluid to temperatures below 50^{o}C before it reaches the shale shaker would significantly reduce mist and vapour levels.

The American Conference of Governmental Industrial Hygienists has recommended a threshold limit value of 5 mg/m^3 for oil mist (Knut Skyberg et al., 1986). Skyberg et.al. (1986) also suggests that the epidemiological studies of Ely et.al. and Goldstein et.al. are insufficient as negative evidence of adverse respiratory effects at levels of 5mg/m^3. Some occupational exposure levels for different countries are shown in Table 1-1.

Table 1-1 Occupational exposure levels (OEL) for 12h work shift

	Oil mist:	**Oil vapour:**
Norway	0.6 mg/m^3	30 mg/m^3
UK		250mg/m^3
USA	5 mg/m^3	

2 Health effects

Exposure to oil mist and vapour occur either by skin contact or inhalation (A. T. Simpson et al., 2003). Structural constraints offshore mean that much for the mud system is confined, exacerbating the potential for exposure (Gardner, 2003). The mixtures are complex and contain many different additives, and each component may contribute to the nature and severity of any health effect (A. T. Simpson et al., 2003).

In a study of mortality and incidence of cancer among workers in a Norwegian cable manufacturing company exposed to mineral oils 1953-84 Rønneberg et.al. (1988) observed a statistically significant excess of lung cancer and deaths from ischemic heart disease in the impregnation, sheathing and installation subcohort. Exposure to mineral oils has been verified for all members of the impregnation, sheathing and installation subcohort through establishment of their work categories. Low viscosity and high viscosity mineral oils must be considered as the most probable causes as excess smoking alone cannot account for the result. (Rønneberg et al., 1988)

In a follow up study of lung function and radiographic signs of pulmonary fibrosis oil exposed workers in a cable manufacturing company Skyberg et.al. (1992) found that the high prevalence of pulmonary fibrosis together with the influence of the duration of exposure suggest a causal relation between oil impregnation work and pulmonary fibrosis. The most plausible cause of the lung fibrosis is suggested to be exposure to low viscosity oil mist and vapours.

Eide (1990) claims that the advantage of low-aromatic base oils, with respect to health hazard, compared with the former diesel oils is mainly due to the increase in of the initial boiling point, making the base oil less volatile. The corresponding aliphatic and naphthenic hydrocarbons, however, may be more efficiently distributed to the brain than the corresponding aromatic hydrocarbons. A reduction in the content of aromatic hydrocarbons becomes particularly important when the upper boiling point is sufficiently high that the base oil otherwise would contain carcinogenic polycyclic aromatic hydrocarbons.

Exposure to oil mist is considered to be of greater risk for adverse health effects. Depending on the size of the aerosols they reach different levels of the respiratory system, the smaller the size the longer down it goes. How an aerosol is generated affects the health effects in the

lungs. A mechanically generated aerosol will have different composition from that of a mist generated by condensation of vapour, and also the size and the lifetime of the droplets may be different. (Eide, 1990)

According to Simpson et al. (2000) occupational exposure to oil mist is associated with eye, nose and throat complaints. In addition there are concerns relating to occupational asthma, allergic alveolitis and other lung diseases, as well as the possibility of cancer.

Svendsen (1997) found that marine engineers exposed to mineral oil mist in engine rooms of ships had a statistically significant increased prevalence of respiratory disorders with symptoms such as mucous membrane irritation and dyspnoea. It was also found a statistically insignificant increase in the prevalence of chronic bronchitis. Results indicative of an increased prevalence of emphysema in the engineers were also found. Direct contact with oils and organic solvents most likely lead to the increased prevalence of skin complaints; in particular hand dermatitis and dry skin

In a study of mortality, symptoms and respiratory function in humans occupationally exposed to oil mist Ely et.al. (1970) found the effect of oil mist exposure or any other job environment, as measured by the number of years on that job, to be not significant.

Skyberg et.al. (1986) demonstrated a significantly increased prevalence of pulmonary fibrosis in a study of 25 cable plant workers exposed to mist and vapour of petroleum distillates. The main exposure factors were mist and vapours of mineral oil and kerosene. Although the exposure levels were consistently lower than the 5 mg/m^3 standard (ACGIH) the actual levels may, however, have been severely underestimated.

The average exposure time for cases in the impregnation department was 25% longer than for non-cases from the same area. Only one of the cases had been exposed for less than ten years. These observations may according to Skyberg et.al. (1986) be suggestive of a possible dose-response relationship. The finding of low levels of fibrogenic dusts in lung tissue according to Skyberg et.al. (1986) implies the possibility of interactions between mineral dusts and petroleum distillates.

The formulation of oil based muds have much in common with the formulation of metalworking fluids and hence there are similar difficulties in respect of sampling and setting appropriate exposure limits (Gardner, 2003).

According to Simpson et al. (2003) dermatitis is by far the most important work-related health effect of metalworking fluids in terms of the number of people affected. According to Steinsvåg et al. (2007) operators handling the mud systems will have some skin exposure to mud.

3 Methods of sampling

This chapter presents some sampling methods that can be used when sampling oil mist and vapour.

3.1 Sampling using filters - sampling of aerosols

Mist is the general term for aerosols of liquids that includes all particle sizes that can keep in air for a certain time. Aerosols are can be sampled using filters. The filter will be placed in a filter holder that is connected to a pump, where a known amount of air is pumped through. The concentration of pollutants in the air can then be determined, and for instance be compared to the occupational exposure level (OEL) for the substance. (The Norwegian Labour Inspection (Authority, 2010)

Filter holders should be chosen depending on what fraction of aerosol it is desirable to test. Traditionally a three part plastic cassette with 37mm or 25mm diameter has been used. These cassettes have however been shown not to satisfy the demands for sampling of the inhalable fraction described in NS-EN 481. The inhalable fraction will be underestimated for aerosols with aerodynamic diameter >15 μm with these cassettes. (The Norwegian Labour Inspection (Authority, 2010)

Pedersen et al. (1984) found that glass fibre filters (37mm) gave 10-15 % lower collection efficiency than Millipore cellulose filters (37mm) with pore size 0.8 μm.

3.2 Sampling using charcoal tubes – sampling of vapour

Sampling of solvents and volatile organic compounds are generally performed using charcoal tubes that makes use of adsorption tubes filled with active charcoal. The charcoal tubes are filled with two charcoal sections, one main part and one control part that is analysed for control of overload. (The Norwegian Labour Inspection (Authority, 2010)

The capacity of the charcoal tubes varies with type of solvent and what mixture of solvents that exists. Therefore it is important to adapt the sampling conditions to avoid overload of the charcoal tube, i.e. sampling time and rate. A sampling rate of 50 ml/min is normal, and a sampling time of 3-4 hours. For some polar and very volatile compounds, e.g. ethanol and ether, it can be necessary with a shoRTer sampling time to avoid overload. The method can

also be used for short time samples, e.g. 15 min. (The Norwegian Labour Inspection (Authority, 2010)

Charcoal tubes will, by normal sampling rates, also gather oil mist, and will therefore not give only the vapour phase, but more or less the sum of vapour and aerosol. For this reason filter and charcoal tube methods, and combinations of these, will not give a correct answer to the distribution between vapour and aerosol. (Malvik & Børresen, 1988)

3.3 Direct reading instruments

Both oil mist and vapour can be sampled using direct reading instruments. Measurement principles and properties of some instruments can be seen in Table 3-1.

Table 3-1 Direct reading instruments: Measuring principles and properties (The Norwegian Labour Inspection (Authority, 2010)

Principle of analysis	**Components**	**Response time**	**Selectivity**
Flame ionization	Volatile organic compounds (VOC)	Fast	Not selective
Photo ionization	Many VOC	Fast	Limited
Electron capture	Halogenated VOC	Fast	Not between components Interference from oxygen and nitrogen compounds
Infrared spectrophotometry	Many VOC and inorganic gasses	A few seconds	Dependent on wavelength
Light scatter - photometry	Aerosols	30-60 s	Low sensitivity for particles >10μm depending on type
Light scatter – particle counting	Aerosols	30-60 s	Depending on type

Direct reading instruments can be a useful tool to evaluate the results of measurements if used parallel to sampling (Galea et al., 2010), but flame and photo ionization detectors does not seem to be suitable for this kind of pollutants. Aerosol monitors based on light scatter on the

other hand are of interest for further testing, as they can be suitable tools to study e.g. the effect of changes in the process. (Malvik & Børresen, 1988)

3.4 Combined sampling

To sample both aerosol/mist and vapour an aerosol filter for collection of particular matter and an adsorbent for collection of vapour can be connected in a series (The Norwegian Labour Inspection (Authority, 2010). Malvik & Børresen (1988) claims that a charcoal tube in series with a filter is the best method to use.

Galea et al. (2010) recommends to use 37 mm closed cassette loaded with glass fibre and cellulose acetate filters with back up charcoal tube (STAMI method) even though sample losses will occur from the filter during sampling for low viscosity base oils and give a substantial underestimation of the oil mist level which can not be quantified.

4 Loss of sample

Sampling and storage are the two phases where most sample is lost.

4.1 Loss during sampling

According to Malvik & Børresen (1988) the absolute evaporation loss during sampling will be dependent on vapour pressure, volume flow and concentration. The particle size will also affect the rate of evaporation. They also claim that several short sampling periods are better than one long to get correct data for the exposure level, but enough oil must be collected so that the quantification can be done with enough accuracy.

By comparing deviations for series with approximately the same flow Pedersen et al. (1984) found that average deviations for these series are practically the same. This shows that the efficiency of accumulation of oil in particle and gas phases does not seem to be considerably dependent on the flow used. A certain increase in evaporation from the particle phase to the gas phase could be observed at increasing fall of pressure over the filter.

Svendsen (1997) found that an IOM sampler with a 25mm filter had the highest collection efficiency of four sampling devices tested.

Galea et al. (2010) recommends that large charcoal tubes are used to minimize the risk of significant breakthrough and underestimation of the oil vapour concentrations when measuring oil mist and vapour which have been generated from drilling fluids which contain base oils of low viscosity.

Simpson & Keen (2007) found that due to evaporation of the volatile oil component traditional filter sampling for drilling mud aerosol is inappropriate. They also considered measurement of vapour as a marker for total exposure. Comparisons with the total airborne oil measurement were mixed, but suggested that although pumped sampling gave inconsistent results when compared with either total airborne oil or diffusive vapour measurements, diffusive sampling of the vapour was representative of total oil.

Simpson & Keen (2007) also point out that traditional filter sampling and gravimetric analysis also does not distinguish between oil and other particulate matter present.

4.2 Loss during storage

Pedersen et al. (1984) found that oil mist transported from a glass fibre filter to a millipore filter when stored in the same filter holder. However the transport seems to be low (<5%) during the 2-3 first hours after exposure and then increase considerably (>60%) after about 1 day of storage. 90-95% of the total collected oil mist on the filters were however still found after 5-8 days of storage.

When comparing plastic holders and "self made" brass holders Pedersen et al. (1984) did not find any essential differences regarding adsorption of oil from the filters. The holders that were used during the storage were extracted using tetrachlorethylene, and most of the oil that had evaporated from the filters was re-found in the extract.

According to Malvik & Børresen (1988) only the vapour pressure and the particle size will affect the evaporation loss during storage. To reduce the losses that can occur during storing and transport the samples should be stored cool after sampling. They found this by freezing the samples immediately after sampling and first weighing, which reduced the loss to nil. Long time storing in room temperature without regular opening of the filter cassette also gave a modest loss of sample.

5 Methods of analysis

There are many methods of analysis that can be applied to oil mist and vapour samples. Some of the most common ones will be presented in this chapter.

5.1 Gravimetric analysis

Gravimetric methods are quantitative methods that are based on determining the mass of a pure compound to which the analyte is chemically related. Gravimetric methods of analysis are based on mass measurements made with an analytical balance. (Skoog et al., 2004) Gravimetric analysis is the most common method of analysis for oil mist and vapour samples. The mass of sample is found by subtracting the mass of the filter before sampling from the mass of the filter after sampling.

Galea et al. (2010) suggests that it is inappropriate to conduct gravimetric as well as oil mist analyses on the same filter as a large component of the oil may evaporate during conditioning of the filter before gravimetric analysis.

5.2 Gas Chromatography (GC)

Chromatography is a separation technique, and in gas chromatography the components of a vaporized sample are separated as a consequence of being partitioned between a mobile gaseous phase and a liquid or a solid stationary phase held in a column. Gas-liquid chromatography is the most widely used method and is usually shortened to gas chromatography (GC). The mobile gaseous phase is inert and does not interact with molecules of the analyte; its only function is to transport the analyte through the column. The liquid phase is immobilized on the surface of an inert solid packing or on the walls of capillary tubing. Depending on how strongly the components are attracted by the stationary phase they will travel through the column at different rates. A chromatogram is then plotted against time. (Skoog et al., 2004)

5.2.1 Flame Ionization Detectors (FID)

The FID is the most widely used and generally applicable detector for gas chromatography. The effluent from the column is directed into small air/hydrogen flame (Figure 5-1). Most organic compounds produce ions and electrons when pyrolyzed at the temperature of an air/hydrogen flame. Detection involves monitoring the current produced by collecting these charge carriers. A few hundred volts applied between the burner tip and a collector electrode located above the flame serve to collect the ions and electrons. The resulting current is then

measured with a picoammeter. It is generally rugged and easy to use. A disadvantage of the FID is that it destroys the sample during the combustion step. (Skoog et al., 2004)

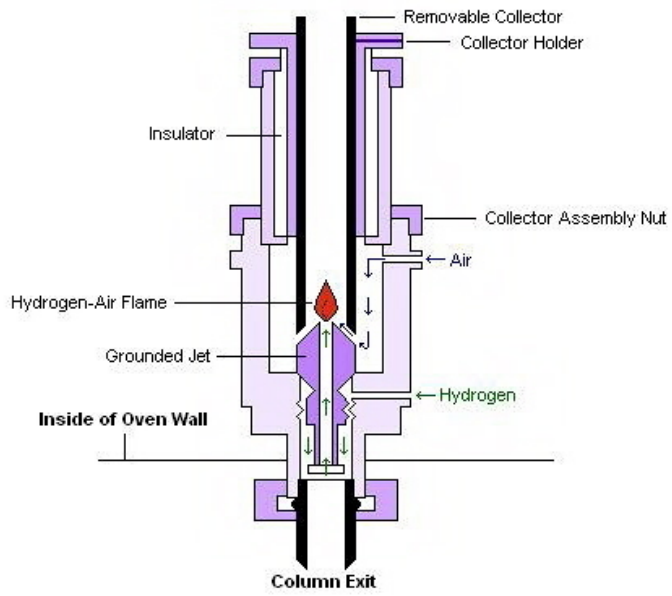

Figure 5-1 Schematic of a typical FID detector (Thet & Woo, 2010)

5.2.2 Mass Spectrometry (MS)

MS is one of the most powerful detectors for GC, and the combination is known as GC/MS. An MS measures the mass to charge ratio (*m/z*) of ions that have been produced form the sample. Usually z=1 and effectively only masses are measured. In the MS, sample molecules enter an ionization source, which ionizes the sample. The fragments produced can also be ionized. The molecular ions then pass to the analyzer; this serves to sort the ions according to *m/z* values. The separated ions are the detected, and a plot of the ion intensity versus *m/z* value is produced by the data system. (Skoog et al., 2004) These days there are computer programs that have mass spectra for many different compounds stored that can interpret the mass spectrum for you. Figure 5-2 shows a schematic of a complete GC/MS system; the sample is injected into the GC and the effluent enters the inlet of the MS (Skoog et al., 2004).

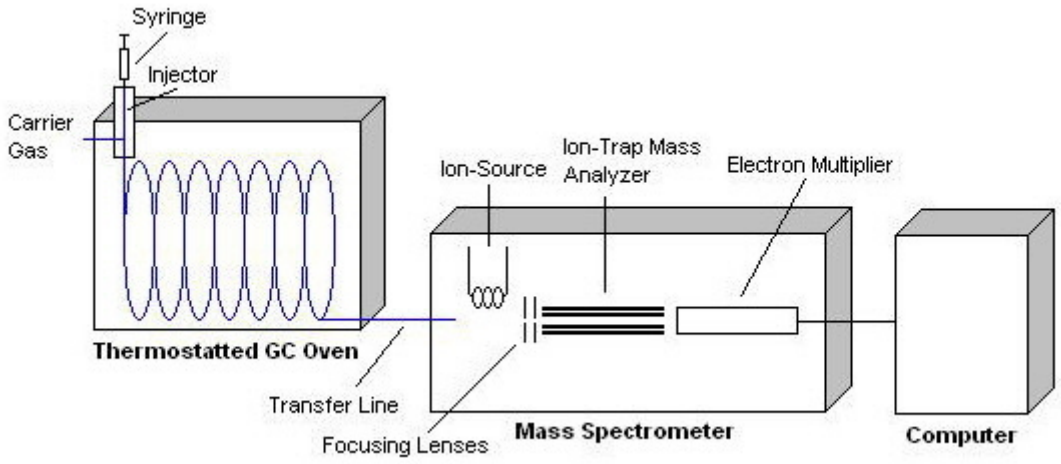

Figure 5-2 Schematic of a typical GC/MS (Thet & Woo, 2010)

5.3 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform IR instruments measure and detect all wavelengths simultaneously. An interferometer is used to produce interference patterns that contain the IR spectral information. To obtain radiant power as a function of wavelength, the interferometer modulates the source signal in such a way that it can be decoded by the mathematical technique of Fourier transformation. (Skoog et al., 2004)

To obtain the spectrum of a sample a reference interferogram of the source with no sample in the light path must first be obtained. The sample is the placed in front of the detector and a second interferogram is obtained. The difference between the second interferogram and the reference interferogram is then computed. Since the difference interferogram depends only on the absorption of radiation by the sample, the fast Fourier transform is performed on the resulting data, which produces the IR spectrum of the sample. (Skoog et al., 2004) The IR spectrum for the sample can then be compared to other IR spectra.

According to Malvik & Børresen (1988) IR analysis should be used as a standard method of analysis for the quantification.

Materials and method

6 Experimental

6.1 General procedure

First filters, one cellulose acetate (CA) (bottom) and one glass fibre (GF) (top), were weighed before and after a sample (3 µl or 1 drop) of base oil (EDC Pearl (B1)/ EDC 95/11 (B2)/ Sipdrill 2.0 (B3)) was added to the filters. The filters were placed in Millipore filter cassettes (37 mm). The filter cassettes were then connected to a charcoal tube and a pump, which were run for 2 hours with a flow of 1.4 l/min. The spiked filters are then weighed again two or three times the first day. The frequency of further weighing can be found in Table 6-1.

Table 6-1 Frequency of weighing from day 2 onwards

Time of storage	**Frequency of weighing**
Day 2	Twice a day
Day 3 – Day 16 (15)	Once a day
Day 16 (15) – Day 24 (23)	Every other day
Day 24 (23) – Day 36 (35)	Every third day
Day 36 (35) – Day 40 (39)	Every fourth day

For the first, third and fourth series the filters were removed from the cassette before weighing, while in the second series the whole cassette with filter is weighed. The first, second and fourth series are stored at room temperature and fridge temperature, while the third series is stored in the freezer. For chemicals and equipment used see appendix I.

6.2 Base oils

The base oils were sent to SINTEF for analysis by GC-MS. Safety data sheets for the base oils can be found in appendix II. Data of viscosity for the base oils are shown in Table 6-2.

Table 6-2 Viscosity data for the base oils

Base oil nr.	**Name**	**Viscosity**
Base oil 1	EDC Pearl	3.5 cSt (20^{o}C)
Base oil 2	EDC 95/11	3.3-3.7 cSt (40^{o}C)

Base oil 3	Sipdrill 2.0	< 2 cSt (40^{o}C)

6.3 Series one (24. Jan. – 4. Mar.)

Six filters are first prepared (3x3 µl and 3x1 drop) and then air drawn through the filter cassettes, with filters, using pumps (2h, 1.4 l/min). The filters where then weighed immediately after the pumps were stopped (0h), two hours and six hours after. These filters were stored at room temperature, in filter cassettes with plugs (RTw 1). On the second day they were weighed after 23 and 28 hours.

Nine more filters were prepared the first day, six (3x3 µl and 3x1 drop) for storing at room temperature in cassettes without plugs (RTwo 1) and three (3x3 µl) for storing, only the filter without filter cassette, in exicator (RTe 1.1). The filters where weighed immediately after and two hours after the pumps were stopped (0h), and on the second day they were weighed after 19 and 24 hours.

On the second day nine more filters were prepared first, six (3x3 µl and 3x1 drop) for storing at refrigerator temperature in cassettes with plugs (RFTw 1) and three (3x1 drop) for storing, only the filter without filter cassette, in exicator (RTe 1.2). The filters where weighed immediately after (0h), two and four hours after the pumps were stopped, and on the second day they were weighed after 19 and 26 hours.

Six more filters were prepared with the three different base oils (3x3 µl and 3x1 drop). The filters where then weighed immediately after the pumps were stopped (0h), and two hours after. These filters were stored at refrigerator temperature, in filter cassettes without plugs (RFTwo 1). On the second day they were weighed after 17 and 24 hours.

RFT samples were stored in closed plastic bags sorted according to base oil. The filters were then weighed as described in Table 6-1. RTe 1.2 and RFT 1 follow the days in parentheses.

