QUALITY CONTROL OF ANALYTICAL DETERMINATIONS IN FAT AND OIL

By

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DECLARATION

The analysis described in this thesis was carried out by my self at the main laboratory of Unilever Sri Lanka, under the supervision of Dr. Ruwan Pathirana and Mr. Baratha Wejesundara, laboratory manager, Uniliver Sri Lanka Limited and Dr. Nirmali Wicramaratne, Head, Department of Physical Sciences, during the industrial training period from 14th February 2005 to 13th June 2005.

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ABSTRACT

The accuracy of an analytical determination following a standard procedure (e.g. a UMA method) can be controlled through the periodic analysis of a check sample. This method describes the use of the Shewhart control chart to judge the result of the check sample. If the results don't falls within the normal variability of the procedure corrective action are recommended to be taken.

The control chart is a decision -making device that gives the user information about quality of product resulting from a manufacturing process. The control chart is constructed in such a way that we can plot the results of assessing the quality of the manufactured product through periodic monitoring of the manufacturing process. Each time the process is monitored, a point is placed on the control chart.

During my training period, the analytical determinations were selected that use for Fats and Oils to determine the quality at main laboratory of Unilever Sri Lanka Limited. Using standard Unilever methods Solid Fat Content Using Pulsed LR NMR instrument , Slip point, Iodine Value (Wijs method), Moisture in Oil/Fat using Karl Fisher were proceed and control charts were constructed.

According to the control chart, the analytical determinations which selected are in control.

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CHAPTER01 - BACKGROUND & INTRODUCTION

The concept of quality control, in its broadest senses, has many facts. Quality control is a concern of most, if not all, of the areas of a business organization. In fact, the life and health of a business depend on the quality of product it produces. Therefore the management is concerned with measuring and controlling the quality of the product. As the consumer judges the quality of a product or service, the most appropriate basic definition of quality is fitness for use.

Management should establish company policy concerning the quality of the product. Customers must base the standard on the performance of the product in actual use. It must be at a level that is acceptable to customers, comparable to that of competitors, and economically feasible from the standpoint of cost of production and service.
The quality of the finished product depends on the quality of the raw materials from which it is made (Wayne W. Daniel and James C. Terrell, 1989).

This concern with good quality-and its measurement and control-is present in a business organization whether or not it has a formal quality-control department. When a business does have a quality-control department, however, that department performs a variety of functions. It collects samples, takes measurements, performs tests, makes arithmetical and statistical computations, keeps records, prepares reports, and makes decisions.

An accurate and precise quantitative analysis of lipids in foods is important for nutritional labelling, to determine whether the food meets the standard of identity and is uniform, and to understand the effects of fats and oils on the functional and nutritional properties of foods.

Accuracy of the quality parameter of the oils and fats depend on the quality of an analytical determination. Therefore the quality of analytical determinations is very important.

Following analytical determinations are normally used to find the quality of Oil and Fats

Solid Fat Content Using Pulsed LR NMR
Slip point
Iodine Value (Wijs method)
Moisture in Oil/Fat using Karl Fisher
Peroxide Value
Lovibond Colour
Fatty Acid Profile using GC
Free Fatty Acid
Moisture in fat spreads & margarine

The accuracy of an analytical determination following a standard procedure (e.g. a UMA method) can be controlled through the periodic analysis of a check sample. This method describes the use oh the Shewhart control chart to judge the result of the check sample.

The control chart is a decision -making device that gives the user information about quality of product resulting from a manufacturing process. The control chart is constructed in such a way that we can plot the results of assessing the quality of the manufactured product through periodic monitoring of the manufacturing process. Each time the process is monitored, a point is placed on the control chart.

During my training period, An accuracy of selected analytical methods in above which are used to determine quality control test methods were studies to determine the standards at the main laboratory of Unilever Sri Lanka Limited.

Fat like butter, margarine, bakery shortening, beef tallow, coca butter appear to be solid, but are infect a mixture of solid and liquid components. The proportion of solid present at various temperature of use is often of interest in relation to the function to be performed, for example the spreading properly of butter . A sample and rapid measurement of the solid fat content (SFC) can be obtained by the use of a nuclear magnetic resonance (NMR) instrument. The measurement depends on the fact that the protons of the combined hydrogen atoms of fat in the liquid state are more mobile than those of the fat in the solid state. The mobile protons are therefore more responsive to a magnetic field and this fact can be used to measure the proportion of liquid fat present. The method is superseding the dilation procedure (Berger, 1982)

The slip point of a fat is the temperature at which a column of fat in an open capillary tube moves up the tube when it is subjected to controlled heating in a water bath. Because of their polymorphic behaviour the slip point of some fats is dependent on the previous treatment of the sample.

Iodine value, also referred to as iodine number, is defined as the number of centigrams iodine absorbed (AOCS Method 1-25) the test is empirical in that the theoretical amount of iodine is never absorbed.
Purchase specifications for shortening products often include iodine value in order to define the product. This is satisfactory as long as the same raw materials are used from batch to batch and the shortening comes from the same refinery with these factors being constant .The iodine value is correlated with the hardness and oxidative stability of the shortening

Moisture determination can be one of the most important analyses performed on a food product and yet one of the most difficult from which to obtain accurate and precise data. One of the most fundamental and important analytical procedures that can be performed on a food product is an assay for the amount of moisture. The dry matter that remains after moisture removal is commonly referred to as total solids. This analytical value is of great economic importance to a food manufacturer because water is inexpensive filler.

1.2. Objectives

Introduce and implement a quality control system for same selected analytical determinations, which are used in Fats and oil analysis.

1.2.1.Specific Aims & Objectives

1 Select suitable check samples for each analysis

2.Certification of check samples from accredited laboratories

3.Evaluations of expiry dates for check samples.

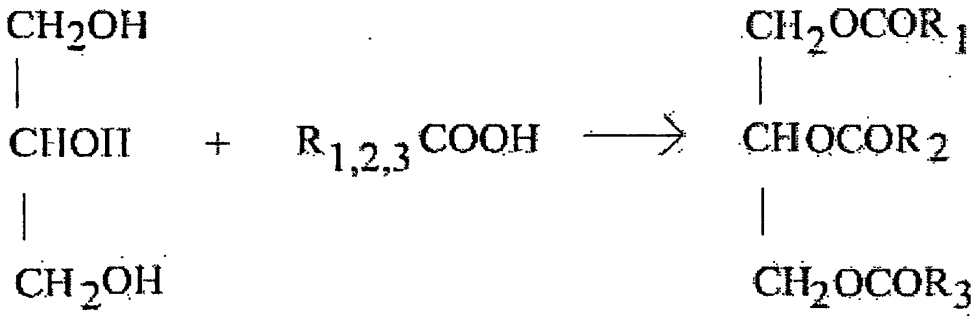
4. Implementation of control charts for ongoing analytical system.

CHAPTER02 - LITERETURE REVEWE

2.1.Fat and Oils

Oils and fats are triglycerides of fatty acids. Most of the physical and chemical parameters of oils and fats are determined by the nature of the side chain of the fatty acid and the unsaturation of the carbon chain.

Therefore each and every crude oil has a characteristic melting point and slip point due to their different side chains and differences in the number of multiple bonds.



Formation of triglycerides

2.2. Crystallization of Fats

During processing of fats, crystallization is often used to modify the properties of the fat. For example, winterization of vegetable oils is needed to ensure that the oil remains a clear liquid even when stored at low temperatures for extended time periods. The process of fractionation of fats to produce components of natural fats with different melting properties also requires control of crystallization to optimize the separation process. Many fats, including palm oil, palm-kernel oil, milk fat, and tallow, are fractionated by crystallization to produce different functional fats.

Fats are made up primarily of triacylglycerols (TAG)s, approximately 98%, with the remainder of the fat being more polar lipids like diacylglycerols (DAGs), monoacylglycerols (MAGs), free fatty acids (FFAs), phospholipids, glycolipids, sterols, and other minor components. In refined fats, these minor lipids are much lower in concentration than in unrefined fats. Although the TAGs form the main crystalline phase, the minor components, or impurities, can often play a large role in how crystallization occurs and crystallization may be substantially different in a refined oil than in the unrefined starting material (Formo and Swern, 1979).

2.2.1.Factors Affecting Experimental Results

2.2.1.1.Tempfature of the sample

The temperature of the sample can strongly affect the results obtained from the Minispec. The following diagram best demonstrates the changes in the FID with temperature.

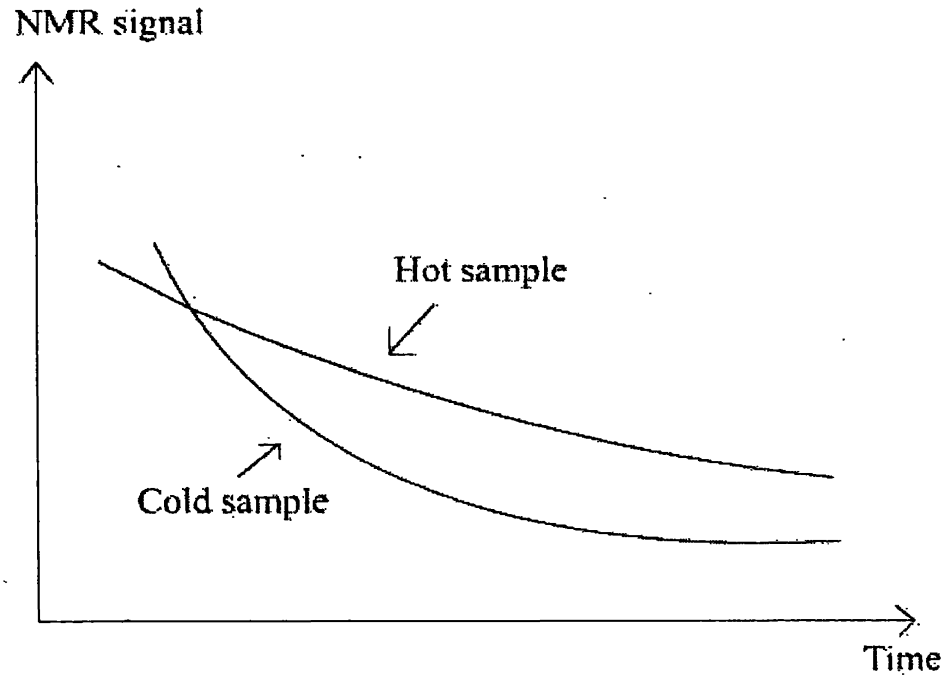


Figure2.1. Effect of Temperature on NMR signal

Because of this temperature effect, all of the samples that are measured and used for calibration (excluding the Plexiglas SFC standards) should be equilibrated to the same temperature. If the samples will be in the sample chamber for more than a few seconds this temperature should be 40 C, the temperature of the magnet (unless a variable temperature probe is being used). Samples, which have all been equilibrated to one temperature, give more accurate and reproducible results.