6.4 Series two (28. Feb. – 8. Apr.)

On the first day six filters are first prepared (3x3 µl and 3x1 drop) for storing at room temperature with plugs (RTw 2), in a closed plastic bag. Six (3x3 µl and 3x1 drop) more filters were prepared the first day for storing at room temperature in cassettes without plugs (RTwo 2), in a closed plastic bag.

On the second day six (3x3 µl and 3x1 drop) more filters were prepared for storing at refrigerator temperature in cassettes with plugs (RFTw 2). Six more filters were then prepared with the three different base oils (3x3 µl and 3x1 drop). These filters were stored at refrigerator temperature, in filter cassettes without plugs (RFTwo 2).

All the filters where weighed straight after the pumps were stopped (0h), and two and six hours after. On the second day they were all weighed after 23 and 28 hours. The filters were then weighed as described in Table 6-1. RFT 2 follows the days in parentheses.

6.5 Series three (15. Mar. – 23. Apr.)

Six filters are first prepared with the three different base oils (3x3 µl and 3x1 drop) for storing at freezer temperature with plugs (FTw 3). Six (3x3 µl and 3x1 drop) more filters were prepared for storing at freezer temperature, in cassettes without plugs (FTwo 3).

All the filters where weighed straight after (0h) the pumps were stopped, and two and six hours after. On the second day they were all weighed after 23 and 28 hours. The filters were then weighed as described in Table 6-1.

6.6 Series four (4. Apr. – 13. May)

In this series double glass fibre filters were used.

Six filters are first prepared (3x3 µl and 3x1 drop) and then air drawn through the filter cassettes, with filters, using pumps (2h, 1.4 l/min). The filters where then weighed immediately after the pumps were stopped (0h) , two hours and six hours after. These filters were stored at room temperature, in filter cassettes with plugs (RTw 4), in a closed plastic bag. On the second day they were weighed after 23 and 28 hours.

Six more filters were prepared (3x3 µl and 3x1 drop) for storing at room temperature in cassettes without plugs (RTwo 4), in a closed plastic bag. The filters where weighed immediately after (0h), and two hours after the pumps were stopped. On the second day they were weighed after 23 and 28 hours.

Six more filters were prepared the first day, six (3x3 µl and 3x1 drop) for storing at room temperature, only the filter without filter cassette, in exicator (RTe 4). The filters where

weighed immediately after (0h), and two and four hours after the pumps were stopped. On the second day they were weighed after 21 and 26 hours

On the second day six more filters were prepared (3x3 µl and 3x1 drop) for storing at refrigerator temperature in cassettes with plugs (RFTw 4), in a closed plastic bag. The filters where weighed immediately after (0h), and two and six hours after the pumps were stopped. On the second day they were weighed after 23 and 28 hours.

Six more filters were prepared with the three different base oils (3x3 µl and 3x1 drop). The filters where then weighed immediately after (0h) the pumps were stopped, and two and four hours after. These filters were stored at refrigerator temperature, in filter cassettes without plugs (RFTwo 4), in a closed plastic bag. On the second day they were weighed after 23 and 28 hours.

The filters were then weighed as described in Table 6-1. RTe 1.2 and RFT 4 follow the days in parentheses. An overview of weighing method and storage for the differerent temperatures is shown Table 6-3.

Table 6-3 Overview of weighing method and storage for the differerent temperatures

Temperature	**Series**	**Method of weighing**	**Storage**
RTw +wo	Series 1	Remove filter before weighing	Cassettes on table in room
	Series 2	Weigh whole cassette with filter	Cassettes in closed plastic bag
	Series 4	Remove filter before weighing	Cassettes in closed plastic bag
RTe	Series 1+4	Remove filter before weighing	Filter in exicator
RFT	Series 1	Remove filter before weighing	Cassettes in closed plastic bag
	Series 2	Weigh whole cassette with filter	Cassettes in closed plastic bag
	Series 4	Remove filter before weighing	Cassettes in closed plastic bag
FT	Series 3	Remove filter before weighing	Cassettes in closed plastic bag

Results

7 GC-MS analysis of base oils

AMDIS GC/MS Analysis Reports for the different base oils can be found in appendices III - V.

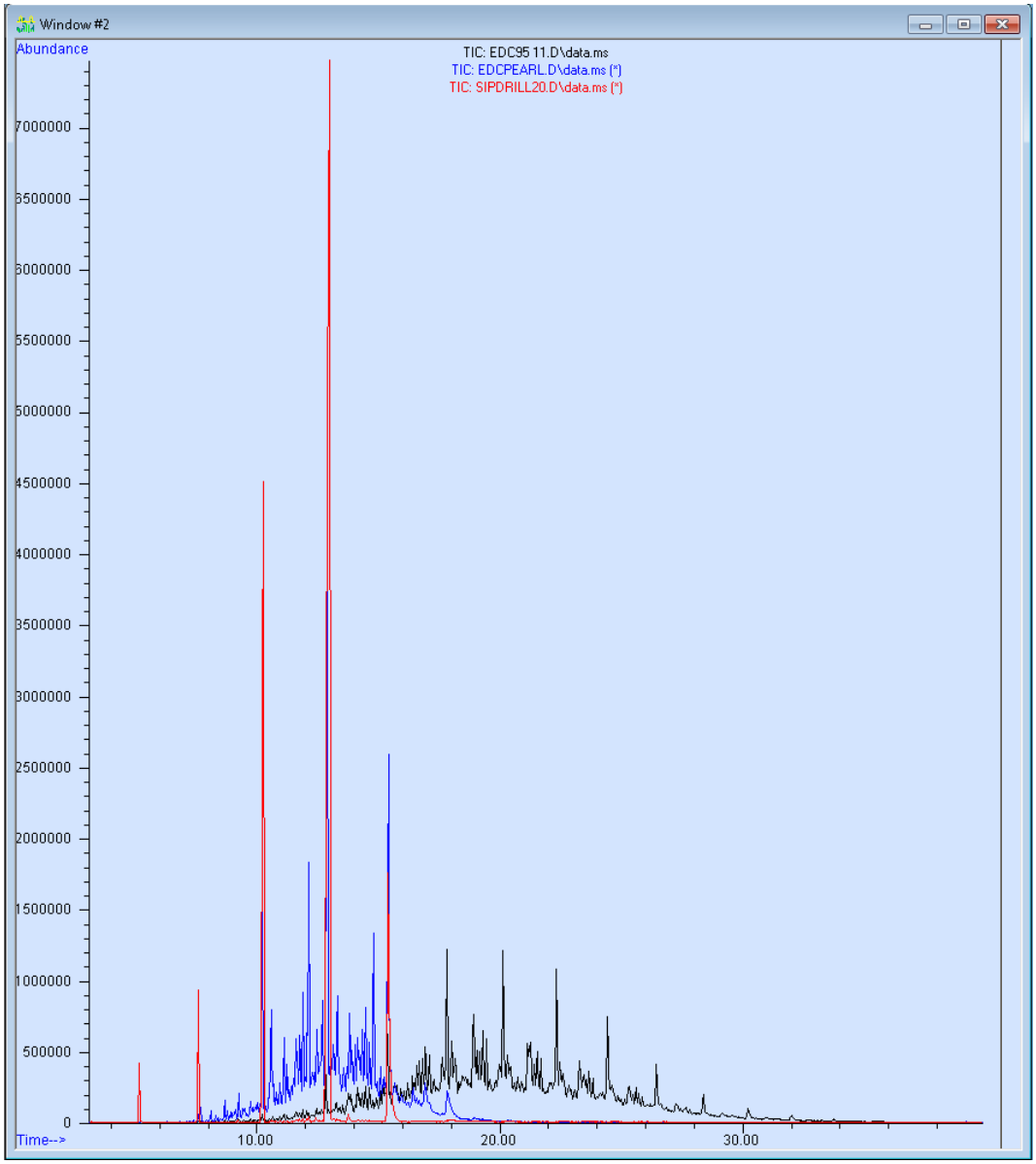

Figure 7-1 GC/MS chromatogram of ECD95/11, ECD Pearl and Sipdrill2 set on top of each other and scaled in relation to each other.

From Figure 7-1 it can be seen that both EDC Pearl and Sipdrill 2.0 contain many of the same major compounds and mostly consist of hydrocarbon chains, though EDC Pearl contains more cyclic and aromatic compounds. EDC 95/11 has more longer hydrocarbon chains with more branching giving it a lower volatility than the two other base oils.

8 Storage tests

Storage was tested in different ways at three temperatures; room temperature, refrigerator temperature and freezer temperature.

8.1 Room temperature

At this temperature storage was tested with and without plugs on filter cassettes, as well as with open cassettes in exicator. The data for storing at room temperature may be found in appendix VI.

8.1.1 Room temperature with plugs (RTw)

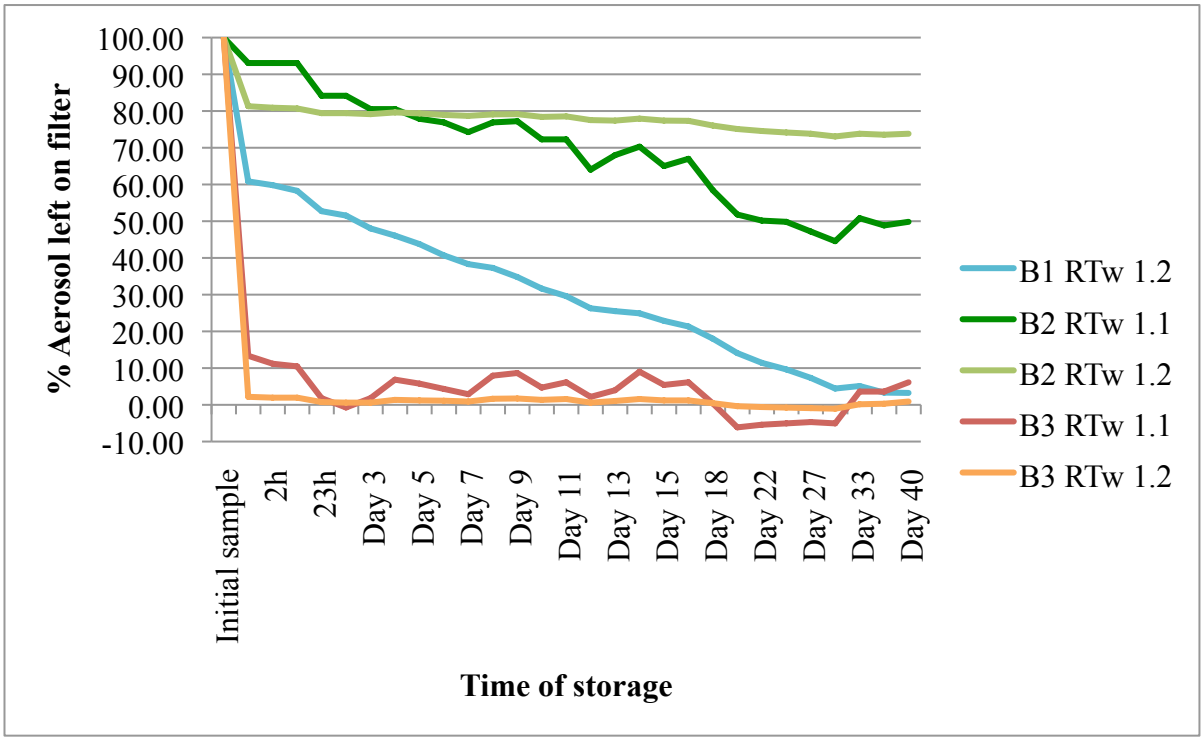

Figure 8-1 Loss of sample when storing at room temperature with plugs, series 1. (Parallel 1 for base oil 1 was discarded due to mistakes in the initial weighing of the filters.)

In both series 1 (Figure 8-1) and 4 (Figure 8-3) base oil 3 lost over 80% of the initial sample during aspiration. Base oil one lost around 40% during aspiration in series 1 (Figure 8-1), and over 70% in series 4 (Figure 8-3), while base oil 2 only lost 20% or less in both series. Base oil 2 still had around 50% or more left on the filters after 40 days in both series 1 (Figure 8-1) and 4 (Figure 8-3). The lower concentration samples generally lost more in percentage than the higher concentration samples.

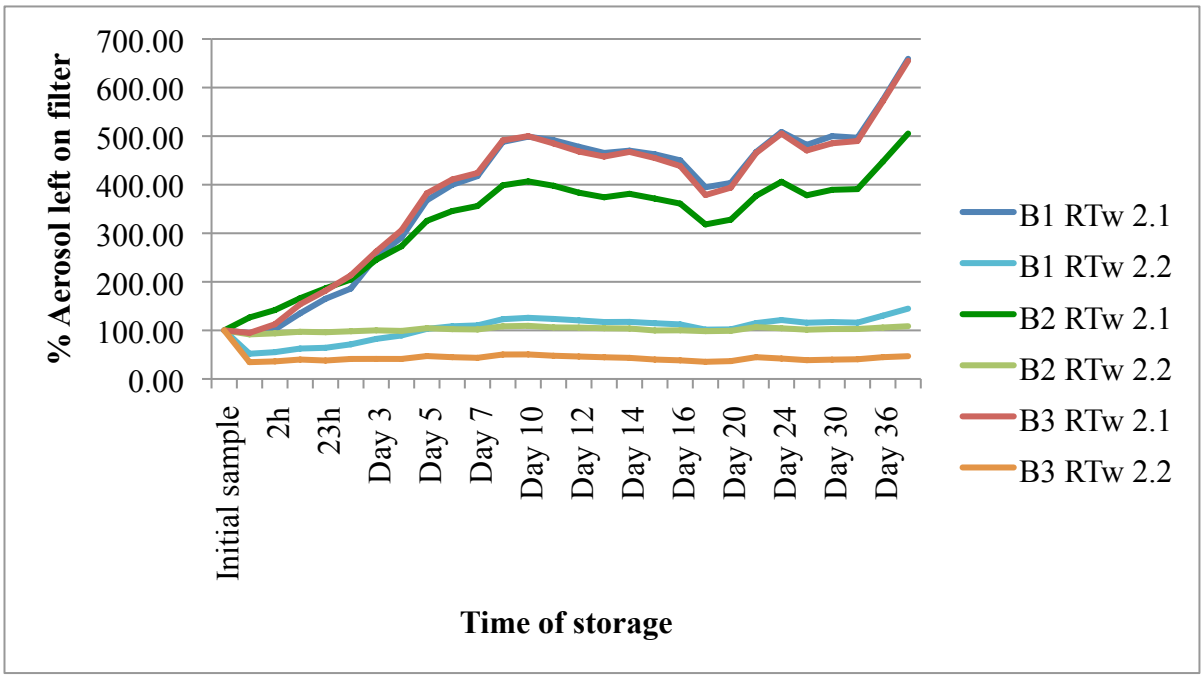

Figure 8-2 Loss of sample when storing at room temperature with plugs, series 2

Figure 8-2 shows that when weighing the whole cassette with filter the samples generally gain more weight during storage, than the weight of the initial sample. The low concentration parallels gains more in percentage than the higher concentration samples.

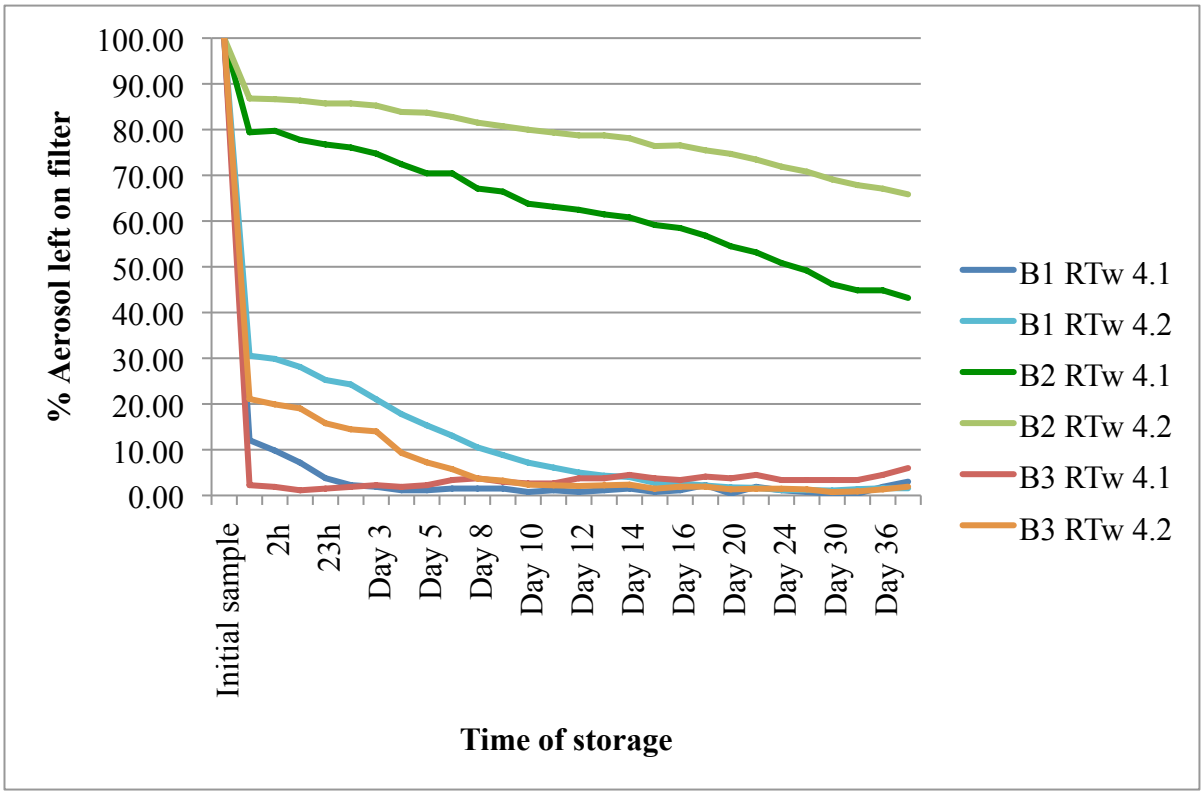

Figure 8-3 Loss of sample when storing at room temperature with plugs, series 4

8.1.2 Room temperature without plugs (RTwo)

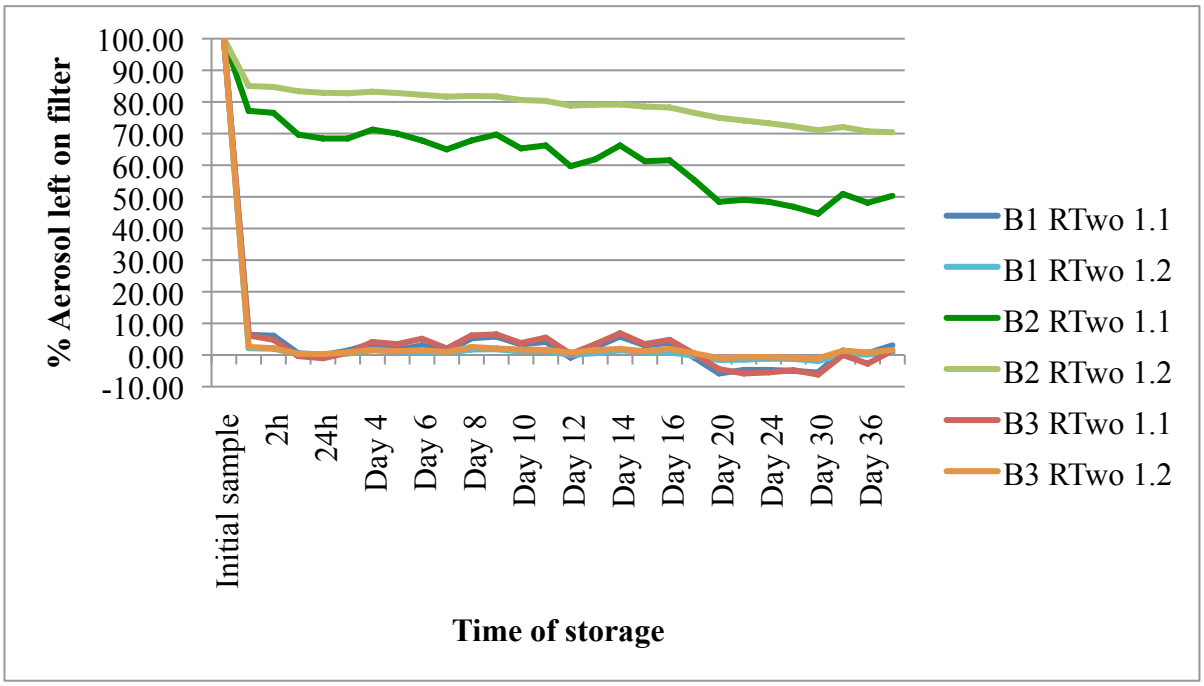

Figure 8-4 Loss of sample when storing at room temperature without plugs, series 1

In series 1 (Figure 8-4) over 90% of sample is lost during aspiration for base oils 1 and 3, while base oil 2 only lost around 15-20%. Base oil 2 only lost around 20% - 30% during storage, most for the low concentration parallel.