2.2.1.2.Height of the sample in the tube

The height of the sample (or amount of sample) and the position of the sample in the tube is also very important when using an absolute probe head. If the sample is not completely located within the coil then the signal will correspond only to the sample found within the coil. Also a homogeneous rf pulse extends out about 0.5 on either side of the centre of the coil so if any sample extends beyond these limits a full 90 degree or 180 degree pulse will not be applied with either of these errors, the signal intensity will not correspond to the total number of protons present so the normalized signal (signal/gram) will no longer be accurate.

Therefore, it is very important to the accuracy of the measurement that the sample have a maximum height of less than 30mm and that it is centred within the coil.

2.2.1.3. Reliability of the calibration samples

For absolute type experiments, the accuracy of the results is governed by the accuracy and precision of the initial calibration standards used to construct the calibration curve.

Thus, it is extremely important that extra time and care is taken in choosing preparing and measuring the standards. They must be completely representative of the samples that will be routinely measured. For example they should be prepared in the same way and their standard values should cover the potential range of the unknown samples. The primary analysis should be done as accurately as possible.

2.2.1.4.Relaxation Delay

If the delay between experiments (Recycle Decay) is not sufficient to allow the net magnetization to return to the equilibrium position, errors with the date acquisition will result. The recycle decay must always be greater than or equal to five times the T1(spin-lattice relaxation constant) of the sample. If a sample is composed of more than one component the Recycle Decay is set to five time the greatest T1 (Herbet Meling and Harald Todl, 1994).

2.3.Slip point

Fats consist of a complex mixture of glycerides and therefore do not have sharp melting points, unlike pure chemical substances.
There are many methods for determination of the melting points, setting points or Slip-point of a fat. These methods often provide different values and even with a single method, different laboratories may obtain different results.

The problems are caused by the fact that fats do not have a melting point but a melting range. Moreover, the melting ranges of a fat dependent on the pre-treatment of the sample, in other words, on its state of crystallisation. The transition temperatures between crystal modifications and rate of transition varies for different fats and this means that its impossible to draft a uniform standard method for applicable to all fats. Any method is bound to be a compromise: The results obtained will have only relative_values and reproducibility will be reasonable only when the method is accurately standardised.
There are Manual and Automatic methods for determination of slip-point. Consequently, the Manual method often leads to non-reproducible results especially between laboratories but also within the same laboratory (Berger, 1982).

2.4. Iodine Value

2.4.1.Principle

The iodine value (or iodine number) is a measure of degree of unsaturation, the number of carbon-carbon double bonds in relation to the amount of fat or oil. Iodine value is defined as the gram iodine absorbed per 100g samples.

A quantity of fat or oil is reacted with a measured amount of iodine (or some other halogen). The amount of iodine left at the end of the reaction is then measured to calculate the amount of unsaturation, the more iodine is absorbed; therefore, the higher the iodine value, the grater the degree of unsaturation .

$$\text{Excess ICl} + \text{R-CH=CH-R} \rightarrow \text{R-CHI-CHCl-R+ Remaining ICl} \quad (1)$$

$$\text{ICl} + 2\,\text{KI} \rightarrow \text{KCl} + \text{KI} + \text{I}_2 \quad (2)$$

$$\text{I}_2 + \text{Starch} + 2\text{Na}_2\text{S}_2\text{O}_3\ (\text{blue}) \rightarrow 2\,\text{NaI} + \text{Starch} + \text{Na}_2\text{S}_4\text{O}_6\ (\text{colourless}) \quad (3)$$

Iodine value is used to characterize oils to follw the hydrogenation process in refining, and as an indication of lipid oxidation, since there is a decline in unsaturation during oxidation. The wij's method is probably more widely used and gives results closer to theoretical values.

2.5.Moisture content of foods

The moisture content of foods varies greatly, water is a major constituent of most food products. The approximate expected moisture of content of a food can affect the choice of the method of measurement. It can also guide the analyst in determining the practical level of accuracy required when measuring moisture content, relative to other food constituents.

Food	Moisture content
Fats & Oil	0.01
Margarine	15.5
Butter	15.5

2.5.1. Karl Fischer titration

The Karl Fischer titration is particularly adaptable to food products that show erratic results when heated or submitted to a vacuum. This is the method of choice for determination of water in many low- moisture foods like dried fruits and vegetables (AOAC Method 967.19 E-G), candies, chocolate (AOAC Method 977.10), roasted coffee, oils and fats (AOAC Method 984.20), or any low- moisture food high in sugar or protein. The method is quite rapid and sensitive and uses no heat.

This method is based on the fundamental reaction described by Bunsen in 1853 involving the reduction of iodine by SO_2 in the presence of water:

$$2\,H_2O + SO2 + I_2 \rightarrow H_2SO_4 + 2HI \qquad (5)$$

This was modified to include methanol and pyridine in a four- component system to dissolve the iodine and SO_2:

$$C_5H_5N \cdot I_2 + C_5H_5N \cdot SO_2 + C_5H_5N + H_2O \rightarrow 2\,C_5H_5N \cdot HI + C_5H_5N \,.\, SO_3 \qquad (6)$$

$$C_5H_5N.\, SO_3 + CH_3OH \rightarrow C_5H_5N\,(H)\,SO_4.\, CH_3 \qquad (7)$$

These reactions show that for each mol of water, 1 mol of iodine, 1 mol of SO_2, 3 mols of pyridine, and 1 mol of methanol are used. For general work, a methanolic solution is used that contains these components in the ratio of 1 iodine: 3 SO_2: 10 pyridine, and at a concentration so that 3.5 mg water = 1ml reagent. A procedure for standardizing this reagent is given below.

In a volumetric titration procedure, iodine and SO_2 in the approximate form are added to the sample in a closed chamber protected from atmospheric moisture. The

excess of I_2 that cannot react with the water can be determined visually. The end point colour is dark red- brown. Some instrumental systems are improved by the inclusion of a potentiometer to electronically determine the endpoint, which increases the sensitivity. Instruments are also available to automatically perform the Karl Fischer moisture analysis by the conductometric method.

The volumetric titration procedure described above is approximate for samples with a moisture content greater ~ 0.03 percent. A second type of titration, referred to as coulometric titration, is ideal for products with very low levels of moisture, from 0.03 percent down to parts per million (ppm) levels. In this method, iodine is electrolytically generated to titrate the water. The amount of iodine required to titrate the water is determined by the current needed to generate the iodine. (Suzanne ,2002)

2.6.Quality Definition (ISO)

"The totality of features and characteristics of a product or service that bare on its ability to satisfy Stated or Implied Needs"

Stated- When contractual agreement exists.

Implied- When produced for assumed needs; as identified by market research and defined.

Needs- Needs may include aspects of usability safety, availability, reliability, maintainability, economics, environment etc.
Needs are usually translated in to features and characteristics with specified criteria.

2.7.Control charts

2.7.1.Control for measurements

If in the data being plotted on a control chart, is reliable the decisions based on the chart is could be innocent .
Since taking the measurements needed for the control chart is itself a process, we can use control chart to detect assignable causes in the measurement procedure.
For example, we can use a control chart to make sure that a measuring device is calibrated properly by running repeated tests on a standard weight or measure.

2.7.2. Decision marking for management

Control charts can be used to help management make policy decisions. A manager might use a control chart to assist in discovering the kinds of changes that need to be made in order to improve the quality of a service. As another example, when a process can be shown to be in statistical control, and when the process is not capable of meeting specifications, a manager has a basis for deciding whether or not to invest in new equipment in order to improve the process (Bruce and Richard, 1997).

2.8.Check Sample

The check sample need not be identical in all aspects to the samples that are the subject of the analytical determination, but it must be sufficiently similar in chemical composition and physical state (solid, liquid, or gas) so that the conclusions drawn from the control chart are relevant to the subsequent analyses. In particular the component that is the subject of a chemical analysis must be present in the check sample in the check sample in the same form as in the daily samples.

The check sample must be stored under conditions that assure the integrity and the long-term stability of the composition. Preferably, the check sample is a suitable Certified Reference Material (CRM), but when that is not available the laboratory can select or compose its own check sample provided it conforms to the requirements stated above.

The laboratory manager must determine the frequency of measuring the check sample and entering a fresh result in the control chart. For a frequent analysis this could be daily, but no less than once a week. For less frequent analyses it is common practice to include a check sample with each sample run (UMA).

CHAPTER 03 - METERIALS & METHODOLOGY

3.1.Determination of Solid Fat Content Using Pulsed LR NMR

3.1.1. Definition

The pulse NMR method is based on the measurements of the ratio of H-Nuclei in the solids to the total number of Nuclei[solids-liquids].The fast and slow decaying signals arising from the H-Nuclei in the solid and liquid phases respectively, are converted into a percentage of solids and is reported as NMR solid percent.

at t ^{0}C$\rightarrow$N$_t$

3.1.1.1. Equipment and Reagents Necessary

Apparatus:

Pulse NMR instrument-Brucker minispecs p20j
Constant temperature baths.
Prescribed glass tubes.
A cooling unit maintained at 0 C filled with a suitable cooling liquid

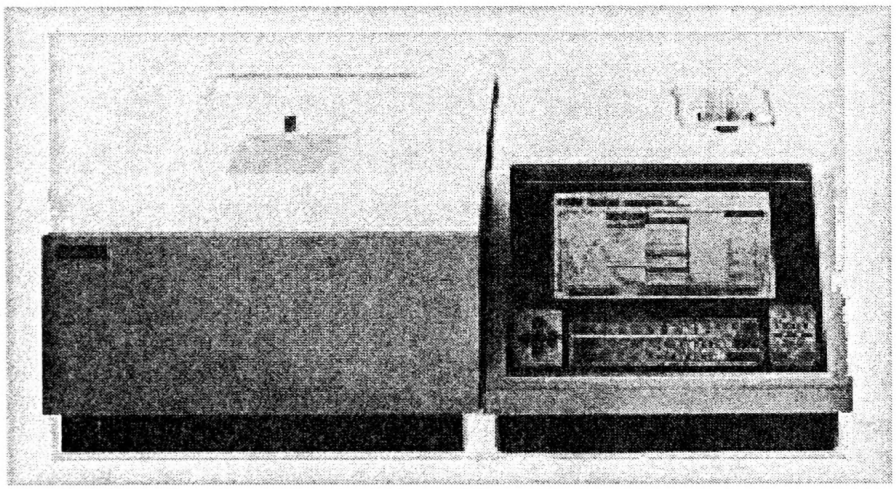


Figure 3.1: Pluse NMR instrument

Reagents:

20% aq. ethylene glycol
Anhydrous Na_2SO_4

3.1.1.2.Check sample for Solid Fat Content

As the check sample, standard fat was used. It was a suitable Certified Reference Material from India.

3.1.1.3. Instrument Calibration and NMR test

Before the Minispec was delivered, a basic instrument calibration was done factory performed, using several Bruker primary standards.
NMS NMR Test routines were allowed the user to check the validity of a current instrument calibration and, if needed, perform a re-calculation of several instrument parameters.

3.1.1.3.1. Instrument calibration

The NMS Instrument calibration was a set of instrument related parameters such as on-resonance magnetic field, instrument gain, detection angles, excitation pulse lengths etc. For SFC instruments additional SFC values were in the parameter list. This basic instrument calibration was factory performed and cannot be repeated by the user. To protect from unintentional cell, a combination of commands were activated the complete fully automatic basic instrument parameter calibration routine.

3.1.1.3.2.NMR Test

To check an existing instrument calibration or to generate new instrument parametervalues, the user can run the NMR Test routine (<CALIBRATE><INSTRUMENT><NMR TEST>).
A complete NMR Test was recommended after relocating the instrument, in case of the instrument had been switched off for an extended period of time or after the system had been working for a while. If the values of the parameters were within certain limits, the test was passed through without stopping. If they were out of limits, the user was been asked whether the new values should be taken into account or not.
The NMR Test was very important, because only a well-tuned instrument can measure accurate results.

3.1.1.3.2.1.Running the NMR Test

The NMR Test routine prompts the user to insert the calibration standard delivered with the NMS as follows:

INSERT NMR TEST SAMPLE#1(0.00% Solid)

After calibration standard no.1 is inserted, the NMR Test starts and the instrument parameters are optimized in the order presented above according to the chosen form (long or short). Follow the instruments prompts during the NMR Test routine.
For SFC instruments, standards no.2 and no.3 were used to calibrate the Minispec for the solid fat content measurements (or similar type experiments). The Minispec was applied a 90 degree pulse to the sample and measured the NMR signal around 0.0085 msec and 0.0675 msec. The curve, as shown in Figure 3., had two components a fast decaying component (solid) and a slow decaying (liquid) component. The signals measured in the two sampling windows were correlated to the known solid\liquid ratio of the standard.

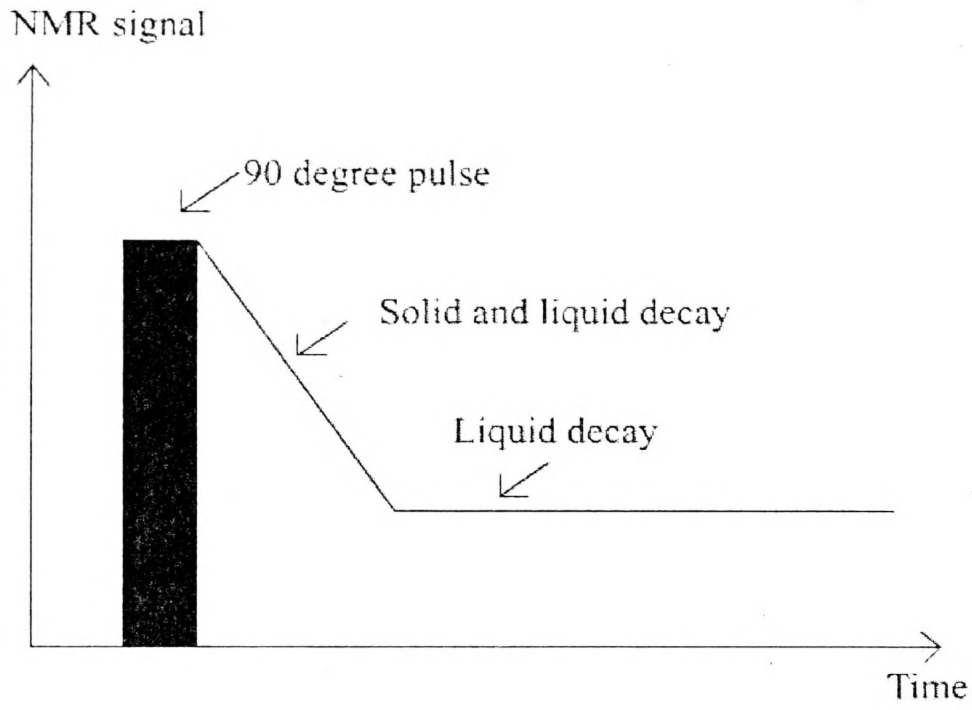


Figure: 3.2.Time evolution of NMR response after a signal 90 degree pulse.

The two standards provided for these tests were made using mineral oil and Plexiglas to simulate the liquid and solid components of edible oil. These samples do not need to be equilibrated to 40 ^{0}C, as they are meat to be analyzed at room temperature. The sample for test no.2 is approximately 30 percent solid and the sample for test no.3 is approximately 70 percent solid (each set was individually prepared so they was vary slightly). The actual percentage should been entered into the dialogue boxes as they appear during the execution of the NMR Test.

The percentage solid (30 percent and 70 percent) of the samples were especially chosen to determine the constraints used in calculating the solid fat content.
If the NMR Test passes without error messages, the NMS was ready for routine measurements.

3.1.1.4.Procedure

All glass tubes were washed, cleaned and dried in an oven prior to use.
Melted samples were filtered to the glass tubes. It was ensured that the sample was free from moisture.

If moisture is present, anhydrous Sodium Sulphate can be used in the filtering process to get rid of moisture. Also glass tubes should be filled with the samples only up to the prescribed level of approximately 3 cm by length.
Melted sample (80 ^{0}C) were left in a content temperature bath of 60 ^{0} C for 30 minutes.
The sample tubes were left for 60 minutes at 0^0 C.

Sample tubes were transferred to the constant water baths of the chosen test temperature and left for 60 minutes.

[All constant temperature baths should be pre-set prior to the commencement of the test as control of temperature is very important].

The Bucker minispecs p20j instrument was calibrated with the use of prescribed the. results was red on the recorder as the 'measure' bulb illuminates.

[Transpiring of sample tubes from the constant temperature baths should be done as quickly as possible, before there could be any changes in temperature.]

Fallowing above standard UMA procedure a training set of 20 initial, was composed for the check sample and control chart was constructed. Then the experiment was done daily and the average of the results during a week was entered to the control chart.

3.2.Determination of Slip point

3.2.1.Introduction

The sample was heated to melt and filtered through a filter paper. A fine capillary with both ends open was used in this experiment.A filtered fat column of approximately 1 cm by length was filled to the capillary and directly crystallised in the unstable from with the use of ice. Slip point was the temperature at which this column of fat starts to rise, after melting under hydrostic pressure.

3.2.1.1. Equipment and Reagents Necessary

Apparatus:

Slip-point apparatus (Glass-water reservoir)

Calibrated thermometer

Slip-point capillaries (open at both ends, length 5 cm, inside diameter 0.9-1.1 mm, wall thickness 0.15-0.20 mm).

Electric heating apparatus (the rate of heating should be such that when water is heated from 5 °C the rise in temperature from 25 °-35 °C takes place in 2.5 - 3 min).

Reagents:

NaCl, Ice, Anhydrous Na_2SO_4

3.2.1.2. Check sample

Standard fat sample (Certified Reference Material from India) was used.

3.2.1.3.Procedure

The fat sample was heated on a water bath.The sample was filtered through a filter paper and filled the capillary with some filtered oil, so that a column was formed by 1 cm of length.The filled end of the capillary was immersed in ice- salt mixture and crystallized the column of oil / fat or oil erectly.

Any water presence in the melting point apparatus was discarded which was previously used and filled it with cool water. So that the starting temperature would be at least 10 ^{0}C below the melting point of the oil / fat or oil blend.

The capillary was attached to a thermometer and immersed in such a way that the entire fat column was below the water surface of the apparatus.

The heating device of the apparatus was switched on and heated at a rate of 3 ^{0}C / minutes and the temperature at which the fat content liquefy to cause the fat column to rise under hydrostatic pressure was red and noted.

Fallowing above standard UMA procedure a training set of 20 initial, was composed for the check sample and control chart was constructed. Then the experiment was done daily and the average of the results during a week was entered to the control chart.

3.3.Determination of Iodine Value (Wijs method)

3.3.1.Chack sample for Iodine value

As the check sample highly purified Sunflower oil was used.

3.3.1.1. Equipment and Reagents Necessary

Apparatus:

Glass stoppered iodine flask 500ml, Pipette 20ml, Burette, Filter paper

Reagents:

0.2N ICl , CCl_4/$CHCl_3$ (Carbon tetrachloride (or) chloroform), 0.2N $Na_2S_2O_3$, 10% Potassium iodide (KI) solution, Starch solution

3.3.1.2.Standardisation of sodium thiosulphate solution

The weigh of 0.20 - 0.25g(W g) of well dried $K_2 Cr_2 O_7$ was dissolved in distilled water.5ml of conc. HCl and 25ml of 10%KI were added and kept in a dark place for half hour. Using starch as indicator, the sample was titrated with prepared sodium thiosulphate solution (V ml). Carryout three determinations to get an average figure.

$K_2Cr_2O_7 + 6\ KI + 14\ HCl \rightarrow KCl + 2\ CrCl_3 + 3\ I_2 + 7\ H_2O$

$2\ Na_2S_2O_3 + I_2 \rightarrow Na_2S_2O_6 + 2\ NaI$

Normality = (1000*W)/(49.03*V)

3.3.1.3.Analysis of Iodine value

The prescribed amount of sample was weighed accurately in to the iodine flask. Weight of sample can be determined as follows.

Weight = 20/Expected IV

The weighed sample was dissolved in an appropriate amount of solvent $CCl_4/CHCl_3$ (10ml). 20ml of wijs solution and 10ml of 10%KI were added accurately and mixed well .The mixture was kept in the dark for about 30-60 minutes. Distilled water was added and titrated with 0.2N $Na_2S_2O_3$ solution, with constant & vigorous shaking. The starch indicator was added when the solution was turned to pale yellow (straw colour). Simultaneously a blank was carried out using the quantity of reagents at the same time under same conditions.

Fallowing above standard UMA procedure a training set of 20 initial, was composed for the check sample and control chart was constructed. Then the experiment was done daily and the average of the results during a week was entered to the control chart.