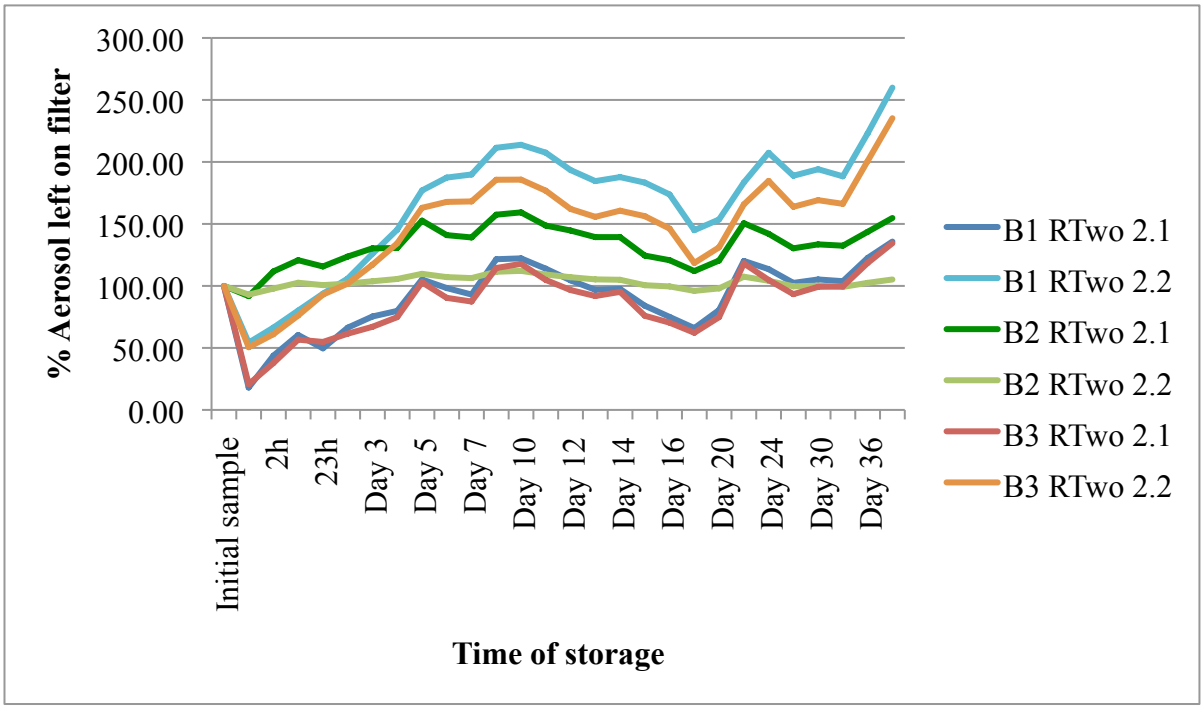

Figure 8-5 Loss of sample when storing at room temperature without plugs, series 2

Figure 8-5 shows that when weighing the whole cassette with filter the samples generally gain more weight during storage, than the weight of the initial sample. The high concentration parallels gains more in percentage than the lower concentration samples.

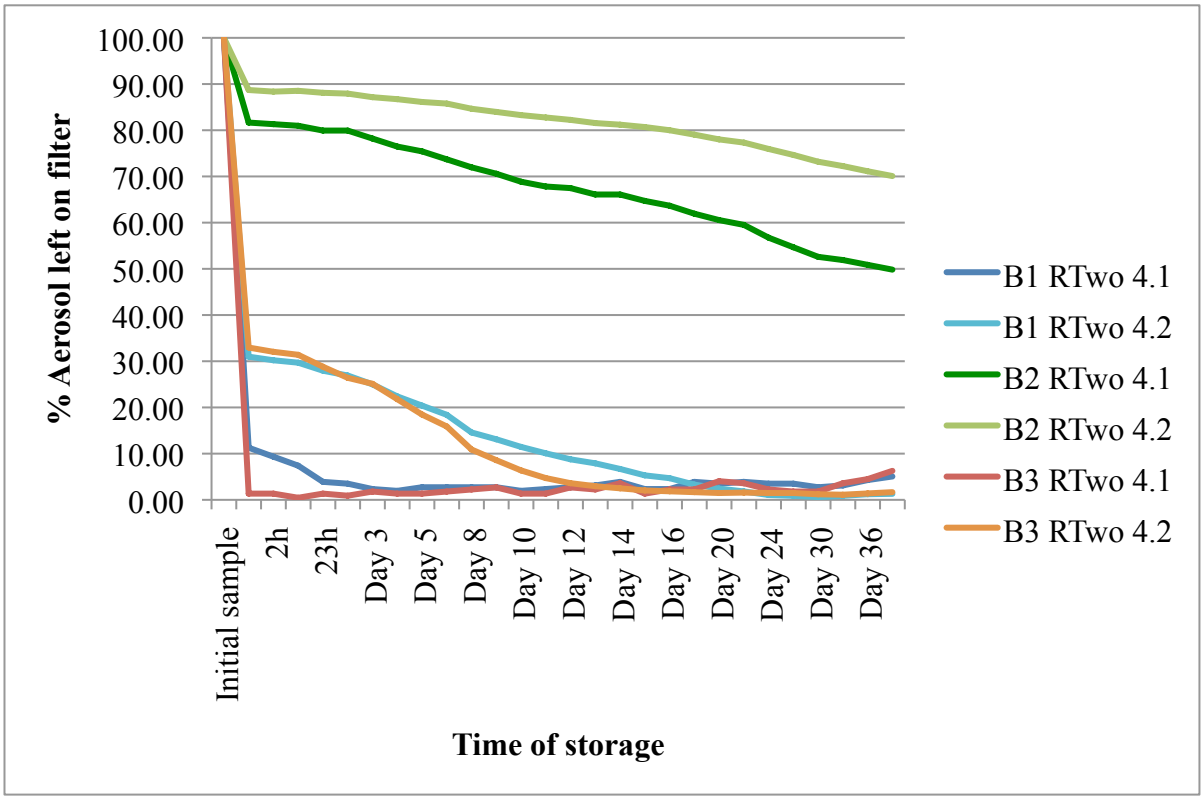

Figure 8-6 Loss of sample when storing at room temperature without plugs, series 4

Series 4 (Figure 8-6) show similar patterns to series 1, though a bit less sample was lost during aspiration, especially for the lower concentration parallels of base oils 1 and 3. Base oils 1 and 3 show similar patterns of loss for similar concentrations. Base oil 2 had a similar amount of loss to that in series 1 (Figure 8-4), but was more stable in this series.

8.1.3 Room temperature in exicator (RTe)

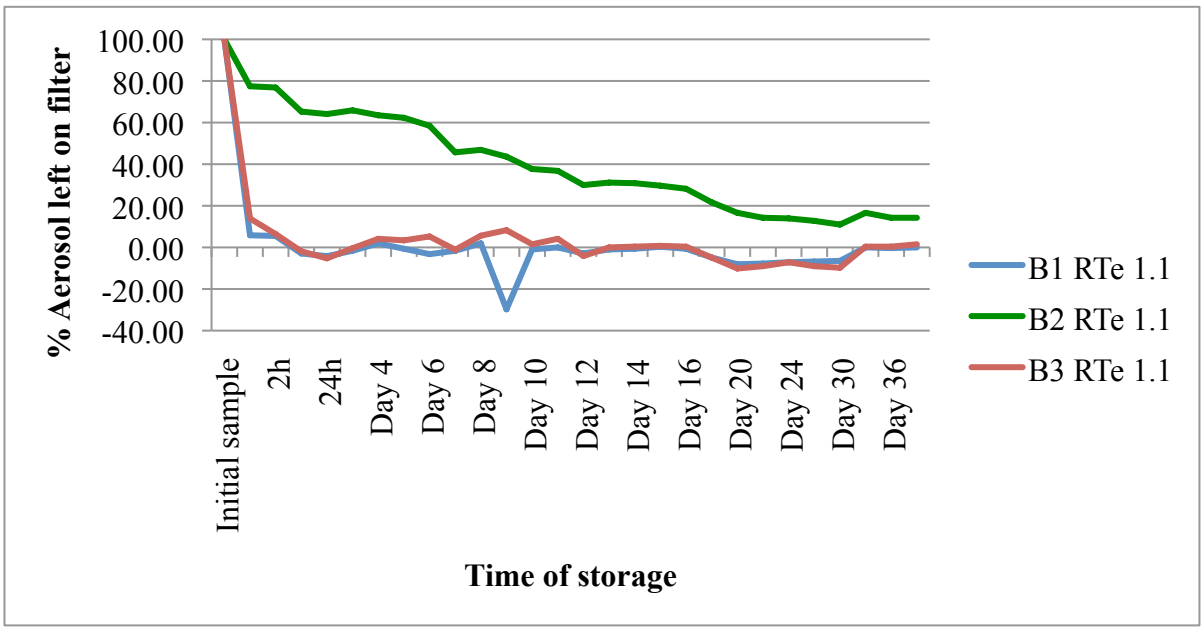

Figure 8-7 Loss of sample when storing at room temperature in exicator, series 1 parallel 1

In series 1 parallel 1 (Figure 8-7) base oils 1 and 3 lose over 80% during aspiration, while base oil 2 loose around 20%. Base oil 2 shows a gradual decline and has less than 20% left after 40 days.

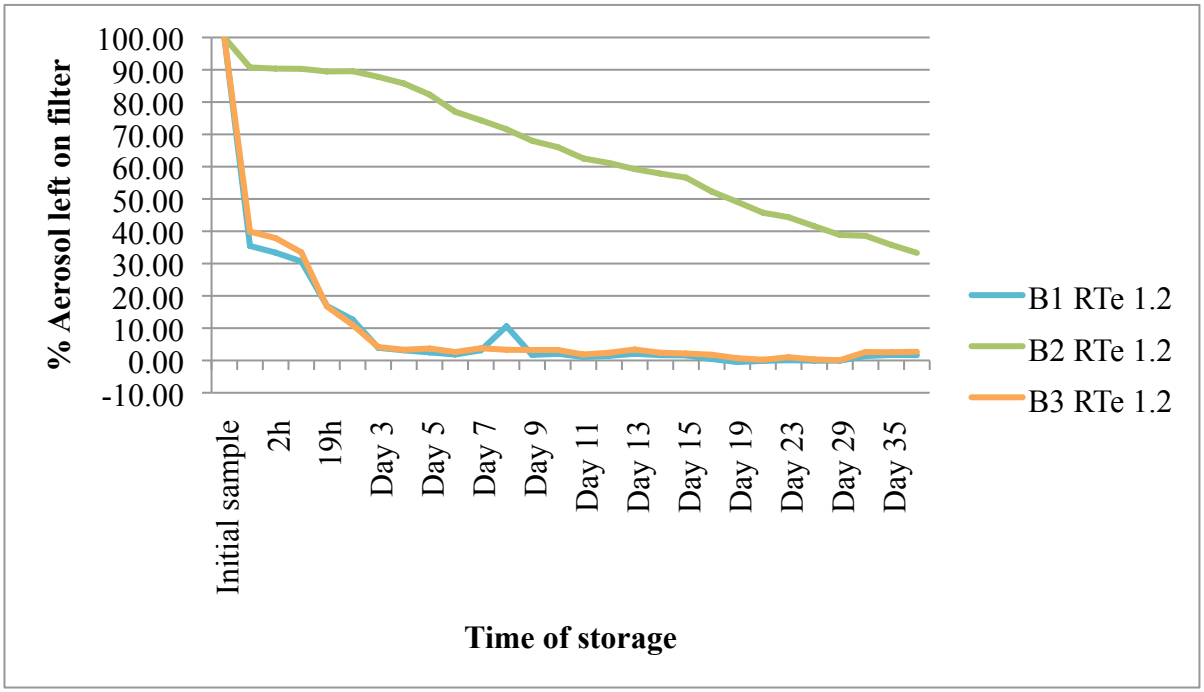

Figure 8-8 Loss of sample when storing at room temperature in exicator, series 1 parallel 2

In parallel 2 (Figure 8-8) base oils 1 and 3 lose over 60% during aspiration, while base oil 2 loose around 10%. Base oil 2 shows a steady decline and has less than 40% left after 40 days.

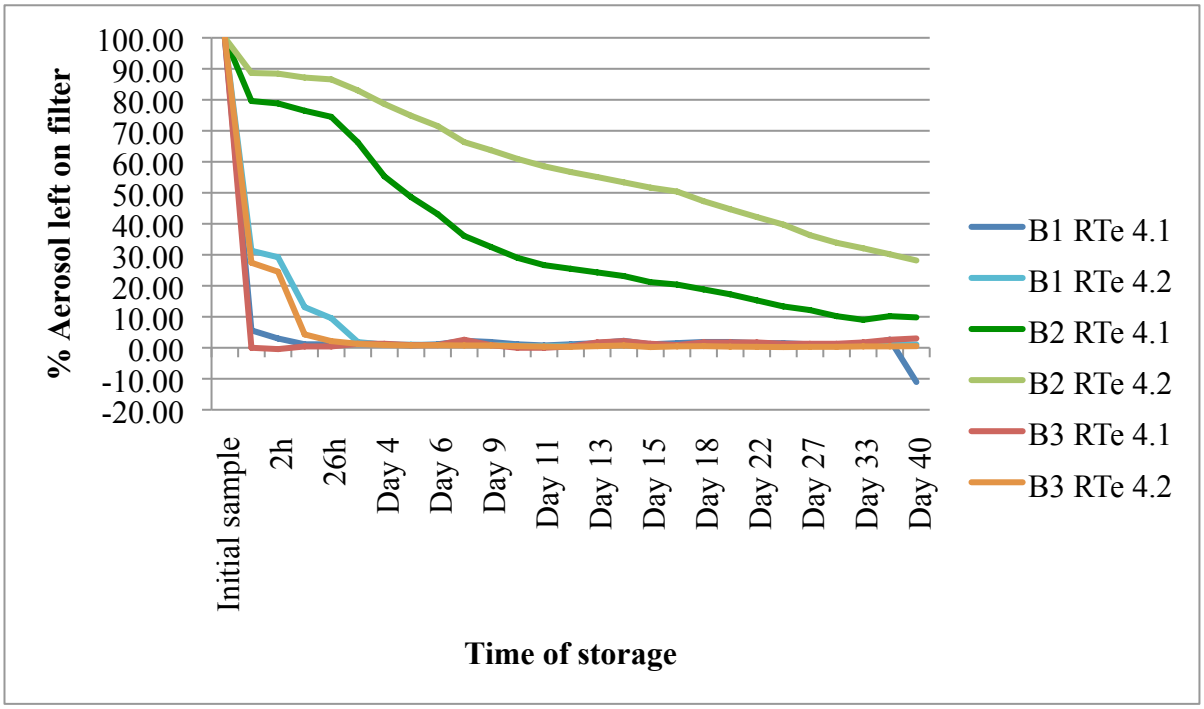

Figure 8-9 Loss of sample when storing at room temperature in exicator, series 4

Series 4 (Figure 8-9) show similar patterns to series 1 (Figure 8-7).

8.2 Refrigerator temperature

At this temperature storage was tested with and without plugs on filter cassettes. The data for storing at refrigerator temperature may be found in appendix VII.

8.2.1 Refrigerator temperature with plugs (RFTw)

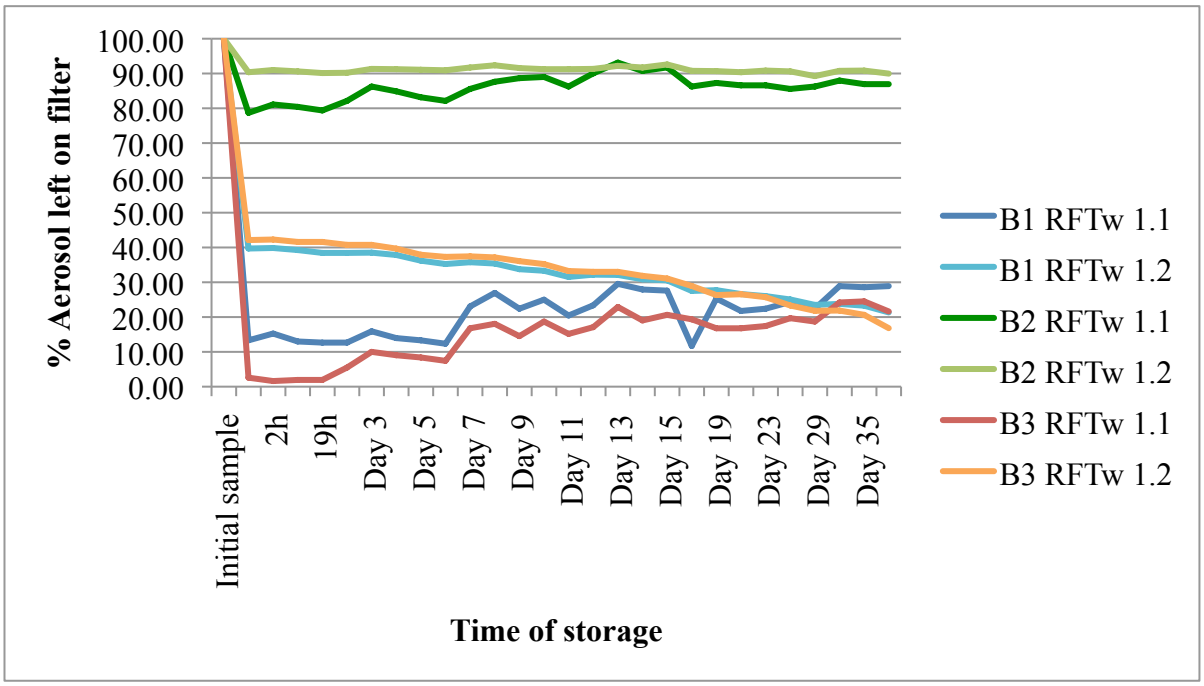

Figure 8-10 Loss of sample when storing at refrigerator temperature with plugs, series 1

Figure 8-10 shows that base oil 2 is fairly stable around 80% -90% throughout the storage period, though some gain for the lower concentration sample. For base oils 1 and 3 around 60% is lost during aspiration for parallel 1, and a steady decline dropping to about 20% at the end of the storage period. Over 80% and 90% is lost during aspiration for parallel 2, but gain around 20% during storage.

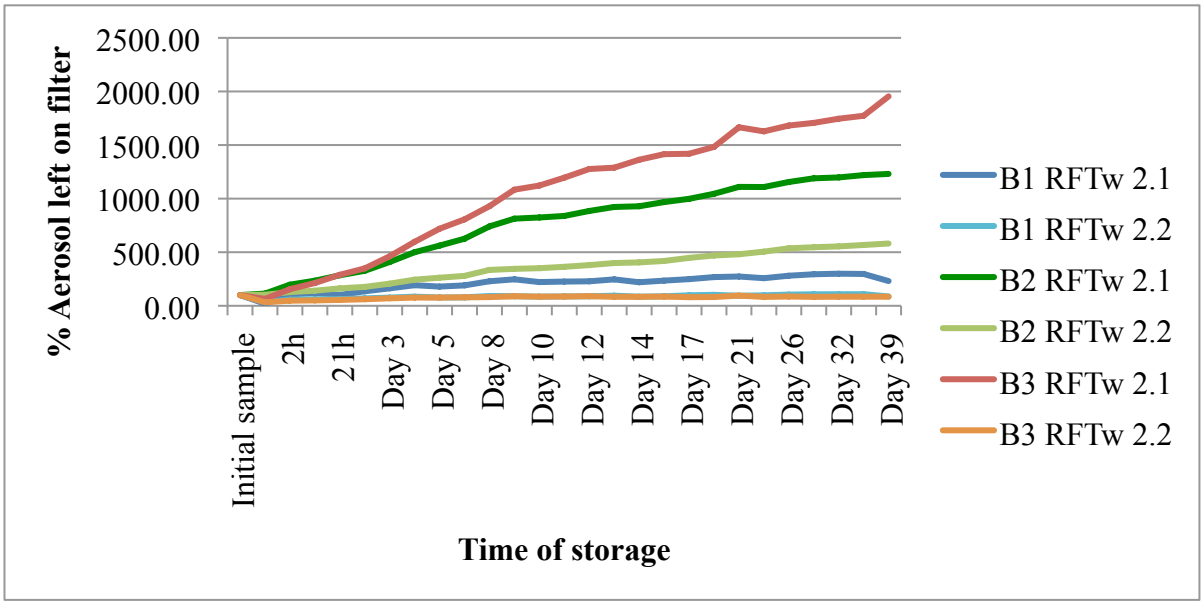

Figure 8-11 Loss of sample when storing at refrigerator temperature with plugs, series 2

Figure 8-11 shows a steady increase, with a higher increase for the lower concentration samples compared to the higher concentration samples.