3.4.Determination of Moisture in Oil/Fat using Karl Fisher

3.4.1. Equipment and Reagents Necessary

Apparatus:

ORION TURBO 2 Blending Karl Fischer Titrator

Micro litre syringe

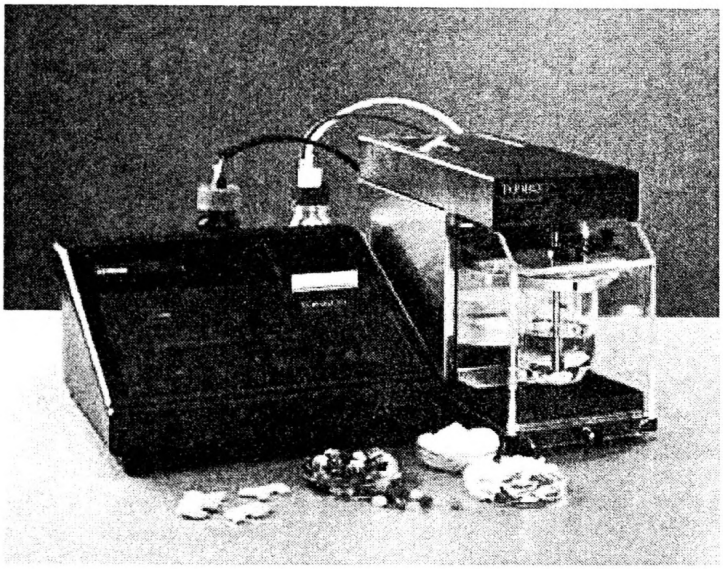


Figure 3.3. ORION TURBO 2 Blending Karl Fischer Titrator

Reagents:

Methanol; Karl Fischer reagent(KFR)

3.4.1.2. Check sample

In this experiment, distilled water used as the check sample.

3.4.1.3. Instruments Calibration

The instrument was switched on and drained out any excess solvent from the titration vessel, if any.It was made sure that the reagent bottle was not empty and it contained adequate amount of reagent.It was made sure that the Silica gel containers were blue in colour.If there was not a colour change in Silica gel with blue, Silica gel was dried.

When the displayer red "IS VESSEL READY", reply was given as " YES" Conditioning was commenced; it was reached to the end point after period of time depending upon the amount of moisture in the solvent. A milligram result of moisture that found was displayed.

After conditioning has completed, the next message was " METHOD I CHANGE IF REQUIRED". The answer was given as "NO".

When the displayer showed " CALIBRATE", the answer "YES" was pressed, and when it was showed " ACCEPT CABRATION MENU?", "YES" Button was pressed.

Conditioning was continued until an end point was reached. Then the displayer was red the "SAMPLE VOLUME = 0.025 ml" and " NEW VOLUME = 0.01 ml".
Enter key was pressed and the displayer was red " SAMPLE ADDED".
The sample port stopper was removed and introduced a 10 micro liter water sample into the titration vessel by way of a microliter cyringe and " YES" button was pressed.

Titration was started by the instrument and when the end point was reached, the result was recorded by the printer as milligrams per steps.

This procedure was repeated for three times. If the results were fallen within 1% CV of their mean value, the displayer would read " CALIBRATION PASSED".
If the "CALIBRATION FAILED" were appeared, a further calibration sample would be requested.

Upon completion a successful calibration, it would be then displayed " ACCEPT SAMPLE MENU" in the screen.

The instrument would be now ready for the titration of samples.

3.4.1.4.Procedure

Known amount of distilled water was injected to the analyser and the reading was observed.

3.5. Construction of Shewart Control Chart

The name of the analytical method (e.g. the UMA number and title), was entered on a blank Shewart chart, the (concentration) units to express the result, and first entry date.

The Conditions for the analytical determination were kept such that the procedure was expected in control, i.e. the instrumentation is working properly, the analyst is familiar with the method; reagents are fresh, facilities are in order etc. Results obtained under such conditions were expected to reflect the variability typical for the analytical method.

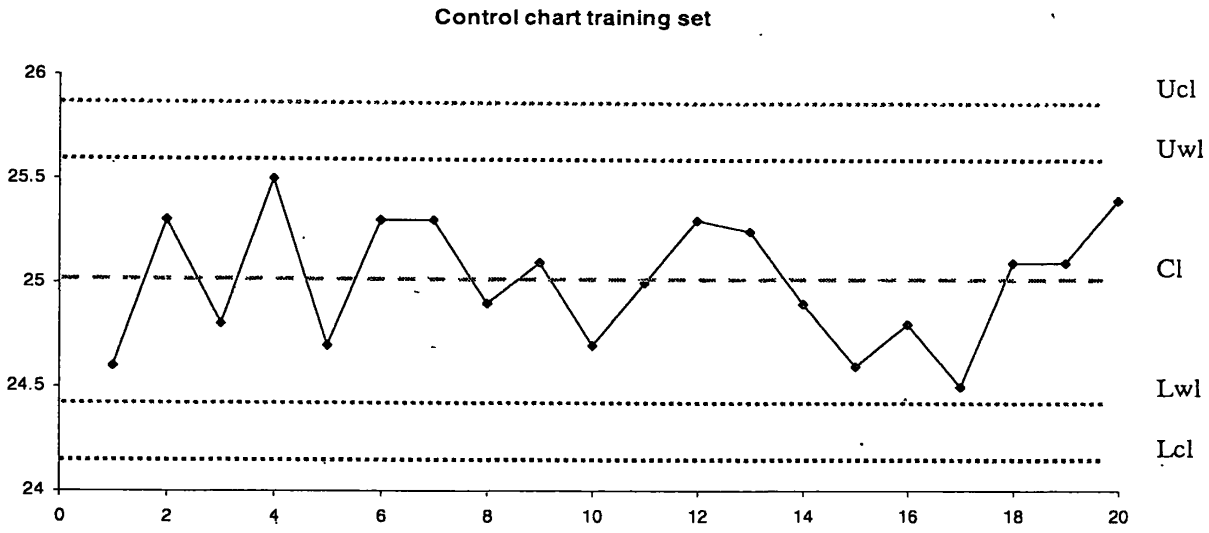
Under these conditions training set of 20 initial were composed, independent observations for the check sample were measured over an interval comparable to the time between this calibrations, i.e. several days.

The average and the standard deviation of the training set were determined.
If the analytical method had been validated in-house, the internal reproducibility was taken as a measure of the standard deviation; in that case the target value in the control chart was derived from the average of six observations for the check sample; for example duplicate measurements over three days.

In either case at the bottom of the control chart were entered calculate from the average and the standard deviation the warning limits (average +2s,and average -2s)and the control limits (average +3s,and average -3s).

All 20 observations were entered from the training set in a graph (see figure 3.).

A green line for the target value, blue line for the warning limits and red lines for the control limits were used.



cl:24.99 ucl: 25.89 lcl: 24.09

Figure 3.4. :Observations of the training set

3.6. Use of the Control Chart

The control chart was accepted new observations after the training set was completed.

New data entered in the control chart reveal whether or not the analytical determination was in control. The warning limits (2s) and the control limits (3s) were used for this decision. From their definition it was statistically expected (on average) one out of every twenty observation were exceed the warning limits and one out of every thousand the control limits. As long as that was the case we consider the method in control.

If, however, the observations for the check sample was exceed the warning or the control limits more frequently, then we conclude that the method was no longer in control. In that case the method can no longer be used for regular analysis until the cause of the out of control situation was identified and removed

Generally speaking there were three major sources for out of control: a continuous drift of the target value, a stepwise change of the target value, or an increase of the

standard deviation. The distinction was relevant, because the nature of the disturbance helps us to identify possible causes and take corrective action. For example, drift was indicated deterioration of gas chromatographic column, and a malfunctioning detector caused an increase of the standard deviation.

When the cause of the disturbance had identified and repaired, a new determination of the check sample should fall within the warning limits and the analyses was resumed. The corrective action taken must annotate in the control chart.

CHAPTER 04 - RESULTS & CALCULATIONS

4.1. Definitions

4.1.1. Standard deviation, s, refers to a signal analysis of the check sample under conditions that the analytical method is in control.

4.1.2. Target value is the long term average of the results obtained for the check sample.

4.1.3. Warning limits are located at **+2s** and **-2s** above and below the target value.

4.1.4. Control limits are located at **+3s** and **-3s** above and below the target value.

4.2. Results of Solid Fat Content Using Pulsed LR NMR

Table 4.1.Check samples results for training sets

Sample number	N-20 (^{0}C)	N-30(^{0}C)	N-35(^{0}C)	N-40(^{0}C)
1	60.9	30.11	17.45	3.17
2	60.9	30.02	17.5	3.16
3	61.02	30.33	17.54	3.18
4	60.94	30.22	17.54	3.1
5	60.9	30.19	17.38	3.62
6	61.55	30.42	17.59	3.16
7	60.48	30.19	17.35	3.06
8	61.16	30.49	17.37	3.28
9	61.14	30.31	17.48	2.93
10	61.23	30.38	17.76	3.07
11	61.08	30.34	17.82	3.07
12	61.23	30.12	17.78	3.05
13	61.02	30.48	17.7	3.13
14	60.96	30.16	17.84	3.04
15	61.6	29.81	17.96	3.23
16	61.79	30.09	17.75	3.13
17	61.66	30.06	17.8	3.12
18	60.95	30.43	17.69	3.02
19	60.95	30.46	17.68	3.06
20	60.96	30.45	17.6	3.08

Table 4.2.Check samples results for Solid fat content

Date	N-20 (^{0}C)	N-30(^{0}C)	N-35(^{0}C)	N-40(^{0}C)
16/03/05	61.17	30.31	17.35	2.89
17/03/05	61.09	30.65	17.82	2.73
18/03/05	61.34	30.82	17.96	2.49
28/03/05	60.85	30.73	17.4	2.45
29/03/05	60.42	30.71	18.19	3.78
30/03/05	60.89	30.88	18.87	3.16
31/03/05	61.05	31.09	17.39	3.04
1/4/05	61.18	30.8	17.75	3.26
4/4/05	61.01	30.59	17.8	2.91
5/4/05	61.04	30.8	16.95	2.56
6/4/05	60.8	30.8	17.63	2.57
7/4/05	60.4	31.15	17.59	2.54
8/4/05	60.7	31.35	18.18	2.6
18/04/05	60.49	31.8	17.82	2.61
19/04/05	60.48	31.32	17.73	2.84
20/04/05	60.82	31.23	18.64	2.48
21/04/05	61.35	31.32	18.2	2.68
22/04/05	60.46	31.37	17.88	2.69
3/5/05	61.05	31.69	18.53	2.4
4/5/05	61.11	30.84	16.53	2.43
5/5/05	60.53	31.25	16.73	2.69
6/5/05	60.7	30.5	16.84	2.78
9/5/05	60.18	30.8	16.89	2.92
10/5/05	60.53	30.87	16.46	2.59
11/5/05	61.29	30.8	16.29	2.55
12/5/05	59.79	30.88	16.66	2.96
13/05/05	60.03	30.5	16.25	2.77
16/05/05	59.99	29.6	16.21	2.54
17/05/05	60.48	29.53	16.17	3
18/05/05	60.17	30.07	16.19	2.73
19/05/05	60.76	29.53	16.38	2.85
20/05/05	60.29	31.22	16.7	2.6
30/05/05	61.17	30.91	16.66	2.56
31/05/05	60.54	30.7	17.26	2.56
1/6/05	60.08	29.69	16.19	2.93
2/6/05	60.45	29.32	16.12	2.89
3/6/05	60.15	29.95	16.61	2.84
6/6/05	60.65	30.64	16.93	2.96
7/6/05	61.06	30.31	17.12	2.68
8/6/05	60.32	29.55	16.71	2.79
9/6/05	60.51	29.77	16.84	2.81

4.2.1. Solid fat content at 20^0C(N-20)

4.2.1.1.Statistical interpretation of the result of N-20

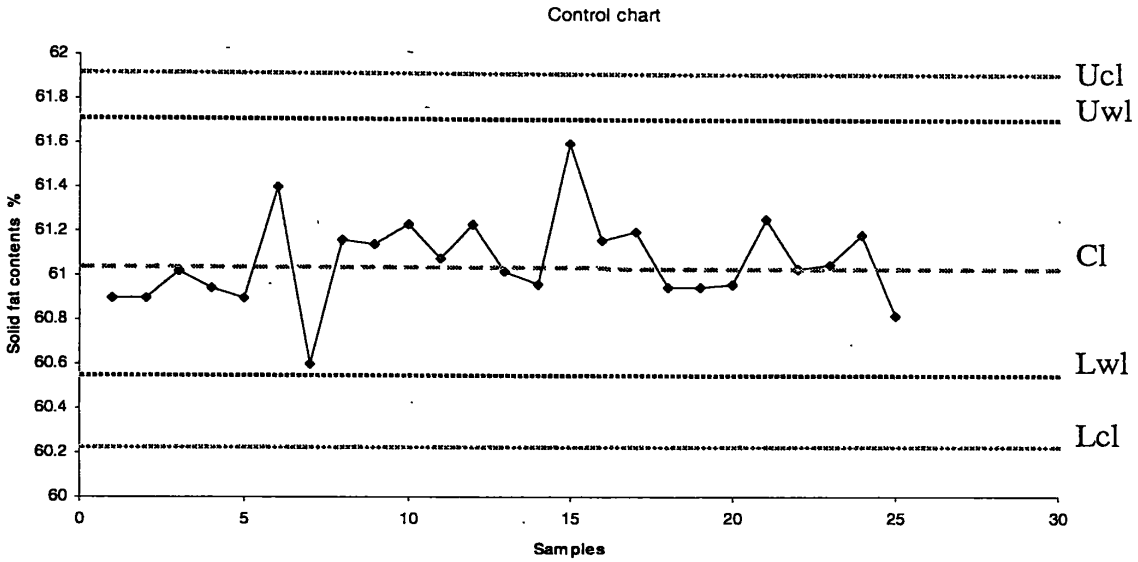


Figure 4.1.Control chart for training set at 20^0C(N-20)

4.2.2. Solid fat content at 30^0C(N-30)

4.2.2.1. Statistical interpretation of the result of N-30

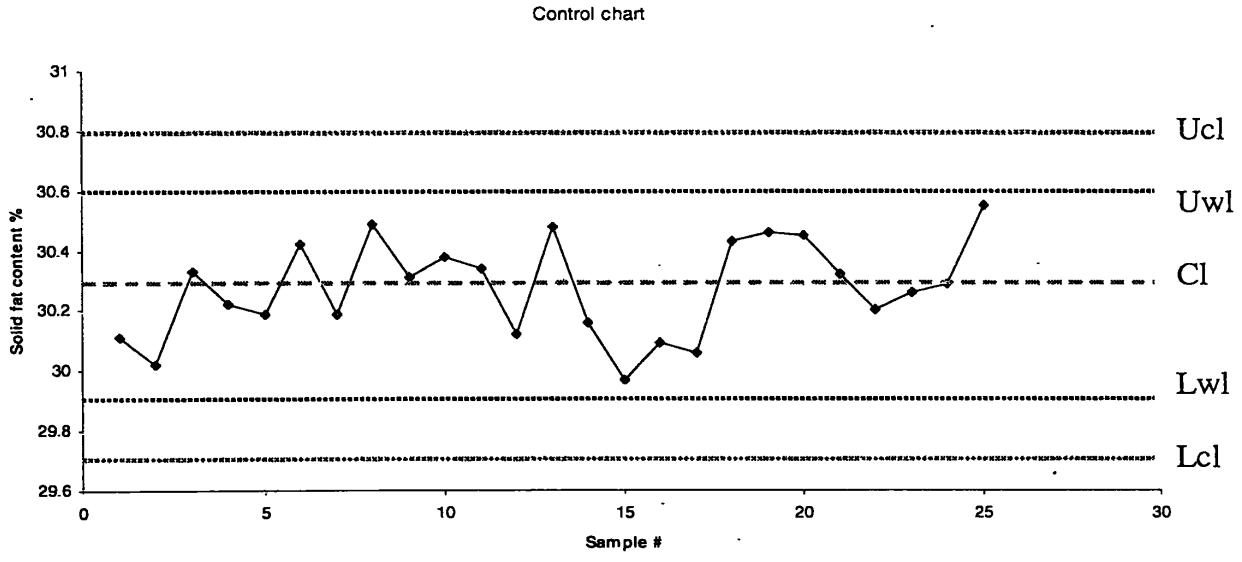


Figure:4.2. Control chart for training set at 30^0C(N-30)

4.2.3. Solid fat content at 35^0C(N-35)

4.2.3.1. Statistical interpretation of the result of N-35

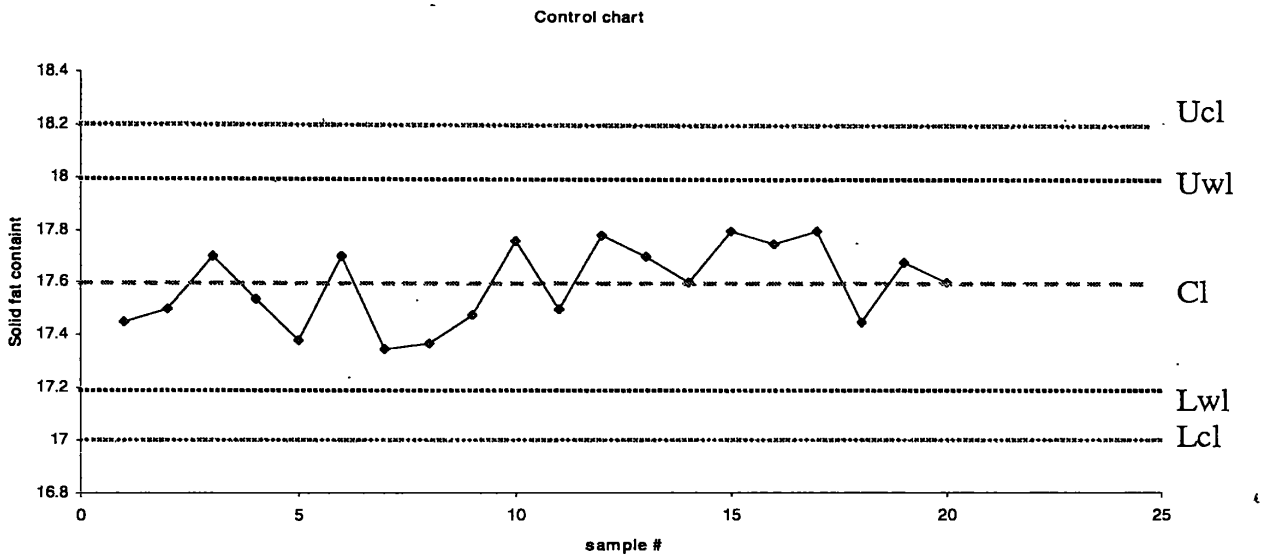


Figure 4.3. Control chart for training set at 35^0C(N-35)

4.2.4. Solid fat content at 40^0C(N-40)

4.2.4.1. Statistical interpretation of the result of N-40

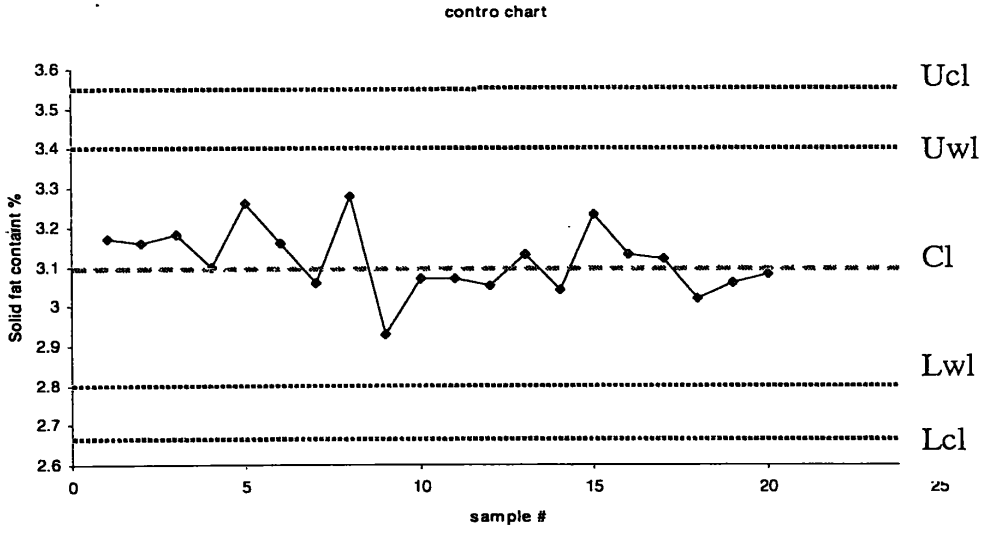


Figure 4.4. Control chart for training set at 40^0C(N-40)

4.3. Results of Slip point

4.3.1. Statistical interpretation of the result of Slip point

Table 4.9.Check samples results for training set

sample #	slip point(40^0C)
1	37.6
2	37.8
3	38.1
4	38.2
5	38.6
6	38.4
7	38.6
8	38.1
9	38.1
10	38.1
11	38.4
12	38.5
13	37.9
14	38.2
15	38.4
16	38.5
17	37.8
18	38.2
19	37.6
20	38.1