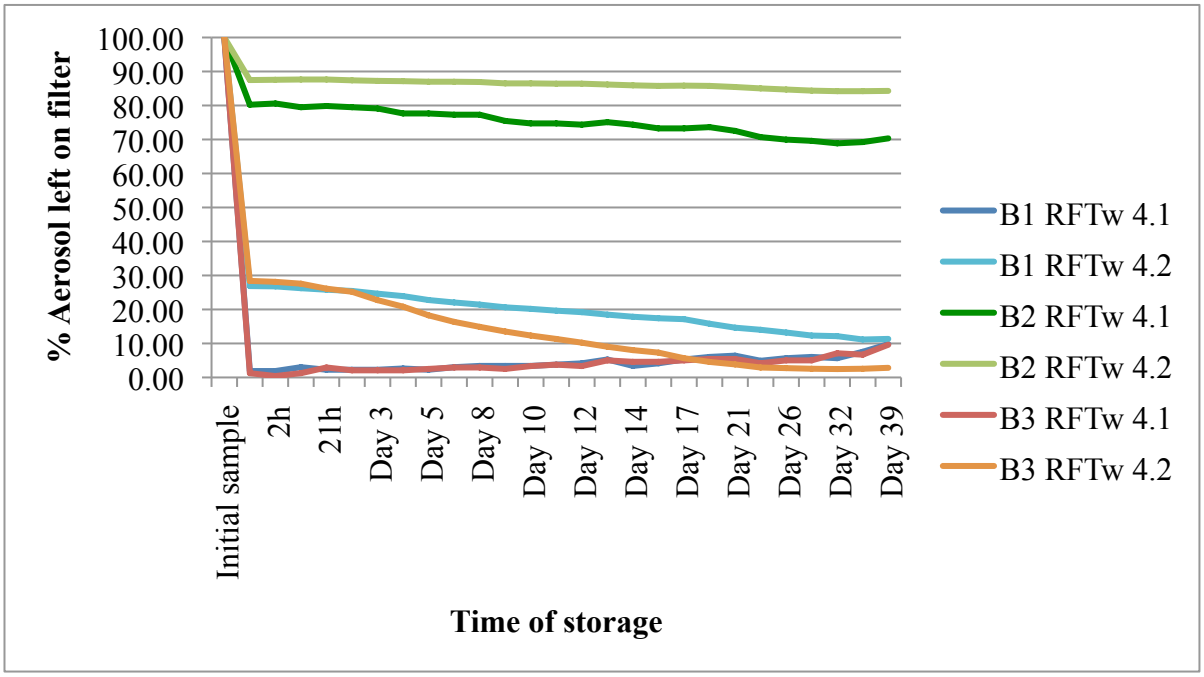

Figure 8-12 Loss of sample when storing at refrigerator temperature with plugs, series 4

Series 4 (Figure 8-12) show similar patterns to series 1, though it is more stable and the low concentration sample of base oil 2 does not increase.

8.2.2 Refrigerator temperature without plugs (RFTwo)

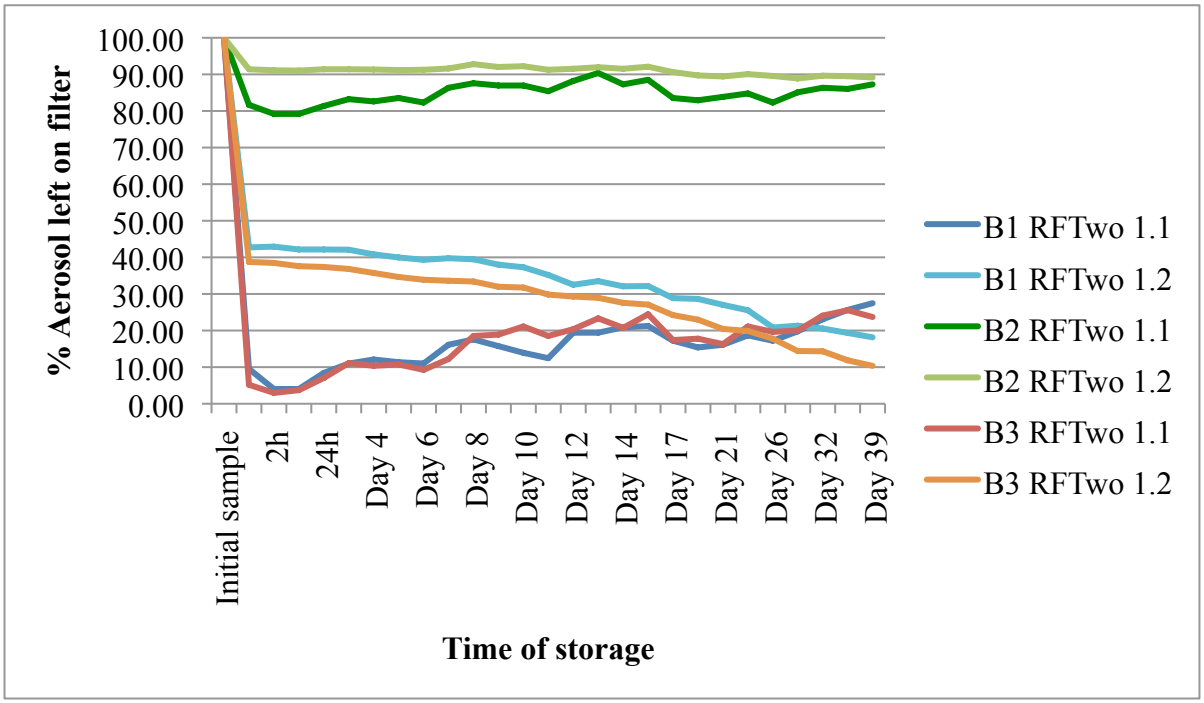

Figure 8-13 Loss of sample when storing at refrigerator temperature without plugs, series 1

Series 1 (Figure 8-13) shows a very similar pattern to series 1 for storing at refrigerator temperature with plugs (Figure 8-10).

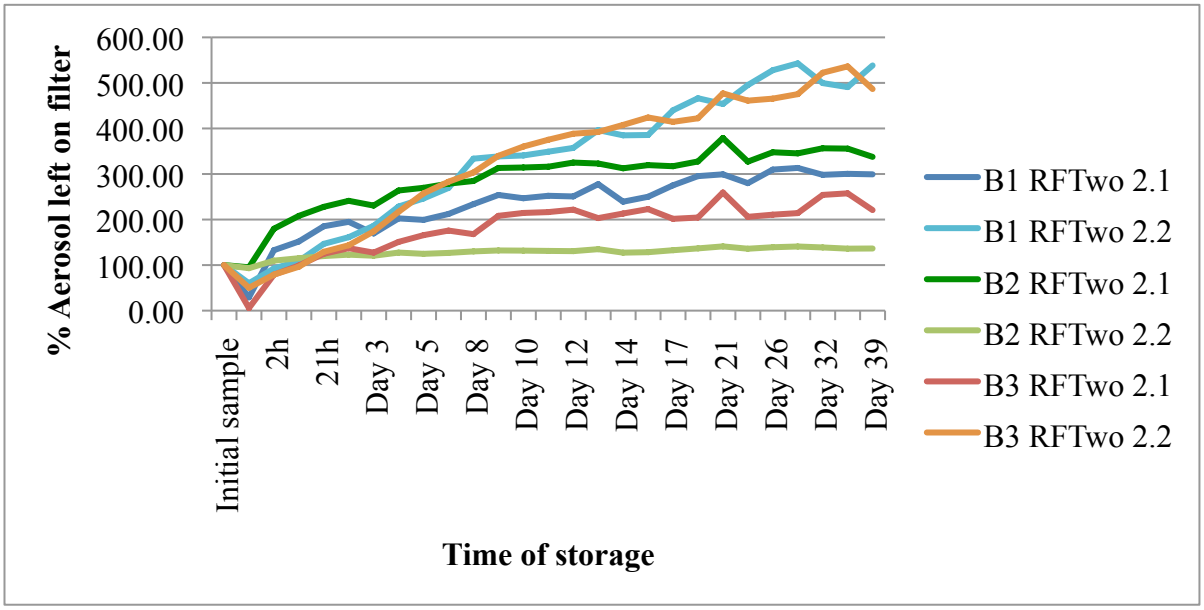

Figure 8-14 Loss of sample when storing at refrigerator temperature without plugs, series 2

Figure 8-14 shows an increase for all samples during storage.

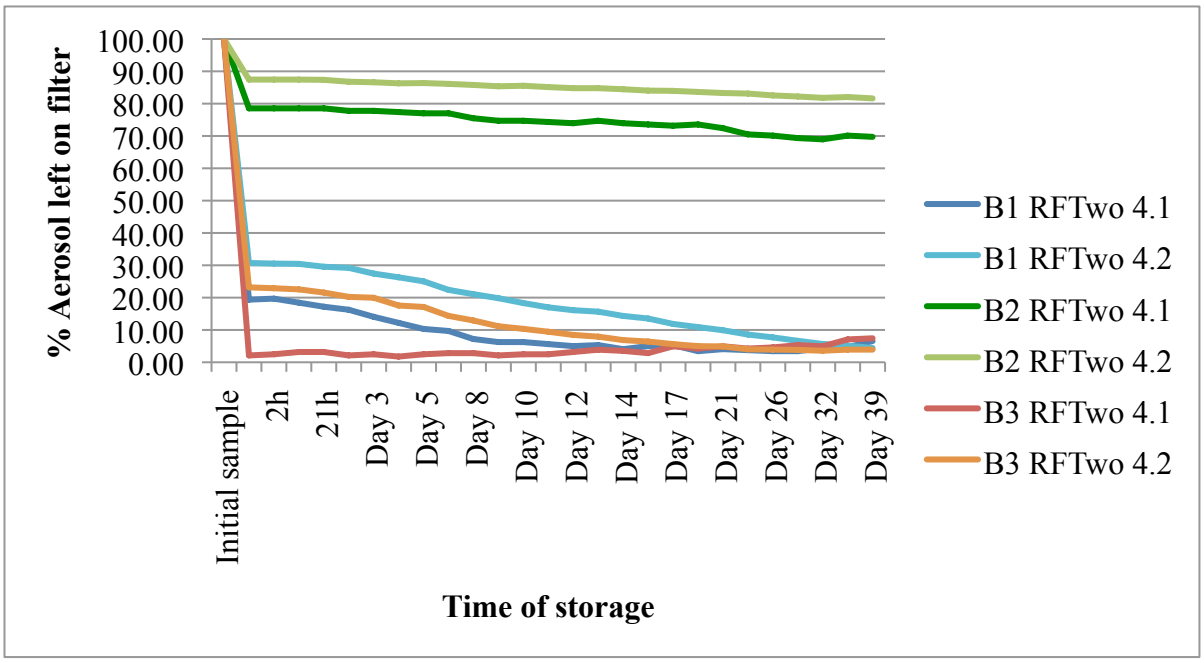

Figure 8-15 Loss of sample when storing at refrigerator temperature without plugs, series 4

Series 4 (Figure 8-15) shows a very similar pattern to series 4 for storing at refrigerator temperature with plugs (Figure 8-12).

8.3 Freezer temperature

At this temperature storage was tested with and without plugs on filter cassettes. The data for storing at freezer temperature may be found in appendix VIII.

8.3.1 Freezer temperature with plugs (FTw)

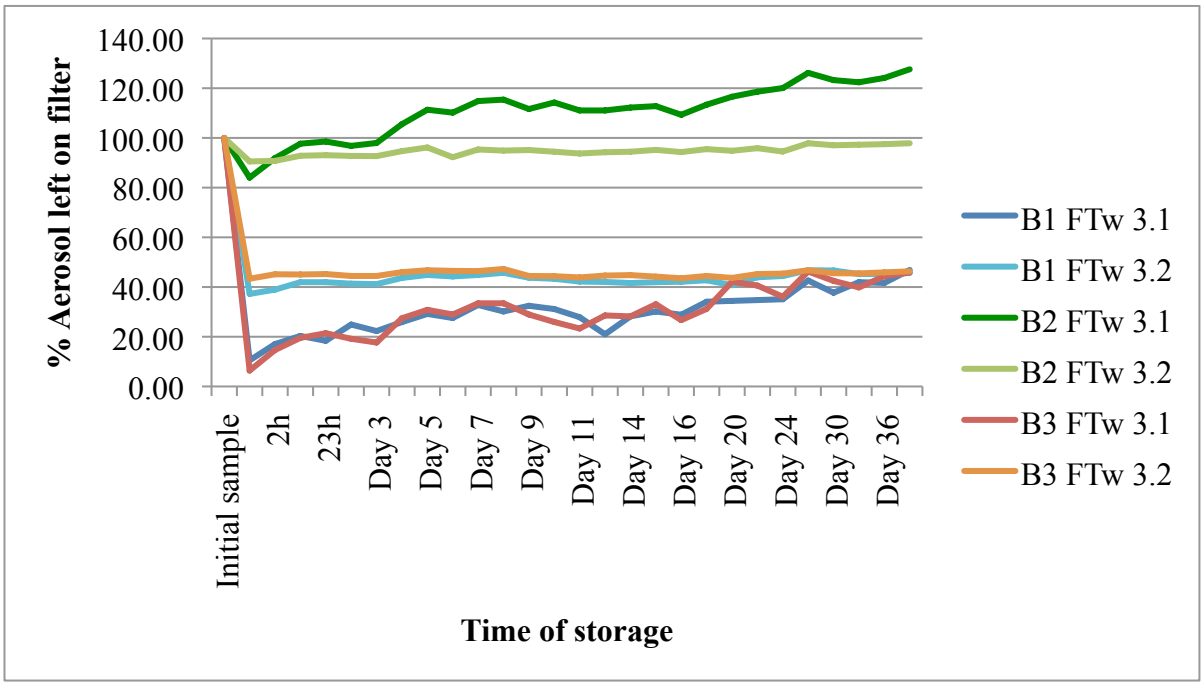

Figure 8-16 Loss of sample when storing at freezer temperature with plugs, series 3

Figure 8-16 shows similar patterns to series 1 (Figure 8-10 and Figure 8-13) for refrigerator temperature, though with a higher increase for the low concentration samples and also an increase for the high concentration samples.

8.3.2 Freezer temperature without plugs (FTwo)

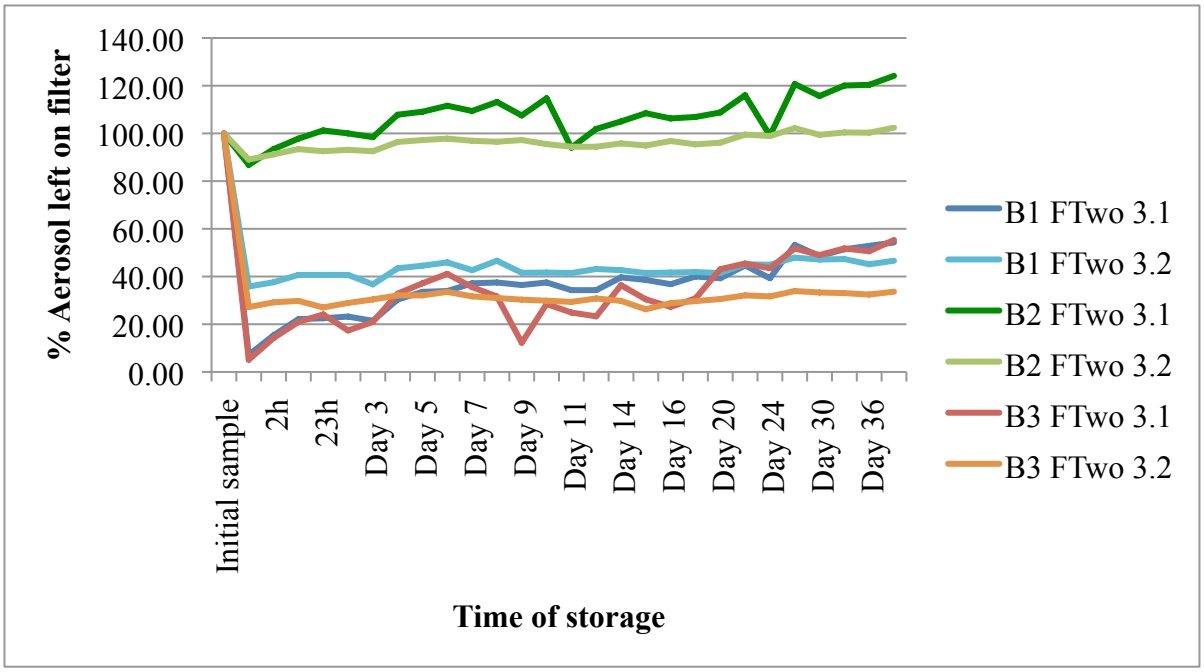

Figure 8-17 Loss of sample when storing at freezer temperature without plugs, series 3

Figure 8-17 shows a similar pattern to Figure 8-16, though the different concentrations for base oils 1 and 3 are closer to each other and have more overlap.

Discussion and conclusion

9 Discussion

Type of base oil

EDC Pearl and Sipdrill 2.0 have similar patterns for similar concentrations, which indicate similar properties for these base oils. This is confirmed by the GC-MS analysis. Most sample is also lost during aspiration for these oils, likely because of low viscosity giving a higher volatility. EDC 95/11 is less volatile and has a much lower fraction of sample lost during aspiration but has a similar pattern of loss to the other two base oils during storage.

Concentration

Samples of similar concentrations tend to have a similar pattern of loss. Though how much is lost during aspiration seem to be based on the properties of the base oil, more seem to be lost from lower concentration samples than higher concentration samples. Lower concentration samples also seem to gain more by percentage than higher concentration samples, most likely because a similar amount of environmental contamination on the different concentrations will give a higher effect for samples with a lower basis. As pointed out by Simpson & Keen (2007) traditional filter sampling and gravimetric analysis also does not distinguish between oil and other particulate matter present.

Method of weighing

From the results of series 2 it is clear that weighing the whole cassette with filter, to avoid loss from taking out the filter, did not have the intended effect. Instead all the contaminations inside and on the outside of the cassette were also weighed, giving a high increases instead of a stable weight.

Filter

Samples have similar patterns for the cellulose acetate plus glass fibre filter combination and double glass fibre filter, but are more stable for double glass fibre filter. Because the filter combinations were used in different series run at different times of the year environmental factors might account for some of these differences. For EDC Pearl and Sipdrill 2.0 more sample is lost during aspiration with a double glass fibre filter than with a cellulose acetate

plus glass fibre filter combination, this is probably due to how the base oil and filter react with each other.

Temperature

Where most sample is not lost during aspiration samples stored in room temperature have a steady drop, but with a higher loss for filter stored in exicator rather than in a filter cassette. In refrigerator temperature the loss is slower except for the lower concentration samples, which increases. When storing at freezer temperature the pattern is similar to the pattern for storing at refrigerator temperature, but with an increase for all samples. Differently to what was found by Malvik & Børresen (1988)

Plug

There does not seem to be much of a difference to the loss of sample in plugging the cassettes or not.

Storage time

Except for samples of EDC 95/11 stored at room temperature most sample is lost during aspiration rather than storage. How long a sample can be stored depends on the volatility of the sample, as high volatility (low viscosity) samples tend to lose more during aspiration.

10 Conclusion

The patterns of loss depend on the properties of the base oil and the concentration of the sample. Most sample is lost during aspiration for high volatility samples, but the degree of loss during storage has similar patterns. Lower concentration samples loose more than higher concentration samples during aspiration.

Filters should be taken out of the cassette before weighing rather than weighing the whole cassette with filter.

Storage on cellulose acetate plus glass fibre filter combination and on double glass fibre filter have similar patterns, but more sample is lost during aspiration with a double glass fibre filter than with a cellulose acetate plus glass fibre filter combination for EDC Pearl and Sipdrill 2.0.

Samples should be stored at refrigerator temperature for higher concentrations (ca. 70 mg/m^3) and at room temperature for low concentrations (ca. 20 mg/m^3). Plugs do not make much difference.

How long a sample can be stored depends on the volatility of the sample, as high volatility (low viscosity) samples tend to lose more during aspiration.

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Appendix I Chemicals and equipment used

Chemicals

- EDC Pearl (M-I Swaco)
- EDC 95/11 (M-I Swaco)
- Sipdrill 2.0 (M-I Swaco)

Equipment

Filters:

- Cellulose acetate:
  - MCE, 0.8μm, 37mm (SKC)
  - Type AA, 0.8μm, 37mm (Millipore)
- Glass fibre:
  - Type A/E Glass fibre filter (Pall corporation – Life sciences)

Pumps:

- Sidekick – 224-52TX Air sampling pump (SKC)
- Standard – Basic air sampling pump (SKC)
- S2500 Air sampler – Constant flow sampler model S2500 (DuPont)
- Personal air sampler – Models 222-3 and 222-4 (SKC)

Other equipment:

- Analytical weight – AE163 (Mettler)
- Mass flow meter – GFM17 (Aalborg)
- Tube, Anasorb CSC (coconut shell charcoal) (SKC)

Appendix II Safety data sheets

EDC Pearl

Xn – Harmful
R65 – Harmful: may cause lung damage if swallowed.
S62 – If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.
Contains: Distillate (petroleum), treated to hydrogen, medium
Viscosity: 3.5 cSt (20^{o}C)

EDC 95/11

Xn – Harmful
R65 – Harmful: may cause lung damage if swallowed.
S62 – If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.
Contains: Distillate (petroleum), treated to hydrogen, medium
Viscosity: 3.3-3.7 cSt (40^{o}C)

Sipdrill 2.0

Xn – Harmful
R65 – Harmful: may cause lung damage if swallowed.
S62 – If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.
Contains: Paraffins (petroleum), normal C5-20
Viscosity: < 2 cSt (40^{o}C)
Boiling point: 210-260^{o}C

Appendix III EDC Pearl GC-MS analysis AMDIS report

AMDIS GC/MS Analysis Report
Organization: SINTEF
Division: BIOTECH

Data: EDCPEARL.D
Library: C:\NIST08\AMDIS32\ONSITE.MSL
Number of Identifications: 72

RT(min) Chemical Name
5.1702 >3,4-Hexanedione, 2,2,5-trimethyl- (ID#:20633038)
5.2373 >Cyclotetrasiloxane, octamethyl- (ID#:556672)
5.2459 >Cyclotetrasiloxane, octamethyl- (ID#:556672)
6.3194 >Naphthalene, decahydro-, trans- (ID#:493027)
7.5874 >Undecane (ID#:1120214)
7.6880 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
8.0949 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
8.3065 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
8.3125 >trans-4a-Methyl-decahydronaphthalene (ID#:2547275)
8.3874 >Cyclohexane, (1-methylbutyl)- (ID#:61208944)
8.6704 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
8.7908 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
8.9190 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
9.0790 >Spiro[3.5]nonan-1-one, 5-methyl-, trans- (ID#:65147560)
9.1099 >Naphthalene, decahydro-2,6-dimethyl- (ID#:1618220)
9.1349 >Undecane, 4-methyl- (ID#:2980690)
9.2200 >Cyclopentasiloxane, decamethyl- (ID#:541026)
9.2553 >Naphthalene, decahydro-2-methyl- (ID#:2958761)
9.2605 >Decalin, syn-1-methyl-, cis- (ID#:EPA-158891)
9.4179 >Undecane, 3-methyl- (ID#:1002433)
9.4953 >Naphthalene, decahydro-2,6-dimethyl- (ID#:1618220)
9.5030 >Naphthalene, decahydro-2,6-dimethyl- (ID#:1618220)
9.9254 >cis,cis-1,6-Dimethylspiro[4.5]decane (ID#:EPA-111724)
10.2222 >Dodecane (ID#:112403)
10.5851 >Undecane, 2,6-dimethyl- (ID#:17301234)
10.6892 >Borane, diethyl(decyloxy)- (ID#:EPA-152343)
10.7821 >Undecane, 4,7-dimethyl- (ID#:17301325)
11.1185 >Cyclohexane, hexyl- (ID#:4292755)
11.2398 >Cyclotetradecane (ID#:295170)
11.5236 >1,1,6,6-Tetramethylspiro[4.4]nonane (ID#:74054925)
11.5993 >Undecane, 2,6-dimethyl- (ID#:17301234)
11.6441 >Undecane, 2,4-dimethyl- (ID#:17312800)
11.6527 >2,3-Dimethyldecane (ID#:17312446)
11.7714 >Dodecane, 4-methyl- (ID#:6117971)
11.8987 >Dodecane, 2-methyl- (ID#:1560970)
11.9907 >Cyclohexane, 1,1'-(1,2-dimethyl-1,2-ethanediyl)bis-, (R*,R*)-(ñ)- (ID#:54889871)
12.0664 >Dodecane, 3-methyl- (ID#:17312571)
12.1464 >Octane, 2,6-dimethyl- (ID#:2051301)

12.3013 >Dodecane, 2,6,10-trimethyl- (ID#:3891983)
12.4681 >Cyclopentane, 1-hydroxymethyl-1,3-dimethyl- (ID#:EPA-156738)
12.4785 >1-Tetradecene (ID#:1120361)
12.6969 >1,1'-Bicyclohexyl (ID#:92513)
12.8974 >Tridecane (ID#:629505)
13.1529 >Oxalic acid, 2-ethylhexyl tetradecyl ester (ID#:EPA-309397)
13.3051 >Dodecane, 2,5-dimethyl- (ID#:56292650)
13.5477 >Tetradecane, 6,9-dimethyl- (ID#:55045131)
13.7593 >Cyclohexasiloxane, dodecamethyl- (ID#:540976)
13.8229 >Heptylcyclohexane (ID#:5617414)
13.9176 >Cyclopentane, hexyl- (ID#:4457005)
14.0483 >1,1'-Bicyclohexyl, 2-methyl-, cis- (ID#:50991087)
14.1429 >Tridecane, 6-methyl- (ID#:13287213)
14.1464 >Tridecane, 6-methyl- (ID#:13287213)
14.2135 >Tridecane, 5-methyl- (ID#:25117311)
14.2195 >Tridecane, 5-methyl- (ID#:25117311)
14.3391 >Tridecane, 4-methyl- (ID#:26730121)
14.4741 >Tridecane, 2-methyl- (ID#:1560969)
14.6341 >Tridecane, 3-methyl- (ID#:6418413)
14.7993 >Dodecane, 2,6,10-trimethyl- (ID#:3891983)
14.9343 >Cyclotetradecane (ID#:295170)
15.0866 >Cyclohexane, 1-methyl-2-pentyl- (ID#:54411017)
15.4126 >Tetradecane (ID#:629594)
15.4874 >Decane, 2,3,5-trimethyl- (ID#:62238113)
15.9648 ?? Caffeine (ID#:58-08-2)
16.4216 >Cyclohexane, octyl- (ID#:1795159)
16.6848 >1-Octanol, 2-butyl- (ID#:3913028)
16.9085 >Heptadecane, 2,6,10,14-tetramethyl- (ID#:18344371)
16.9257 ?? Tetracosane (ID#:646-31-1)
16.9257 >Tridecane (ID#:629505)
17.0926 >Heptadecane, 2,6-dimethyl- (ID#:54105678)
17.8297 >Undecane, 5-methyl- (ID#:1632708)
17.8383 >Pentadecane (ID#:629629)
18.9411 >1-Azabicyclo[3.1.0]hexane (ID#:285767)

Appendix IV EDC 95/11 GC-MS analysis AMDIS report

AMDIS GC/MS Analysis Report
Organization: SINTEF
Division: BIOTECH

Data: EDC95 11.D
Library: C:\NIST08\AMDIS32\ONSITE.MSL
Number of Identifications: 103

RT(min) Chemical Name
5.2366 >Cyclotetrasiloxane, octamethyl- (ID#:556672)
7.6116 >3,4-Hexanedione, 2,2,5-trimethyl- (ID#:20633038)
7.6916 >trans-Decalin, 2-methyl- (ID#:EPA-152473)
9.2228 >Cyclopentasiloxane, decamethyl- (ID#:541026)
9.4258 >Octane, 2,2-dimethyl- (ID#:15869871)
9.7492 >Cyclohexane, 1-methyl-3-pentyl- (ID#:54411028)
10.2112 >Dodecane (ID#:112403)
10.5785 >Undecane, 2,6-dimethyl- (ID#:17301234)
11.1135 >Cyclohexane, hexyl- (ID#:4292755)
11.3182 >1,3-Dimethyladamantan-5-carboxylic acid, ethyl ester (ID#:63263092)
11.3982 >Dimethylphosphinic fluoride (ID#:753708)
11.4120 >Thiophen-2-methylamine, N,N-dibutyl- (ID#:EPA-310353)
11.5935 >Dodecane, 6-methyl- (ID#:6044719)
11.7629 >Dodecane, 4-methyl- (ID#:6117971)
11.8894 >Undecane, 3-methyl- (ID#:1002433)
12.0511 >Dodecane, 3-methyl- (ID#:17312571)
12.4511 >Cyclohexane, 1-isopropyl-1-methyl- (ID#:16580260)
12.6696 >Cyclohexane, (1-methylpropyl)- (ID#:7058017)
12.8348 >Tridecane (ID#:629505)
13.3999 >Decane, 3,3,5-trimethyl- (ID#:62338130)
13.5117 >trans-(2-Chlorovinyl)dimethylethoxysilane (ID#:EPA-139715)
13.5392 >Hexadecane (ID#:544763)
13.5436 >Heptadecane (ID#:629787)
13.6924 >Tridecane, 3-methyl- (ID#:6418413)
13.7595 >Cyclohexasiloxane, dodecamethyl- (ID#:540976)
13.8085 >Heptylcyclohexane (ID#:5617414)
13.8807 >Perhydrophenalene, (3aà, 6aà, 9aà, 9bá)- (ID#:40250644)
14.1371 >Tridecane, 6-methyl- (ID#:13287213)
14.2093 >Tridecane, 5-methyl- (ID#:25117311)
14.3298 >Tridecane, 4-methyl- (ID#:26730121)
14.4657 >Tridecane, 2-methyl- (ID#:1560969)
14.6274 >Tridecane, 3-methyl- (ID#:6418413)
14.7857 >Dodecane, 2,6,11-trimethyl- (ID#:31295564)
15.0833 >Cyclohexane, 1-methyl-2-propyl- (ID#:4291796)
15.3844 >Tetradecane (ID#:629594)
15.6734 >Undecane, 3-methyl- (ID#:1002433)
15.7586 >Octane, 5-ethyl-2-methyl- (ID#:62016186)
15.9177 >Undecane, 4,7-dimethyl- (ID#:17301325)

15.9418 >Naphthalene, 2-butyldecahydro- (ID#:6305528)
16.0596 >Hexadecane (ID#:544763)
16.2024 >Tetradecane, 3-methyl- (ID#:18435228)
16.4157 >Cyclohexane, octyl- (ID#:1795159)
16.5895 >Octane, 2,4,6-trimethyl- (ID#:62016379)
16.5947 >Decane, 6-ethyl-2-methyl- (ID#:62108218)
16.6781 >10-Methylnonadecane (ID#:56862625)
16.8080 >Tetradecane, 4-methyl- (ID#:25117242)
16.9362 >Tetradecane, 2-methyl- (ID#:1560958)
17.1005 >Tetradecane, 3-methyl- (ID#:18435228)
17.6140 >Cyclohexane, 1-methyl-3-propyl- (ID#:4291809)
17.8239 >Pentadecane (ID#:629629)
18.0054 >Cycloheptasiloxane, tetradecamethyl- (ID#:107506)
18.0278 >Tetradecane, 5-methyl- (ID#:25117322)
18.1731 >Nonadecane (ID#:629925)
18.3237 >Undecane, 5,7-dimethyl- (ID#:17312833)
18.8295 >1,2-Bis(diethylamino)-1,2-bis(4-cyanophenyl)ethane (ID#:EPA-193300)
18.9060 >2,3-Dimethyldecane (ID#:17312446)
19.0419 >Pentadecane, 5-methyl- (ID#:25117333)
19.1667 >Tridecane, 3-ethyl- (ID#:13286732)
19.2966 >Pentadecane, 2-methyl- (ID#:1560936)
19.4540 >Pentadecane, 3-methyl- (ID#:2882964)
19.5839 >Sulfurous acid, 2-ethylhexyl hexyl ester (ID#:EPA-309202)
19.8290 >2-Decene, 5-methyl-, (Z)- (ID#:74645866)
19.9959 >Cyclohexane, ethyl- (ID#:1678917)
20.0140 >Cyclopentane, 1-hydroxymethyl-1,3-dimethyl- (ID#:EPA-156738)
20.1378 >Hexadecane (ID#:544763)
20.3021 >Nonane, 2-methyl-5-propyl- (ID#:31081171)
20.4380 >Pentadecane, 2,6,10,14-tetramethyl- (ID#:1921706)
20.4552 >Pentadecane, 2,6,10,14-tetramethyl- (ID#:1921706)
20.5198 >2-Butyl-3,4,5,6-tetrahydropyridine (ID#:1462948)
20.7331 >Heptadecane, 4-methyl- (ID#:26429118)
20.8776 >Octane, 2,4,6-trimethyl- (ID#:62016379)
20.8957 >Hexadecane (ID#:544763)
21.1468 >Decane, 5-propyl- (ID#:17312628)
21.4161 >Hexadecane, 4-methyl- (ID#:25117264)
21.5374 >Pentadecane, 3-methyl- (ID#:2882964)
21.6922 >Hexadecane, 3-methyl- (ID#:6418435)
21.8479 >Cyclooctasiloxane, hexadecamethyl- (ID#:556683)
22.3399 >Heptadecane (ID#:629787)
22.3425 >Heptadecane (ID#:629787)
22.4578 >Hexadecane (ID#:544763)
22.5963 >Octatetracontane, 1-iodo- (ID#:40710701)
23.2595 >Decane, 5-propyl- (ID#:17312628)
23.2767 >Decane, 2-methyl- (ID#:6975980)
23.6655 >Heptadecane, 2-methyl- (ID#:1560890)
23.8263 >Heptadecane, 3-methyl- (ID#:6418446)
24.4371 >Octadecane (ID#:593453)
24.6126 >Undecane (ID#:1120214)
24.6186 >Dodecane, 2,6,10-trimethyl- (ID#:3891983)

24.6367 >Tridecane (ID#:629505)
25.3059 >Eicosane, 10-methyl- (ID#:54833237)
25.4693 >Octadecane, 5-methyl- (ID#:25117355)
25.7033 >Undecane, 3,8-dimethyl- (ID#:17301303)
25.8642 >Octadecane, 3-methyl- (ID#:6561440)
26.4482 >Nonadecane (ID#:629925)
27.2517 >Tridecane, 6-propyl- (ID#:55045108)
27.6620 >Nonadecane, 2-methyl- (ID#:1560867)
27.6697 >Nonadecane, 2-methyl- (ID#:1560867)
28.3768 ? Tetracosane (ID#:646-31-1)
28.3768 >Heneicosane (ID#:629947)
30.2340 >Nonane, 3-methyl-5-propyl- (ID#:31081182)
32.0146 ?? Hexacosane (ID#:630-01-3)
32.0146 >Hexadecane (ID#:544763)
33.7109 >Nonane, 1-iodo- (ID#:4282422)

Appendix V Sipdrill 2.0 GC-MS analysis AMDIS report

AMDIS GC/MS Analysis Report
Organization: SINTEF
Division: BIOTECH

Data: SIPDRILL20.D
Library: C:\NIST08\AMDIS32\ONSITE.MSL
Number of Identifications: 20

RT(min) Chemical Name
3.2151 >Hexane, 2,4-dimethyl- (ID#:589435)
5.1609 >Decane (ID#:124185)
5.2374 >1-[2,4-Bis(trimethylsiloxy)phenyl]-2-[(4-trimethylsiloxy)phenyl]propan-1-one (ID#:102907880)
7.5883 >Undecane (ID#:1120214)
9.2245 >Cyclopentasiloxane, decamethyl- (ID#:541026)
10.2653 >Dodecane (ID#:112403)
10.5784 >Undecane, 2,6-dimethyl- (ID#:17301234)
11.6167 >Undecane, 2,6-dimethyl- (ID#:17301234)
11.7586 >Undecane, 2,3-dimethyl- (ID#:17312775)
11.8868 >Dodecane, 2-methyl- (ID#:1560970)
12.0511 >Dodecane, 3-methyl- (ID#:17312571)
12.0597 >Dodecane, 3-methyl- (ID#:17312571)
12.1259 >Nonane, 3-methyl- (ID#:5911046)
12.4330 >Naphthalene, 1,2,3,4-tetrahydro-6-methyl- (ID#:1680519)
12.9551 >Tridecane (ID#:629505)
13.0024 >Tridecane (ID#:629505)
13.7517 >Cyclohexasiloxane, dodecamethyl- (ID#:540976)
14.4837 >1-Methyl-2-n-hexylbenzene (ID#:1595104)
15.4041 >Tetradecane (ID#:629594)

Appendix VI Data for results at room temperature

Room temperature with plugs

Data for storing at room temperature with plugs are shown tables A- 1-6.

Table A – 1 Storage data for room temperature with plugs series 1 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTw 1.2	B1 RTw 1.2	B2 RTw 1.1	B2 RTw 1.1	B2 RTw 1.2	B2 RTw 1.2
Initial sample	11.72	100.00	3.03	100.00	14.82	100.00
0h	7.13	60.84	2.82	93.07	12.05	81.31
2h	7.01	59.81	2.82	93.07	11.99	80.90
6h	6.83	58.28	2.82	93.07	11.96	80.70
23h	6.18	52.73	2.55	84.16	11.77	79.42
28h	6.04	51.54	2.55	84.16	11.77	79.42
Day 3	5.63	48.04	2.44	80.53	11.73	79.15
Day 4	5.40	46.08	2.44	80.53	11.80	79.62
Day 5	5.13	43.77	2.36	77.89	11.76	79.35
Day 6	4.77	40.70	2.33	76.90	11.70	78.95
Day 7	4.49	38.31	2.25	74.26	11.66	78.68
Day 8	4.37	37.29	2.33	76.90	11.72	79.08
Day 9	4.08	34.81	2.34	77.23	11.73	79.15
Day 10	3.71	31.66	2.19	72.28	11.62	78.41
Day 11	3.47	29.61	2.19	72.28	11.64	78.54
Day 12	3.08	26.28	1.94	64.03	11.49	77.53
Day 13	2.99	25.51	2.06	67.99	11.47	77.40
Day 14	2.92	24.91	2.13	70.30	11.55	77.94
Day 15	2.68	22.87	1.97	65.02	11.47	77.40
Day 16	2.5	21.33	2.03	67.00	11.46	77.33
Day 18	2.11	18.00	1.77	58.42	11.27	76.05
Day 20	1.65	14.08	1.57	51.82	11.13	75.10
Day 22	1.34	11.43	1.52	50.17	11.05	74.56
Day 24	1.13	9.64	1.51	49.83	10.99	74.16
Day 27	0.86	7.34	1.43	47.19	10.94	73.82
Day 30	0.52	4.44	1.35	44.55	10.83	73.08
Day 33	0.6	5.12	1.54	50.83	10.94	73.82
Day 36	0.39	3.33	1.48	48.84	10.90	73.55
Day 40	0.38	3.24	1.51	49.83	10.94	73.82

Table A – 2 Storage data for room temperature with plugs series 1 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss
Time	B3 RTw 1.1	B3 RTw 1.1	B3 RTw 1.2	B3 RTw 1.2
Initial sample	2.77	100.00	13.31	100.00
0h	0.37	13.36	0.29	2.18
2h	0.31	11.19	0.26	1.95
6h	0.29	10.47	0.26	1.95
23h	0.05	1.81	0.1	0.75
28h	-0.02	-0.72	0.08	0.60
Day 3	0.05	1.81	0.08	0.60
Day 4	0.19	6.86	0.18	1.35
Day 5	0.16	5.78	0.16	1.20
Day 6	0.12	4.33	0.15	1.13
Day 7	0.08	2.89	0.12	0.90
Day 8	0.22	7.94	0.22	1.65
Day 9	0.24	8.66	0.23	1.73
Day 10	0.13	4.69	0.18	1.35
Day 11	0.17	6.14	0.21	1.58
Day 12	0.06	2.17	0.08	0.60
Day 13	0.11	3.97	0.14	1.05
Day 14	0.25	9.03	0.21	1.58
Day 15	0.15	5.42	0.16	1.20
Day 16	0.17	6.14	0.16	1.20
Day 18	0.01	0.36	0.06	0.45
Day 20	-0.17	-6.14	-0.05	-0.38
Day 22	-0.15	-5.42	-0.08	-0.60
Day 24	-0.14	-5.05	-0.1	-0.75
Day 27	-0.13	-4.69	-0.12	-0.90
Day 30	-0.14	-5.05	-0.14	-1.05
Day 33	0.1	3.61	0.02	0.15
Day 36	0.1	3.61	0.04	0.30
Day 40	0.17	6.14	0.12	0.90

Table A – 3 Storage data for room temperature with plugs series 2 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTw 2.1	B1 RTw 2.1	B1 RTw 2.2	B1 RTw 2.2	B2 RTw 2.1	B2 RTw 2.1
Initial sample	3.61	100.00	13.48	100.00	5.60	100.00
0h	3.41	94.46	7.00	51.93	7.09	126.61
2h	3.68	101.94	7.44	55.19	7.94	141.79
6h	4.88	135.18	8.44	62.61	9.31	166.25
23h	5.96	165.10	8.65	64.17	10.45	186.61
28h	6.72	186.15	9.61	71.29	11.47	204.82
Day 3	9.08	251.52	11.12	82.49	13.74	245.36
Day 4	10.51	291.14	12.07	89.54	15.30	273.21
Day 5	13.28	367.87	13.90	103.12	18.23	325.54
Day 6	14.43	399.72	14.62	108.46	19.36	345.71
Day 7	15.08	417.73	14.87	110.31	19.94	356.07
Day 9	17.62	488.09	16.59	123.07	22.33	398.75
Day 10	18.01	498.89	16.97	125.89	22.78	406.79
Day 11	17.75	491.69	16.66	123.59	22.28	397.86
Day 12	17.26	478.12	16.26	120.62	21.48	383.57
Day 13	16.80	465.37	15.80	117.21	20.95	374.11
Day 14	16.97	470.08	15.84	117.51	21.35	381.25
Day 15	16.70	462.60	15.47	114.76	20.80	371.43
Day 16	16.25	450.14	15.14	112.31	20.22	361.07
Day 18	14.25	394.74	13.69	101.56	17.82	318.21
Day 20	14.57	403.60	13.76	102.08	18.37	328.04
Day 22	16.87	467.31	15.51	115.06	21.12	377.14
Day 24	18.36	508.59	16.35	121.29	22.74	406.07
Day 27	17.40	481.99	15.61	115.80	21.18	378.21
Day 30	18.05	500.00	15.80	117.21	21.81	389.46
Day 33	17.93	496.68	15.63	115.95	21.89	390.89
Day 36	20.73	574.24	17.56	130.27	25.05	447.32
Day 40	23.80	659.28	19.52	144.81	28.30	505.36