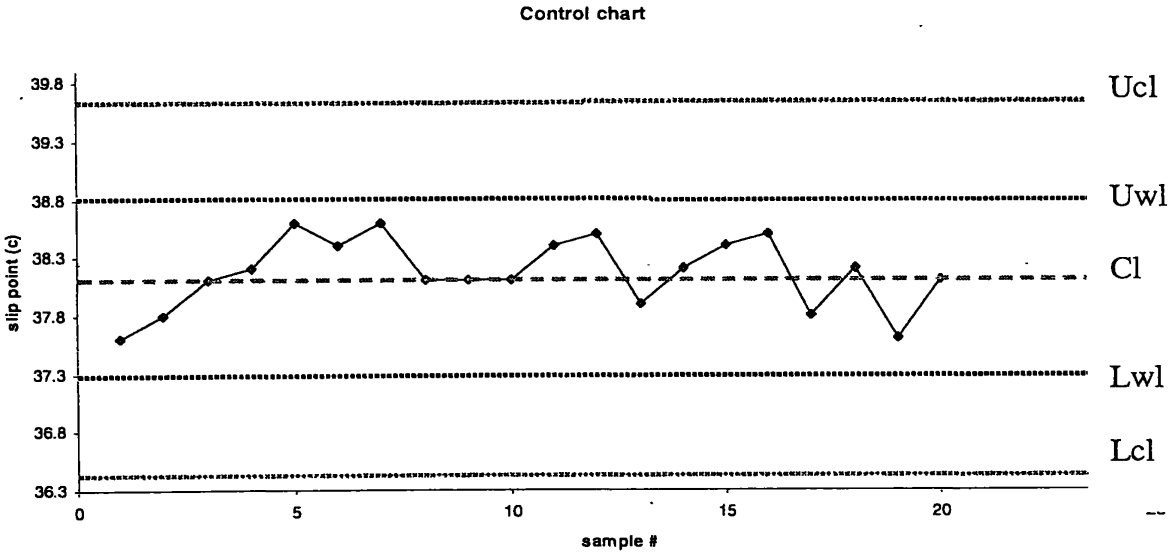


Figure 4.5: Control chart for training set

Table 4.10.Check sample results for Slip point

Date	Slip point (40^0C)	Average
19/04/05	38.6	
20/04/05	38.4	
21/04/05	38	
22/04/05	38.2	38.3
3/05/05	38.6	
4/05/05	38.6	
5/05/05	38.4	
8/05/05	38.5	
9/05/05	37.8	38.4
10/05/05	37.6	
11/05/05	37.9	
12/05/05	38.5	
13/05/05	38.1	
16/05/05	37.6	37.9
17/05/05	37.9	
18/05/05	38.1	
19/05/05	38.6	
20/05/05	38.2	
30/05/05	37.8	38.2
31/05/05	38.4	
1/06/05	38.5	
2/06/05	37.8	
3/06/05	37.9	38.2
6/06/05	38.1	
7/06/05	37.6	
8/06/05	37.9	
9/06/05	38.5	38.0

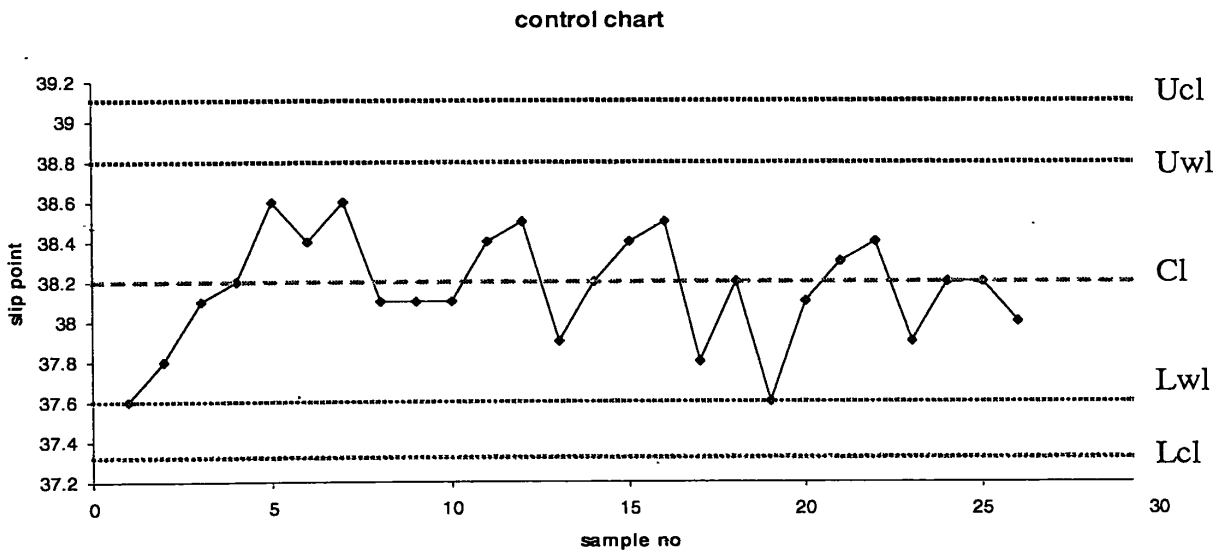


Figure 4.6.Control chart for slip point determination

4.4.Results of Iodine Value (Wijs method)

4.4.1. Calculated iodine values from wij's method

Weight of sample in grams = W,

Volume of thiosulphate used for sample = V_S,

Volume of thiosulphate used for blank = V_B,

Normality of thiosulphate = N,

$IV = [(V_B-V_S)*N*126.9*100]/[W*1000]$

$IV = [12.69*N*(V_B-V_S)]/W$

Example

$V_S = 18.0ml$,

$V_B = 26.5ml$,

W 0.1613g,

N = 0.1999,

$IV = [12.69*0.1939*(26.5-18.0)]/0.1613$

IV = 136.9

4.4.2. Statistical interpretation of the result of Iodine value

Table 4.11.Check samples results for training set

sample #	weight of oil (w g)	volume of titrant(ml)	Iodine value
1	0.1679	17.4	130.3
2	0.1873	17.3	127.2
3	0.173	17.5	137.7
4	0.187	16.6	137.1
5	0.1613	18	136.5
6	0.1712	17.45	136.9
7	0.22	14.8	137.7
8	0.1504	18.7	134.3
9	0.249	13.1	139.4
10	0.1646	17.5	141.6
11	0.1843	16.7	137.7
12	0.1522	18	144.4
13	0.2227	14.3	141.8
14	0.1602	17.4	128.6
15	0.1496	18.8	133.3
16	0.2151	14.7	140.1
17	0.1236	19.6	136.4
18	0.1534	18.4	137.1
19	0.1625	17.3	138.2
20	0.1701	16.9	129.9

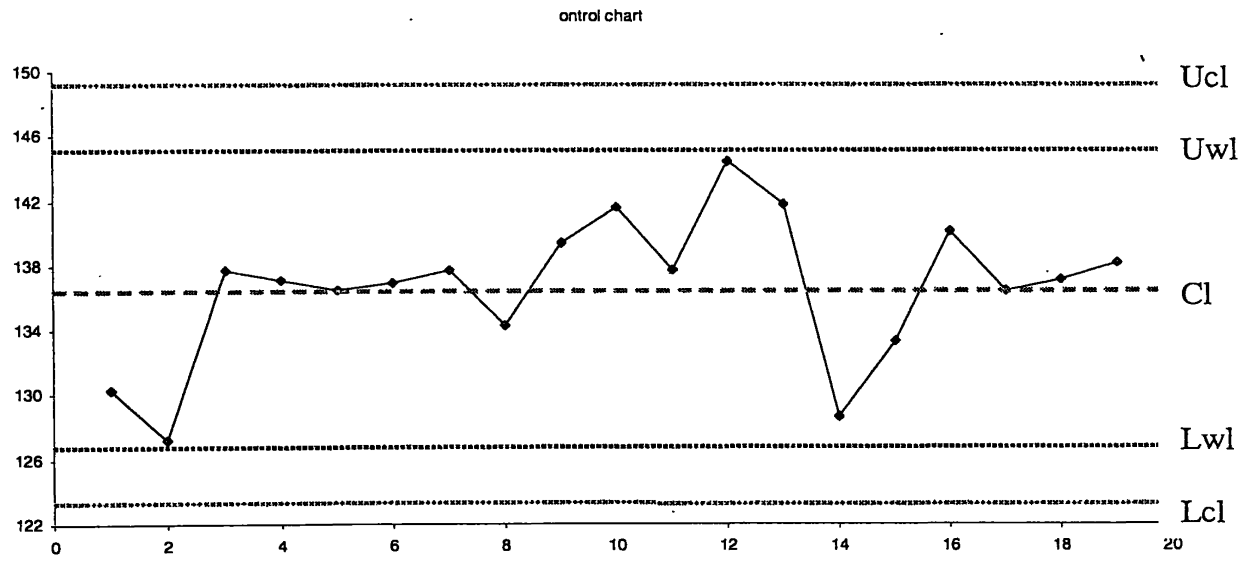
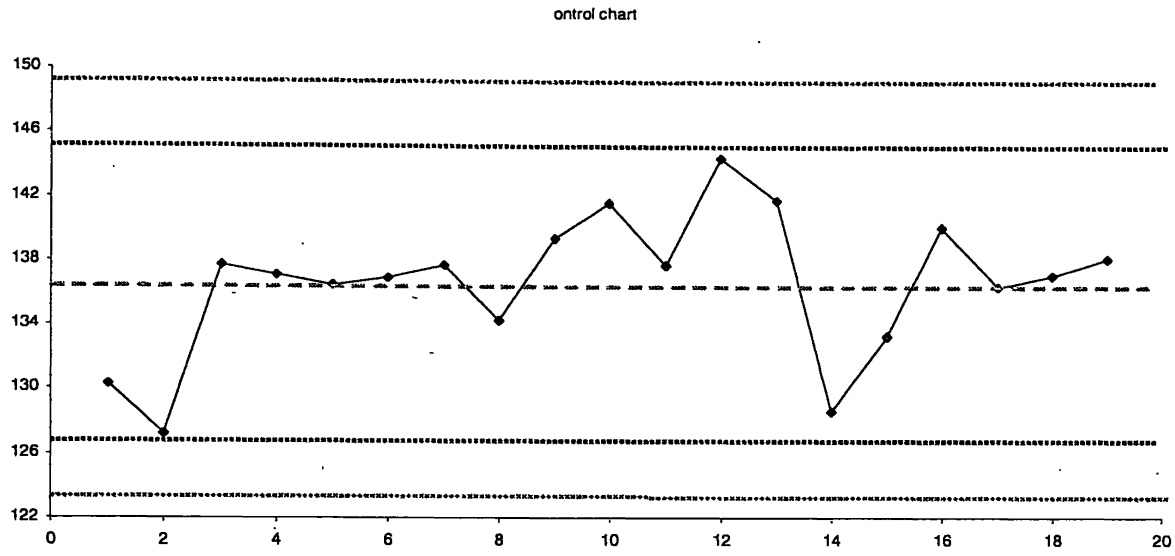