Table A – 4 Storage data for room temperature with plugs series 2 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RTw 2.2	B2 RTw 2.2	B3 RTw 2.1	B3 RTw 2.1	B3 RTw 2.2	B3 RTw 2.2
Initial sample	13.13	100.00	3.66	100.00	10.73	100.00
0h	12.05	91.77	3.47	94.81	3.71	34.58
2h	12.33	93.91	4.12	112.57	3.88	36.16
6h	12.76	97.18	5.63	153.83	4.29	39.98
23h	12.63	96.19	6.65	181.69	4.07	37.93
28h	12.89	98.17	7.81	213.39	4.42	41.19
Day 3	13.14	100.08	9.60	262.30	4.43	41.29
Day 4	12.99	98.93	11.21	306.28	4.42	41.19
Day 5	13.72	104.49	13.99	382.24	5.06	47.16
Day 6	13.46	102.51	15.03	410.66	4.81	44.83
Day 7	13.38	101.90	15.52	424.04	4.67	43.52
Day 9	14.23	108.38	17.98	491.26	5.40	50.33
Day 10	14.36	109.37	18.30	500.00	5.42	50.51
Day 11	13.94	106.17	17.75	484.97	5.13	47.81
Day 12	13.89	105.79	17.14	468.31	4.96	46.23
Day 13	13.71	104.42	16.76	457.92	4.79	44.64
Day 14	13.64	103.88	17.11	467.49	4.66	43.43
Day 15	13.11	99.85	16.65	454.92	4.29	39.98
Day 16	13.15	100.15	16.05	438.52	4.11	38.30
Day 18	12.92	98.40	13.86	378.69	3.79	35.32
Day 20	13.01	99.09	14.42	393.99	3.94	36.72
Day 22	14.02	106.78	17.03	465.30	4.81	44.83
Day 24	13.70	104.34	18.49	505.19	4.51	42.03
Day 27	13.32	101.45	17.22	470.49	4.15	38.68
Day 30	13.49	102.74	17.76	485.25	4.27	39.79
Day 33	13.50	102.82	17.93	489.89	4.35	40.54
Day 36	13.90	105.86	20.92	571.58	4.81	44.83
Day 40	14.26	108.61	23.98	655.19	5.03	46.88

Table A – 5 Storage data for room temperature with plugs series 4 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTw 4.1	B1 RTw 4.1	B1 RTw 4.2	B1 RTw 4.2	B2 RTw 4.1	B2 RTw 4.1
Initial sample	2.65	100.00	10.18	100.00	3.01	100.00
0h	0.32	12.08	3.11	30.55	2.39	79.40
2h	0.26	9.81	3.04	29.86	2.40	79.73
6h	0.19	7.17	2.86	28.09	2.34	77.74
23h	0.10	3.77	2.57	25.25	2.31	76.74
28h	0.06	2.26	2.47	24.26	2.29	76.08
Day 3	0.05	1.89	2.14	21.02	2.25	74.75
Day 4	0.03	1.13	1.81	17.78	2.18	72.43
Day 5	0.03	1.13	1.56	15.32	2.12	70.43
Day 6	0.04	1.51	1.33	13.06	2.12	70.43
Day 8	0.04	1.51	1.07	10.51	2.02	67.11
Day 9	0.04	1.51	0.90	8.84	2.00	66.45
Day 10	0.02	0.75	0.73	7.17	1.92	63.79
Day 11	0.03	1.13	0.62	6.09	1.90	63.12
Day 12	0.02	0.75	0.51	5.01	1.88	62.46
Day 13	0.03	1.13	0.44	4.32	1.85	61.46
Day 14	0.04	1.51	0.41	4.03	1.83	60.80
Day 15	0.02	0.75	0.28	2.75	1.78	59.14
Day 16	0.03	1.13	0.27	2.65	1.76	58.47
Day 18	0.06	2.26	0.22	2.16	1.71	56.81
Day 20	0.01	0.38	0.18	1.77	1.64	54.49
Day 22	0.05	1.89	0.17	1.67	1.60	53.16
Day 24	0.03	1.13	0.12	1.18	1.53	50.83
Day 27	0.02	0.75	0.12	1.18	1.48	49.17
Day 30	0.01	0.38	0.11	1.08	1.39	46.18
Day 33	0.01	0.38	0.14	1.38	1.35	44.85
Day 36	0.05	1.89	0.16	1.57	1.35	44.85
Day 40	0.08	3.02	0.16	1.57	1.30	43.19

Table A – 6 Storage data for room temperature with plugs series 4 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RTw 4.2	B2 RTw 4.2	B3 RTw 4.1	B3 RTw 4.1	B3 RTw 4.2	B3 RTw 4.2
Initial sample	6.44	100.00	2.67	100.00	6.78	100.00
0h	5.59	86.80	0.06	2.25	1.43	21.09
2h	5.58	86.65	0.05	1.87	1.35	19.91
6h	5.56	86.34	0.03	1.12	1.29	19.03
23h	5.52	85.71	0.04	1.50	1.07	15.78
28h	5.52	85.71	0.05	1.87	0.98	14.45
Day 3	5.49	85.25	0.06	2.25	0.95	14.01
Day 4	5.40	83.85	0.05	1.87	0.63	9.29
Day 5	5.39	83.70	0.06	2.25	0.49	7.23
Day 6	5.33	82.76	0.09	3.37	0.39	5.75
Day 8	5.25	81.52	0.10	3.75	0.25	3.69
Day 9	5.20	80.75	0.08	3.00	0.22	3.24
Day 10	5.15	79.97	0.07	2.62	0.16	2.36
Day 11	5.11	79.35	0.07	2.62	0.15	2.21
Day 12	5.07	78.73	0.10	3.75	0.14	2.06
Day 13	5.07	78.73	0.10	3.75	0.15	2.21
Day 14	5.03	78.11	0.12	4.49	0.16	2.36
Day 15	4.92	76.40	0.10	3.75	0.10	1.47
Day 16	4.93	76.55	0.09	3.37	0.13	1.92
Day 18	4.86	75.47	0.11	4.12	0.13	1.92
Day 20	4.81	74.69	0.10	3.75	0.09	1.33
Day 22	4.73	73.45	0.12	4.49	0.10	1.47
Day 24	4.63	71.89	0.09	3.37	0.10	1.47
Day 27	4.56	70.81	0.09	3.37	0.09	1.33
Day 30	4.45	69.10	0.09	3.37	0.05	0.74
Day 33	4.37	67.86	0.09	3.37	0.06	0.88
Day 36	4.32	67.08	0.12	4.49	0.09	1.33
Day 40	4.24	65.84	0.16	5.99	0.13	1.92

Room temperature without plugs

Tables A- 7 – 12 shows data for storing at room temperature without plugs.

Table A – 7 Storage data for room temperature withot plugs series 1 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTwo 1.1	B1 RTwo 1.1	B1 RTwo 1.2	B1 RTwo 1.2	B2 RTwo 1.1	B2 RTwo 1.1
Initial sample	3.60	100.00	12.08	100.00	3.20	100.00
0h	0.23	6.39	0.26	2.15	2.47	77.19
2h	0.22	6.11	0.24	1.99	2.45	76.56
19h	0.02	0.56	0.05	0.41	2.23	69.69
24h	0.00	0.00	0.03	0.25	2.19	68.44
Day 3	0.05	1.39	0.06	0.50	2.19	68.44
Day 4	0.13	3.61	0.18	1.49	2.28	71.25
Day 5	0.06	1.67	0.14	1.16	2.24	70.00
Day 6	0.11	3.06	0.11	0.91	2.17	67.81
Day 7	0.04	1.11	0.08	0.66	2.08	65.00
Day 8	0.19	5.28	0.21	1.74	2.17	67.81
Day 9	0.21	5.83	0.22	1.82	2.23	69.69
Day 10	0.12	3.33	0.13	1.08	2.09	65.31
Day 11	0.15	4.17	0.15	1.24	2.12	66.25
Day 12	-0.03	-0.83	0.00	0.00	1.91	59.69
Day 13	0.09	2.50	0.07	0.58	1.98	61.87
Day 14	0.21	5.83	0.18	1.49	2.12	66.25
Day 15	0.11	3.06	0.12	0.99	1.96	61.25
Day 16	0.14	3.89	0.10	0.83	1.97	61.56
Day 18	-0.03	-0.83	-0.03	-0.25	1.77	55.31
Day 20	-0.21	-5.83	-0.19	-1.57	1.55	48.44
Day 22	-0.17	-4.72	-0.18	-1.49	1.57	49.06
Day 24	-0.17	-4.72	-0.14	-1.16	1.55	48.44
Day 27	-0.18	-5.00	-0.15	-1.24	1.50	46.88
Day 30	-0.20	-5.56	-0.23	-1.90	1.43	44.69
Day 33	0.05	1.39	0.03	0.25	1.63	50.94
Day 36	0.02	0.56	0.02	0.17	1.54	48.12
Day 40	0.11	3.06	0.10	0.83	1.61	50.31

Table A – 8 Storage data for room temperature without plugs series 1 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RTwo 1.2	B2 RTwo 1.2	B3 RTwo 1.1	B3 RTwo 1.1	B3 RTwo 1.2	B3 RTwo 1.2
Initial sample	14.03	100.00	2.91	100.00	13.43	100.00
0h	11.93	85.03	0.18	6.19	0.34	2.53
2h	11.89	84.75	0.14	4.81	0.29	2.16
19h	11.70	83.39	-0.01	-0.34	0.05	0.37
24h	11.63	82.89	-0.03	-1.03	0.04	0.30
Day 3	11.61	82.75	0.02	0.69	0.11	0.82
Day 4	11.68	83.25	0.12	4.12	0.22	1.64
Day 5	11.62	82.82	0.10	3.44	0.16	1.19
Day 6	11.54	82.25	0.15	5.15	0.20	1.49
Day 7	11.46	81.68	0.06	2.06	0.13	0.97
Day 8	11.49	81.90	0.18	6.19	0.34	2.53
Day 9	11.47	81.75	0.19	6.53	0.28	2.08
Day 10	11.31	80.61	0.11	3.78	0.23	1.71
Day 11	11.27	80.33	0.16	5.50	0.22	1.64
Day 12	11.06	78.83	0.01	0.34	0.10	0.74
Day 13	11.10	79.12	0.10	3.44	0.21	1.56
Day 14	11.11	79.19	0.20	6.87	0.26	1.94
Day 15	11.02	78.55	0.10	3.44	0.16	1.19
Day 16	10.98	78.26	0.14	4.81	0.26	1.94
Day 18	10.74	76.55	0.01	0.34	0.07	0.52
Day 20	10.52	74.98	-0.13	-4.47	-0.13	-0.97
Day 22	10.40	74.13	-0.17	-5.84	-0.09	-0.67
Day 24	10.28	73.27	-0.16	-5.50	-0.09	-0.67
Day 27	10.14	72.27	-0.14	-4.81	-0.12	-0.89
Day 30	9.97	71.06	-0.18	-6.19	-0.14	-1.04
Day 33	10.11	72.06	0.00	0.00	0.18	1.34
Day 36	9.92	70.71	-0.08	-2.75	0.11	0.82
Day 40	9.88	70.42	0.04	1.37	0.21	1.56

Table A – 9 Storage data for room temperature without plugs series 2 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTwo 2.1	B1 RTwo 2.1	B1 RTwo 2.2	B1 RTwo 2.2	B2 RTwo 2.1	B2 RTwo 2.1
Initial sample	2.88	100.00	10.86	100.00	3.76	100.00
0h	0.52	18.06	5.89	54.24	3.45	91.76
2h	1.26	43.75	7.21	66.39	4.20	111.70
6h	1.74	60.42	8.70	80.11	4.54	120.74
23h	1.43	49.65	10.18	93.74	4.35	115.69
28h	1.91	66.32	11.50	105.89	4.65	123.67
Day 3	2.17	75.35	13.68	125.97	4.90	130.32
Day 4	2.30	79.86	15.79	145.40	4.91	130.59
Day 5	3.03	105.21	19.22	176.98	5.74	152.66
Day 6	2.83	98.26	20.35	187.38	5.30	140.96
Day 7	2.68	93.06	20.62	189.87	5.23	139.10
Day 9	3.50	121.53	22.96	211.42	5.92	157.45
Day 10	3.52	122.22	23.22	213.81	5.99	159.31
Day 11	3.28	113.89	22.53	207.46	5.59	148.67
Day 12	3.01	104.51	21.01	193.46	5.44	144.68
Day 13	2.79	96.87	20.04	184.53	5.24	139.36
Day 14	2.81	97.57	20.39	187.75	5.24	139.36
Day 15	2.42	84.03	19.91	183.33	4.68	124.47
Day 16	2.16	75.00	18.87	173.76	4.54	120.74
Day 18	1.90	65.97	15.74	144.94	4.21	111.97
Day 20	2.32	80.56	16.68	153.59	4.53	120.48
Day 22	3.46	120.14	19.92	183.43	5.66	150.53
Day 24	3.27	113.54	22.52	207.37	5.34	142.02
Day 27	2.95	102.43	20.51	188.86	4.90	130.32
Day 30	3.03	105.21	21.08	194.11	5.02	133.51
Day 33	2.99	103.82	20.46	188.40	4.98	132.45
Day 36	3.53	122.57	24.22	223.02	5.40	143.62
Day 40	3.91	135.76	28.23	259.94	5.82	154.79

Table A – 10 Storage data for room temperature without plugs series 2 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RTwo 2.2	B2 RTwo 2.2	B3 RTwo 2.1	B3 RTwo 2.1	B3 RTwo 2.2	B3 RTwo 2.2
Initial sample	12.52	100.00	2.70	100.00	10.86	100.00
0h	11.64	92.97	0.56	20.74	5.49	50.55
2h	12.25	97.84	1.02	37.78	6.63	61.05
6h	12.84	102.56	1.53	56.67	8.27	76.15
23h	12.60	100.64	1.48	54.81	10.10	93.00
28h	12.76	101.92	1.66	61.48	11.06	101.84
Day 3	12.99	103.75	1.81	67.04	12.72	117.13
Day 4	13.22	105.59	2.02	74.81	14.59	134.35
Day 5	13.74	109.74	2.78	102.96	17.70	162.98
Day 6	13.41	107.11	2.44	90.37	18.22	167.77
Day 7	13.31	106.31	2.36	87.41	18.26	168.14
Day 9	13.95	111.42	3.09	114.44	20.16	185.64
Day 10	14.04	112.14	3.18	117.78	20.17	185.73
Day 11	13.67	109.19	2.83	104.81	19.20	176.80
Day 12	13.40	107.03	2.61	96.67	17.60	162.06
Day 13	13.18	105.27	2.48	91.85	16.91	155.71
Day 14	13.14	104.95	2.57	95.19	17.45	160.68
Day 15	12.59	100.56	2.05	75.93	16.97	156.26
Day 16	12.47	99.60	1.90	70.37	15.88	146.22
Day 18	12.02	96.01	1.68	62.22	12.87	118.51
Day 20	12.27	98.00	2.02	74.81	14.24	131.12
Day 22	13.45	107.43	3.19	118.15	18.00	165.75
Day 24	13.04	104.15	2.83	104.81	20.06	184.71
Day 27	12.48	99.68	2.52	93.33	17.79	163.81
Day 30	12.54	100.16	2.68	99.26	18.37	169.15
Day 33	12.42	99.20	2.69	99.63	18.05	166.21
Day 36	12.83	102.48	3.20	118.52	21.80	200.74
Day 40	13.17	105.19	3.63	134.44	25.55	235.27

Table A – 11 Storage data for room temperature without plugs series 4 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTwo 4.1	B1 RTwo 4.1	B1 RTwo 4.2	B1 RTwo 4.2	B2 RTwo 4.1	B2 RTwo 4.1
Initial sample	2.57	100.00	11.52	100.00	2.89	100.00
0h	0.29	11.28	3.57	30.99	2.36	81.66
2h	0.24	9.34	3.48	30.21	2.35	81.31
4h	0.19	7.39	3.42	29.69	2.34	80.97
23h	0.10	3.89	3.22	27.95	2.31	79.93
28h	0.09	3.50	3.10	26.91	2.31	79.93
Day 3	0.06	2.33	2.88	25.00	2.26	78.20
Day 4	0.05	1.95	2.58	22.40	2.21	76.47
Day 5	0.07	2.72	2.35	20.40	2.18	75.43
Day 6	0.07	2.72	2.12	18.40	2.13	73.70
Day 8	0.07	2.72	1.68	14.58	2.08	71.97
Day 9	0.07	2.72	1.51	13.11	2.04	70.59
Day 10	0.05	1.95	1.32	11.46	1.99	68.86
Day 11	0.06	2.33	1.16	10.07	1.96	67.82
Day 12	0.07	2.72	1.01	8.77	1.95	67.47
Day 13	0.08	3.11	0.91	7.90	1.91	66.09
Day 14	0.10	3.89	0.77	6.68	1.91	66.09
Day 15	0.06	2.33	0.61	5.30	1.87	64.71
Day 16	0.06	2.33	0.54	4.69	1.84	63.67
Day 18	0.10	3.89	0.38	3.30	1.79	61.94
Day 20	0.09	3.50	0.28	2.43	1.75	60.55
Day 22	0.10	3.89	0.21	1.82	1.72	59.52
Day 24	0.09	3.50	0.12	1.04	1.64	56.75
Day 27	0.09	3.50	0.10	0.87	1.58	54.67
Day 30	0.07	2.72	0.07	0.61	1.52	52.60
Day 33	0.08	3.11	0.10	0.87	1.50	51.90
Day 36	0.11	4.28	0.14	1.22	1.47	50.87
Day 40	0.13	5.06	0.15	1.30	1.44	49.83

Table A – 12 Storage data for room temperature without plugs series 4 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RTwo 4.2	B2 RTwo 4.2	B3 RTwo 4.1	B3 RTwo 4.1	B3 RTwo 4.2	B3 RTwo 4.2
Initial sample	11.60	100.00	2.23	100.00	10.83	100.00
0h	10.29	88.71	0.03	1.35	3.57	32.96
2h	10.25	88.36	0.03	1.35	3.47	32.04
4h	10.27	88.53	0.01	0.45	3.40	31.39
23h	10.22	88.10	0.03	1.35	3.12	28.81
28h	10.20	87.93	0.02	0.90	2.86	26.41
Day 3	10.11	87.16	0.04	1.79	2.72	25.12
Day 4	10.06	86.72	0.03	1.35	2.36	21.79
Day 5	9.99	86.12	0.03	1.35	2.00	18.47
Day 6	9.95	85.78	0.04	1.79	1.72	15.88
Day 8	9.82	84.66	0.05	2.24	1.18	10.90
Day 9	9.74	83.97	0.06	2.69	0.93	8.59
Day 10	9.66	83.28	0.03	1.35	0.69	6.37
Day 11	9.60	82.76	0.03	1.35	0.51	4.71
Day 12	9.54	82.24	0.06	2.69	0.39	3.60
Day 13	9.46	81.55	0.05	2.24	0.32	2.95
Day 14	9.42	81.21	0.08	3.59	0.27	2.49
Day 15	9.36	80.69	0.03	1.35	0.22	2.03
Day 16	9.28	80.00	0.05	2.24	0.20	1.85
Day 18	9.17	79.05	0.05	2.24	0.18	1.66
Day 20	9.05	78.02	0.09	4.04	0.16	1.48
Day 22	8.97	77.33	0.08	3.59	0.17	1.57
Day 24	8.81	75.95	0.05	2.24	0.16	1.48
Day 27	8.66	74.66	0.04	1.79	0.16	1.48
Day 30	8.49	73.19	0.04	1.79	0.13	1.20
Day 33	8.38	72.24	0.08	3.59	0.12	1.11
Day 36	8.25	71.12	0.10	4.48	0.15	1.39
Day 40	8.13	70.09	0.14	6.28	0.18	1.66

Room temperature in exicator

Data for storing at room temperature in exicator are shown tables A- 13 – 16.