Figure 4.7.Control chart for training set for iodine value

Table 4.12.Check sample results for Iodine value

Date	weight of oil (w g)	volume of titrant	Iodine value	Average
22/04/05	0.1679	17.4	130.3	130.3
3/04/05	0.1873	17.3	127.2	
4/5/05	0.173	17.5	137.7	
5/5/05	0.187	16.6	137.1	
6/5/05	0.1613	18	136.5	
9/5/05	0.1712	17.45	136.9	135.1
10/5/05	0.22	14.8	137.7	
11/5/05	0.1504	18.7	134.3	
12/5/05	0.249	13.1	139.4	
13/05/05	0.1646	17.5	141.6	
16/05/05	0.1843	16.7	137.7	138.1
17/05/05	0.1522	18	144.4	
18/05/05	0.2227	14.3	141.8	
19/05/05	0.1602	17.4	128.6	
20/05/05	0.1496	18.8	133.3	137
30/05/05	0.2151	14.7	140.1	
31/05/05	0.1236	19.6	136.4	
1/6/05	0.1534	18.4	137.1	
2/6/05	0.1625	17.3	138.2	
3/6/05	0.1701	16.9	129.9	136.3
6/6/05	0.249	13.1	139.4	
7/6/05	0.1646	17.5	141.6	
8/6/05	0.1504	18.7	134.3	
9/6/05	0.249	13.1	139.4	
10/6/05	0.2227	14.3	141.8	139.3



4.5. Results of Moisture in Oil/Fat using Karl Fisher

Table 4.5.1Check samples results for training set

sample #	moisture(mg)
1	10.14
2	10.13
3	10.12
4	10.2
5	10.1
6	9.99
7	9.95
8	9.98
9	10.14
10	9.96
11	9.89
12	9.91
13	10.19
14	10.21
15	9.98
16	10.28
17	10.21
18	10.24
19	10.17
20	9.98

4.5.1. Statistical interpretation of the result of Moisture

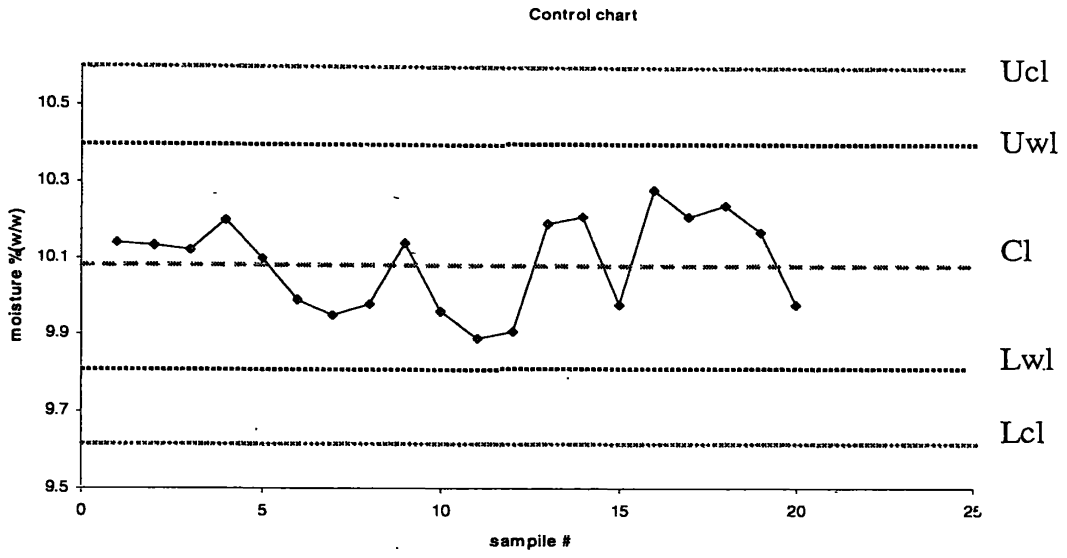


Figure 4.8.Control chart for training set

Table 4.14.Check sample results for Moisture in Oil/Fat using Karl Fisher

Date	Moisture(mg)	Average
4/05/05	10.1	
5/05/05	9.98	
8/05/05	10.12	
9/05/05	10.2	
10/05/05	10.2	
11/05/05	9.89	10.1
12/05/05	9.91	
13/05/05	10.19	
16/05/05	9.89	
17/05/05	9.91	
18/05/05	10.19	
19/05/05	10.2	9.98
20/05/05	10.1	
30/05/05	9.99	
31/05/05	9.95	
1/06/05	10.2	
2/06/05	10.1	10.0
3/06/05	9.99	
6/06/05	9.95	
7/06/05	9.91	
8/06/05	10.19	
9/06/05	10.1	10.1

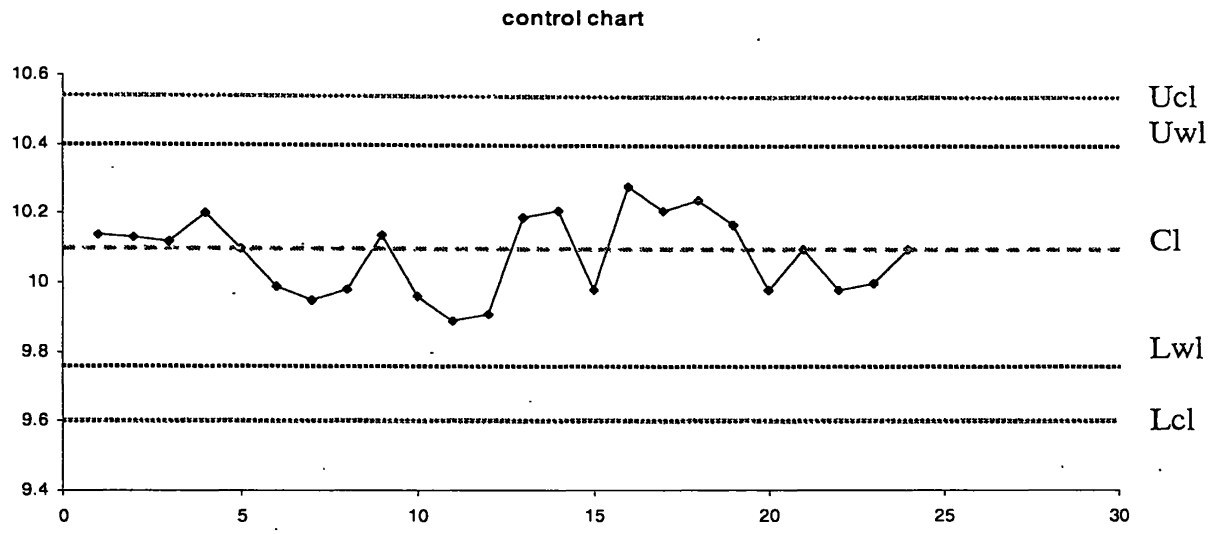


Figure 4.9.Control chart for moisture

CHAPTER 05- DISCUSSION

5.1. Discussion

During the period of the four-month the observations obtained from the check samples using standard UMA methods were placed between control limits in the control charts.

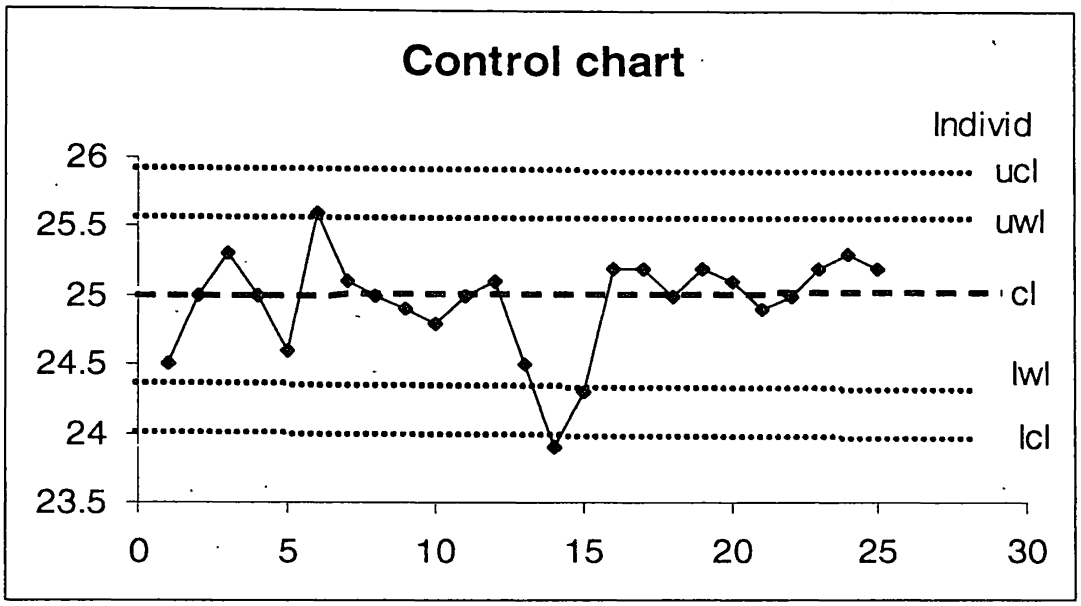
5.2.Identification of out-of-control situations

One observation exceeds either control limit (see Figure 5.1.)

Since the chance that an observation exceeds the control limit (3s) as a result of purely random variability is significantly small (1 out of 1000), one must be alert and vifslle every time such a result is observed. The actions taken depend on the previous observations.

If the 10 previous results for the check sample are evenly distributed around the target value, it is likely that we have encountered a purely random variation. Repeat the analysis for the check sample and if the result falls within the warning limits (2s), ignore the outlying observation and proceed with the analysis. Enter an appropriate remark on the control chart. If the repeat of the check sample did not fall within the warning limits, Then which signifies in that is out-of-control it was recommended to fault identify the cause.

If the 10 results for the check sample preceding the observation exceeding the control limit are not evenly distributed around the target value, but are decidedly one-sided in the same direction, then we are probably observing a strong drift. This constitutes again an out-of –control situation that must be inspected and remedied before we continue with the analyses.