Table A - 13 Storage data for room temperature in exicator series 1 parallell 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTe 1.1	B1 RTe 1.1	B2 RTe 1.1	B2 RTe 1.1	B3 RTe 1.1	B3 RTe 1.1
Initial sample	3.09	100.00	3.37	100.00	2.66	100.00
0h	0.18	5.83	2.61	77.45	0.37	13.91
2h	0.17	5.50	2.59	76.85	0.17	6.39
19h	-0.09	-2.91	2.20	65.28	-0.05	-1.88
24h	-0.13	-4.21	2.16	64.09	-0.14	-5.26
Day 3	-0.05	-1.62	2.22	65.88	-0.01	-0.38
Day 4	0.06	1.94	2.14	63.50	0.11	4.14
Day 5	-0.02	-0.65	2.10	62.31	0.09	3.38
Day 6	-0.10	-3.24	1.97	58.46	0.14	5.26
Day 7	-0.05	-1.62	1.54	45.70	-0.03	-1.13
Day 8	0.06	1.94	1.58	46.88	0.15	5.64
Day 9	-0.92	-29.77	1.47	43.62	0.22	8.27
Day 10	-0.03	-0.97	1.27	37.69	0.04	1.50
Day 11	0.00	0.00	1.24	36.80	0.11	4.14
Day 12	-0.09	-2.91	1.01	29.97	-0.11	-4.14
Day 13	-0.03	-0.97	1.05	31.16	0.00	0.00
Day 14	-0.02	-0.65	1.04	30.86	0.01	0.38
Day 15	0.01	0.32	1.00	29.67	0.02	0.75
Day 16	-0.02	-0.65	0.95	28.19	0.01	0.38
Day 18	-0.15	-4.85	0.73	21.66	-0.13	-4.89
Day 20	-0.25	-8.09	0.56	16.62	-0.27	-10.15
Day 22	-0.24	-7.77	0.48	14.24	-0.24	-9.02
Day 24	-0.22	-7.12	0.47	13.95	-0.19	-7.14
Day 27	-0.21	-6.80	0.43	12.76	-0.24	-9.02
Day 30	-0.20	-6.47	0.37	10.98	-0.26	-9.77
Day 33	0.00	0.00	0.56	16.62	0.01	0.38
Day 36	-0.01	-0.32	0.48	14.24	0.01	0.38
Day 40	0.00	0.00	0.48	14.24	0.04	1.50

Table A - 14 Storage data for room temperature in exicator series 1 parallell 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTe 1.2	B1 RTe 1.2	B2 RTe 1.2	B2 RTe 1.2	B3 RTe 1.2	B3 RTe 1.2
Initial sample	14.05	100.00	13.69	100.00	12.99	100.00
0h	4.98	35.44	12.42	90.72	5.18	39.88
2h	4.69	33.38	12.37	90.36	4.92	37.88
4h	4.31	30.68	12.36	90.28	4.35	33.49
19h	2.37	16.87	12.25	89.48	2.17	16.71
26h	1.78	12.67	12.26	89.55	1.43	11.01
Day 3	0.55	3.91	12.02	87.80	0.54	4.16
Day 4	0.44	3.13	11.74	85.76	0.43	3.31
Day 5	0.35	2.49	11.27	82.32	0.48	3.70
Day 6	0.26	1.85	10.54	76.99	0.34	2.62
Day 7	0.44	3.13	10.18	74.36	0.49	3.77
Day 8	1.49	10.60	9.80	71.59	0.43	3.31
Day 9	0.24	1.71	9.31	68.01	0.42	3.23
Day 10	0.29	2.06	9.04	66.03	0.42	3.23
Day 11	0.15	1.07	8.56	62.53	0.24	1.85
Day 12	0.20	1.42	8.37	61.14	0.31	2.39
Day 13	0.29	2.06	8.11	59.24	0.44	3.39
Day 14	0.23	1.64	7.92	57.85	0.31	2.39
Day 15	0.22	1.57	7.75	56.61	0.28	2.16
Day 17	0.06	0.43	7.16	52.30	0.23	1.77
Day 19	-0.07	-0.50	6.72	49.09	0.09	0.69
Day 21	-0.02	-0.14	6.26	45.73	0.03	0.23
Day 23	0.01	0.07	6.07	44.34	0.13	1.00
Day 26	-0.01	-0.07	5.69	41.56	0.04	0.31
Day 29	-0.01	-0.07	5.32	38.86	0.01	0.08
Day 32	0.19	1.35	5.28	38.57	0.34	2.62
Day 35	0.24	1.71	4.90	35.79	0.33	2.54
Day 39	0.23	1.64	4.56	33.31	0.35	2.69

Table A - 15 Storage data for room temperature in exicator series 4 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RTe 4.1	B1 RTe 4.1	B1 RTe 4.2	B1 RTe 4.2	B2 RTe 4.1	B2 RTe 4.1
Initial sample	2.67	100.00	12.97	100.00	2.55	100.00
0h	0.15	5.62	4.06	31.30	2.03	79.61
2h	0.08	3.00	3.79	29.22	2.01	78.82
21h	0.03	1.12	1.70	13.11	1.95	76.47
26h	0.03	1.12	1.24	9.56	1.90	74.51
Day 3	0.04	1.50	0.24	1.85	1.69	66.27
Day 4	0.03	1.12	0.13	1.00	1.41	55.29
Day 5	0.02	0.75	0.13	1.00	1.24	48.63
Day 6	0.03	1.12	0.12	0.93	1.10	43.14
Day 8	0.06	2.25	0.12	0.93	0.92	36.08
Day 9	0.05	1.87	0.12	0.93	0.83	32.55
Day 10	0.03	1.12	0.08	0.62	0.74	29.02
Day 11	0.02	0.75	0.07	0.54	0.68	26.67
Day 12	0.03	1.12	0.09	0.69	0.65	25.49
Day 13	0.04	1.50	0.10	0.77	0.62	24.31
Day 14	0.06	2.25	0.11	0.85	0.59	23.14
Day 15	0.03	1.12	0.08	0.62	0.54	21.18
Day 16	0.04	1.50	0.08	0.62	0.52	20.39
Day 18	0.05	1.87	0.09	0.69	0.48	18.82
Day 20	0.05	1.87	0.10	0.77	0.44	17.25
Day 22	0.04	1.50	0.10	0.77	0.39	15.29
Day 24	0.04	1.50	0.08	0.62	0.34	13.33
Day 27	0.03	1.12	0.09	0.69	0.31	12.16
Day 30	0.02	0.75	0.10	0.77	0.26	10.20
Day 33	0.02	0.75	0.08	0.62	0.23	9.02
Day 36	0.07	2.62	0.14	1.08	0.26	10.20
Day 40	-0.29	-11.05	0.14	1.08	0.25	9.80

Table A - 16 Storage data for room temperature in exicator series 4 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 Rte 4.2	B2 Rte 4.2	B3 Rte 4.1	B3 Rte 4.1	B3 Rte 4.2	B3 Rte 4.2
Initial sample	13.49	100.00	2.32	100.00	13.45	100.00
0h	11.96	88.66	0.00	0.00	3.69	27.43
2	11.93	88.44	-0.01	-0.43	3.30	24.54
21h	11.76	87.18	0.01	0.43	0.58	4.31
26h	11.68	86.58	0.01	0.43	0.29	2.16
Day 3	11.20	83.02	0.03	1.29	0.17	1.26
Day 4	10.61	78.65	0.03	1.29	0.12	0.89
Day 5	10.10	74.87	0.02	0.86	0.10	0.74
Day 6	9.65	71.53	0.02	0.86	0.11	0.82
Day 8	8.95	66.35	0.06	2.59	0.10	0.74
Day 9	8.60	63.75	0.02	0.86	0.10	0.74
Day 10	8.22	60.93	0.00	0.00	0.07	0.52
Day 11	7.90	58.56	0.00	0.00	0.05	0.37
Day 12	7.65	56.71	0.01	0.43	0.04	0.30
Day 13	7.43	55.08	0.04	1.72	0.07	0.52
Day 14	7.20	53.37	0.05	2.16	0.10	0.74
Day 15	6.97	51.67	0.03	1.29	0.03	0.22
Day 16	6.80	50.41	0.02	0.86	0.06	0.45
Day 18	6.38	47.29	0.04	1.72	0.07	0.52
Day 20	6.03	44.70	0.04	1.72	0.05	0.37
Day 22	5.69	42.18	0.04	1.72	0.04	0.30
Day 24	5.36	39.73	0.03	1.29	0.03	0.22
Day 27	4.90	36.32	0.03	1.29	0.04	0.30
Day 30	4.57	33.88	0.03	1.29	0.04	0.30
Day 33	4.33	32.10	0.04	1.72	0.06	0.45
Day 36	4.07	30.17	0.06	2.59	0.06	0.45
Day 40	3.80	28.17	0.07	3.02	0.07	0.52

Appendix VII Data for results at refrigerator temperature

Refrigarator temperature with plugs

Data for storing at refrigerator temperature with plugs are shown tables A- 17 – 22 .

Table A – 17 Storage data for refrigerator temperature with plugs series 1 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RFTw 1.1	B1 RFTw 1.1	B1 RFTw 1.2	B1 RFTw 1.2	B2 RFTw 1.1	B2 RFTw 1.1
Initial sample	3.08	100.00	13.45	100.00	2.91	100.00
0h	0.41	13.31	5.34	39.70	2.29	78.69
2h	0.47	15.26	5.36	39.85	2.36	81.10
4h	0.40	12.99	5.28	39.26	2.34	80.41
19h	0.39	12.66	5.17	38.44	2.31	79.38
26h	0.39	12.66	5.17	38.44	2.39	82.13
Day 3	0.49	15.91	5.18	38.51	2.51	86.25
Day 4	0.43	13.96	5.09	37.84	2.47	84.88
Day 5	0.41	13.31	4.87	36.21	2.42	83.16
Day 6	0.38	12.34	4.74	35.24	2.39	82.13
Day 7	0.71	23.05	4.81	35.76	2.49	85.57
Day 8	0.83	26.95	4.76	35.39	2.55	87.63
Day 9	0.69	22.40	4.54	33.75	2.58	88.66
Day 10	0.77	25.00	4.48	33.31	2.59	89.00
Day 11	0.63	20.45	4.24	31.52	2.51	86.25
Day 12	0.72	23.38	4.33	32.19	2.62	90.03
Day 13	0.91	29.55	4.32	32.12	2.71	93.13
Day 14	0.86	27.92	4.15	30.86	2.64	90.72
Day 15	0.85	27.60	4.11	30.56	2.67	91.75
Day 17	0.36	11.69	3.70	27.51	2.51	86.25
Day 19	0.78	25.32	3.73	27.73	2.54	87.29
Day 21	0.67	21.75	3.58	26.62	2.52	86.60
Day 23	0.69	22.40	3.50	26.02	2.52	86.60
Day 26	0.75	24.35	3.37	25.06	2.49	85.57
Day 29	0.69	22.40	3.16	23.49	2.51	86.25
Day 32	0.89	28.90	3.20	23.79	2.56	87.97
Day 35	0.88	28.57	3.13	23.27	2.53	86.94
Day 39	0.89	28.90	2.87	21.34	2.53	86.94

Table A – 18 Storage data for refrigerator temperature with plugs series 1 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RFTw 1.2	B2 RFTw 1.2	B3 RFTw 1.1	B3 RFTw 1.1	B3 RFTw 1.2	B3 RFTw 1.2
Initial sample	12.56	100.00	3.10	100.00	13.03	100.00
0h	11.35	90.37	0.08	2.58	5.49	42.13
2h	11.43	91.00	0.05	1.61	5.51	42.29
4h	11.38	90.61	0.06	1.94	5.42	41.60
19h	11.32	90.13	0.06	1.94	5.42	41.60
26h	11.33	90.21	0.17	5.48	5.31	40.75
Day 3	11.47	91.32	0.31	10.00	5.31	40.75
Day 4	11.46	91.24	0.28	9.03	5.17	39.68
Day 5	11.44	91.08	0.26	8.39	4.94	37.91
Day 6	11.42	90.92	0.23	7.42	4.86	37.30
Day 7	11.52	91.72	0.52	16.77	4.88	37.45
Day 8	11.60	92.36	0.56	18.06	4.84	37.15
Day 9	11.50	91.56	0.45	14.52	4.70	36.07
Day 10	11.46	91.24	0.58	18.71	4.59	35.23
Day 11	11.46	91.24	0.47	15.16	4.33	33.23
Day 12	11.47	91.32	0.53	17.10	4.30	33.00
Day 13	11.58	92.20	0.71	22.90	4.30	33.00
Day 14	11.52	91.72	0.59	19.03	4.15	31.85
Day 15	11.63	92.60	0.64	20.65	4.05	31.08
Day 17	11.40	90.76	0.60	19.35	3.77	28.93
Day 19	11.39	90.68	0.52	16.77	3.43	26.32
Day 21	11.35	90.37	0.52	16.77	3.46	26.55
Day 23	11.41	90.84	0.54	17.42	3.35	25.71
Day 26	11.38	90.61	0.61	19.68	3.04	23.33
Day 29	11.21	89.25	0.58	18.71	2.84	21.80
Day 32	11.40	90.76	0.75	24.19	2.85	21.87
Day 35	11.41	90.84	0.76	24.52	2.69	20.64
Day 39	11.30	89.97	0.67	21.61	2.19	16.81

Table A – 19 Storage data for refrigerator temperature with plugs series 2 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RFTw 2.1	B1 RFTw 2.1	B1 RFTw 2.2	B1 RFTw 2.2	B2 RFTw 2.1	B2 RFTw 2.1
Initial sample	3.66	100.00	11.87	100.00	3.50	100.00
0h	0.73	19.95	4.24	35.72	3.99	114.00
2h	3.84	104.92	6.81	57.37	6.90	197.14
4h	4.44	121.31	7.31	61.58	8.20	234.29
21h	3.71	101.37	7.25	61.08	9.97	284.86
26h	4.95	135.25	8.25	69.50	11.43	326.57
Day 3	5.96	162.84	9.13	76.92	14.31	408.86
Day 4	7.03	192.08	10.13	85.34	17.50	500.00
Day 5	6.56	179.23	9.20	77.51	19.66	561.71
Day 6	6.97	190.44	9.34	78.69	21.91	626.00
Day 8	8.38	228.96	10.89	91.74	25.92	740.57
Day 9	9.04	246.99	10.64	89.64	28.46	813.14
Day 10	8.10	221.31	10.13	85.34	28.84	824.00
Day 11	8.27	225.96	10.37	87.36	29.34	838.29
Day 12	8.36	228.42	10.56	88.96	30.97	884.86
Day 13	9.02	246.45	11.25	94.78	32.27	922.00
Day 14	8.04	219.67	10.10	85.09	32.50	928.57
Day 15	8.60	234.97	10.51	88.54	33.87	967.71
Day 17	9.10	248.63	11.64	98.06	34.93	998.00
Day 19	9.80	267.76	12.13	102.19	36.58	1045.14
Day 21	10.00	273.22	11.18	94.19	38.83	1109.43
Day 23	9.43	257.65	11.60	97.73	38.79	1108.29
Day 26	10.27	280.60	12.41	104.55	40.43	1155.14
Day 29	10.77	294.26	12.74	107.33	41.63	1189.43
Day 32	10.96	299.45	12.80	107.83	41.92	1197.71
Day 35	10.87	296.99	12.85	108.26	42.68	1219.43
Day 39	8.45	230.87	10.35	87.19	43.07	1230.57

Table A – 20 Storage data for refrigerator temperature with plugs series 2 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RFTw 2.2	B2 RFTw 2.2	B3 RFTw 2.1	B3 RFTw 2.1	B3 RFTw 2.2	B3 RFTw 2.2
Initial sample	12.89	100.00	3.12	100.00	11.89	100.00
0h	13.26	102.87	2.01	64.42	3.88	32.63
2h	16.47	127.77	4.83	154.81	5.70	47.94
4h	18.18	141.04	6.63	212.50	5.98	50.29
21h	20.93	162.37	9.03	289.42	6.52	54.84
26h	22.75	176.49	10.89	349.04	7.25	60.98
Day 3	26.63	206.59	14.41	461.86	8.31	69.89
Day 4	31.43	243.83	18.63	597.12	9.11	76.62
Day 5	33.78	262.06	22.44	719.23	9.14	76.87
Day 6	35.97	279.05	25.15	806.09	9.39	78.97
Day 8	43.14	334.68	29.00	929.49	9.66	81.24
Day 9	44.43	344.69	33.81	1083.65	10.53	88.56
Day 10	45.21	350.74	35.01	1122.12	10.16	85.45
Day 11	46.90	363.85	37.30	1195.51	10.27	86.38
Day 12	48.93	379.60	39.82	1276.28	10.62	89.32
Day 13	51.42	398.91	40.21	1288.78	9.96	83.77
Day 14	52.20	404.97	42.52	1362.82	9.99	84.02
Day 15	53.92	418.31	44.14	1414.74	10.21	85.87
Day 17	57.64	447.17	44.26	1418.59	9.56	80.40
Day 19	60.49	469.28	46.25	1482.37	9.74	81.92
Day 21	61.98	480.84	51.97	1665.71	11.33	95.29
Day 23	65.14	505.35	50.78	1627.56	9.87	83.01
Day 26	69.16	536.54	52.48	1682.05	10.30	86.63
Day 29	70.42	546.31	53.25	1706.73	9.90	83.26
Day 32	71.44	554.23	54.46	1745.51	10.08	84.78
Day 35	73.14	567.42	55.32	1773.08	10.09	84.86
Day 39	74.91	581.15	60.97	1954.17	10.07	84.69

Table A – 21 Storage data for refrigerator temperature with plugs series 4 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RFTw 4.1	B1 RFTw 4.1	B1 RFTw 4.2	B1 RFTw 4.2	B2 RFTw 4.1	B2 RFTw 4.1
Initial sample	2.65	100.00	11.20	100.00	2.73	100.00
0h	0.05	1.89	3.01	26.88	2.19	80.22
2h	0.05	1.89	3.00	26.79	2.20	80.59
4h	0.08	3.02	2.94	26.25	2.17	79.49
21h	0.06	2.26	2.89	25.80	2.18	79.85
26h	0.06	2.26	2.85	25.45	2.17	79.49
Day 3	0.06	2.26	2.76	24.64	2.16	79.12
Day 4	0.07	2.64	2.68	23.93	2.12	77.66
Day 5	0.06	2.26	2.55	22.77	2.12	77.66
Day 7	0.08	3.02	2.47	22.05	2.11	77.29
Day 8	0.09	3.40	2.40	21.43	2.11	77.29
Day 9	0.09	3.40	2.31	20.63	2.06	75.46
Day 10	0.09	3.40	2.26	20.18	2.04	74.73
Day 11	0.10	3.77	2.20	19.64	2.04	74.73
Day 12	0.11	4.15	2.15	19.20	2.03	74.36
Day 13	0.14	5.28	2.07	18.48	2.05	75.09
Day 14	0.09	3.40	2.00	17.86	2.03	74.36
Day 15	0.11	4.15	1.95	17.41	2.00	73.26
Day 17	0.14	5.28	1.92	17.14	2.00	73.26
Day 19	0.16	6.04	1.77	15.80	2.01	73.63
Day 21	0.17	6.42	1.64	14.64	1.98	72.53
Day 23	0.13	4.91	1.57	14.02	1.93	70.70
Day 26	0.15	5.66	1.48	13.21	1.91	69.96
Day 29	0.16	6.04	1.38	12.32	1.90	69.60
Day 32	0.15	5.66	1.36	12.14	1.88	68.86
Day 35	0.20	7.55	1.25	11.16	1.89	69.23
Day 39	0.26	9.81	1.27	11.34	1.92	70.33

Table A – 22 Storage data for refrigerator temperature with plugs series 4 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RFTw 4.2	B2 RFTw 4.2	B3 RFTw 4.1	B3 RFTw 4.1	B3 RFTw 4.2	B3 RFTw 4.2
Initial sample	12.22	100.00	2.39	100.00	10.95	100.00
0h	10.69	87.48	0.03	1.26	3.11	28.40
2h	10.70	87.56	0.01	0.42	3.08	28.13
4h	10.71	87.64	0.03	1.26	3.02	27.58
21h	10.71	87.64	0.07	2.93	2.86	26.12
26h	10.68	87.40	0.05	2.09	2.76	25.21
Day 3	10.66	87.23	0.05	2.09	2.49	22.74
Day 4	10.65	87.15	0.05	2.09	2.28	20.82
Day 5	10.63	86.99	0.06	2.51	2.00	18.26
Day 7	10.63	86.99	0.07	2.93	1.79	16.35
Day 8	10.62	86.91	0.07	2.93	1.63	14.89
Day 9	10.57	86.50	0.06	2.51	1.48	13.52
Day 10	10.57	86.50	0.08	3.35	1.35	12.33
Day 11	10.56	86.42	0.09	3.77	1.24	11.32
Day 12	10.56	86.42	0.08	3.35	1.12	10.23
Day 13	10.53	86.17	0.12	5.02	0.99	9.04
Day 14	10.50	85.92	0.11	4.60	0.88	8.04
Day 15	10.48	85.76	0.11	4.60	0.80	7.31
Day 17	10.49	85.84	0.12	5.02	0.62	5.66
Day 19	10.48	85.76	0.13	5.44	0.50	4.57
Day 21	10.44	85.43	0.13	5.44	0.42	3.84
Day 23	10.39	85.02	0.10	4.18	0.32	2.92
Day 26	10.35	84.70	0.12	5.02	0.30	2.74
Day 29	10.31	84.37	0.12	5.02	0.28	2.56
Day 32	10.29	84.21	0.17	7.11	0.27	2.47
Day 35	10.29	84.21	0.16	6.69	0.28	2.56
Day 39	10.30	84.29	0.23	9.62	0.31	2.83

Refrigerator temperature without plugs

Data for storing at refrigaretor temperature without plugs are shown tables A- 23 – 28.