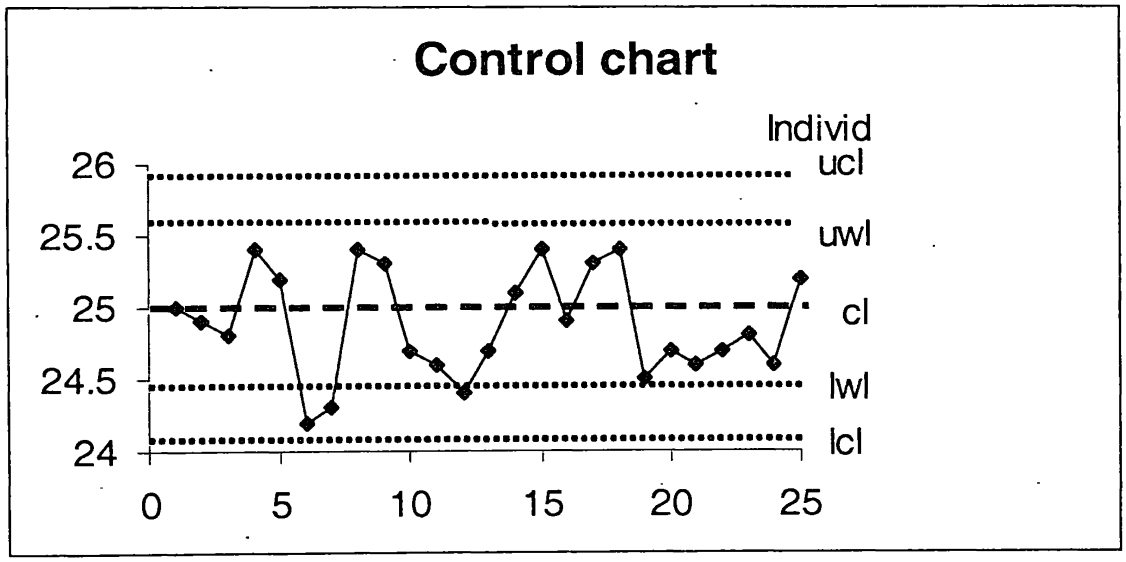


individ: cl:25 ucl:25.9 lcl:24.1

Figure 5.1.Result for the check sample with one observation exceeding the 3s limit.

Two successive observations fall within the warning (2s) and the control (3s) limit at the same side of the target value (see Figure 5.2.)

This situation may point to a drift in the target value, although we remain within the control limits that call for immediate action.



individ: cl:25 ucl:25.9 lcl:24.1

Figure 5.2.Result for the check sample with two successive observation between the 2s and 3s limit.

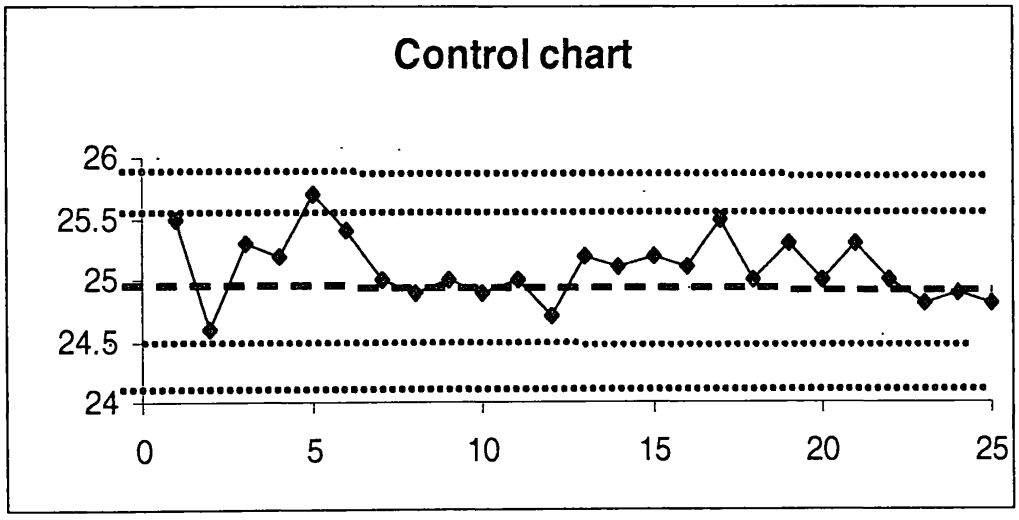
If the 5 previous results for the check sample are evenly distributed around the target value, repeat the analysis for the check sample and if the result falls between

the warning limits, ignore the two irregular results frequency of check sample analysis may be temporarily increased.

If the 5 previous results all lie on the same side of the target value as the two exceeding the warning limit, there is a serious indication of progressive drift, that warrants further inspection and repair.

Eight successive observations fall on the same side of the target value(see Figure 5.3.)

This is a clear warning that the target value may be drifting. Since the observations remain the warning limit there is no immediate reason for alarm, but the frequency of analyzing the check sample should be raised and if the observation continue on the same side of the target value, there is a clear risk that first the warning limit and eventually the control limit will be exceeded. To avoid a future out-of –control situation it is prudent to discontinue the analyses and address potential causes of the drift.

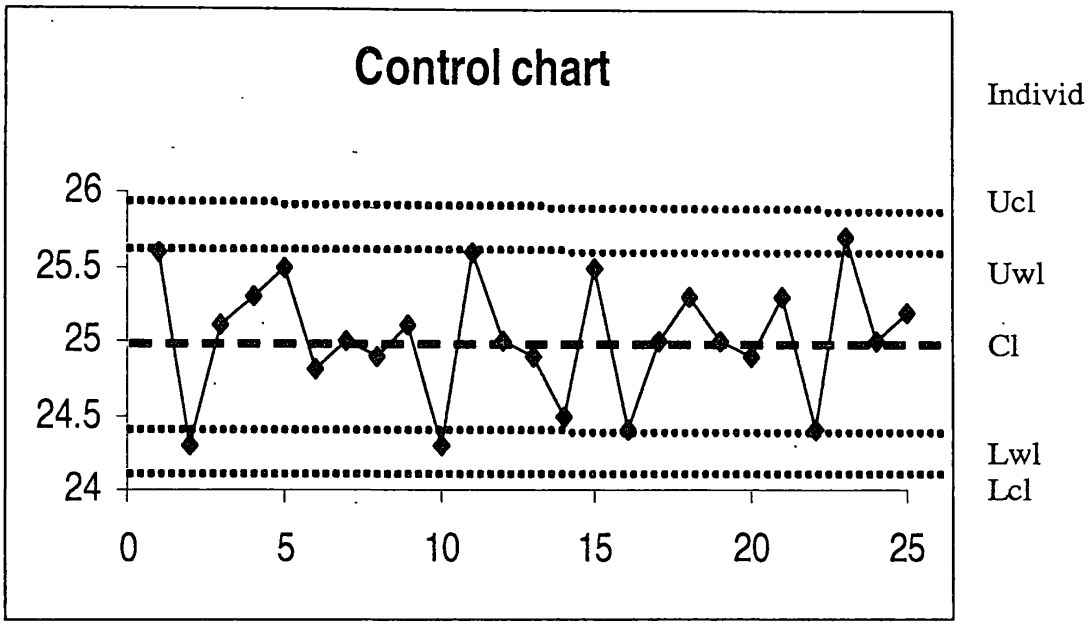


individ: cl:25 ucl:25.9 lcl:24.1

Figure 5.3.Result for the check sample with 8 successive observations above the target value

The observations follow a regular pattern (see Figure 5.4.)

This situation indicates a periodic disruption of the measurement system. The cause must be sought in possible periodic variations of external conditions, e.g. the temperature, the electric power act. . It warrants closer inspection and repair.



individ: cl:25 ucl:25.9 lcl:24.1

Figure 5.4.Result for the check sample demonstrating a regular pattern

5.3.Consequences of out-of-control situations

Evaluation of the analytical method

If an out-of-control situation has occurred further analyses must be postponed until the cause has been identified and removed. Frequent causes for out-of-control are:

The check sample has deteriorated and no longer conforms to the original composition. Replace by a fresh check sample.

There is a failure in the instrumentation. Test the equipment, readjust or replace malfunctioning parts, and verify proper operation. Thereafter run a check sample to assure that its result falls within the warning limits.

There has been a sudden change in the conditions, e.g. a reagent has been altered, a new analyst has taken over the analysis etc. Make sure that the conditions return to

normal and run a check sample to assure that the results falls within the warning limits.

The parameters of the control chart have changed. Return training set as describe in section 5 and determine the new target value and standard deviation. It is the responsibility of the laboratory manager to decide whether the new data are acceptable to design a new control chart.

Reporting analytical results preceding the out-of-control situation

As soon as an out-of –control situation is observed the regular analytical determinations of ordinary samples must be discontinued until the cause of the out-of-control has been identified and removed. The question remains, however, what must be done with the result of the analyses that have been performed since the previous determination of the check sample. Two important situations can be distinguished.

If the tolerance for the analytical result is much wider than the variability of the method, e.i. the precision (standard deviation) is more than adequate, then the analytical results can still be released. This conclusion does not remove the need to remedy the observed out-of-control situation.

If the tolerance for the analytical result is similar to the variability of the method or, in fact, the precision of the method is the decisive criterion, then the recipient of the analytical results must be alerted that the data reported since the previous determination of the check sample may be in error. Generally, the systematic deviation will not be larger than 2s. It is for the recipient to decide whether that is acceptable or whether the suspect samples need to be re-analysed

5.4.Updating the control charts

5.4.1.New check sample

The control chart has been constructed on the basis of 20 observations from the training set, but the two parameters, the average and the standard deviation, cannot be

expected to remain the same at infinitum. One clear case arises when a new check sample is introduced. Although the standard deviation of the analytical method is expected to remain the same (since it is a property of the method), it is equally likely that the target value has changed. Analyse the new check sample at least six times over several days to determine the new target value.

5.4.2.Periodic update of the control chart after 25 new data points

After 25 fresh results of the check sample the parameters of of the control chart need to be verified. Before we do so we remove from the data set only those observations that exceed the control (3s) limits for an identified cause

If the remaining fresh results are evenly distributed around the target value within the existing control (3s) limits with no more than one point outside the control limits without an identified cause, then we add the data to the training set and recalculate the average and the standard deviation from the total data. Henceforth they form the basis for the control chart.

If the fresh results are again evenly distributed around the target value, but two or more data exceed the control (3s) limit without a clearly identified cause, then first calculate the average and the standard for the (maximum 25) fresh data only. Inspect the results and remove any observation that exceeds the new 3s limits. Add the remaining data to the training set and recalculate the average and the standard deviation from the total data. Henceforth they form the basis for the control chart.

If the fresh data are not randomly distributed, but demonstrate a one-sided bias or a pattern, then the characteristics of the analytical method appear to have changed over the time interval that the 25 results for the check sample have been collected. The fresh data cam not be amalgamated with the data from the training set and the analytical method needs to be further investigated.

Upon further collections of results for the check sample proceed in the same way after every new set of 25 data points.

CHAPTER 06- CONCLUSSIONS

According to the results, the analytical determinations, which measure the quality, are in control.

There introduced check samples are better to implement a quality control system for selected analytical determinations, which are used in fat & oil analysis.

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