Table A – 23 Storage data for refrigerator temperature without plugs series 1 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RFTwo 1.1	B1 RFTwo 1.1	B1 RFTwo 1.2	B1 RFTwo 1.2	B2 RFTwo 1.1	B2 RFTwo 1.1
Initial sample	2.73	100.00	14.21	100.00	3.22	100.00
0h	0.26	9.52	6.07	42.72	2.63	81.68
2h	0.11	4.03	6.10	42.93	2.55	79.19
17h	0.11	4.03	5.99	42.15	2.55	79.19
24h	0.23	8.42	5.99	42.15	2.62	81.37
Day 3	0.30	10.99	5.98	42.08	2.68	83.23
Day 4	0.33	12.09	5.80	40.82	2.66	82.61
Day 5	0.31	11.36	5.68	39.97	2.69	83.54
Day 6	0.30	10.99	5.59	39.34	2.65	82.30
Day 7	0.44	16.12	5.65	39.76	2.78	86.34
Day 8	0.48	17.58	5.61	39.48	2.82	87.58
Day 9	0.43	15.75	5.40	38.00	2.80	86.96
Day 10	0.38	13.92	5.30	37.30	2.80	86.96
Day 11	0.34	12.45	5.00	35.19	2.75	85.40
Day 12	0.53	19.41	4.62	32.51	2.84	88.20
Day 13	0.53	19.41	4.76	33.50	2.91	90.37
Day 14	0.57	20.88	4.56	32.09	2.81	87.27
Day 15	0.58	21.25	4.57	32.16	2.85	88.51
Day 17	0.47	17.22	4.10	28.85	2.69	83.54
Day 19	0.42	15.38	4.07	28.64	2.67	82.92
Day 21	0.44	16.12	3.84	27.02	2.70	83.85
Day 23	0.51	18.68	3.63	25.55	2.73	84.78
Day 26	0.47	17.22	2.96	20.83	2.65	82.30
Day 29	0.54	19.78	3.03	21.32	2.74	85.09
Day 32	0.63	23.08	2.92	20.55	2.78	86.34
Day 35	0.70	25.64	2.75	19.35	2.77	86.02
Day 39	0.75	27.47	2.58	18.16	2.81	87.27

Table A – 24 Storage data for refrigerator temperature without plugs series 1 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RFTwo 1.2	B2 RFTwo 1.2	B3 RFTwo 1.1	B3 RFTwo 1.1	B3 RFTwo 1.2	B3 RFTwo 1.2
Initial sample	14.29	100.00	2.70	100.00	14.58	100.00
0h	13.06	91.39	0.14	5.19	5.65	38.75
2h	13.02	91.11	0.08	2.96	5.61	38.48
17h	13.01	91.04	0.10	3.70	5.48	37.59
24h	13.06	91.39	0.19	7.04	5.45	37.38
Day 3	13.06	91.39	0.30	11.11	5.37	36.83
Day 4	13.05	91.32	0.28	10.37	5.21	35.73
Day 5	13.03	91.18	0.29	10.74	5.05	34.64
Day 6	13.04	91.25	0.25	9.26	4.94	33.88
Day 7	13.09	91.60	0.33	12.22	4.90	33.61
Day 8	13.26	92.79	0.50	18.52	4.87	33.40
Day 9	13.15	92.02	0.51	18.89	4.66	31.96
Day 10	13.18	92.23	0.57	21.11	4.63	31.76
Day 11	13.04	91.25	0.50	18.52	4.35	29.84
Day 12	13.08	91.53	0.55	20.37	4.27	29.29
Day 13	13.14	91.95	0.63	23.33	4.22	28.94
Day 14	13.08	91.53	0.56	20.74	4.02	27.57
Day 15	13.16	92.09	0.66	24.44	3.95	27.09
Day 17	12.95	90.62	0.47	17.41	3.53	24.21
Day 19	12.82	89.71	0.48	17.78	3.35	22.98
Day 21	12.78	89.43	0.44	16.30	2.98	20.44
Day 23	12.87	90.06	0.57	21.11	2.90	19.89
Day 26	12.80	89.57	0.53	19.63	2.59	17.76
Day 29	12.70	88.87	0.54	20.00	2.10	14.40
Day 32	12.81	89.64	0.65	24.07	2.09	14.33
Day 35	12.79	89.50	0.69	25.56	1.73	11.87
Day 39	12.74	89.15	0.64	23.70	1.51	10.36

Table A – 25 Storage data for refrigerator temperature without plugs series 2 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RFTwo 2.1	B1 RFTwo 2.1	B1 RFTwo 2.2	B1 RFTwo 2.2	B2 RFTwo 2.1	B2 RFTwo 2.1
Initial sample	2.78	100.00	12.33	100.00	2.87	100.00
0h	0.81	29.14	7.41	60.10	2.73	95.12
2h	3.69	132.73	11.41	92.54	5.16	179.79
4h	4.22	151.80	13.43	108.92	5.97	208.01
21h	5.15	185.25	18.08	146.63	6.54	227.87
26h	5.42	194.96	19.88	161.23	6.92	241.11
Day 3	4.71	169.42	22.97	186.29	6.62	230.66
Day 4	5.63	202.52	28.18	228.55	7.57	263.76
Day 5	5.54	199.28	30.37	246.31	7.74	269.69
Day 6	5.90	212.23	33.22	269.42	8.00	278.75
Day 8	6.50	233.81	41.14	333.66	8.17	284.67
Day 9	7.06	253.96	41.75	338.61	8.99	313.24
Day 10	6.86	246.76	42.01	340.71	9.02	314.29
Day 11	7.01	252.16	43.03	348.99	9.07	316.03
Day 12	6.97	250.72	44.05	357.26	9.33	325.09
Day 13	7.72	277.70	48.88	396.43	9.27	323.00
Day 14	6.65	239.21	47.46	384.91	8.97	312.54
Day 15	6.95	250.00	47.55	385.64	9.17	319.51
Day 17	7.65	275.18	54.24	439.90	9.10	317.07
Day 19	8.21	295.32	57.50	466.34	9.40	327.53
Day 21	8.32	299.28	55.95	453.77	10.88	379.09
Day 23	7.78	279.86	61.07	495.30	9.39	327.18
Day 26	8.61	309.71	65.09	527.90	9.98	347.74
Day 29	8.71	313.31	66.94	542.90	9.91	345.30
Day 32	8.29	298.20	61.60	499.59	10.23	356.45
Day 35	8.35	300.36	60.52	490.84	10.21	355.75
Day 39	8.32	299.28	66.38	538.36	9.69	337.63

Table A – 26 Storage data for refrigerator temperature without plugs series 2 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RFTwo 2.2	B2 RFTwo 2.2	B3 RFTwo 2.1	B3 RFTwo 2.1	B3 RFTwo 2.2	B3 RFTwo 2.2
Initial sample	13.91	100.00	3.18	100.00	11.50	100.00
0h	12.96	93.17	0.18	5.66	5.77	50.17
2h	15.27	109.78	2.51	78.93	9.10	79.13
4h	16.01	115.10	3.15	99.06	11.08	96.35
21h	16.67	119.84	3.97	124.84	14.86	129.22
26h	17.09	122.86	4.36	137.11	16.51	143.57
Day 3	16.77	120.56	4.05	127.36	20.01	174.00
Day 4	17.73	127.46	4.80	150.94	24.96	217.04
Day 5	17.35	124.73	5.27	165.72	29.61	257.48
Day 6	17.63	126.74	5.59	175.79	32.55	283.04
Day 8	18.09	130.05	5.34	167.92	34.88	303.30
Day 9	18.38	132.14	6.62	208.18	39.09	339.91
Day 10	18.33	131.78	6.82	214.47	41.43	360.26
Day 11	18.24	131.13	6.88	216.35	43.15	375.22
Day 12	18.17	130.63	7.05	221.70	44.67	388.43
Day 13	18.77	134.94	6.46	203.14	45.11	392.26
Day 14	17.71	127.32	6.78	213.21	46.90	407.83
Day 15	17.85	128.32	7.09	222.96	48.77	424.09
Day 17	18.45	132.64	6.41	201.57	47.67	414.52
Day 19	19.01	136.66	6.50	204.40	48.57	422.35
Day 21	19.62	141.05	8.25	259.43	54.86	477.04
Day 23	18.91	135.95	6.55	205.97	53.02	461.04
Day 26	19.36	139.18	6.70	210.69	53.53	465.48
Day 29	19.59	140.83	6.81	214.15	54.66	475.30
Day 32	19.30	138.75	8.08	254.09	60.07	522.35
Day 35	18.93	136.09	8.19	257.55	61.67	536.26
Day 39	18.98	136.45	7.02	220.75	55.95	486.52

Table A – 27 Storage data for refrigerator temperature without plugs series 4 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 RFTwo 4.1	B1 RFTwo 4.1	B1 RFTwo 4.2	B1 RFTwo 4.2	B2 RFTwo 4.1	B2 RFTwo 4.1
Initial sample	3.20	100.00	11.30	100.00	2.61	100.00
0h	0.62	19.37	3.47	30.71	2.05	78.54
2h	0.63	19.69	3.45	30.53	2.05	78.54
4h	0.59	18.44	3.44	30.44	2.05	78.54
21h	0.55	17.19	3.34	29.56	2.05	78.54
26h	0.52	16.25	3.30	29.20	2.03	77.78
Day 3	0.45	14.06	3.10	27.43	2.03	77.78
Day 4	0.39	12.19	2.97	26.28	2.02	77.39
Day 5	0.33	10.31	2.83	25.04	2.01	77.01
Day 7	0.31	9.69	2.53	22.39	2.01	77.01
Day 8	0.23	7.19	2.38	21.06	1.97	75.48
Day 9	0.20	6.25	2.24	19.82	1.95	74.71
Day 10	0.20	6.25	2.07	18.32	1.95	74.71
Day 11	0.18	5.62	1.92	16.99	1.94	74.33
Day 12	0.16	5.00	1.82	16.11	1.93	73.95
Day 13	0.17	5.31	1.77	15.66	1.95	74.71
Day 14	0.13	4.06	1.62	14.34	1.93	73.95
Day 15	0.16	5.00	1.53	13.54	1.92	73.56
Day 17	0.17	5.31	1.34	11.86	1.91	73.18
Day 19	0.11	3.44	1.23	10.88	1.92	73.56
Day 21	0.13	4.06	1.12	9.91	1.89	72.41
Day 23	0.12	3.75	0.97	8.58	1.84	70.50
Day 26	0.11	3.44	0.87	7.70	1.83	70.11
Day 29	0.11	3.44	0.75	6.64	1.81	69.35
Day 32	0.14	4.38	0.64	5.66	1.80	68.97
Day 35	0.15	4.69	0.58	5.13	1.83	70.11
Day 39	0.21	6.56	0.50	4.42	1.82	69.73

Table A – 28 Storage data for refrigerator temperature without plugs series 4 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 RFTwo 4.2	B2 RFTwo 4.2	B3 RFTwo 4.1	B3 RFTwo 4.1	B3 RFTwo 4.2	B3 RFTwo 4.2
Initial sample	12.03	100.00	2.83	100.00	11.22	100.00
0h	10.52	87.45	0.06	2.12	2.60	23.17
2h	10.52	87.45	0.07	2.47	2.57	22.91
4h	10.52	87.45	0.09	3.18	2.53	22.55
21h	10.51	87.36	0.09	3.18	2.42	21.57
26h	10.44	86.78	0.06	2.12	2.27	20.23
Day 3	10.42	86.62	0.07	2.47	2.24	19.96
Day 4	10.38	86.28	0.05	1.77	1.97	17.56
Day 5	10.39	86.37	0.07	2.47	1.92	17.11
Day 7	10.36	86.12	0.08	2.83	1.61	14.35
Day 8	10.32	85.79	0.08	2.83	1.45	12.92
Day 9	10.27	85.37	0.06	2.12	1.25	11.14
Day 10	10.29	85.54	0.07	2.47	1.16	10.34
Day 11	10.24	85.12	0.07	2.47	1.06	9.45
Day 12	10.20	84.79	0.09	3.18	0.95	8.47
Day 13	10.20	84.79	0.11	3.89	0.89	7.93
Day 14	10.16	84.46	0.10	3.53	0.77	6.86
Day 15	10.11	84.04	0.08	2.83	0.72	6.42
Day 17	10.10	83.96	0.14	4.95	0.63	5.61
Day 19	10.06	83.62	0.12	4.24	0.56	4.99
Day 21	10.02	83.29	0.14	4.95	0.55	4.90
Day 23	10.00	83.13	0.12	4.24	0.45	4.01
Day 26	9.93	82.54	0.13	4.59	0.44	3.92
Day 29	9.89	82.21	0.15	5.30	0.43	3.83
Day 32	9.84	81.80	0.14	4.95	0.40	3.57
Day 35	9.87	82.04	0.20	7.07	0.44	3.92
Day 39	9.82	81.63	0.21	7.42	0.44	3.92

Appendix VIII Data for results at freezer temperature

Freezer temperature with plugs

Data for storing at freezer temperature with plugs are shown tables A- 29 – 30.

Table A - 29 Storage data for freezer temperature with plugs series 3 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 FTw 3.1	B1 FTw 3.1	B1 FTw 3.2	B1 FTw 3.2	B2 FTw 3.1	B2 FTw 3.1
Initial sample	3.05	100.00	11.68	100.00	3.44	100.00
0h	0.32	10.49	4.35	37.24	2.89	84.01
2h	0.52	17.05	4.55	38.96	3.16	91.86
6h	0.62	20.33	4.90	41.95	3.36	97.67
23h	0.56	18.36	4.90	41.95	3.39	98.55
28h	0.76	24.92	4.83	41.35	3.33	96.80
Day 3	0.68	22.30	4.81	41.18	3.37	97.97
Day 4	0.79	25.90	5.10	43.66	3.63	105.52
Day 5	0.89	29.18	5.24	44.86	3.83	111.34
Day 6	0.84	27.54	5.17	44.26	3.79	110.17
Day 7	1.00	32.79	5.24	44.86	3.95	114.83
Day 8	0.92	30.16	5.34	45.72	3.97	115.41
Day 9	0.99	32.46	5.11	43.75	3.84	111.63
Day 10	0.95	31.15	5.06	43.32	3.93	114.24
Day 11	0.85	27.87	4.93	42.21	3.82	111.05
Day 12	0.64	20.98	4.91	42.04	3.82	111.05
Day 14	0.86	28.20	4.86	41.61	3.86	112.21
Day 15	0.92	30.16	4.90	41.95	3.88	112.79
Day 16	0.88	28.85	4.92	42.12	3.76	109.30
Day 18	1.04	34.10	4.99	42.72	3.90	113.37
Day 20	1.05	34.43	4.76	40.75	4.01	116.57
Day 22	1.06	34.75	5.13	43.92	4.08	118.60
Day 24	1.07	35.08	5.19	44.43	4.13	120.06
Day 28	1.30	42.62	5.46	46.75	4.34	126.16
Day 30	1.15	37.70	5.45	46.66	4.24	123.26
Day 33	1.28	41.97	5.28	45.21	4.21	122.38
Day 36	1.27	41.64	5.29	45.29	4.27	124.13
Day 40	1.43	46.89	5.33	45.63	4.39	127.62

Table A - 30 Storage data for freezer temperature with plugs series 3 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 FTw 3.2	B2 FTw 3.2	B3 FTw 3.1	B3 FTw 3.1	B3 FTw 3.2	B3 FTw 3.2
Initial sample	13.52	100.00	2.66	100.00	11.72	100.00
0h	12.24	90.53	0.17	6.39	5.08	43.34
2h	12.27	90.75	0.39	14.66	5.29	45.14
6h	12.55	92.83	0.52	19.55	5.28	45.05
23h	12.58	93.05	0.57	21.43	5.30	45.22
28h	12.54	92.75	0.51	19.17	5.21	44.45
Day 3	12.53	92.68	0.47	17.67	5.21	44.45
Day 4	12.80	94.67	0.73	27.44	5.39	45.99
Day 5	13.00	96.15	0.82	30.83	5.48	46.76
Day 6	12.47	92.23	0.77	28.95	5.45	46.50
Day 7	12.89	95.34	0.89	33.46	5.44	46.42
Day 8	12.83	94.90	0.89	33.46	5.54	47.27
Day 9	12.86	95.12	0.77	28.95	5.21	44.45
Day 10	12.77	94.45	0.69	25.94	5.20	44.37
Day 11	12.67	93.71	0.62	23.31	5.14	43.86
Day 12	12.74	94.23	0.76	28.57	5.23	44.62
Day 14	12.77	94.45	0.75	28.20	5.25	44.80
Day 15	12.87	95.19	0.88	33.08	5.18	44.20
Day 16	12.75	94.30	0.71	26.69	5.10	43.52
Day 18	12.91	95.49	0.83	31.20	5.21	44.45
Day 20	12.82	94.82	1.12	42.11	5.12	43.69
Day 22	12.96	95.86	1.08	40.60	5.30	45.22
Day 24	12.78	94.53	0.96	36.09	5.32	45.39
Day 28	13.23	97.86	1.23	46.24	5.48	46.76
Day 30	13.12	97.04	1.13	42.48	5.34	45.56
Day 33	13.15	97.26	1.06	39.85	5.33	45.48
Day 36	13.18	97.49	1.18	44.36	5.38	45.90
Day 40	13.23	97.86	1.22	45.86	5.43	46.33

Freezer temperature without plugs

Data for storing at freezer temperature without plugs are shown tables A- 31 – 32.

Table A - 31 Storage data for freezer temperature without plugs series 3 – part 1

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B1 FTwo 3.1	B1 FTwo 3.1	B1 FTwo 3.2	B1 FTwo 3.2	B2 FTwo 3.1	B2 FTwo 3.1
Initial sample	2.80	100.00	10.08	100.00	3.19	100.00
0h	0.20	7.14	3.61	35.81	2.77	86.83
2h	0.43	15.36	3.79	37.60	2.98	93.42
6h	0.62	22.14	4.10	40.67	3.12	97.81
23h	0.63	22.50	4.10	40.67	3.23	101.25
28h	0.65	23.21	4.10	40.67	3.19	100.00
Day 3	0.60	21.43	3.70	36.71	3.14	98.43
Day 4	0.85	30.36	4.38	43.45	3.44	107.84
Day 5	0.94	33.57	4.49	44.54	3.48	109.09
Day 6	0.95	33.93	4.63	45.93	3.56	111.60
Day 7	1.04	37.14	4.30	42.66	3.49	109.40
Day 8	1.05	37.50	4.70	46.63	3.61	113.17
Day 9	1.02	36.43	4.19	41.57	3.43	107.52
Day 10	1.05	37.50	4.20	41.67	3.66	114.73
Day 11	0.96	34.29	4.18	41.47	3.00	94.04
Day 12	0.96	34.29	4.35	43.15	3.25	101.88
Day 14	1.11	39.64	4.30	42.66	3.35	105.02
Day 15	1.08	38.57	4.18	41.47	3.46	108.46
Day 16	1.03	36.79	4.20	41.67	3.39	106.27
Day 18	1.12	40.00	4.22	41.87	3.41	106.90
Day 20	1.10	39.29	4.18	41.47	3.47	108.78
Day 22	1.25	44.64	4.55	45.14	3.70	115.99
Day 24	1.10	39.29	4.53	44.94	3.16	99.06
Day 28	1.49	53.21	4.83	47.92	3.85	120.69
Day 30	1.36	48.57	4.75	47.12	3.69	115.67
Day 33	1.44	51.43	4.77	47.32	3.83	120.06
Day 36	1.48	52.86	4.55	45.14	3.84	120.38
Day 40	1.52	54.29	4.70	46.63	3.96	124.14

Table A - 32 Storage data for freezer temperature without plugs series 3 – part 2

	Weight [mg]	% loss	Weight [mg]	% loss	Weight [mg]	% loss
Time	B2 FTwo 3.2	B2 FTwo 3.2	B3 FTwo 3.1	B3 FTwo 3.1	B3 FTwo 3.2	B3 FTwo 3.2
Initial sample	10.03	100.00	2.53	100.00	11.65	100.00
0h	8.93	89.03	0.13	5.14	3.17	27.21
2h	9.15	91.23	0.36	14.23	3.40	29.18
6h	9.37	93.42	0.53	20.95	3.47	29.79
23h	9.28	92.52	0.61	24.11	3.15	27.04
28h	9.34	93.12	0.44	17.39	3.37	28.93
Day 3	9.28	92.52	0.53	20.95	3.54	30.39
Day 4	9.67	96.41	0.83	32.81	3.74	32.10
Day 5	9.75	97.21	0.94	37.15	3.74	32.10
Day 6	9.81	97.81	1.04	41.11	3.91	33.56
Day 7	9.72	96.91	0.90	35.57	3.69	31.67
Day 8	9.68	96.51	0.80	31.62	3.61	30.99
Day 9	9.75	97.21	0.31	12.25	3.53	30.30
Day 10	9.58	95.51	0.72	28.46	3.48	29.87
Day 11	9.47	94.42	0.63	24.90	3.42	29.36
Day 12	9.47	94.42	0.59	23.32	3.59	30.82
Day 14	9.61	95.81	0.92	36.36	3.47	29.79
Day 15	9.52	94.92	0.77	30.43	3.06	26.27
Day 16	9.71	96.81	0.69	27.27	3.36	28.84
Day 18	9.57	95.41	0.78	30.83	3.46	29.70
Day 20	9.64	96.11	1.09	43.08	3.56	30.56
Day 22	9.98	99.50	1.15	45.45	3.74	32.10
Day 24	9.92	98.90	1.10	43.48	3.69	31.67
Day 28	10.25	102.19	1.31	51.78	3.95	33.91
Day 30	9.97	99.40	1.24	49.01	3.88	33.30
Day 33	10.07	100.40	1.31	51.78	3.85	33.05
Day 36	10.06	100.30	1.28	50.59	3.78	32.45
Day 40	10.27	102.39	1.40	55.34	3.92	33.65